

ORIGINAL RESEARCH PAPER

**MODELING AND OPTIMIZATION OF PHENOLIC COMPOUNDS  
EXTRACTION FROM PRICKLY PEAR (*OPUNTIA FICUS-INDICA*)  
SEEDS VIA ULTRASOUND-ASSISTED TECHNIQUE**

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Optimal conditions of total phenolic contents (TPC) and antioxidant activity (AA) from *Opuntia ficus-indica* (*OFI*) seeds using ultrasound-assisted extraction (USAE) and response surface methodology (RSM) were investigated. The Box–Behnken design was used to investigate the effects of three independent variables: acetone concentration (40-80%), ultrasonic time (5-15 min) and sonication amplitude (40-100%). The experimental values of TPC and AA under the optimal conditions were  $4.70 \pm 0.13$  mg GAE/g DW and  $2.15 \pm 0.02$  mg GAE/g DW, respectively. High Performance Liquid Chromatography analysis in optimized conditions revealed the presence of 10 and 9 phenolic compounds respectively, which were then identified and quantified. The effect of optimum extraction conditions using USAE on cell surface changes of *OFI* seeds powder was observed by scanning electron microscopy.

**Keywords:** *Opuntia ficus-indica* seeds, phenolic compounds, antioxidant activity, ultrasound, Box-Behnken design

### **Introduction**

Cactus (*Opuntia ficus-indica*, *OFI*), commonly known as prickly pear, belongs to the *Cactaceae* family (Sawaya *et al.*, 1983). Originated from Mexico, it was introduced into the Mediterranean countries at the beginning of the sixteenth century (Inglese *et al.*, 1995). Under optimal conditions, annual production can reach 50 tons of dry matter per hectare (Bensadón *et al.*, 2010). Habibi *et al.* (2005) reported that the amount of seeds is important as it ranges from 20 to 40% dry-weight of the whole fruit, depending on the cultivar.

In recent years, the research has been directed towards natural antioxidants. Indeed, *OFI* seeds were shown to be rich in phenolic compounds (Chaalal *et al.*, 2013). These compounds have received substantial attention due to their beneficial effects on human health, such as a protective action against cancer, cardiovascular, and chronic degenerative diseases, which is attributed to their antioxidant activity (Ranic *et al.*, 2014).

The first step of processing is “extraction”, which involves separation of phytochemicals from the cellular matrix. The “ideal” extraction method must provide high extraction rates and should be time saving and non-destructive. There are various methods for extracting phenolic compounds such as leaching-out extraction (Zhang *et al.*, 2007). Ultrasound-assisted extraction (USAE) is an inexpensive, simple, rapid, effective extraction technique that uses ultrasonic waves to generate cavitations in the solvent, which allows a high penetration of solvent into the raw plant materials (Teng and Choi, 2014). Ultrasound can also reduce the operating temperature allowing the extraction of thermolabile substances (Wang and Weller, 2006). It has been successfully used for the extraction of some bioactive compounds from plant materials (Tomšik *et al.*, 2016). As USAE used low energy and less solvent consumption, and high extraction efficiency, it is preferable to conventional extraction methods (Ying *et al.*, 2011).

The different structure of phenolic compounds can be affected by various extraction parameters such as extraction time, temperature, solvent composition, solid/liquid ratio, and their interactions (Fattahi and Rahimi, 2016; Ilaiyaraja *et al.*, 2015; Wei *et al.*, 2015). Therefore, optimization of the extraction protocol is required. Some authors have carried out optimization by statistical approaches; response surface methodology (RSM) such Box-Behnken design (BBD) is a mathematical and statistical tool to reduce measurements, where it allows to modeling the extraction processes by given a mathematical equation, improving the statistical interpretation possibility and indicating the interaction between variables (Bachir bey *et al.*, 2014; Cheok *et al.*, 2012; Spigno and De Faveri, 2009).

In the present study, the optimization of total phenolic contents (TPC) extraction process and antioxidant activity (AA) using DPPH• from *Opuntia ficus-indica* seeds using ultrasound-assisted extraction (USAE) was performed using Box-Behnken design (BBD). The first step was to study the effects of solvent concentration, amplitude and extraction time for USAE on the extraction efficiency of TPC and AA and the interaction between factors were determined using response surface methodology. Then, the phenolic compounds profile using HPLC–ESI-MS and powders cell change using Scanning Electron Microscopy (SEM) were analyzed to well understand the influence USAE on the *OFI* seed powders.

