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Optimization of torrefaction conditions on antioxidant activity of prickly pear seeds extract using response surface methodology and chemometric analysis

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ABSTRACT

The prickly pear (*Opuntia ficus-indica* L., Cactaceae) seeds can be used as an alternative food product by the torrefaction process. Torrefaction is a pre-treatment process of biomass into solid fuel within a temperature range of 60–200 °C. This study aimed to investigate the effect of torrefaction parameters on the antioxidant potential of prickly pear seeds, which is considered a food by-product, by using the Central Composite Design approach (CCD).The best torrefaction parameters found to have the optimal phytochemical contents were: 200 °C and 50 min with 104.86 ± 1.94 GAE/g extract for Total Phenolic Content (TPC), 200 °C and 50 min with 81.23 ± 0.90 mg QE/g extract for Total Flavonoid Content (TFC), 200 °C and 50 min with IC_{50} = 90.66 ± 2.09 μg/ml for free radical scavenging activity (DPPH assay), 200 °C and 50 min with IC_{50} = 124.9 ± 4 μg/ml for radical cation total antioxidant capacity (ABTS assay). Our results demonstrated an increase of antioxidant activities with the increase of torrefaction parameters. Therefore, torrefaction has proven to be a value-added process to exploit prickly pear seed biomass and use it as a source of antioxidant food additives.

1. Introduction

Prickly pear (*Opuntia ficus-indica* L.) is a spiny, succulent shrub native to Central America where it is used as an essential dietary nutrient by the indigenous populations [1,2]. This plant belongs to the Cactaceae family and finds its natural habitat especially in the arid (Africa) and semi-arid continents (South and Central America) [3]. Moreover, biomass (*Opuntia ficus-indica*L) is rich in antioxidant compounds such as flavonoids and tannins, and fatty acids, as well as macro- and micronutrients such as sugars, pigments, and vitamin C; for that, it is recommended in human food [4,5]. A portion of these chemical antioxidants can be found in the seeds, which constitute approximately 3–15 % of the flesh of prickly pears [6]. These seeds are frequently deemed waste subsequent to pulp processing [7]; [8]. The seeds are high in oil (98.8 g/kg) as well as linoleic and oleic acids [7]; [6], along with other compounds such as phenols [9]. Presently, seed oils are utilized as natural food preservatives as a result of their antibacterial and antioxidant properties [10], and they might also offer health benefits to humans [11]. The torrefaction is an important pre-treatment that is done before extraction of bioactive compounds because it can modify the nutritional value as

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well as the texture and color of food biomass [12,13]. Moreover, torrefaction can influence the antioxidant activity due to the tothermal degradation of phytoconstituents and the formation of Maillard reaction products (MRP) [14,15].Various studies indicated that the antioxidant compounds increased during torrefaction [16,17] [18]; [19]. Chandrasekara et al. [20] found that elevated torrefaction temperatures led to an increase in phenolic content. Vujasinovic et al. [21] demonstrated the roasting of naked pumpkin seeds (90,110,130 °C) led to a significant increase in the content of phospholipids (from 0.005 to 0.463 %), total phenolic compounds (from 4.63 to 19.60 mg/kg) and total tocopherols (from 265.79 to 350.98 mg/kg) in the oil. In addition, Cisneros et al. [22] conducted a study on sacha inchi oil, finding that roasting improved its oxidative stability and antioxidant capacity by promoting the formation of phenolic compounds. Moreover, Yu et al. [23] found that torrefaction at 175 °C for 5 min increased the Total Phenolic Content (TPC) in both water and ethanol extracts from peanut skin by 35.9 % compared to the raw sample. Additionally, Locatelli et al. [24] indicated that the TPC from hazelnut skin extracts increased after heating at 180 °C for 20 min. And, Kim et al. [25] showed a significant increase (p-value <0.05) in total phenolic content (TPC) with heat treatment.

In the present study, Response Surface Methodology (RSM) was used to optimize the effect of torrefaction on the antioxidant activities of biomass from prickly pear seeds. RSM is a statistical method useful for optimization, based on Central Composite Design (CCD). It aims to establish an empirical model to optimize operational conditions during torrefaction (time and temperature) to enhance the antioxidant activities of biomass. This approach offers the advantage of requiring fewer experimental trials to evaluate the effects of several factors and their interactions [26]. The purpose of this study was to develop a torrefaction model applicable to the extraction of antioxidant compounds from prickly pear seed biomass. To achieve this, the Central Composite Design (CDD) approach was applied to evaluate the impact of torrefaction treatment (temperature and time) on maximizing the content of antioxidant compounds in terms of Total Phenolic Content (TPC), Total Flavonoid Content (TFC), and Total Condensed Tannins content (TTC), as well as the antioxidant activity (measured by DPPH and ABTS radical scavenging). Finally, Principal Component Analysis (PCA) was used to evaluate the correlations between the phenolic contents and their antioxidant activities.

