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OPTIMIZED MICROWAVE-ASSISTED EXTRACTION OF TOTAL PHENOLIC AND FLAVONOID CONTENTS FROM DEFATTED SEEDS AND PRESS RESIDUE OF *OPUNTIA FICUS-INDICA* (L.) MILL

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Abstract

Phenolic content of *Opuntia ficus indica* (*OFI*) was estimated in samples of seeds and press residue (cake) generated through mechanical oil extraction process. A microwave-assisted extraction (MAE) method was investigated for the optimization of the extracted total phenolic (TPC) and total flavonoid contents (TFC). The influence of extraction parameters on the yields of TPC, TFC were modelled using a second-order regression equation. The optimal MAE conditions were: 61.65 % ethanol concentration, 633.42 W microwave power, 3.59 min irradiation time and 46.28 mL/g solid-to-liquid ratio (SLR) for phenolics extraction, and 54.97 %, 580.04 W, 3.20 min and 47.40 mL/g for flavonoids extraction from defatted seeds powder. The extracts obtained under these conditions showed a TPC and TFC of 416.311±1.154 mg gallic acid equivalent (GAE) per100 g dry weight (DW) and 52.67±0.72 mg quercetin equivalent (QE)/100 g DW, respectively. Regarding press residue, the optimal conditions were 60.26%, 643.89 W, 3.85 min and 47.28 mL/g for phenolics extraction, and 67.96 %, 464.41 W, 2.46 min and 41.04 mL/g for flavonoids extraction; with a TPC and TFC of 400.11±3.14 mg

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GAE/100 g DW and 38.75±0.16 mg QE/100g DW, respectively. The results of the present study confirmed that *OFI* seeds and press residues could be considered as a non-negligible source of phenolics with a good antioxidant activity and revealed that MAE is a reliable method for optimizing yields of recovered phytochemicals from such agricultural products derivatives and by-products, with an intent to be scaled-up for industrial, nutraceutical or pharmaceutical applications.

Keywords: antioxidants, Microwave-Assisted Extraction (MAE), *Opuntia ficus-indica*, press residue, RSM-modelling, seeds

Introduction

Cactus pear, the fruit of the nopal cactus, is produced by a plant that belongs to the genus *Opuntia* and to the family of Cactaceae (Kuti, 2004). *Opuntia ficus-indica* (*OFI*) an indigenous species from the American continent has been spread in many other regions of the world including Africa and Australia. It was introduced into the Mediterranean area during the 16th century (Vignon *et al*., 2004). Due to its agro-ecologic characteristics, the cactus pear has been for long time an important crop for populations of the semi-arid and arid zones of the planet, thanks to its special adaptive mechanism and capacity to produce biomass; which made it relevant to those areas since the accessibility of the other vegetables is limited (Morales *et al.*, 2012). Thus, a larger interest has been devoted recently for increasing their cultivation and diverse research has been conducted to enlarge the industrialization of the fruit as well as the cladodes exploitation (El-Gharras *et al.*, 2006). This plant has been used to prevent soil erosion in arid areas and as a forage substitute during periods of dryness in North Africa (Habibi *et al.*, 2009). It is principally cultivated for the production of fruits (Bensadón *et al.*, 2010). Moreover, cladodes are used as a vegetable and in industry for the extraction of mucilage and pectin (Felkai-Haddache *et al.*, 2016) or even for the treatment of divers' effluents (Adjeroud *et al.*, 2015). The edible fruit is essentially consumed fresh or converted into juice (Terki *et al.*, 2018) or marmalades (Habibi, 2004). This food transformation engenders a large quantity of by-products (seeds and peels). The amount of seeds is important as it varies from 20 to 40 % per dry weight of the whole fruit, depending on the cultivars and are typically disposed in the food industry such as waste after pulp extraction (Habibi *et al.*, 2002). Seeds, considered as potential by-products, have a high content of unsaturated fatty acids, in particular polyunsaturated fatty acids (PUFAs), whose intake reduces the risk of developing cardiovascular, inflammatory and autoimmune illness (Simopoulos, 2002). Currently, the cactus is the subject of many investigations, for its content in bioactive compounds, well known for their therapeutic properties. They are more efficient antioxidants than vitamins; because flavonoids and phenolic compounds are, generally, able to delay the pro-oxidative effects on proteins, DNA and lipids by the generation of stable radicals (Feugang *et al.*, 2006). It has been indicated that a diet rich in cacti is positively correlated with a reduced risk of diseases in relation with oxidative stress; such as diabetes, cancer, cardiovascular and neurodegenerative illness (Osuna-martínez *et al.*, 2014). Until now, many conventional extraction techniques have been indicated for the extraction

of polyphenols from *OFI* seeds such as solvent extraction (Chaalal *et al.*, 2013; Chougui *et al.*, 2013). The inconvenient of the conventional methods include longer extraction time, and larger solvent consumption. Recently, with the development of the ‘‘Green chemistry’’ concept, environment-friendly techniques are becoming interesting. The technique of extraction of bioactive compounds with the aid of microwaves is one of the upcoming extraction techniques that could permit high reproducibility in shorter times, simplified manipulation, reduced solvent consumption and temperature and decreased energy input (Dahmoune *et al.*, 2015).

Consequently, the principal objectives of this investigation were: to study the effects of different parameters (Solvent concentration, microwave power, irradiation time, and solid-to-liquid ratio (SLR)) on the extraction efficiency, in terms of recovery and antioxidant activity of total phenolic and total flavonoid contents, from *OFI* seeds powder and press residue using MAE; to optimize the MAE conditions by response surface methodology (RSM) using Box-Behnken design (BBD); to compare optimized MAE results between the two samples: *OFI* seeds and its press residue.

Materials and methods

Chemical reagents

Folin–Ciocalteu and aluminium chloride were from Biochem, Chemopharma (Montreal, Quebec), sodium carbonate and sulfuric acid were from Biochem, Chemopharma (Georgia, USA), gallic acid was from Biochem-chemopharma (UK), acetone, ethanol, methanol and butanol were from Prolabo (CE), all other chemicals were from Sigma Chemical (Sigma–Aldrich GmbH, Germany).

Plant material

The intact seeds and press residue originating from the oil recovery process were obtained from oil producers in Bejaia, about 300 Km East of Algiers. Each sample was reduced into powder by grinding for 5 min using a blender type A11 basic (IKA, Germany), interspersed with three stops of 30s in order to minimize thermal degradation of thermosensitive molecules. The powders were passed through standard 500 µm mesh size sieve and only the fraction with particle size < 500 µm, representing 92%, was used. The powder was defatted using a Soxhlet apparatus (Behr, Labor-Tecnick, Dusseldorf, Germany) and was stored in airtight bags until use.