## Materials and methods

### *Plant material*

The mature prickly pear (*Opuntia ficus-indica*) harvested in Seddouk, Bejaia department, (North of Algeria). Fruits were washed carefully and peeled with

knife. The seeds were removed from the pulp, washed with distilled water, dried in ventilated oven (Binder, Germany) at 40°C during 24 h, then ground, and sieved (diameter of the sieve gate 250 µm). The seed powder had undergone a delipidation in order to remove lipids using Soxhlet (Beher, Germany) during 4 hours using hexane as solvent with ratio 20g: 150 mL, where the oil percentage yield was 8%. Then the *OFI* seed powder was dried again in the same condition. Then stored at 4°C in airtight bags until further use.

#### **Chemical reagents**

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

#### **Ultrasound-assisted extraction (USAE)**

Table 1 represents coded and actual values for Box-Behnken design of ultrasound-assisted extraction (USAE).

One gram of powder seeds was placed in a glass flask containing 25 ml of acetone. The suspension was exposed to acoustic waves using an ultrasonic apparatus (Sonics Vibra cell, VCX 130 PB, U.S.A.), with of frequency 40 kHz and power 130W. Under variations of solvent concentration, irradiation duration and sonication amplitude (Table 2). The extracts were centrifuged at 4500 rpm for 10 min (Nüve, Turkey). The experiments were done in triplicate.

#### **Determination of total phenolic contents (TPC)**

The total phenolic compound content of *OFI* extracts was determined according to Velioglu *et al.* (1998). Extract (1.5 mL) was mixed with 1.5 mL Folin–Ciocalteu reagent. After 5 min, 1.5 mL of sodium carbonate (6%) was added. The mixture was incubated in darkness for 1 hour and then the absorbance was measured at 750 nm (Uvline 9400, Secomam, Alès, France). Gallic acid was used as standard for the calibration curve. The results are expressed as milligrams gallic acid equivalents (GAE) per gram of dry weight (DW).

#### **Determination of antioxidant activity (DPPH radical scavenging assay)**

The scavenging capacity for the radical 1,1-diphenyl-2-picrylhydrazyl (DPPH) was used to determine the antioxidant activity according to Molyneux (2004). An aliquot (200 µL) of the extract was added to 1 mL of methanolic DPPH• solution (60 µM). The decolorization process was estimated at 515 nm after 30 min. The antioxidant capacity was expressed as milligrams gallic acid equivalents (GAE) per gram of dry weight (DW).

#### **Experimental Design and Statistical Analyses**

Influence of process parameters, i.e.  $X_1$ ,  $X_2$  and  $X_3$  were investigated using response surface methodology. The JMP software (Version 10, SAS) was used to establish mathematical models and to obtain the optimal conditions of TPC extraction and AA. In the present study, three-level three-factorial Box–Behnken experimental

design (BBD) were applied to investigate and validate extraction process parameters affecting the extraction of phenolic compounds from *OFI* seeds. 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 three factors chosen in the current study were designated as  $X_1$  acetone concentration (40-80% V/V),  $X_2$  for ultrasonic time (5-15 min), and  $X_3$  for sonication amplitude.

The variables were coded according to the equation 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 the variable;  $X_0$  was the value of  $X$  at the center point and  $\Delta X$  was the step change. The experiments were performed according to the design of experiments shown in Tables 1 and 2. The output results were fitted to a second-order polynomial equation, according to the model in Eq. (3).

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

where  $Y$  represents the responses values (TPC and AA);  $B_0$  is a constant coefficient;  $B_i$ ,  $B_{ii}$  and  $B_{ij}$  are the coefficients of the linear, quadratic, and interaction terms, respectively, and  $X_i$  and  $X_j$  represents the actual independent variables. The regression coefficients of individual linear, quadratic and interaction terms were determined using analysis of variance. In order to visualize the relationship between the response and experimental levels of each factor and to deduce the optimum conditions, the three dimensional response surface plots of ultrasound-assisted extraction (USAE) were generated in Figure 1.