2. Materials and methods

2.1. Main raw biomass, chemicals and reagents

The biomass material, composed of prickly pear seeds, was collected between June and July 2019 in Taza city, located in the north-eastern Morocco. The chemicals and reagents used in this study are: Ascorbic acid ($C_6H_8O_6$, Molar Mass: 176.12 g/mol, Density:1.65 g/cm^3, Solubility: Highly soluble in water (330 g/L at 20 °C), Purity: 99 %), 2-thiobarbituric acid (TBA) ($C_4H_4N_2O_2S$, Molar Mass: 144.15 g/mol, Density: 1.43 g/cm^3, Solubility: Sparingly soluble in water, Purity: ≥98 %), aluminum chloride ($AlCl_3$, Molar Mass: 133.34 g/mol, Density: 2.48 g/cm^3, Solubility: Soluble in ethanol, chloroform, and carbon tetrachloride, Purity: 99.999 % trace metals basis), 2,2-diphenyl-1-pycridazil ($C_{18}H_{12}N_5O_6$, Purity: 90 %, Molar Mass: 394.32 g/mol, Density: 1.4 g/cm^3, Solubility: Soluble in organic solvents such as ethanol, methanol, and acetone), 2,2′-azino-bis (3-ethyl benzthiazoline–6-sulphonic acid) ($C_{18}H_{18}N_4O_6S_4$, Molar Mass: 548.68 g/mol, Solubility: Highly soluble in water. Purity: ≥98 % (HPLC)), sodium carbonate (Na_2CO_3, Molar Mass: 105.99 g/mol, Density:2.54 g/cm^3 (anhydrous), Solubility:Soluble in water, forming a mildly alkaline solution.), sodium hydroxide solution (NaOH, 50 % solutions, Density: 1.53 g/cm^3 at 20 °C, Solubility: Completely miscible with water, producing a highly exothermic reaction Purity: ≥99.5 %), methanol (CH_3OH, Appearance: Colorless liquid, Density: 0.792 g/cm^3 at 20 °C, Boiling Point: 64.7 °C, Solubility: Completely miscible with water and most organic solvents, Purity:99.8 %), Potassium phosphate dibasic (K_2HPO_4, crystalline powder, Molar Mass:174.18 g/mol, Density: 2.44 g/cm^3, Solubility: Soluble in water. Purity: ≥98.0 %), Phosphate de potassium monobasic (KH_2PO_4, Apparence: Poudre cristalline blanche, Masse Molaire: Environ 136.09 g/mol, Densité: Environ 2.34 g/cm^3, Solubilité: Soluble dans l'eau, Purity ≥99.0 %), ethylenediamine tetra acetic acid (EDTA)($C_{10}H_{16}N_2O_8$, appearance: usually as a white crystalline powder, Molar mass: 292.24 g/mol, Solubility: Soluble in water, Purity: 99.4–100.6 %). sulfuric acid (H_2SO_4, Appearance: Colorless to slightly yellow viscous liquid, Molar Mass 98.08 g/mol, Density: 1.84 g/cm^3 for concentrated sulfuric acid (98 %), Solubility: Highly soluble in water, with strong exothermic reaction, Purity: 99.999 %), and phenol (C_6H_5OH, Appearance: Colorless to white crystalline solid, Molar Mass: 94.11 g/mol, Density: Around 1.07 g/cm^3 for liquid phenol, Solubility: Soluble in water to some extent, miscible with organic solvents such as ethanol and ether), and potassium persulphate ($K_2S_2O_8$, Appearance: White crystalline powder, Molar Mass: 270.32 g/mol, Density: 2.477 g/cm^3, Solubility: Soluble in water, forming a clear solution, Purity: ≥99.0 %), Folin–Ciocalteu's phenol reagent (Folin–Ciocalteu's phenol reagent is a mixture of phosphomolybdic and phosphotungstic acids, which undergo a redox reaction with phenolic compounds.), sodium acetate (CH_3COONa, Molar Mass:82.03 g/mol, Density: 1.528 g/cm^3, Solubility: Soluble in water, Purity: ≥99.0 %), ethanol (C_2H_5OH, Molar Mass: 46.07 g/mol, Density: 0.789 g/cm^3, Boiling Point: 78.37 °C, Solubility: Miscible with water in all proportions, ferrous sulfate (C_2H_5OH, Molar Mass: 46.07 g/mol, Density: 0.789 g/cm^3, Boiling Point: 78.37 °C, Solubility: Miscible with water in all proportions), ferric chloride($FeCl_3$, Molar Mass: 162.2 g/mol for the anhydrous form, Density:2.9 g/cm^3 for the anhydrous form, Solubility: Highly soluble in water), and 2,4,6-Tris (1-pyridyl)-5-triazine (TPTZ)($C_{15}H_{12}N_6$, Molar Mass: 276.3 g/mol, Melting Point: 163–165 °C, Solubility: sparingly soluble in water) were purchased from.Sigma-Aldrich Co. (St. Louis, MO, USA).

Table 1
Levels of variables of the roasting condition by Central Composite Design approach(CCD).

Extraction process	Independent variables	Level		
		(-1)	(0)	(+1)
Maceration process	X_1 torrefaction temperature (°C)	60	130	200
	X_2 torrefaction time (min)	10	30	50

2.2. Torrefaction and extraction of seed biomass and experimental design

Once the biomass (*Opuntia ficus-indica* L) was collected, the seeds were isolated and then dried in the dark at room temperature for 3 days.

The torrefaction of biomass was done on forced hot-air convection according to the following protocol. Conventional roasting: seeds were torrefied at approximately 60–200 °C for 10–50 min in a traditional oven and roaster (Table 1).

The seeds were gently stirred with the sand to achieve homogeneous heating. The roasted kernels were immediately sieved out of the hot sand and cooled. After sifting the hot sand, the torrefied seeds were cooled and kept at 4 °C in polyethylene bags until needed for a maximum period of 1 week. After that, the roasted seeds have been ground into powder in a swing-type electric grain miller and sieved through a 35-μm,. Then, 40 g of roasted seeds were macerated with the ethanol solvent for two days. After that, the solvent was evaporated using a rotary evaporator (Rotavapor R-100, BUCHI, Boiling point 79 °C at 1013 mbar), and the ethanolic extract (seed extract) was stored at −4 °C for further analysis.

A Central Composite Design approach (CCD) was used to optimize the effects of torrefaction factors on the responses TPC, TFC, TTC, ABTS (IC_{50}), and DPPH (IC_{50})) of the model's design (Y). This approach consisted of 11 experimental assays with 3 levels (−1, 0, and +1) of independent variables (torrefaction temperature and torrefaction time) (Table 1). The regression coefficients (β) were calculated by the

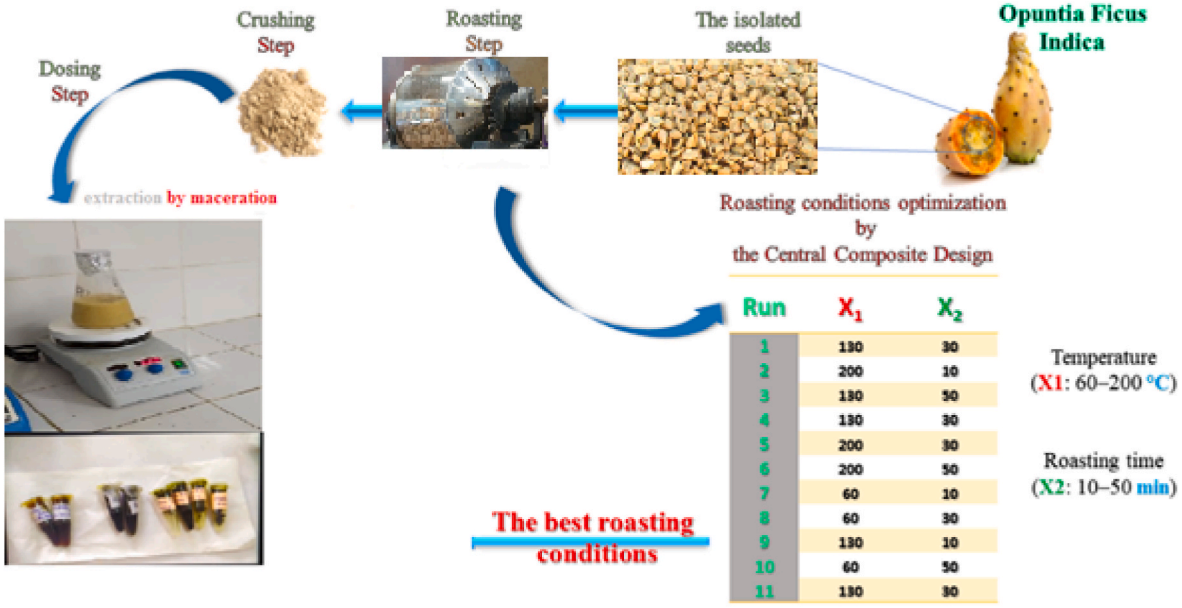

Fig. 1. Raw material, equipment, and work process.

adjustment of the experimental data to a second-order polynomial model; this model was used for response surface analysis as follows:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j \quad \text{(Eq 1)}$$

Where Y is the response variable, Xi and Xj are the independent variables. β0 (constant), βi (linear), βii (quadratic), and βij (interactive) are the regression coefficients of the model. JMP11 (SW) software was used to determine these coefficients by analysis of variance (ANOVA). The estimation of the adequacy of the polynomial equation to the responses was done by the *p*-value of the model, coefficient of determination (R^2), coefficient of adjustment (R^2adj), and *p*-value of lack of fit. The desirability function was applied for multi-response optimization [27,28].