Microwave-assisted extraction

Phenolic compounds from powders of *OFI* seeds and press residues were extracted using a domestic digital microwave oven system (2450 MHz, 230 V, 50 Hz, Samsung Model NN-S674MF, Kuala Lumpur, Malaysia). The microwave was equipped with a digital control system for power (adaptable from 100 ~ 1000 W) and time of irradiation. This apparatus was modified in a way allowing for the condensation of the vapors produced over extraction in the sample.

For the extraction, *OFI* sample (seeds or their press residue powder) was suspended in a 500 mL flask containing the extracting solvent (pure water, ethanol, acetone, and methanol). After that, the suspension was irradiated at regular intervals

according to oven operation. Depending on the test, a different solvent, irradiation time, microwave power and SLR were applied (Tables 1 and 3). After irradiation, the flask was taken out and cooled in ice water bath (4°C). The solution obtained was filtered through Whatman No. 1 paper, and collected in a volumetric flask. The extract was stored at (4°C) until further use.

Determination of the total phenolic content (TPC)

The phenolic compounds contents of the extracts were determined by Folin–Ciocalteau method according to Singleton & Rossi (1965) with slight modification. 500 µL of aqueous extract were mixed with 2.5 mL of Folin–Ciocalteau reagent (10 %). After incubation for 2 minutes at 27°C, 2 mL of sodium carbonate solution (Na_2CO_3, 7.5 %) was added and again incubated at 50°C for 15 min in dark. After that, a UV–Vis spectrophotometer (Model: SpectroScan 50, Nicosia, Cyprus) has been used to measure the absorbance at 760 nm. In order to estimate the concentration of TPC in the sample, the absorbance of the extract was compared to a standard curve of gallic acid. The results were expressed as mg of gallic acid equivalents per 100 gram of sample dry weight basis (mg GAE/100 g DW).

Determination of the flavonoids content

The total flavonoids content was determined in accordance with the method of Djeridane *et al.*, (2006) which consists in mixing two equal volumes of sample extract and Aluminium chlorides ($AlCl_3$, 2 % in methanol). The mixture was incubated 15 min in the dark at room temperature. After that, the absorbance was measured at 430 nm. The results were expressed in mg quercetin equivalents/100 g of sample dry weight basis (mg QE/100 g DW).

Determination of the antioxidant activity

Radical scavenging activity

The scavenging capacity against the radical 1,1-diphenyl-2-picrylhydrazyl (DPPH) was determined according to Amrane-Abider *et al*. (2023). *OFI* seed extract (200 µl) was added to 1000 µl of methanolic DPPH solution (60 µM). The mixture was incubated 30 min at room temperature in a dark place. The decolorizing process was recorded at 515 nm. The antioxidant activity was evaluated using a standard curve for the concentrations of the standard compound (Gallic acid) and DPPH radical scavenging rates were plotted on it to determine the equivalent concentration of gallic acid that induced the same rate of radical scavenging. The DPPH radical scavenging activity of the sample was expressed as mg gallic acid equivalents/g DW (Hwang & Lee 2023).

Ferric Reducing Antioxidant Power (FRAP)

The ferric reducing antioxidant power of extracts was evaluated according to the method of Oyaizu (1986). 1 mL of sample was mixed with 2.5 mL of phosphate buffer (0.2 M, pH 6.6) and 2.5 mL of potassium ferricyanide 1 % (w/v). The mixture was incubated in a water bath at 50°C for 20 minutes. After that, 2.5 mL of a trichloroacetic acid solution 10 % (w/v) was added, and then the mixture was centrifuged at 5000 rpm for 15 min. A 2.5 mL aliquot of the upper layer was combined with 2.5 mL of distilled water and 0.5 mL of 0.1 % (w/v) solution of ferric

chloride. The absorbance was measured spectrophotometrically at 700 nm. The results were expressed as mg gallic acid equivalent (GAE) per 100 g of sample dry weight (DW).

Experimental optimization

Preliminary trials

In order to optimize the microwave procedure, the influences of the process parameters were initially separately investigated in single-factor experiments to limit the total experimental work (Table 1). When one variable was not studied, it was kept constant. Ethanol concentration, microwave power, irradiation time, and SLR were selected as independent variables, whereas TPC and TFC yields were selected in response.

The constant values for irradiation time, SLR, ethanol concentration and microwave power were 120 s, 20 mL/g, 50 % and 500 W, respectively.

Experimental design and statistical analyses

To determinate the preliminary range of extraction variables, the influence of the process parameters i.e. X_1, X_2 X_3 and X_4 were investigated using single-factor-test (Table 2).

From on the single-factor experimental results, the factors influencing the extraction process were selected for designing experiments employing response surface methodology (RSM). The JMP software (10.0.0 version, SAS Institute) package was used to establish mathematical models and to obtain the optimal conditions for TPC and TFC extraction. To study and validate extraction process parameters affecting the extraction of phenolic compounds from *OFI* seeds, Box–Behnken experimental design (BBD) was applied in the present investigation. The number of experiments (N) required for the development of BBD is defined in Eq. (1).

$$N = 2k(k-1) + C_0 \quad (1)$$

Where k is the number of factors and C_0 is the number of center points. The factor levels were coded as −1, 0 and 1 for low, center point or middle and high values, respectively. The four factors chosen for this study (Table 2) were designated as X_1 for ethanol concentration (50 ~ 100 %), X_2 for microwave power (400 ~ 800 Watt), X_3 for extraction time (2 ~ 4 minutes) and X_4 for SLR (30 ~ 50 mL/g). The variables were coded according to the equation (2) established by (Song et al., 2011):

$$x_i = \frac{(X_i - X_0)}{\Delta X} \quad (2)$$

Where x_i was a coded value of the variable; X_i was the actual value of variable; X_0 was the value of X at the center point and ΔX was the step change. The experiments were realized according to the design of experiments shown in Tables 1 and 3. The output results were fitted to a second-order polynomial equation, according to the model in Eq. (3).

$$Y = B_0 + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} X^2 + \sum_{i>j}^{k} B_{ij} X_i X_j + E \quad (3)$$

Where Y represents the response function (TPC and TFC); B_0 is a constant coefficient; B_i, B_{ii} and B_{ij} are the coefficients of the linear, quadratic and interactive terms, respectively, and X_i and X_j represent the actual independent variables. Using analysis of variance, the regression coefficients of individual linear, quadratic and interaction terms were determined. The three dimensional response surface plots and contour plots from the fitted polynomial equation were used in the aim to visualize the relationship between the response and experimental levels of each factor and to deduce the optimum conditions.