Analysis of variance (ANOVA) was performed for response variable using the full models where  $p$ -values (partitioned into linear and interaction factors) indicated whether the terms were significant or not;  $p$  values < 0.05 were regarded as significant. Fischer's test was used for determination the type of the model equation. The determination coefficient  $R^2$  and adjusted  $R^2$  were also calculated (Table 3).

#### **High-performance liquid chromatography–mass spectrometry (HPLC–ESI-MS) analysis**

The *OFI* seed extracts obtained in the optimal conditions had undergone the solvent evaporation using rotavapor (BÜCHI, Switzerland) and lyophilisation (Freeze Drier Alpha 1–4 LD; Christ, Osterode am Harz, Germany), then the identification of phenolic compounds was obtained by ultrahigh-performance

liquid chromatography–mass spectrometry with electrospray ionization (UPLC–ESI-MS). The equipment was Xevo G2 mass spectrometer consisting of a hexapole, a collision cell and a time of flight analyzer (QTOF) supplied by Waters (Milford, MA, USA). The electrospray probe was used in positive (ESI+) and negative (ESI–) modes as well as sensitivity analyzer mode. The mass range considered was from 10 to 1,000 Da. The corona voltage was 2.5 kV for (ESI+) and 0.5 kV for (ESI–). The sampling cone voltage was optimized between 20 and 50 V. Finally, 30 V was selected for the screening because more peaks were detected. Other MS parameters were as follows: the source temperature was 150 °C, the desolvation gas temperature 450 °C and the desolvation gas flow 650 Lh<sup>-1</sup>. MSE mode was selected for the acquisition, and collision ramp energy from 5 to 40 V was used. MassLynx v.4.1 software (Waters, Milford MA, USA) was used to analyze the samples and CromaLynx (Waters, Milford MA, USA) was used to deconvolve the spectra. Figure 2 and Table 4 shows the phenolic profile from *OFI* seeds in optimum extract. Quantitative data for *OFI* seed phenolic compounds were obtained by calibration curves obtained from known standards. Both calibration curve equation and determination coefficient of standards were as follows:

- protocatechuic acid ( $y=73.504x+697.780$ ,  $R=0.989$ );
- lupin isoflavone ( $y=186.471x+143$ ,  $R=0.998$ );
- ellagic acid ( $y=159.252x+325.781$ ,  $R=0.969$ );
- isohamnetin ( $y=100.920x$ ,  $R=0.999$ );
- myricetin ( $y=1731.624x-18.247$ ,  $R=0.998$ );
- Chlorogenic acid ( $y=2001.198x$ ,  $R=0.999$ );
- ferulic acid glucoside ( $y=5198.875x-35.984$ ,  $R=0.997$ );
- isorhamnetin 3-O-(6"-O-feruloyl)-glucoside ( $y=5036x-168.47$ ,  $R=0.999$ );
- ferulic acid 4-glucuronide ( $y=6046x-44.89$ ,  $R=0.997$ );
- 1,2 Di-O-sinapoyl glucose ( $y=1287.2x-33.42$ ,  $R=0.999$ ).

#### **Scanning electron microscopy (SEM) analysis**

In order to well understand the influence USAE on the *OFI* seed powders cell change. The scanning electron microscopy (SEM) analysis (Quanta 200, FEI company) was carried out for *OFI* seed powders before and after USAE under the optimal conditions of both TPC and AA as shown in Figure 3. The powdered samples of USAE residues were collected and dried until constant mass in an oven at 50°C before SEM analysis. The samples were fixed on the specific carbon film substrate, and their shape and surface characteristics were observed by using GSED detector in environmental mode (ESEM).

## **Results and discussion**

### **Optimization of USAE**

According to the results shown in Table 2, the total phenolic compound contents and antioxidant activity of prickly pear seeds varied from 2.39 to 4.47 mg GAE/g DW and from 1.16 to 2.09 mg GAE/g DW, respectively, confirming the influence of the optimized parameters (solvent concentration, sonication time and radiation amplitude). Table 2 shows that the experimental data are not far from the predicted

values. This table shows also that the experimental conditions which give an extract which have the best antioxidant activity (run 14), does not give the highest total phenolic content. This justifies the importance of studying both TPC and AA.