2.3. Determination of total phenolic content (TPC)

The TPC of seed extracts was determined by the method of El Idrissi et al. [29]. Briefly, 200 µL of extract were added to 1.5 mL of Folin Ciocalteu reagent (10 %). After 4 min, 1.5 ml of 5 % sodium carbonate (Na_2CO_3) were added to the solution. The tubes were placed in darkness for 2 h. Gallic acid was used as a standard for the calibration curve. The results were read at 750 nm, using an LLG-uniSPEC 2 spectrophotometer (LLG Labware, Meckenheim, Germany). The concentration of TPC, expressed in mg equivalent of a gallic acid per g of extract (mg GAE/g extract), was deduced according to the calibration interval established with gallic acid (0–100 µg/mL).

2.4. Determination of Total Flavonoid Content (TFC)

The TFC was quantified according to the method of El Moudden et al. [30]. One ml of extract was added to 1 ml of a solution of $AlCl_3$ (2 % in methanol). Then, the absorbance was read at 430 nm after 10 min of reaction. The TFC was obtained using a linear regression equation deduced from the calibration curve and expressed in mg equivalent of quercetin per g of extract (mg QE/g extract). The quercetin calibration range was 0–100 µg/mL.

2.5. Determination of Total Condensed Tannins Content (TTC)

The TTC was determined following the method of Eddahhaouiet al. [31]. The extract was diluted in ethanol, then, fifty mL of extract (50–600 mg/mL) were added to 3 mL of 4 % methanolic vanillin solution and 1.5 mL of H_2SO_4. After 15 min, the absorbance was read at 430 nm. TTC was presented as mg catechin equivalent (CE) per g of extract (mg CE/g extract).

2.6. Free radical scavenging activity (DPPH assay)

The antioxidant activity of seed extracts was evaluated according to the method described by Li et al. [32]. The extract was diluted in ethanol, then,one ml of each extract (50–1000 µg/mL) was added to 1 mL of a methanolic solution of DPPH at 0.1 mM and incubated for 30 min at 27 °C. DPPH and methanol were used as controls. The absorbance was read at 517 nm. The antioxidant activity was obtained by the following equation: inhibition (%) = 1_ [$A_{sample}/A_{control}$] × 100, where A_{sample} and $A_{control}$ were the absorbances of the sample and control, respectively.

2.7. Radical cation total antioxidant capacity (ABTS assay)

The radical cation capacity of extracts was quantified according to the method of Yim et al. [26]. Eighty µL of 140 mM potassium persulfate (K_2S2O_8) were mixed with 5 mL of 7 mM ABTS solution (diluted in ethanol). Then, the mixture was stored in the dark at room temperature for 16 h. The absorbance of the ABTS-containing mixture was adjusted with ethanol to 0.70 ± 0.05 at 734 nm. Ten µL of extracts at different concentrations were added to 1 ml of ABTS reagent (100–1000 µg/mL). The absorbance was read against the blank reagent at 734 nm. The inhibition capacity was quantified according to the following equation: inhibition (%) = 1_ [$A_{extract}/A_{control}$] × 100, where $A_{extract}$ and $A_{control}$ were the absorbances of the extract and control, respectively.

2.8. Principal Component Analysis (PCA)

The correlations between the responses (TPC, TFC, TTC, ABTS ($1/IC_{50}$), DPPH ($1/IC_{50}$)) and the 11 extraction runs were established by PCA. This technique allowed to graphiucally represent the variation of responses from the 11 extraction runs according to the nature of torrefaction factors [33]. The softwares used in this study were: JMP 11 (SW) (CCD approach), and XLSTAT 2014 (PCA); the software IBM SPSS Statistics 21 was applied to express the data in means ± standard error of the mean and for the signification of data (Tukey test and student t-test

Table 2
Experimental design and results of TPC, TFC, TTC, and antioxidant activity by DPPH and ABTS assayson prickle pear seed extracts.

Run	X_1 (°C)	X_2 (min)	TPC	TFC	TTC	DPPH IC_{50}	ABTS IC_{50}
1	130	30	23.43 ± 1.23^{a}	53.71 ± 0.90^{ah}	6.8 ± 0.20^{a}	217.34 ± 2.96^{a}	410.62 ± 4.70^{a}
2	200	10	46 ± 0.56^{b}	49.14 ± 1.04^{h}	1.2 ± 0.30^{b}	149.72 ± 2.89^{b}	287.76 ± 2.09^{b}
3	130	50	41.85 ± 1.05^{b}	70.21 ± 0.79^{b}	4.6 ± 0.70^{ae}	144.10 ± 3.03^{b}	320.83 ± 4.93^{c}
4	130	30	24.27 ± 1.20^{a}	57.21 ± 1.20^{a}	6.4 ± 0.95^{a}	212.93 ± 4.03^{a}	402.92 ± 3.98^{a}
5	200	30	59.86 ± 0.94^{d}	56.34 ± 0.96^{a}	1 ± 0.17^{b}	120.30 ± 2.18^{d}	240.013 ± 3.997^{d}
6	200	50	104.86 ± 1.94^{e}	81.23 ± 0.90^{c}	1.4 ± 0.23^{b}	90.663 ± 2.093^{e}	124.9 ± 4^{e}
7	60	10	21.57 ± 1.07^{af}	28.86 ± 1.10^{d}	0.4 ± 0.05^{b}	458.69 ± 3.70^{f}	624.736 ± 3.11^{f}
8	60	30	15.57 ± 1.10^{f}	30.43 ± 0.43^{de}	1.8 ± 0.33^{be}	375.92 ± 4.98^{g}	471.67 ± 2.90^{g}
9	130	10	20.71 ± 0.71^{af}	34.43 ± 1.03^{e}	2.1 ± 0.30^{be}	290.47 ± 3.79^{h}	546.48 ± 3.21^{i}
10	60	50	15.42 ± 1.02^{f}	63.29 ± 0.90^{g}	2.2 ± 0.60^{be}	265.94 ± 4.02^{i}	367.54 ± 2.10^{j}
11	130	30	26.23 ± 1.20^{a}	57.89 ± 1.09^{a}	6.1 ± 0.90^{a}	215.92 ± 3.02^{a}	413.97 ± 4.02^{a}

Data are presented as the average values ± standard deviation of the two replicates (mean ± SD, n = 2).the means followed by similar The mean followed by similar letters exposing in the column are not different ($P < 0.05$).TPC (mgGAE/gextract); TFC(mgQE/gextract); TTC(mgCE/gextract); DPPH IC_{50} (μg/ml); ABTS IC_{50} (μg/ml); X_1torrefactiontemperature(°C); X_2torrefaction time(min).
Total Phenolic Content (TPC); (Total Flavonoid Content (TFC); Total Condensed Tannins Content(TTC); free radical scavenging activity (DPPH assay); radical cation total antioxidant capacity (ABTS assay).

at *p*-value = 0.05) [34]. Hierarchical cluster analysis (HCA) was used to analyze the interconnectivity of all extracts into clusters based on determined bioactive component properties. Furthermore, the XLSTAT 2014 program generates dendrograms to depict the commonalities among the examined extracts. Square Euclidean measurements were used to calculate similarities between extracts. The gaps between clusters are calculated using an analysis of variance approach.