Analysis of variance (ANOVA) was performed for response variable using the statistical XLSTAT software for windows. The full models where *p*-values (partitioned into linear and interaction factors) showed whether the terms were significant or not; *p* values < 0.05 were regarded as significant (Table 4).

Results and discussion

Single-factor experiments

Effect of ethanol concentration

The effects of diverse extraction parameters (ethanol ratio, irradiation time, microwave power and SLR) on the TPC and TFC yields were studied using the one-variable-at-a-time approach. Figure 1 indicates that yields of TPC and TFC vary depending on the type of solvent. Ethanol induced significantly higher yields compared to other solvents. Ethanol has several advantages over the other solvents, including higher extraction efficiency, environmental compatibility and lower toxicity and cost. Yet, the extraction efficiency could be affected by the ratio of ethanol in the water (Wu *et al*., 2012); thus, different ratios of aqueous ethanol were tested. Table 1 indicates that yields of TPC and TFC increased significantly when the concentration of ethanol was raised up from 30 to 50 %. At higher concentrations the yields were statistically similar for both 50 and 70 %. At the ultimate concentration (100 %) the yields were statistically lower.

For the extraction of phenolic compounds from other plant sources, a similar effect was observed (Pan, *et al*., 2003; Spigno, *et al*., 2007; Li *et al*., 2012). This could be explained by the fact that with the increase in ethanol concentration, solvent polarity diminished and molecular movement decreased, leading to light dissolution of phenolic compounds as a consequence of the attenuation of the diffusion coefficient and reduced solubility (Yang *et al*., 2009).

The positive effect of intermediate aqueous ethanol ratio could have allowed an increased solubility of phenolic compounds and a declined heating of the mixture with a limited thermal degradation of the recovered compounds. For the RMS trials, the concentration range 50 % ~ 100 % was chosen and 50 % was fixed for the other single-factor experiments.

Table 1. Parameterization of microwave-assisted extraction (MAE) conditions of polyphenols and flavonoids from *Opuntia ficus indica* seed and press residue powders.

Parameter		Defatted seeds		Press residue	
		TPC (mg GAE/100g DW)	TFC (mg QE/100g DW)	TPC (mg GAE/100g DW)	TFC (mg QE/100g DW)
Ethanol concentration (%)	30	265.76 ± 1.16^{b}	29.58 ± 0.31^{b}	257.92 ± 1.91^{b}	24.60 ± 0.81^{b}
	50	306.45 ± 0.88^{a}	38.81 ± 0.48^{a}	300.89 ± 0.76^{a}	36.35 ± 0.42^{a}
	70	308.22 ± 1.16^{a}	24.86 ± 0.27^{d}	303.42 ± 1.16^{a}	22.29 ± 0.64^{c}
	100	150.21 ± 0.08^{c}	28.49 ± 0.47^{c}	136.15 ± 2.61^{c}	20.59 ± 0.45^{d}
Microwave Power (Watt)	200	243.01 ± 2.32^{d}	27.48 ± 0.36^{d}	228.35 ± 1,16^{d}	24.91 ± 0.87^{c}
	400	274.86 ± 2.66^{c}	29.58 ± 0.31^{c}	265.76 ± 2.87^{c}	27.07 ± 0.16^{b}
	500	311.00 ± 1.58^{a}	35.77 ± 0.79^{a}	303.42 ± 2.44^{a}	32.57 ± 0.63^{a}
	700	283.70 ± 2.87^{b}	33.10 ± 0.09^{b}	280.16 ± 1.58^{b}	23.18 ± 0.51^{d}
	800	231.89 ± 0.76^{e}	27.90 ± 0.24^{d}	220.76 ± 1.58^{e}	26.07 ± 0.48^{bc}
Extraction time (min)	1	276.37 ± 1.16^{de}	31.10 ± 0.51^{b}	243.26 ± 2.01^{d}	27.90 ± 0.40^{b}
	1.5	285.98 ± 0.44^{c}	29.48 ± 0.09^{c}	265.50 ± 2.44^{c}	27.54 ± 0.72^{b}
	2	310.50 ± 1.16^{b}	29.58 ± 0.31^{c}	303.67 ± 2.32^{b}	24.97 ± 0.51^{c}
	3	351.44 ± 2.19^{a}	39.65 ± 0.31^{a}	344.11 ± 1.52^{a}	37.08 ± 0.79^{a}
	4	277.89 ± 0.44^{d}	20.40 ± 0.51^{d}	242.50 ± 1.31^{d}	16.94 ± 0.55^{d}
	5	274.60 ± 0.44^{e}	18.41 ± 0.47^{e}	240.99 ± 0.76^{d}	15.00 ± 0.48^{e}
Solid-to-Liquid (mL/g)	10	197.00 ± 2.01^{e}	39.76 ± 0.96^{e}	193.97 ± 0.76^{e}	33.57 ± 0.79^{d}
	20	351.44 ± 2.19^{c}	49.41 ± 0.94^{c}	344.11 ± 1.52^{c}	43.22 ± 0.66^{c}
	30	362.31 ± 0.76^{b}	61.05 ± 0.63^{b}	358.27 ± 1.16^{b}	54.97 ± 0.48^{b}
	40	389.86 ± 1.16^{a}	63.57 ± 0.31^{a}	379.00 ± 1.31^{a}	59.38 ± 0.79^{a}
	50	290.53 ± 1.58^{d}	46.58 ± 0.31^{d}	267.53 ± 3.47^{d}	41.54 ± 0.31^{c}

Results are reported as means ± standard deviation (S.D). $^{a\text{-}e}$: different letters indicate statistically significant differences at $p < 0.05$ according to ANOVA and Tukey's post-hoc test.