**Table 1.** Coded and actual values for Box-Behnken design of ultrasound-assisted extraction (USAE)

Factors		Coded levels		
		-1	0	+1
$X_1$	Acetone concentration (% V/V)	40	60	80
$X_2$	Sonication time (min)	5	10	15
$X_3$	Sonication amplitude (%)	40	70	100

-1 (low), 0 (center point) and +1 (high), were the code of factor levels

**Table 2.** Box–Behnken design matrix (in coded level of three variables) experimental data and predicted values for total phenolic contents and antioxidant activity in *OFI* extracts using ultrasound-assisted extraction (USAE)

Run	TPC (mg GAE/g DW)			AA (mg GAE/g DW)	
	Pattern $X_1X_2X_3$	Experimental data	Predicted data	Experimental data	Predicted data
1	000	4.42	4.43	2.04	2.03
2	+0	2.84	2.89	1.45	1.46
3	000	4.47	4.43	2.01	2.03
4	0--	2.39	2.45	1.19	1.21
5	+0-	3.45	3.32	1.51	1.48
6	0++	3.53	3.46	1.98	1.96
7	-0-	2.40	2.33	1.21	1.25
8	000	4.40	4.43	2.04	2.03
9	0+-	3.71	3.65	1.74	1.77
10	-0+	3.20	3.32	1.42	1.45
11	--0	2.40	2.33	1.16	1.10
12	--0	3.35	3.29	1.42	1.41
13	++0	3.29	3.35	1.83	1.89
14	+0+	3.32	3.32	2.09	2.05
15	0+-	4.00	4.05	1.77	1.74

As shown in Table 3, the F values of both TPC and antioxidant activity models were high, suggesting that variations of response measured were due to factors affecting. This result was confirmed by the low value of  $p < 0.05$  ( $p = 0.0002^*$  and  $0.0001^*$  respectively); hence, both models were significant. Lacks of fit of both models were insignificant ( $p > 0.05$ ), confirming the model validity.

In the present study, the determination coefficients ( $R^2$ ) of the models were of 0.99; the values of the adjusted determination coefficients ( $_{\text{adjusted}} R^2$ ) were of 0.97 and 0.98 for TPC and AA respectively. These findings showed a close agreement between the experimental results and the theoretical values envisaged by the polynomial models.

The fitted mathematical models for TPC and antioxidant activity (AA) were given in Eqs. 4 and 5, respectively.

$$\text{TPC} = -13.303 + 0.2920X_1 + 0.8450X_2 + 0.114X_3 - 0.0003X_1X_3 - 0.0030X_2X_3 - 0.0021X_1^2 - 0.0250X_2^2 - 0.0005X_3^2 \quad (4)$$

$$\text{AA} = -3.664 + 0.0980X_1 + 0.2390X_2 + 0.0240X_3 + 0.0002X_1X_3 - 0.0004X_2X_3 - 0.0008X_1^2 - 0.0090X_2^2 - 0.0002X_3^2 \quad (5)$$

**Table 3.** Analysis of variance (ANOVA) for the fitted quadratic polynomial models

		USAE	
		TPC	AA
<b>DF</b>	Model	9	9
	Lack of fit	3	3
	Pure error	2	2
<b>Sum of Squares</b>	Model	6.440	1.587
	Lack of fit	0.059	0.013
	Pure error	0.002	0.006
<b>F value</b>	Model	58.108	63.552
	Lack of fit	15.122	14.750
<b>p value</b>	<b>Model</b>	0.0002*	<0.0001*
	<b>Lack of fit</b>	0.0627	0.0642
	<b>R<sup>2</sup></b>	0.99	0.99
	<b>Adj. R<sup>2</sup></b>	0.97	0.98