3. Results and discussion

Conventional roasting is a widely used method for the heat treatment of various types of food products. The process involves subjecting the raw material to controlled heat in order to obtain specific flavor, aroma, texture, and color characteristics (Fig. 1). The conventional roasting process is as follows:

Before roasting, the raw material undergoes cleaning and sorting to remove impurities. Afterwards, this cleaned raw material is loaded into the roasting equipment. Common roasting equipment includes rotary drum roasters, fluidized bed roasters, and hot air roasters. Once loaded, the roasting equipment applies heat to the raw material.

During roasting, heat is transferred to the raw material, causing various chemical reactions such as Maillard reactions. After roasting, the heat source is turned off, and the roasted material is quickly cooled to stop the cooking process and stabilize the product. Cooling can be achieved using ambient air, water quenching, or specialized cooling equipment [35].

In addition, roasting was performed in a stainless-steel drum roaster using sand as a heating medium. A diesel burner was used to provide the heat for the roaster. Overheating was avoided, and roasting was controlled so that roasted seeds' acceptable flavor was retained [35].

The optimization of the torrefaction conditions was achieved in 11 randomized trials to evaluate the effects of different torrefaction factors on the studied responses: TPC, TFC, TTC, DPPH, and ABTS inhibition (Table 2).

3.1. Interpretation of the response surface model of TPC

3.1.1. Second-order polynomial model

In this study, the TPC of extracts from prickly pear seed biomass varied from 15.42 ± 1.02 to 104.86 ± 1.94 mg GAE/g extract. According to the results of ANOVA for TPC (Table 3), the model was significant (p-value <0.0001). The lack of fit was not significant (p-value = 0.0862), showing that the model equation was adequate for predicting the values of the response. Additionally, the R^2 value was 0.990054 and the adjusted determination coefficient Ra^2_{dj} was 0.980107, showing that the model adequately describes the combination between all factors studied. According to Li et al. [32], when the determination coefficient is more than 0.75, the model is adequate. The equation Eq (2) was determined according to a reduced regression model; it can predict the effects of factor variables on the content of TPC.

$$TPC = 24.093684 + 26.36X_1 + 12.308333X_2 + 16.2525X_1{*}X_2 + 14.445789X_1{*}X_1 + 8.0107895X_2{*}X_2 \quad \text{(Eq 2)}$$

Torrefaction temperature (X_1) had a significant positive linear effect (p-value<0.0001), as well as its quadratic $X_1{*}X_1$, which had a significant positive effect (*p*-value<0.05) on TPC. Moreover, the linear effect of the torrefaction time X_2 and its quadratic effects $X_2{*}X_2$ were shown to have a significant positive effect (p-value <0.05). Furthermore, the interaction

Table 3
Anova data of the regression coefficient and the terms of the model(Total Phenolic Content (TPC)).

Source	Coef	Sumofsquare	Degree offreedom	Meansquare	F-value	p-value
TPC						
Model		7047.1123	5	1409.42	99.5379	<0.0001[a]
Constant	24.093684					<0.0001[a]
X_1	26.36	4169 .0976	1	4169 .0976	294.4350	<0.0001[a]
X_2	12.308333	908.9704	1	908.9704	64.1944	0.0005[a]
X_1 [a] X_2	16.2525	1056.5750	1	1056.5750	74.6187	0.0003[a]
X_1 [a] X_1	14.445789	528.6581	1	528.6581	37.3355	0.0017[a]
X_2 [a] X_2	8.0107895	162.5710	1	162.5710	11.4813	0.0195[a]
Residual		70.7983	5	14.16		
Lack of fit		66.669210	3	22.2231	10.7642	0.0862
Pure Error		4.129067	2	2.0645		
TotalError		70.798277	5			
R^2		0.990054				
$Radj^2$						

[a] Significant at p-value<0.05.

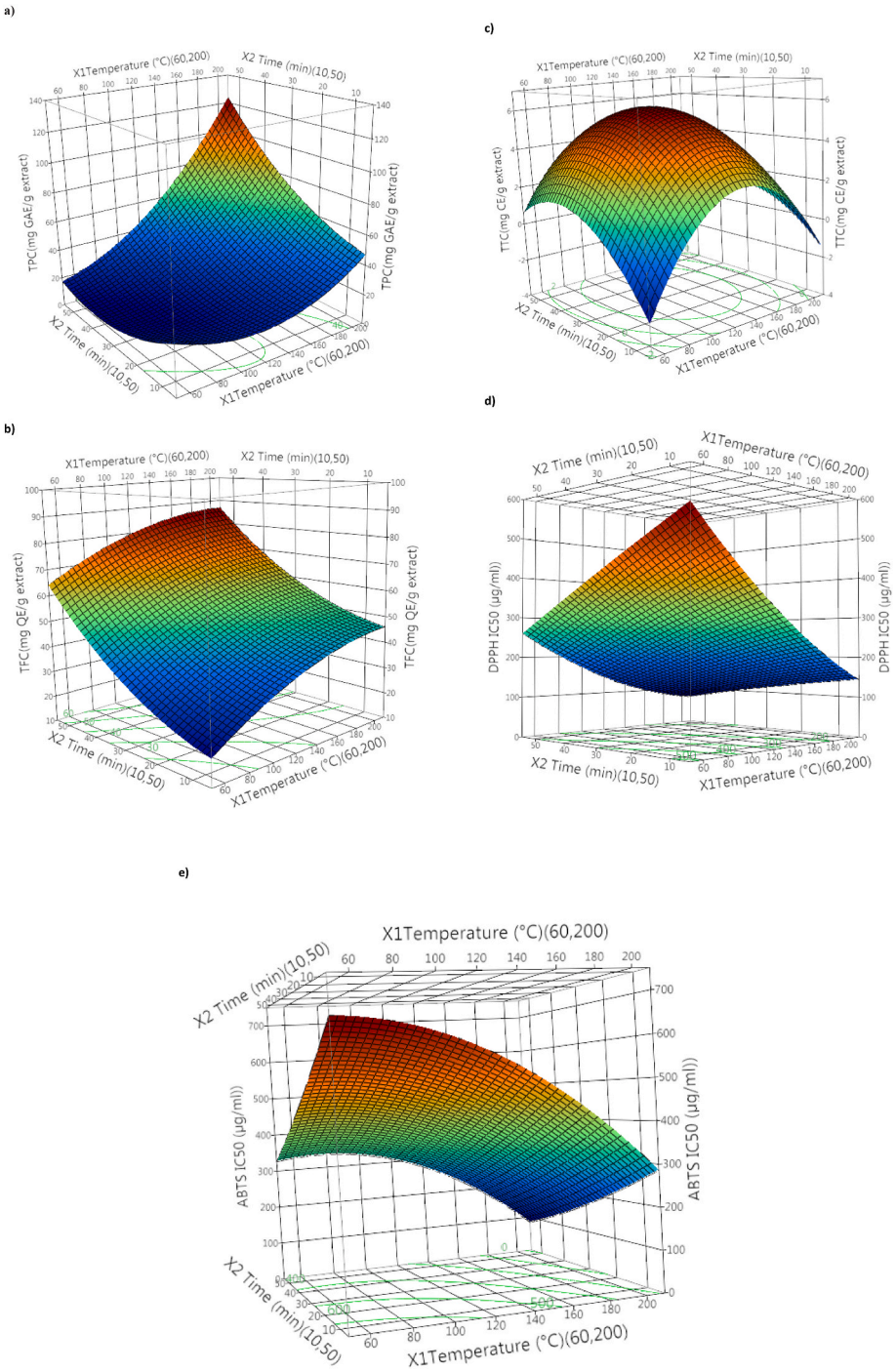

Fig. 2. Response surface plotof the effect of roasting conditions on of seed biomass extracts (a): Total Phenolic Content (TPC); (b): Total Flavonoid Content (TFC); (c): Total Condensed Tannins Content(TTC); (d): free radical scavenging activity (DPPH); (e): radical cation total antioxidant capacity (ABTS assay).

effect between the two parameters studied, X_1*X_2 was significant (p-value<0.05) on TPC (see Fig. 1).