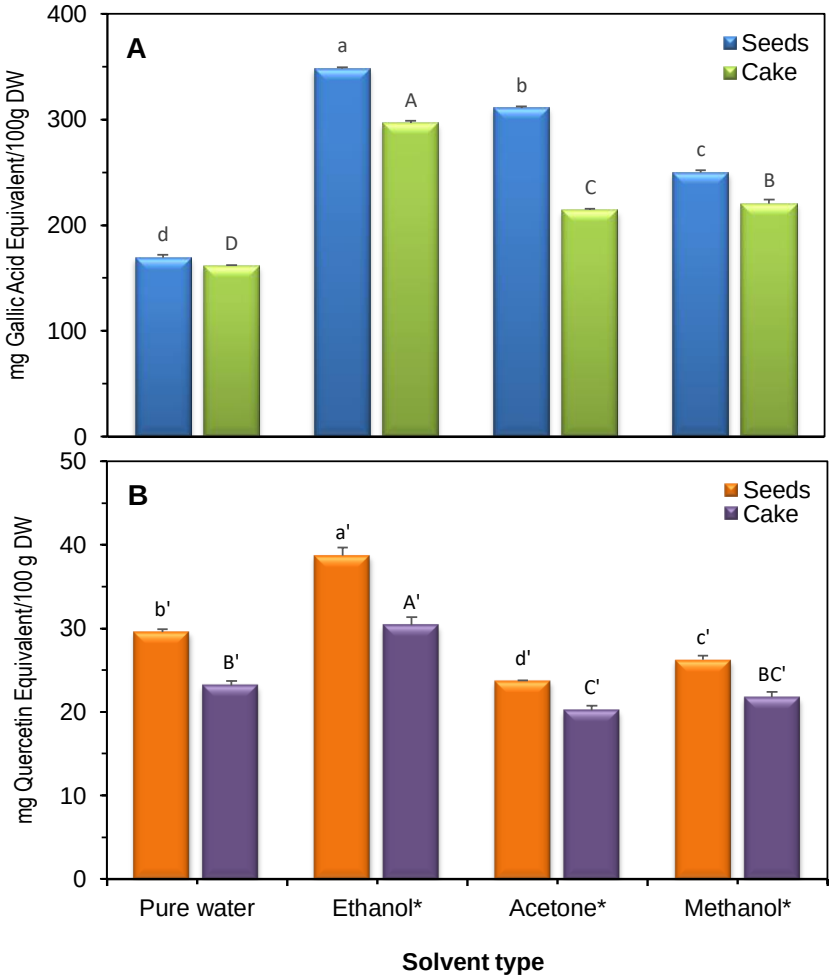

Figure 1. Effect of solvent type on microwave assisted extraction of total phenolic (**A**) and total flavonoids (**B**) content from *Opuntia ficus indica* defatted seed and cake powders. *Solvent/water mixture (50%; v/v).

Heating effect with consequent increase of mass transfer phenomena probably induces an enhancement of phenols recovery, up to a certain power density value and then, to thermal degradation of bioactive compounds at more elevated densities (Li *et al*., 2012). The range 400 ~ 800 W was selected for the RSM study, whereas the 500 W was used for the rest of single-factor trials.

To examine the effect of irradiation time on the TPC and TFC yields, experiments were carried-out in the time range (1 ~ 5 min).

Effect of microwave power and irradiation time

The yield increased with increasing microwave power from 200 to 500 W and then lightly decreased for higher powers (700 and 800 W).

It must be indicated that the operating temperature could not be regulated in the employed equipment. It is known that the temperature increases with microwave power for a constant sample size (Spigno and De Faveri, 2009). The main effect of microwaves is the heating effect. As it has been reported by Kappe *et al.* (2013),

Table 2 Range of coded and actual values for Box-Behnken Design

Factor	Level		
	-1	0	+1
X_1	50	75	100
X_2	400	600	800
X_3	2	3	4
X_4	30	40	50

X_1, ethanol concentration (% v/v, solvent/water); X_2, power (Watt); X_3, time (min); X_4, SLR (mL/g).

As shown in Table 1, when the extraction time increased from 1 to 3 min, a significant increase in extraction efficiency was observed, followed by a significant decrease beyond 4 min. Continuous irradiation for a long time without temperature control probably induced a thermal deterioration of phenolic compounds (Yang *et al.*, 2009). Shorter extraction time was also favorable to reduce energy costs, for RMS trials the 2 ~ 4 min range was selected, whereas, 2 min was kept for the single-factor trials dealing with a varying SLR.

Effect of solid-to-liquid ratio (SLR)

Increasing the SLR from 10 to 40 (mL/g) affected the yields of TPC and TFC in seeds samples which significantly increased from 197.01 % to 389.86 % and from 39.76 % to 63.57 %, respectively. For the press residue samples, yields of TPC and TFC increased from 193.97 % to 379.00 % and from 33.57 % to 59.38 %, respectively. Though, upon reaching the 50 mL/g ratio yields dropped in both samples. This could be explained by the fact that increasing SLR may have upraised the diffusivity of the solvent into cells and enhanced the desorption of the phenolic compounds from the cells which led to increased yield (Spigno and De Faveri, 2009). As previously reported by Bhuyan *et al.* (2015), when the SLR increases, suspension density increases, resulting in less effective solvation of the liberated cellular contents. The 30–50 mL/g SLR range was selected for the RSM tests.

Modeling and fitting the models using response surface methodology (RSM)

Fitting the models for TPC and TFC of the *OFI* sample extracts (seeds and press residue) is crucial to clarify how exactly the RSM mathematical model can predict ideal variances and represent the correlations between the selected parameters of microwave extraction (Bhuyan *et al.*, 2015). The responses (TPC and TFC of seeds and press residue) of each run of the experimental design were presented in Table 3. The extraction yield of TPC ranged from 135.61 to 379.84 mg GAE/100 g of defatted

dry weight (DW) for *OFI* seeds, whereas the content in the press residue ranged from 125.02 to 375.26 mg GAE/100 g DW.

While the content of TFC ranged from 27.80 to 55.48 mg QE/100 g DW and from 24.42 to 50.53 mg QE/100 g DW for extract samples of defatted seeds and press residue, respectively. The phenolic content of press residue was slightly lower compared to that of seeds.

This difference could be explained by the thermosensibility of phenolic compounds and to a partial transfer into the oil during pressing.

Even though the solubility of phenolic compounds in the oil was poor, a small amount could have been transferred into the oil during processing.

These findings are in accordance with an already, published study demonstrating that grape seed processing may modify their polyphenol and flavonoid contents (Maier *et al.*, 2009). The polyphenol and flavonoid contents in the *OFI* seeds obtained in the current study exhibited quite higher values than those reported by Chaalal *et al.* (2013) and Chougui *et al.* (2013). This shows that MAE is a good alternative to conventional solvent extraction for the recovery of phenolic compounds from *OFI* seeds, it gave higher yields of active substances and a better antioxidant quality as well as it reduced the time of the extraction process.

The regression coefficient and analysis of variance of the intercept, linear, quadratic and interaction terms of the model for TPC and TFC for (seeds and press residue) were summarized in Table 4.

As indicated, the regression parameters of the surface response analysis of the models, the linear, quadratic and interaction terms had significant effects.

In fact, the ANOVA treatment of the set parameters for optimal extraction yields of phenolic and flavonoid content from seed powder and press residue samples showed that the model was strongly significant at the level of $p < 0.0001$.