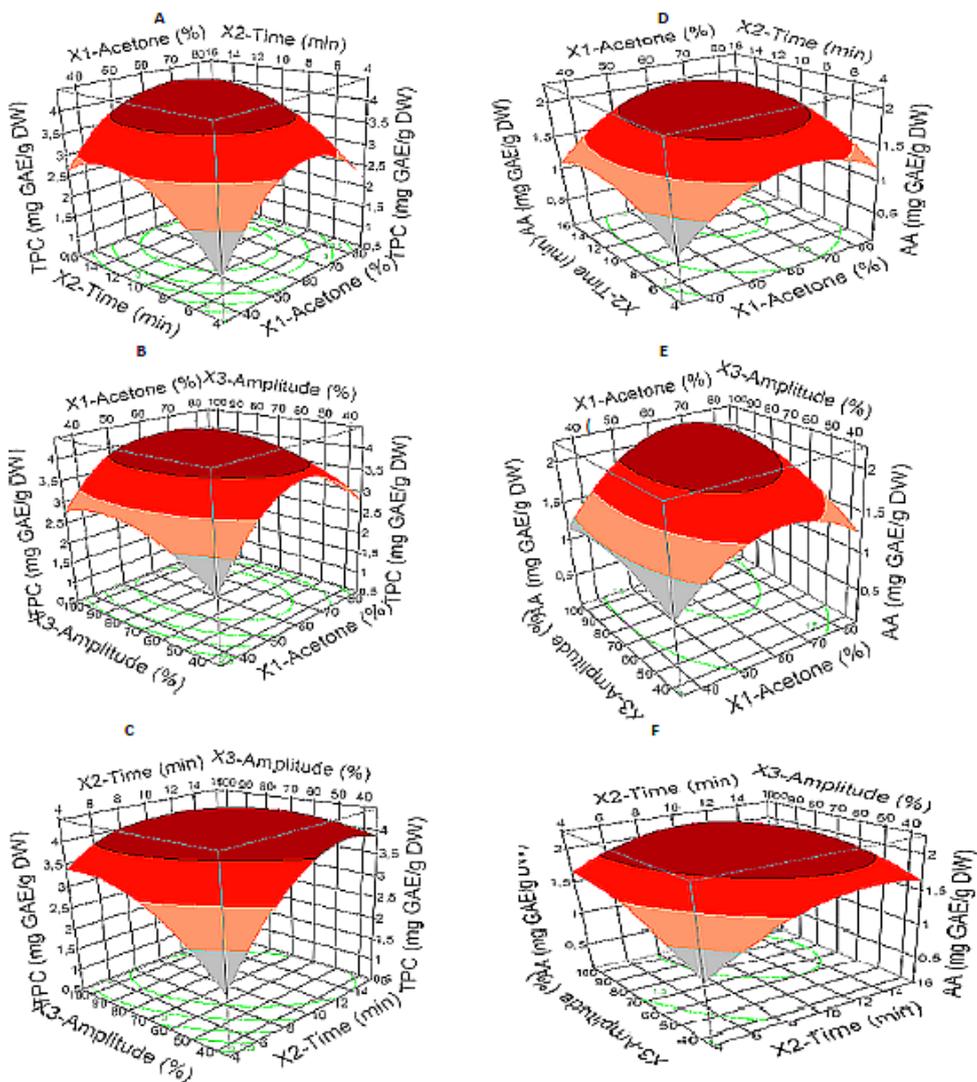
The equations 4 and 5 showed that the three linear factors were significant at  $p < 0.05$  level for TPC extraction and antioxidant activity. Regarding interactions between factors and quadratic effect, acetone concentration-amplitude, time-amplitude interactions and the three quadratic factors affected significantly ( $p < 0.05$ ) both TPC extraction and antioxidant activity. Furthermore, generally the linear factors influence positively the both responses TPC and AA, while interaction factors and quadratic influence negative these responses.

The results of optimized conditions of ultrasound-assisted extraction (USAE) were as follows: 61.37%, 11.37 min, 70.61% and 68.54%, 11.42 min, 93.44% for ( $X_1$ ,  $X_2$  and  $X_3$ ) TPC and AA respectively, where their predicted and experimental responses were 4.49 and 2.17;  $4.70 \pm 0.13$  and  $2.15 \pm 0.02$  mg GAE/g DW. This result shows that the experimental and the predicted responses were close. This suggests that the models could function well for the forecast of antioxidant extraction by USAE from the prickly pear seeds.