3.1.2. *Response Surface Methodology (RSM) analysis*

The effects of both torrefaction temperature and time and their reciprocal interactions on TPC can be visualized on the generating 3D response surface plots shown in Fig. 2. According to Fig. 2a, the TPC content increased when the torrefaction temperature (X_1) increased at a torrefaction time (X_2) fixed; it also increased rapidly when the torrefaction time(X2) exceeds 30 min. These increases in the total content of phenolic compounds can be attributed to the thermal degradation of complex molecules, the formation of new phenolic compounds, the release of phenolic compounds from cellular structures, the increased solubility of phenolic compounds, and the contribution of Maillard reaction products. These factors collectively contribute to the overall increase in phenolic content and highlight the influence of roasting on bioactive compounds present in the raw material [36]; [37].

Thus, the highest values of TPC were found at the strong levels of torrefaction, high temperature (X_1) and torrefaction time (X_2). However, to get optimization of all variables together the desirability function (d) was adopted to obtain the maximum response of TPC in the seed biomass extract. Notably, the maximum response precision is obtained when the desirability is close to 1 (Fig. 3) [27,28,38]. Therefore, the optimal conditions were determined by using the statistical discovery software

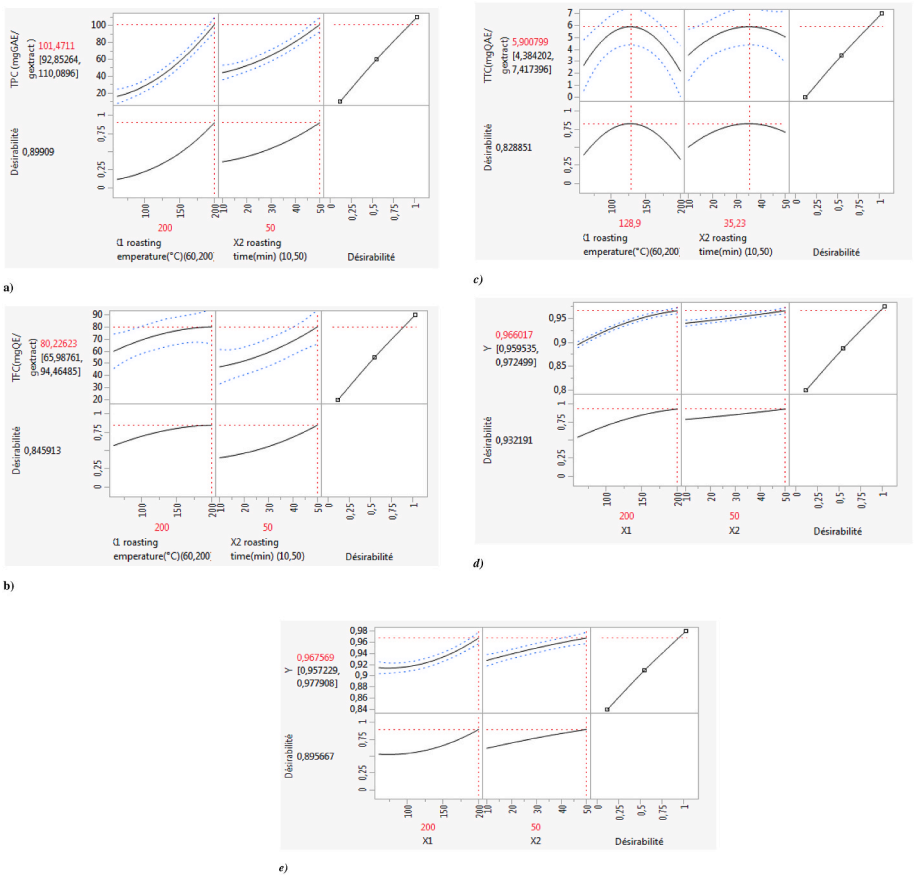

Fig. 3. Desirability results for:(a):Total Phenolic Content (TPC); (b): Total Flavonoid Content (TFC); (c): Total Condensed Tannins Content(TTC); (d): free radical scavenging activity (DPPH); (e): radical cation total antioxidant capacity (ABTS assay).

(JMP) prediction profiler. The results of optimal roasting conditions were when the desirability value (d = 0.89) was close to 1 (Fig. 3a). In this case, these conditions are 200 °C and 50 min, with predicted response = 101.4711 mg GAE/g extract, as well as the experimental value being 104.86 ± 1.94 mg GAE/g extract.

Therefore, the experimental and predicted responses were close. Hence, these results suggest that the model may be valid for the prediction of phenolic content in the torrified extracted seed biomass. These results confirm those reported in some studies. Chandrasekara et al. [20] showed that the torrefaction at high temperature (130 °C) for 33 min had increased the phenolic content relative to raw cashew seed (testa, nuts). Also, Yu et al. (2005) have found that the TPC (in both water and ethanol) from peanut skin was increased by 35.9 % with torrefaction at 175 °C for 5 min relative to the raw sample. Locatelli et al. [24] indicated that the TPC from the extract soluble in hazelnut skin increased at 180 °C for 20min, while Yin et al. [39] showed that TPC increased 3.4 times at temperature of 140 °C for 180 min. Moreover, Kim et al [25] showed that the TPC significantly increased (p-value<0.05) with heat treatment. Accordingly, the increase in the content of phenolic compounds can be explained by the following causes.

1. During the torrefaction, the phenolics can be degraded/polymerized, giving rise to compounds more soluble in ethanol and water; at the

Table 4
Anova data of the regression coefficient and the terms of the model (Total Flavonoid Content (TFC)).

Source	Coef	Sum of square	Degree of freedom	Mean square	F-value	p-value
TFC						
Model		2510.9388	5	502.188	12.9938	0.0069[a]
Constant	52.860526					<0.0001[a]
X_1	10.688333	685.4428	1	685.4428	17.7354	0.0084[a]
X_2	17.05	1744.2150	1	1744.2150	45.1306	0.0011[a]
$X_1 * X_2$	−0.585	1.3689	1	1.3689	0.0354	0.8581
$X_1 * X_1$	−4.361316	48.1867	1	48.1867	1.2468	0.3149
$X_2 * X_2$	4.5736842	52.9938	1	52.9938	1.3712	0.2944
Residual		193.2410	5	38.648		
Lack of fit		183.17941	3	61.0598	12.1372	0.0771
Pure Error		10.06160	2	5.0308		
TotalError		193.24101	5			
R^2		0.92854				
$Radj^2$					0.85708	

[a] Significant at $p < 0.05$.