In the case of extraction of total phenolic content from seed sample, all linear parameters (ethanol concentration (X_1), irradiation time (X_3) and liquid-to-solid ratio (X_4)) and their quadratic parameters were strongly significant at the level of $p \leq 0.0001$, except for microwave power (X_2) and its quadratic parameter which were significant at the level $p < 0.01$. Also, the interaction parameter (X_1X_4) was notable at $p < 0.01$, while (X_1X_3) and (X_2X_4) were significant at $p < 0.05$, whereas the interaction parameters (X_1X_2, X_2X_3 and X_3X_4) were not significant ($p > 0.05$).

For the extraction of the total phenolic content from the press residue sample, the linear parameters (ethanol concentration (X_1), irradiation time (X_3) and liquid-to-solid ratio (X_4) and the quadratic parameter (X_1X_1) were strongly relevant at the level $p < 0.0001$. Moreover, the quadratic parameters (X_3X_3 and X_4X_4) were highly significant at the level of $p < 0.001$. The linear parameter for microwave power (X_2) was very significant at $p < 0.01$. The interactions (X_1X_3) and (X_1X_4) were significant at the level ($p < 0.05$).

Table 3 Box– Behnken Design with the observed responses and predicted values for yield of TPC and TFC referred to dry weight (DW) of *OFI* seeds powder and press residue using microwave assisted extraction. GAE: Gallic acid equivalents.

Run	Pattern $X_1 X_2 X_3 X_4$	Defatted seed powder				Press residue			
		TPC (mg GAE/100 g DW)		TFC (mg QE/ 100g DW)		TPC (mg GAE/100 g DW)		TFC (mg QE/ 100g DW)	
		Observed	Prediction	Observed	Prediction	Observed	Prediction	Observed	Prediction
1	+0+0	161.83 ± 2.69	155.98	36.37 ± 2.79	37.02	136.87 ± 0.47	136.40	32.21 ± 0.99	33.77
2	+00-	141.30 ± 6.44	140.24	32.46 ± 0.45	31.22	125.02 ± 1.42	128.23	29.73 ± 3.18	31.92
3	00-+	345.02 ± 0.47	344.53	48.39 ± 0.39	48.75	323.18 ± 3.32	327.34	31.44 ± 0.23	31.16
4	+−00	135.61 ± 1.79	144.24	27.80 ± 0.23	27.13	128.18 ± 3.59	133.38	42.90 ± 1.38	40.95
5	0+−0	330.55 ± 2.70	338,24	44.98 ± 2.30	44.71	330.76 ± 3.59	337.34	31.21 ± 1.18	32.52
6	00−−	309.23 ± 2.78	311,83	48.53 ± 2.68	48.12	308.91 ± 2.17	313.28	38.82 ± 0.60	37.44
7	00++	376.21 ± 5.02	375.84	50.36 ± 0.79	50.09	370.00 ± 2.74	365.65	44.43 ± 5.80	44.47
8	0000	375.52 ± 2.34	377.84	47.62 ± 1.42	46.70	365.56 ± 1.91	362.78	37.64 ± 0.45	37.24
9	0−0−	315.39 ± 1.10	311.36	37.68 ± 0.23	39.69	316.65 ± 1.42	313.08	34.75 ± 0.23	34.23
10	0+0−	347.77 ± 0.95	343.23	46.55 ± 1.82	48.19	343.35 ± 3.01	343.01	35.24 ± 1.38	34.39
11	0−0+	369.89 ± 3.01	364.93	46.42 ± 1.04	45.44	360.99 ± 0.72	354.95	37.41 ± 2.19	38.51
12	++00	160.25 ± 0.72	164.32	27.93 ± 0.39	28.43	159.78 ± 1.19	152.29	24.42 ± 0.60	23.07
13	+0-0	153.05 ± 4.39	144.28	30.68 ± 1.42	30.46	138.24 ± 3.98	131.92	30.95 ± 1.64	31.01
14	0−+0	350.30 ± 2.61	349.87	44.60 ± 2.75	44.88	347.62 ± 0.72	347.40	37.64 ± 0.60	37.41
15	−+00	372.89 ± 0.99	366.50	49.18 ± 1.42	49.17	371.79 ± 0.99	366.60	50.53 ± 1.77	51.15
16	−00+	375.89 ± 2.24	384.20	49.26 ± 1.38	50.50	370.21 ± 2.43	373.37	47.34 ± 3.16	46.23
17	0++0	370.36 ± 5.34	375.01	47.47 ± 0.82	46.96	356.04 ± 1.66	365.28	42.88 ± 1.18	42.38
18	−0+0	378.89 ± 2.14	378.17	51.80 ± 0.60	52.68	375.26 ± 1.71	375.20	45.92 ± 5.41	46.09
19	−−00	357.73 ± 0.82	355.89	47.08 ± 0.82	45.91	347.62 ± 3.68	355.12	35.23 ± 2.19	35.24
20	0000	378.16 ± 0.27	377.84	46.12 ± 0.91	46.70	362.73 ± 0.27	362.78	37.73 ± 1.59	37.24
21	0+0+	369.21 ± 7.26	363.74	42.83 ± 0.83	41.48	358.21 ± 4.67	355.39	35.61 ± 2.75	36.38
22	00+−	331.72 ± 1.19	334.46	52.71 ± 2.58	51.68	329.61 ± 3.28	322.46	32.98 ± 0.68	31.93
23	0000	379.84 ± 5.49	377.84	46.36 ± 0.39	46.70	360.05 ± 3.32	362.78	36.34 ± 3.87	37.24
24	−0−0	339.56 ± 0.72	335.92	54.30 ± 0.82	54.32	335.09 ± 2.24	329.17	42.36 ± 1.20	41.05
25	−00−	319.34 ± 4.74	323.62	55.48 ± 1.38	54.51	326.29 ± 1.19	326.78	35.16 ± 0.39	36.77
26	0−−0	330.08 ± 1.92	332.69	41.70 ± 0.68	42.23	327.71 ± 2.61	324.84	37.88 ± 1.27	39.47
27	+00+	150.77 ± 6.09	153.75	33.29 ± 1.04	34.27	130.00 ± 4.57	135.88	29.24 ± 0.68	28.72

X_1, ethanol concentration (% v/v, solvent/water); X_2, power (Watt); X_3, time (minutes); X_4, SLR (mL/g).

In the case of the extraction of the total flavonoid content from seed sample, the linear parameter (X_1) and the quadratic parameters (X_1X_1 and X_2X_2) were strongly relevant at the level of $p < 0.0001$. The interaction parameter (X_2X_4) was highly notable at the level of $p < 0.001$. The linear parameters (X_3) and (X_4), the quadratic parameter (X_3X_3) and the interaction parameters (X_1X_3 and X_2X_4) were significant at the level of $p < 0.01$. The linear parameter (X_2) and the interaction parameter (X_1X_4) were significant at the level $p < 0.05$.