### Response surface plots

Figure 1 presents a three-dimensional response surface for the independent variables (acetone concentration, sonication time and amplitude). Summarizing, total phenolic content (Figure 1 A, B and C) and antioxidant activity of the extracts (Figure 1(D, E, and F) revealed a similar trend for the three response surfaces. Figure 1 (A and D) and showed that increase of acetone concentration increased the TPC extraction and antioxidant activity. The polarity played an important role in

antioxidant extraction. Hence, the extraction of this latter depends on their solubility in solvent. Water was a polar solvent with 78.3 as dielectric constant at 20°C. While acetone was less polar solvent than water with 20.7 as dielectric constant at the same temperature. The increase of solvent concentration caused a decrease in its polarity, which favored the extraction of less polar components (Chok *et al.*, 2012).



**Figure 1.** Response surface plots showing the effects of acetone concentration, extraction time and sonication amplitude on TPC extraction (A, B and C) and antioxidant activity (D, E and F) from prickly pear seeds.

In addition, the sonication duration is another significant parameter in the USAE; where the extraction of bioactive compounds increased with the sonication time up

to a certain limit (Fan *et al.*, 2012). During sonication, the cavitation process causes the swelling of cells or the breakdown of cell walls, which allow high diffusion rates across the cell wall (Khan *et al.*, 2010).

Figure 1 (B and E) allows still tracking that low and high acetone concentrations and sonication amplitude led to a decrease both TPC and antioxidant activity. The results indicated that initially, the extraction efficiency was improved by sonication amplitude increasing, but decreased at a high sonication amplitude. The enhancement in extraction obtained by using ultrasound was mainly attributed to the effects of acoustic cavitations produced in the solvent by the passage of the ultrasonic waves. Ultrasounds also exert a mechanical effect, allowing a better penetration of solvent into the vegetal matrix, increasing the contact surface area between solid and liquid phase, and improve the mass transfer rate of target compounds into the extraction medium. Hence, the solute quickly diffuses from solid phase to the solvent (Toma *et al.*, 2001; Wang *et al.*, 2008). As regards the interactions between sonication time and amplitude, Figure 1 (C and F) shows that both TPC and the DPPH• scavenging effect increased with increasing time-amplitude. According to Jerman *et al.* (2010), increasing of radiation amplitude produced a great number of cavitations bubbles and therefore improved extraction efficiency of present in the sample.

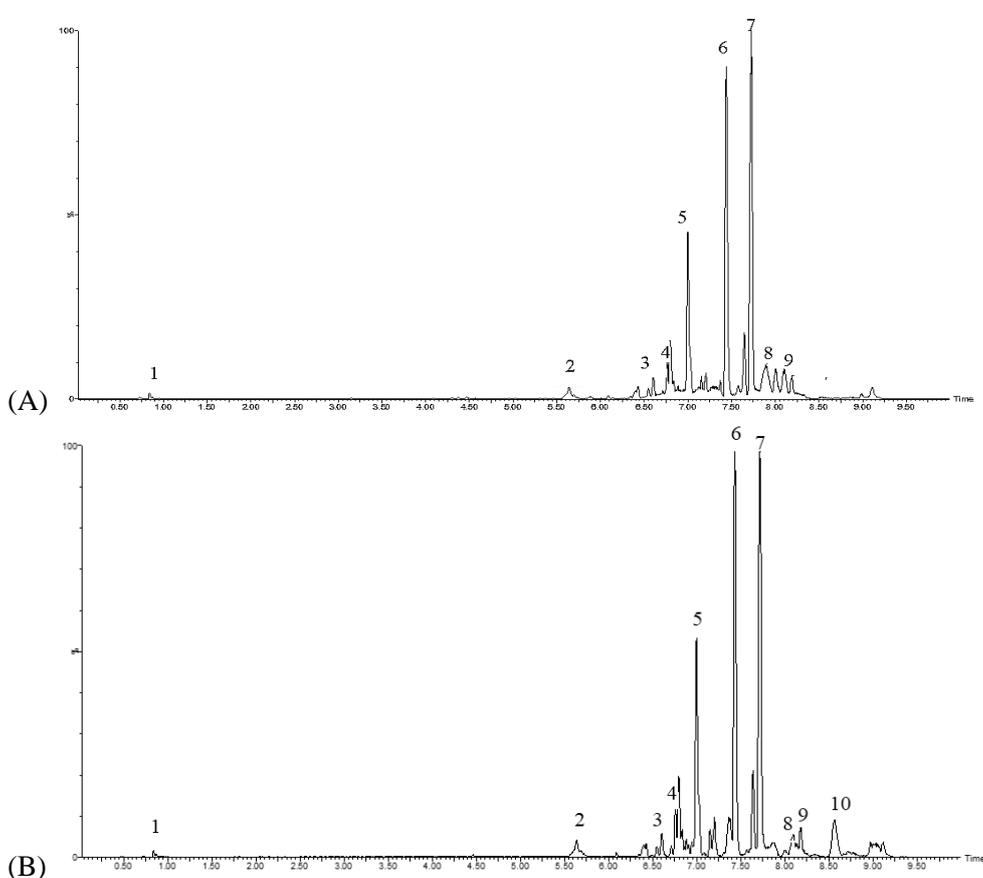
#### ***Phenolic compounds profile and powders cell surface of the optimized extract***