Table 5
Anova data of the regression coefficient and the terms of the model(Total Condensed Tannins Content(TTC)).

Source	Coef	Sum of square	Degree of freedom	Mean square	F-value	p-value
TTC						
Model		50.697863	5	10.1396	7.4215	0.0231[a]
Constant	5.8052632					0.0002[a]
X_1	−0.133333	0.106667	1	0.106667	0.0781	0.7911
X_2	0.75	3.375000	1	3.375000	2.4703	0.1768
$X_1 * X_2$	−0.4	0.640000	1	0.640000	0.4684	0.5241
$X_1 * X_1$	−3.463158	30.383439	1	30.383439	22.2386	0.0053[a]
$X_2 * X_2$	−1.513158	5.800439	1	5.800439	4.2455	0.0944
Residual		6.831228	5	1.3662		
Lack of fit		6.5845614	3	2.19485	17.7961	0.0537
Pure Error		0.2466667	2	0.12333		
TotalError		6.8312281	5			
R^2		0.881256				
$Radj^2$					0.762512	

[a] Significant at $p < 0.05$.

Table 6
Anova data of the regression coefficient and the terms of the model(free radical scavenging activity (DPPH assay)).

Source	Coef	Sum of square	Degree of freedom	Mean of square	F-value	p-value
DPPH						
Model		124190.70	5	24838.1	479.9081	<0.0001[a]
Constant	217.23774					<0.0001[a]
X_1	−123.3112	91233.863	1	91233.863	1762.768	<0.0001[a]
X_2	−66.36283	26424.154	1	26424.154	510.5521	<0.0001[a]
$X_1 * X_2$	33.42325	4468.455	1	4468.455	86.3369	0.0002[a]
$X_1 * X_1$	28.110658	2001.863	1	2001.863	38.6788	0.0016[a]
$X_2 * X_2$	−2.714342	18.665	1	18.665	0.3606	0.5743
Residual		258.78	5	51.8		
Lack of fit		248.64531	3	82.8818	16.3558	0.0582
Pure Error		10.13487	2	5.0674		
TotalError		258.78018	5			
R^2		0.997921				
$Radj^2$					0.995841	

[a] Significant at $p < 0.05$.

Table 7
Anova data of the regression coefficient and the terms of the model(radical cation total antioxidant capacity (ABTS assay)).

Source	Coef	Sum of square	Degree of freedom	Mean square	F-value	p-value
ABTS						
Model		193571.35	5	38714.3	107.1061	<0.0001[a]
Constant	415.29474					<0.0001[a]
X_1	−135.2122	109693.98	1	109693.98	303.4772	<0.0001[a]
X_2	−107.6177	69489.37	1	69489.37	192.2479	<0.0001[a]
$X_1 * X_2$	23.584	2224.82	1	2224.82	6.1551	0.0558
$X_1 * X_1$	−68.64034	11935.79	1	11935.79	33.0213	0.0022[a]
$X_2 * X_2$	9.1731579	213.17	1	213.17	0.5898	0.4772
Residual		1807.29	5	361.5		
Lack of fit		1743.0803	3	581.027	18.0991	0.0528
Pure Error		64.2050	2	31.103		
TotalError		1807.2853	5			
R^2		0.99075				
$Radj^2$					0.9815	

[a] Significant at $p < 0.05$.

Table 8
Predicted and experimental results at conditions optimal.

	X_1 torrefaction temperature (°C)	X_2 torrefaction time (min)	Predicted value	Experimental value
TFC (mg QE/g extract)	200 °C	50min	80.22623[a]	81.23 ± 0.90[a]
TTC (mg CE/g extract)	128.9 °C	34.92min	5.901[a]	6.12 ± 0.95[b]
TPC (mg GAE/g extract)	200 °C	50min	101.4711[a]	104.86 ± 1.94[a]
DPPH IC_{50} (μg/ml)	200 °C	50min	86.3845[a]	90.663 ± 2.09[a]
ABTS IC_{50} (μg/ml)	200 °C	50min	130.581[a]	124.9±4[a]

Data are presented as the average values ± standard deviation of the two replicates (mean ± SD, n = 2). The mean followed by similar lettersexposing in the line are not different ($P < 0.05$)(student t-test).
Total Phenolic Content (TPC); (Total Flavonoid Content (TFC); Total Condensed Tannins Content(TTC); free radical scavenging activity (DPPH assay); radical cation total antioxidant capacity (ABTS assay).

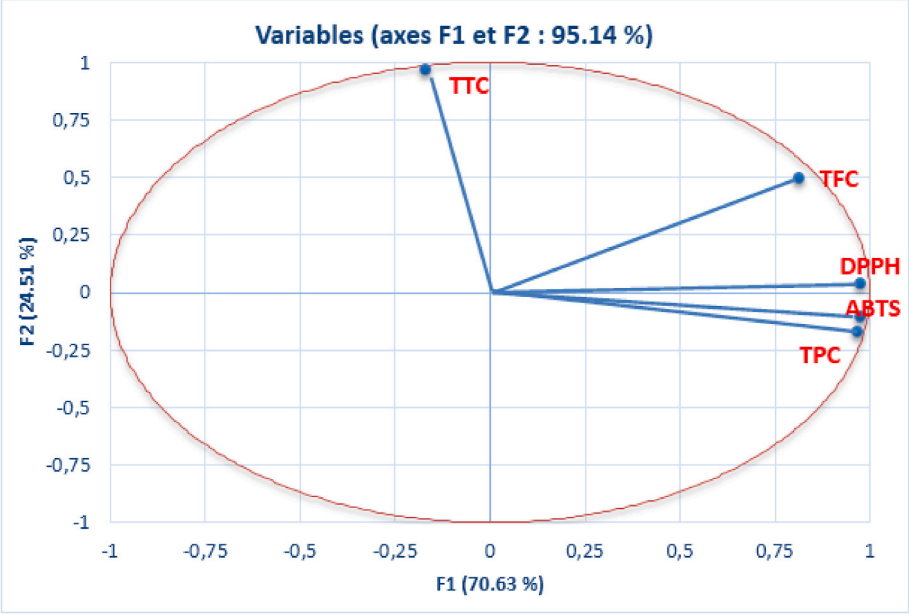

Fig. 4. Principal Component Analysis factorial plan carried out on the values of Total Phenolic Content (TPC); Total Flavonoid Content (TFC); Total Condensed Tannins Content(TTC); free radical scavenging activity (DPPH assay); radical cation total antioxidant capacity (ABTS assay).for the different extracts from prickly pear seed biomass.

same time, they can react with the folin-ciocalteu in the alkaline medium [23].