Furthermore, for the extraction of total flavonoid content from the press residue, the linear parameters (X_1) and the interaction parameters (X_1X_2 and X_3X_4) were strongly relevant at the level $p < 0.0001$. While the linear parameters (X_3) and (X_4) and the interaction parameter (X_1X_4) were significant at the level $p < 0.01$. The quadratic parameter (X_4X_4) and the interaction parameter (X_2X_3) were significant at the level ($p < 0.05$). The determined coefficients (R^2) were 0.99 for both models (TPC from seeds and press residue), 0.98 and 0.96 for TFC from seeds and press residue, respectively, which means that the sample variations for the microwave extraction efficiency of seeds and press residue were attributed to the independent variables. Though, a large value of R^2 does not always imply that the regression model is a valid one (Karazhiyan *et al.*, 2011).

In a good statistical model, R^2_{adj} should be comparable to R^2. As shown in Table 4, R^2 and R^2_{adj} values for the models did not differ greatly. The lack of fit test determines if the model is adequate to describe the experimental data or whether another model should be selected. The absence of any lack of fit for all ($p > 0.05$) equally reinforces the reliability of all models.

Eq. (4), (5), (6) and (7) show the relationship between independent variables for the extraction of TPC and TFC of seeds (Y_1 and Y_2, respectively) and press residue (Y_3 and Y_4, respectively).

$$Y_1 = 377.84 - 103.46X_1 + 7.67X_2 + 13.49X_3 + 18.52X_4 - 7.63X_1X_3 - 11.77X_1X_4 - 8.27X_2X_4 - 107.73X_1^2 - 12.37X_2^2 - 16.52X_3^2 - 19.66X_4^2 \quad (4)$$

$$Y_2 = 46.70 - 9.88X_1 + 1.14X_2 + 1.23X_3 + 2.05X_1X_3 + 1.76X_1X_4 - 3.11X_2X_4 - 5.06X_1^2 - 3.98X_2^2 + 1.98X_3^2 \quad (5)$$

$$Y_3 = 362.78 - 109.01X_1 + 7.59X_2 + 12.62X_3 + 13.56X_4 - 10.38X_1X_3 - 9.73X_1X_4 - 105.74X_1^2 - 13.87X_3^2 - 15.98X_4^2 \quad (6)$$

$$Y_4 = 37.23 - 5.59X_1 + 1.95X_3 + 1.56X_4 - 8.45X_1X_2 - 3.17X_1X_4 + 2.97X_2X_3 + 4.71X_3X_4 - 1.52X_4^2 \quad (7)$$

Analysis of the response surface model and contour plots

The effects of the independent variables and their mutual interaction on the efficiency of the TPC and TFC extraction yields for seeds and press residue can also be seen on three-dimensional response surface profiles of multiple non-linear regression models.

Table 4 Analysis of variance (ANOVA) for the fitted quadratic polynomial model for optimization of extraction parameters.

Coefficient	Defatted seeds				Press residue			
	TPC		TFC		TPC		TFC	
	Estimate	Prob >[t]	Estimate	Prob >[t]	Estimate	Prob >[t]	Estimate	Prob >[t]
β_0	377.84	< 0.0001*	46.69	< 0.0001*	362.78	< 0.0001*	37.24	< 0.0001*
β_1	-103.46	< 0.0001*	-9.88	< 0.0001*	-109.01	< 0.0001*	-5.59	< 0.0001*
β_2	7.67	0.0022*	1.14	0.0118*	7.59	0.0026*	-0.23	0.6484
β_3	13.49	< 0.0001*	1.23	0.0076*	12.63	< 0.0001*	1.69	0.0055*
β_4	18.52	< 0.0001*	-0.24	0.5430	13.56	< 0.0001*	1.57	0.0050*
β_{12}	2.37	0.5012	-0.49	0.4731	1.86	0.6027	-8.45	< 0.0001*
β_{13}	-7.64	0.0452*	2.05	0.0095*	-10.39	0.0113*	-0.57	0.4758
β_{14}	-11.77	0.0049*	1.77	0.0208*	-9.74	0.0159*	-3.17	0.0018*
β_{23}	4.89	0.1774	-0.10	0.8805	1.342	0.7059	2.19	0.0472*
β_{24}	-8.27	0.0324*	-3.11	0.0005*	-7.37	0.0552	-0.57	0.4763
β_{34}	2.17	0.5369	-0.55	0.4203	6.53	0.0844	4.71	< 0.0001*
β_{11}	-107.73	< 0.0001*	-5.06	< 0.0001*	-105.74	< 0.0001*	0.07	0.9187
β_{22}	-12.37	0.0013*	-3.98	< 0.0001*	-5.19	0.1097	0.43	0.5533
β_{33}	-16.52	0.0001*	1.98	0.0049*	-13.87	0.0006*	0.81	0.2752
β_{44}	-19.66	< 0.0001*	0.98	0.1125	-15.98	0.0002*	-1.66	0.0328*
***P* of model >F**		< 0.0001*		< 0.0001*		< 0.0001*		< 0.0001*
Lack of fit		0.0818		0.2724		0,1246		0.1826
R^2	0.99		0.98		0.99		0.96	
R^2_{Adj}	0.99		0.97		0.99		0,93	

*Significantly different at $p < 0.05$; β_0: intercept; β_1, β_2, $\beta_3$and β_4: linear regression coefficients for ethanol concentration, microwave power, irradiation time and SLR; β_{12}, β_{13},β_{14},β_{23}, β_{24} **and** β_{34}: regression coefficients for interaction between ethanol concentration × microwave power, ethanol concentration × irradiation time, ethanol concentration × SLR, microwave power x irradiation time, microwave power x SLR and irradiation time x SLR; β_{11}, β_{22}, β_{33} and β_{44}: quadratic regression coefficients for ethanol concentration × ethanol concentration, microwave power × microwave power, irradiation time x irradiation time and SLR × SLR.

Figures 2, and 3 illustrate the three-dimensional (3D) plots and their respective contour plots. They show the type of interactions between two tested variables and the relationship between responses and experiment levels of each variable, by maintaining the other two independent variables at zero level. In this study, the chosen response surface plots (3D) were those whose interactions between the independent variables were significant.

In the case of TPC, the significant interactions between the independent variables are illustrated in Figure 2 (A, B, C) and Figure 3(A, B) for seeds powder and press residue samples, respectively. Figure 2A shows the effect of ethanol concentration (X_1) and extraction time (X_3) on the extracted TPC yield for defatted seeds. The recovery of TPC from seeds of *OFI* increased with the increase in ethanol concentration (50 ~ 61 %) and extraction time (2 ~ 3.5 min) after which a decrease of the TPC with further increase in ethanol concentration took place.