The two *OFI* seeds optimum (TPC and AA) were compared on the phytochemical compounds profile and cell change.

**Table 4.** Phenolic profile of *Opuntia ficus-indica* seed at antioxidant activity (AA) and total phenolic contents (TPC) optimum.

Phenolic compound	ID-Cod	M-H	optimal AA		optimal TPC
			Retention time (min)	Concentration (µg/g)	Concentration (µg/g)
Protocatechuic acid	72	153	0.94	2.108±0.02	2.144±0.01
Lupiniso flavone	5319901	351	5.64	60.165±0.10	64.419±0.12
Ellagic acid	5281855	301	6.43	35.501±0.68	6.010±0.01
Isohamnetin	15817847	315	6.76	74.379±0.04	73.741±0.56
Myricetin	5281672	317	6.80	428.141±0.81	197.189±0.06
Chlorogenic acid	1794427	353	7.44	1148.409±1.01	885.310±0.03
Ferulic acid glucoside	13916049	355	7.72	1366.242±0.05	96.327±0.92
Isorhamnetin 3-O-(6"-O-feruloyl)-glucoside)	90657785	653	7.89	288.584±0.99	67.142±0.17
Ferulic acid 4-glucuronide	6443140	369	8.19	68.807±0.05	67.142±0.04
1,2 Di-O-sinapoyl glucose	5280665	591	8.57	-	174.823±0.48