2. The bound bioactive molecules can be released during roasting[40].

3.2. Interpretation of the response surface model of TFC

3.2.1. Second-order polynomial model

Our results showed that the TFC of extracts from prickle pear seed biomass varied from 28.86 ± 1.10 to 81.23 ± 0.90 mg QE/g extract. Moreover, ANOVA was used to verify the adequacy and the significance of the model. Table 4 showed that the F-value is large (12. 9938) and the *p*-value is small (0.0069), which confirms the validity of the model. Additionally, the lack of fit was not significant (p-value: 0.0771, which confirms that the optimization model of the TFC response was significant [41]. The coefficients of determination R^2 and adjusted showed high values ($R^2 = 0.92854$ and $R^2_{adj} = 0.85708$). These values indicated that the model was valid. The equation that combines the relationship between the variables and prediction TFC was described below.

$$TFC = 52.860526 + 10.688333X_1 + 17.05X_2 \quad \text{(Eq 3)}$$

Torrefaction time (X_2) had a positive significant linear effect (p-value = 0.0011 < 0.05) on TFC, and it does not have a significant quadratic effect (p-value = 0.2944). As well, the torrefaction temperature (X_1) had a significant positive linear (p-value = 0.0084), but its quadratic effect is not significative (p-value = 0.2944). Moreover, the not significant interaction effect between the two parameters studied

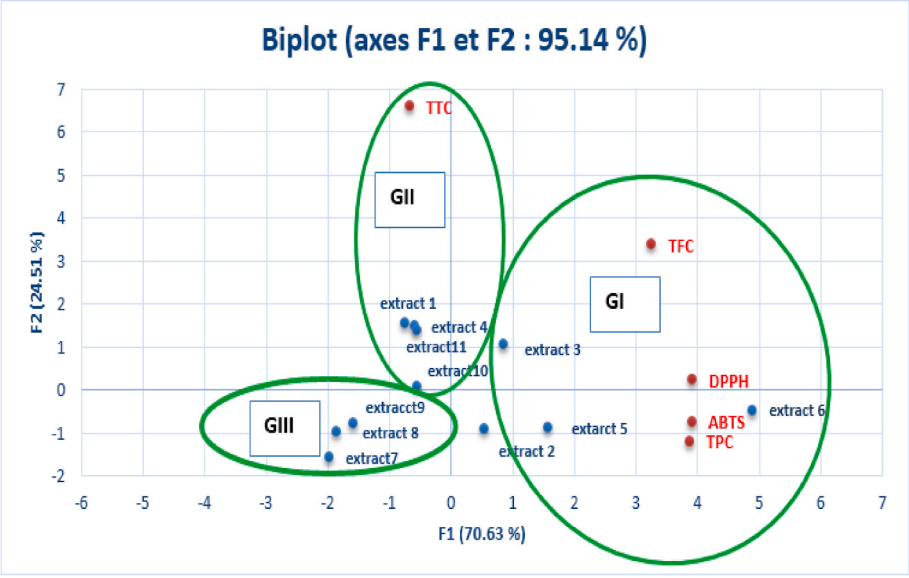

Fig. 5. Projection on the factorial plan (F1 × F2) of the individual's variable.

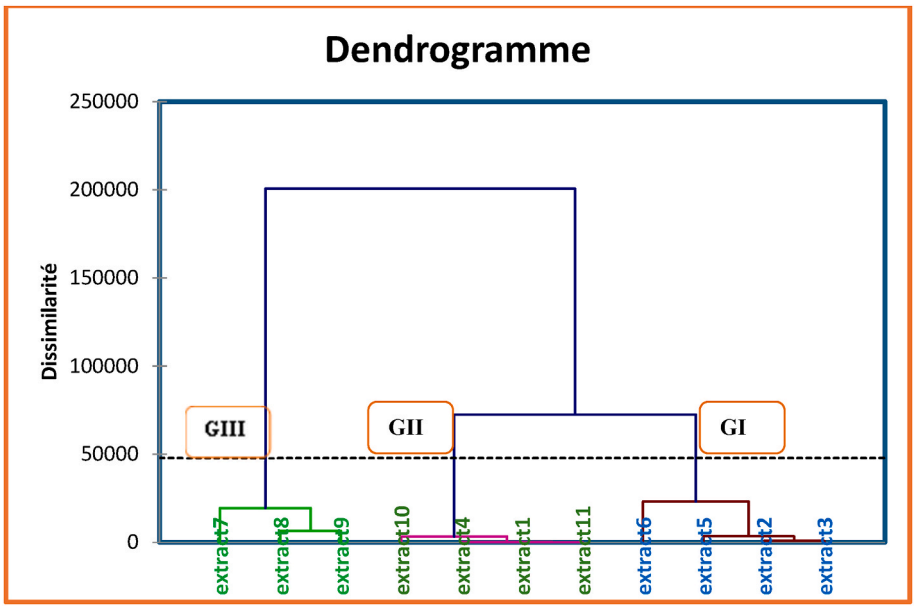

Fig. 6. Dendrogram of the extracts tested founded by HCA using antioxidant capacity.

was observed (Table 4).

3.2.2. Response Surface Methodology(RSM)analysis

Fig. 2b shows the response surface plot of torrefaction temperature and time on TFC. The latter increased with the increase of both torrefaction time and temperature. Accordingly, the highest TFC yield was detected in regions of high torrefaction temperature and time. This increase can be attributed to several factors that influence the chemical and physical properties of the torrefied material, such as thermal degradation and release of bound phenolic compounds; when the roasting temperature increases, the thermal energy supplied to the material enhances the degradation of the complex polymer structures. , such as lignin and cellulose, which often contain phenolic compounds.

Then, this degradation facilitates the release of phenolic compounds that were previously bound to these structures ([42]; 2010; [43]).

Consequently, the optimum extraction of TFC was at a torrefaction temperature of 200 °C and a torrefaction time of 50 min with the predicted response = 80.22623 (mg QE/g extract), and the desirability was 0.84 (Fig. 3b). Our results are consistent with the study of Lin et al [44] who reported that the TFC increased significantly after 5 min of the torrefaction, as the flavonoid aglycones and acids are increased according to torrefaction temperature and time. Furthermore, Kumar et al. [45] mentioned that, the fractions of sugar in flavonoids glycosides have an important role in antioxidant capacity, as well as the aglcycones having a higher effect on the antioxidant capacity than the glycosides.

3.3. Interpretation of the response surface model of TTC

3.3.1. Second-order polynomial model

Table 5 shows the coefficients of regression and their significance for the TTC. The regression model was significant (p-value = 0.0231). Also, the determination coefficients (R^2) for the TTC response variable (0.881256) and the lack-of-fit values (0.0537) were not significant ($p > 0.05$), which indicates that the model can explain all data. So, the response variable was included in roasting optimization. Besides, the R^2_{adjs} was 0.762512, which indicated that 76.25 % of the variability was estimated by the model. Therefore, the second-order polynomial model was applied (Eq (4)).

$$TTC = 5.8052632 - 0.133333X_1 + 0.75X_2 \quad \text{(Eq 4)}$$

According to the results (Table 5) the quadratic effect of the roasting temperature ($X_1 * X_1$) was positive and significant (p-value<0.05) for the optimization of the TTC response. On the other hand, its linear effect X_1 had no significance (p-value>0.05). As well as the linear and quadratic effects of roasting time (X_2 and $X_2 * X_2$) were not significant because their p-value was respectively: 0.1768, and 0.0944. The interaction effect ($X_1 * X_2$) was not significant because its p-value was 0.5241.