Usually, the polarity of ethanol–water mixture would increase continually with the addition of water to ethanol. More polar phenolic compounds may be extracted according to ‘‘like dissolves like’’ principle. Thereby, it could be seen that phenolics recovery using 50% ethanol was higher than 100 % ethanol (Karazhiyan *et al.*, 2011).

Figure 2B demonstrates the effect of ethanol concentration (X_1) and SLR (X_4) on the TPC recovery from seeds powder. It first increased significantly ($p < 0.01$) with increasing ethanol concentration and SLR up to the optimum, and then the TPC decreased with further increase in ethanol concentration. Figure 2C shows the effect of microwave power (X_2) and SLR (X_4) on the TPC yield for seeds. Raising the microwave power and SLR led to an optimal TPC yield, followed by a declined yield with further increase of these independent variables. This decline could be assigned to the increase in the SLR that slowed down mass transfer resulting from the lower heating efficiency under microwave conditions and the solubility of polyphenols.

Figure 3A-B displays the interactions between the amount of ethanol concentration and each of the two other factors (irradiation time and SLR) on the recovery of TPC from the press residue of *OFI* originating from the oil recovery process. The yield of TPC increased with the increase of ethanol concentration from (50 ~ 67.96 %), irradiation time from (2 ~ 2.5 min) and SLR from (30 ~ 41 mL/g) and nearly reached a peak at the 67.96 % ethanol concentration. After that, additional ethanol concentration with irradiation time and ratio caused negative effects on the yield of TPC.

In the case of TFC, the significant interactions between independent variables were ethanol concentration with two other factors (irradiation time and SLR) and microwave power and SLR for the seeds matrix, also interaction of ethanol concentration with both two factors (microwave power and SLR) and irradiation time with both microwave power and SLR for the press residue matrix. Figure 2A’-C’ and Figure 3(C-F) show that increasing the value of each process variable improved the extraction of TFC up to an optimum, over which the yield started to decrease with constant increase of the process variables values.

The surface plots indicates that the maximum TFC was achieved at an ethanol concentration of 55 %, irradiation time of 3.20 min, microwave power of 580 W and SLR of 47 mL/g, and ethanol concentration of 68 %, irradiation time of 2.46 min, microwave power of 464 W and SLR of 41 mL/g for seeds and press residue, respectively. Which means that, moving away from these points, in any direction, would induce the regression of extracted TFC.

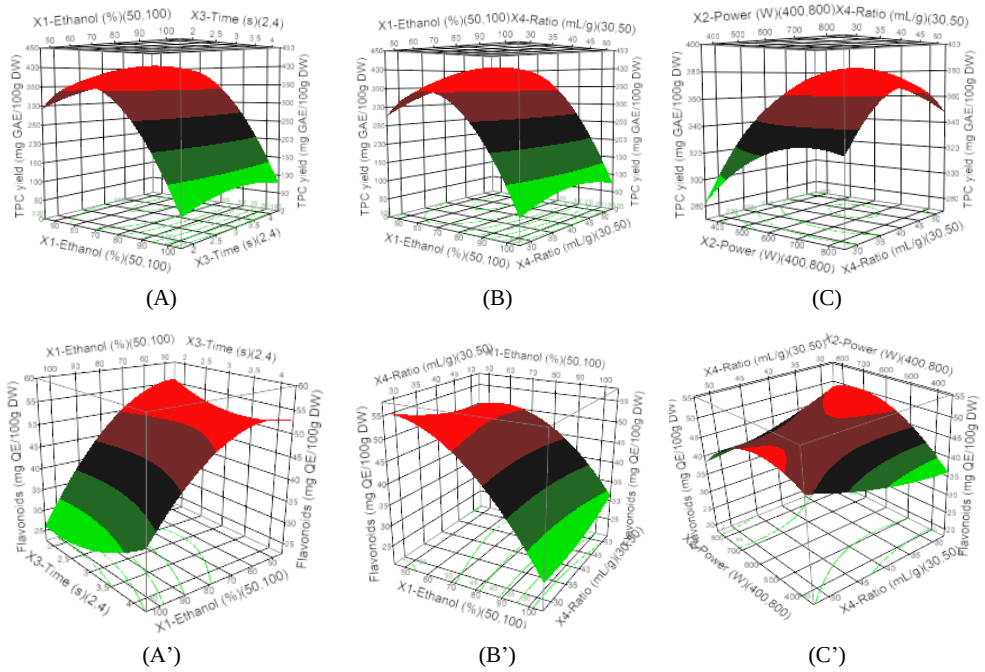

Figure 2. Response surface plots for the effect of ethanol concentration and irradiation time (A, A'); ethanol concentration and solid-to-liquid ratio (B, B'); microwave power and solid-to-liquid ratio (C, C') on the Total Phenolic (A, B, C) and Total Flavonoids Content (A'B'C') of *Opuntia ficus indica* defatted seeds.

Optimization and validation of the models

On the basis of the RSM predictive models, the ideal MAE conditions for the extraction of TPC and TFC from *OFI* seeds and press residue were presented in Table 5. The antioxidant activity (DPPH and FRAP) was studied under these optimal conditions. Validation experiments were realized under the previous optimal conditions to validate the adequacy of the models.

The results obtained were not significantly different from those predicted at $p > 0.05$ using a paired t-test (Hossain *et al.*, 2012), confirming that the models were adequate to reflect optimization (Wang *et al.*, 2010). The strong correlation between the actual and predicted results confirms the effectiveness of the response surface models to reflect the expected optimizations. Therefore, these conditions were recommended for future extractions of TPC, TFC from *OFI* seeds and press residue. These findings

also justified that RSM is a reliable and effective tool for modeling and optimizing extraction conditions.