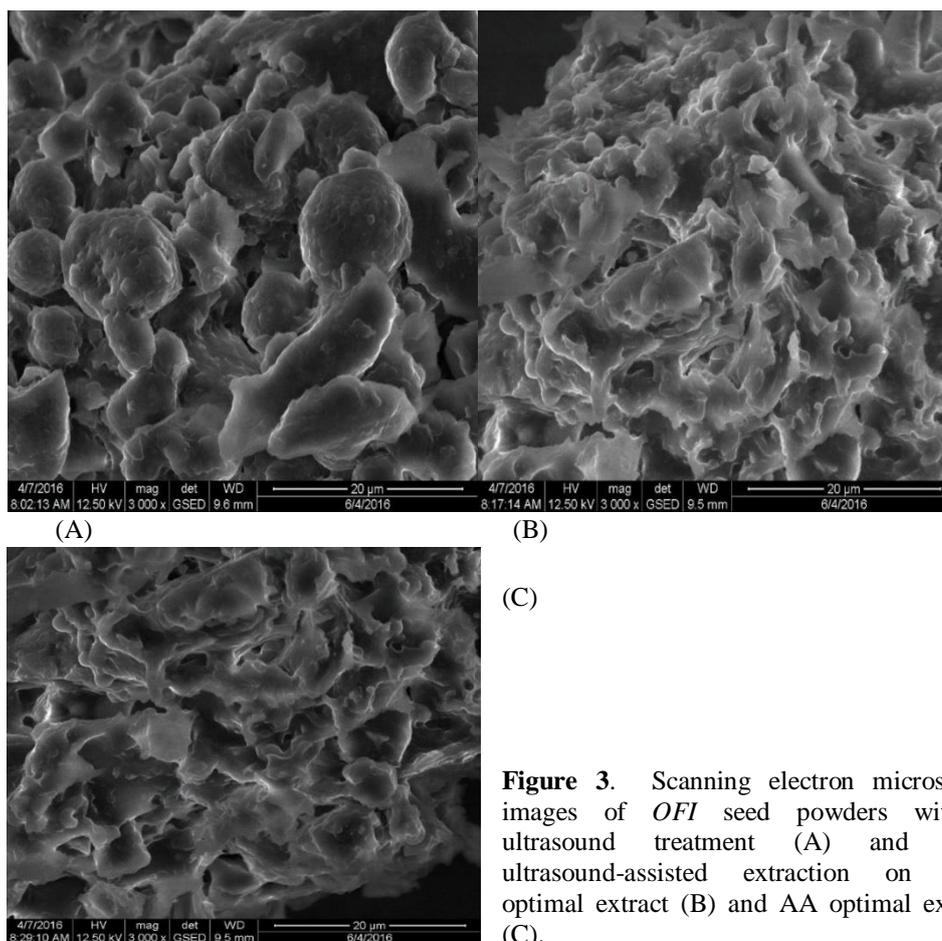
Figure 2 and Table 4 show the phenolic profile of *OFI* seed extracts under TPC and AA optimal conditions. Except 1,2 Di-O-sinapoyl glucose, the two extracts have the same profile, this result could be attributed to the close condition such as shows previously (Optimization of USAE part). However, the amount was different. Most phenolic compounds in antioxidant activity under optimal conditions exhibit a higher amount than TPC optimal extract. Moreover, ferulic acid glucoside was a dominant *OFI* seed compound. However, the phenolic compounds identify by HPLC represent approximately 35% of the total phenolic measured by spectrometric method.



**Figure 2.** HPLC profile of *OFI* seeds for (A) AA USAE, (B) TPC USAE extract. (1) Protocatechuic acid; (2) Lupinisoflavone ;(3) Ellagic acid (4) Isohamnetin ; (5) Myricetin ; (6) Chlorogenic acid ; (7) Ferulic acid glucoside; (8) Isorhamnetin 3-O-(6''-O-feruloyl)-glucoside) ; (9) Ferulic acid 4-glucuronide; (10) 1,2 Di-O-sinapoyl glucose.

Figure3 shows cell surface changes of *OFI* seed powders observed by scanning electron microscope (SEM) before (A) and after ultrasound-assisted extraction of TPC optimal extract (B) and AA optimal extract (C). The SEM results show big differences before and after USAE. However, no difference between the two

USAE extracts (TPC and AA). Figure 3 shows clearly that ultrasound assisted extraction caused serious and considerable damage to the cell structure surface of *OFI* seed powder. It is well known that ultrasounds disrupted vegetal cells via cavitation bubble collapse produced by the passage of an ultrasonic wave (Kong et al., 2010). This way, a maximum compound of the internal cells would be extracted.



**Figure 3.** Scanning electron microscope images of *OFI* seed powders without ultrasound treatment (A) and after ultrasound-assisted extraction on TPC optimal extract (B) and AA optimal extract (C).

## Conclusion

*Opuntia ficus-indica* seeds can be considered as an interesting source of natural antioxidants. Implemented response surface methodology was useful in terms of reducing time. The applied of second-order polynomial models gave a satisfactory description of the experimental data, showing that acetone concentration, extraction time, and sonication amplitude affected significantly both TPC extraction and antioxidant activity of USAE. This study provides that ultrasound-assisted extraction process is an effective technique for natural bioactive compound extraction and antioxidant activity. Ultrasonic wave disrupted *OFI* seed cells and

allowed the extraction of 9 and 10 compounds on AA and TPC extracts, respectively. Ferulic acid glucoside was a dominant *OFI* seed compound. The presented data can be used as a guideline for establishing full-scale, cost-effective and resource-effective food industry. Further studies may determine the possibility to use *OFI* seeds as an additive in the food industry and cosmetic sector.

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