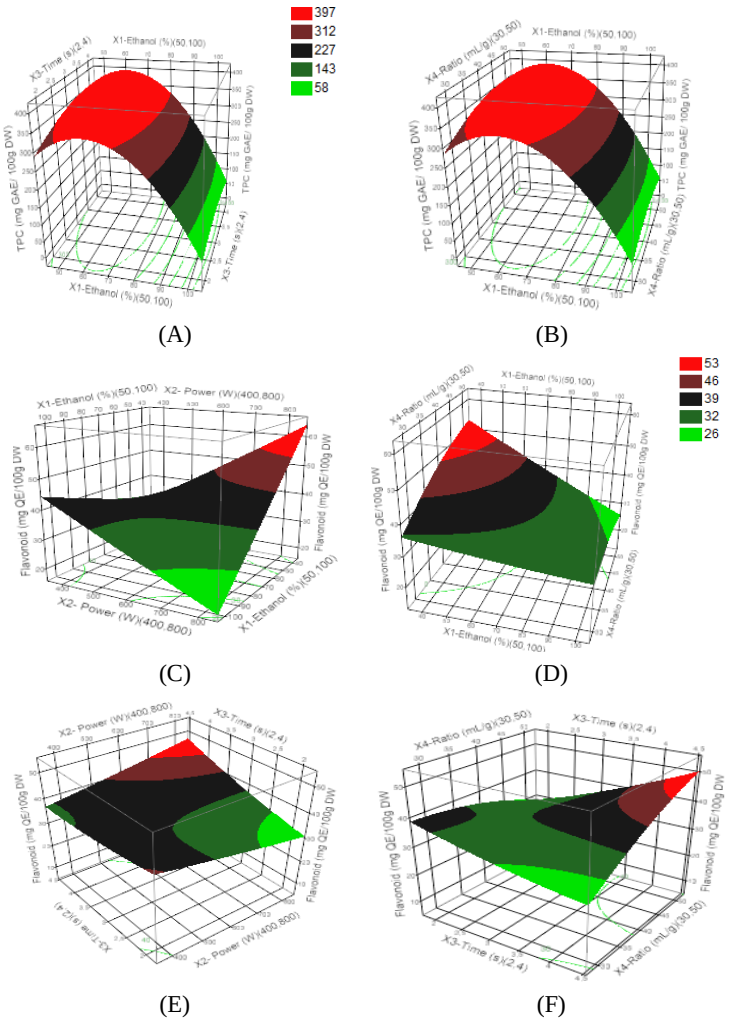

Figure 3. Response surface plots for the effect of ethanol concentration and irradiation time (A); ethanol concentration and solid-to-liquid ratio (B); ethanol concentration and microwave power (C); ethanol concentration and solid-to-liquid ratio (D); irradiation time and solid-to-liquid ratio (E); irradiation time and microwave power (F) on the total phenolic content (A and B) and total flavonoid content (C, D, E, F) of *Opuntia ficus indica* press residue.

Antioxidant activity

To evaluate the antioxidant and the antiradical activities of the *OFI* seeds extracts, the reducing power and DPPH free radicals tests were used. The values were expressed as mg gallic acid equivalent per 100 g of dry maters. The mean total antioxidant activity of samples was presented in the Table 5.

Data on antioxidant activities of *OFI* press residue originating from oil recovery process is not available in the specialized literature. The press residue of *OFI* exhibited lower reducing power (241.03 mg GAE/100 g) than seeds. Similarly, the DPPH scavenging potential of seeds (186.39 mg GAE/100 g) was stronger than that of press residue (162.21 mg GAE/100 g).

Table 5. Estimated optimum conditions, predicted and experimental values of responses and antioxidant activities.

		Defatted seeds		Press residue	
		TPC[a]	**TFC[b]**	**TPC[a]**	**TFC[b]**
Optimum conditions	**Ethanol (%)**	61.65	54.97	60.26	67.96
	Power (W)	633.42	580.04	643.89	464.41
	Time (min)	3.59	3.20	3.85	2.46
	SLR (mL/g)	46.28	47.40	47.28	41.04
Maximum value	**Predicted**	415.95	50.63	406.08	37.75
	Actual	416.31 ± 1.15	052.67 ± 0.72	400.11 ± 3.14	038.75 ± 0.16
Antioxidant activity	**DPPH[a]**	186.39 ± 1.91	157.72 ± 2.39	162.21 ± 1.69	119.94 ± 5.42
	FRAP[a]	325.55 ± 7.84	283.13 ± 2.66	241.03 ± 3.87	235.83 ± 4.41

Units: [a](mg GAE/100 g DW), [b](mg QE/ 100g DW).

The ferric reducing antioxidant power in seeds was 325.55 GAE/100 g and 283.13 GAE/100 g for the extract of the optimum TPC and TFC, respectively. Chougui *et al.* (2013) noticed a lower activity varying from 32.3 mg to 51.3 mg AAE/100 g for the red and orange varieties, respectively. Moreover, Chaalal *et al.* (2013), reported that, depending on the variety, the reducing power and DPPH of the ground seeds ranged, from 1861.55 µg AAE/g to 1978.16 µg AAE/g and 891.38 µg AAE/g to 1146.99 µg AAE/g, respectively. These results are lower than those of the current study (Table 5).

Extracts obtained from experiments with optimal conditions for phenolics extraction presented a higher antioxidant activity compared to those from experiments with optimal conditions for flavonoids extraction. It is well established that phenolic compounds exert antioxidant activity (Chougui *et al.*, 2013). This result may imply that the optimal conditions for the extraction of polyphenols have generated more antioxidants in amount and quality, molecules that are efficient and resistant to extraction conditions. While it is known that some flavonoids are of a thermosensitive nature which would suggest the possibility of the lower resistance of these molecules to extraction conditions beyond a certain degree of applied energy, thus affecting the amount and capacity of these molecules in terms of antioxidant activity.

Conclusions

The aim of this investigation was to better study and to compare the optimization parameters of phenolic compounds extraction from *Opuntia ficus indica* seeds and press residue with Microwave Assisted Extraction (MAE) method using Box-

Behnken Design (BBD) of Response Surface Methodology (RSM). The results indicated that the mathematical models used in the present study worked well for the prediction of total phenolic content (TPC) and total flavonoids content (TFC).

It was concluded that the optimal extraction conditions for seeds were: 61 % ethanol concentration, 700 Watt (W) microwave power, 3.82 minutes (min) irradiation time and 46 mL/g Solid-to-Liquid Ratio (SLR) for TPC extraction, and : 50 %, 800 W, 4 min and 50 mL/g for TFC extraction.

The optimal conditions for press residue were: 60 % ethanol concentration, 500 W microwave power, 3.86 min irradiation time and 49 mL/g SLR for TPC extraction, and: 50 %, 800W, 4 min and 50 mL/g for TFC extraction.

The current investigation highlights the efficiency of MAE and its importance as an alternative method of phenolic compounds extraction. It clearly demonstrates that the press residues originating from the oil recovery process to be a phytochemical-rich by-product with a noticeable antioxidant activity. Also, it emphasizes the need for further study to isolate and characterize the phytochemical profile of cactus pear seeds press residue. Thus, *Opuntia ficus indica* by-products as a good and economic source of natural antioxidants could be an alternative exploitable source of ingredients of functional or enriched foods.

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