3.3.2. Response Surface Methodology (RSM) analysis

The 3D of the response surface of regression Eq (3) was constructed using RSM to illustrate the effects of the torrefaction temperature and time and their interaction on the TTC (Fig. 2c). Accordingly, the TTC content increased with increasing roasting temperature (between 60 and 130 °C) and roasting duration (between 10 and 35 min). Beyond this range, it decreased rapidly. This increase and decrease can be explained by several factors, including thermal degradation, release of bound compounds, and subsequent degradation and polymerization of tannins: Thermal Degradation of Cellular Structures: During the initial roasting phase, applied heat breaks down complex cellular structures and lignin matrices, releasing tannins that were previously bound or trapped in these structures. Thermal transformation: Heat can cause chemical reactions that form new tannic compounds. Decomposition of tannins: Prolonged exposure to higher temperatures beyond 35 min can result in thermal degradation of tannin compounds. Tannins are heat sensitive and can break down into simpler, non-tannin molecules, thereby reducing TCT [46].

The optimum extraction of TTC was a torrefaction temperature of 128.9 °C and a torrefaction time of 34.92 min yield ing5.901 (mg CE/g extract) of predicted response, with a desirability of 0.82(Fig. 3c). These results are similar to those reported by Lin et al. [44], who showed that during the torrefaction of almond (Prunus dulcis) kernel at 200 °C for 20 min the TTC was the highest.

3.4. Interpretation of the response surface model of free radical scavenging activity (DPPH assay)

3.4.1. Second-order polynomial model

The ANOVA results from DPPH assay content based on the RSM design are reported in Table 6. The p-value of the model was <0.0001 which indicated that the model describes well the response. Moreover, the R^2 and R^2_{adj} were 0.997921 and 0.995841, respectively, which confirms the adequacy of the model [32]. Additionally, the lack of fit

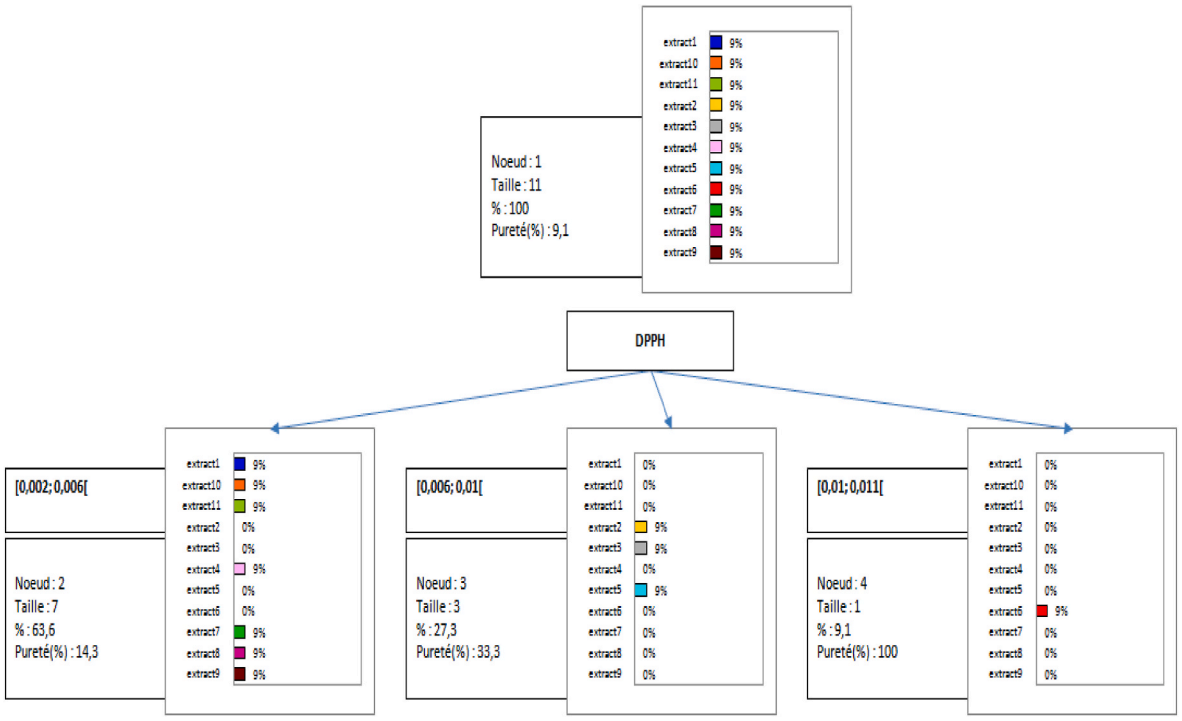

Fig. 7. Classification tree obtained from CHAID for samples extracted.

(p-value>0.05) confirms also the adequacy of the model for prediction of the antioxidant power of extracts for seed-torrified biomass. Therefore, the second-order polynomial model was applied (eq (5)).

$$DPPH(IC_{50}) = 217.23774\text{-}123.3112X_1\text{-}66.36283X_{2+}33.42325X_1{*}X_2{+}28.110658X_1{*}X_1 \quad \text{(Eq 5)}$$

Based on statistical analyses of ANOVA for DPPH, the torrefaction temperature (X_1) and torrefaction time (X_2) had a negative significant linear effect on the IC_{50} of the DPPH assay because their p-value was<0.0001. Also, their interaction wassignificant for DPPH because the p-value was 0.0002*. On the contrary, their quadratic effects were not significant (p-value >0.005).

3.4.2. Response Surface Methodology(RSM) analysis

The response surface (3D) of regression Eq (5) was constructed using RSM to illustrate the effects of torrefaction temperature (X_1) and torrefaction time (X_2) and their interaction X_1*X_2 on the IC_{50} of the DPPH assay (Fig. 2d.). We know that the antioxidant power is inversely proportional to the value of the IC_{50}. We observed that the increase in the antioxidant power was linear with the increase of the torrefaction time (X_2) and torrefaction temperature (X_1). The optimum was observed at a temperature of 200 °C and time of 50 min giving a 96.60 % inhibition (IC_{50} of 86.3845 μg/mL predicted response, d = 0.93 (Fig. 3d). Our results confirm those of Lin et al. [44] who reported that the highest antioxidant capacity of ethanolic extracts from almond kernel was observed at 200 °C for 20. Moreover, Chandrasekra et al. [20] reported that the scavenging capacity of DPPH radical increased significantly with the increase of the torrefaction temperature for a phenolic extract from raw shelled cashews with testa, probably due to Maillard reaction products (MRPs). The Maillard reaction is a complex series of chemical reactions that occur between reducing sugars and amino acids when food is heated, leading to the formation of Maillard reaction products (MRPs). These reactions are responsible for the development of flavor, color, and aroma in a wide range of cooked foods. MRPs also include a variety of compounds with antioxidant properties, which can increase the total phenolic content in foods subjected to heat treatment [47,48].

Indeed, during torrefaction, a reaction between the reducing sugars and amino acids can be done; this reaction can produce MRPs, contributing to TPC, flavor, antioxidant activity, and color of food [20]. In addition, the Maillard reaction intermediate products (MRP) had a strong antioxidant power, due to the presence of reductone-like structures [49].

3.5. Interpretation of the response surface model of radical cation total antioxidant capacity (ABTS assay)

3.5.1. Second-order polynomial model

Experimental modeling results for antioxidant power by ABTS assay were shown in Table 7. From the model analysis, the R^2 and R^2_{adj} were 0.99075 and 0.9815 respectively, with no lack of fit (p-value = 0. 0528). The model was significant sinceits p-valuewas <0.0001, thus the following model equation (Eq (6)) was acceptable to predict the antioxidant power by ABTS assay.

$$ABTS\ (IC_{50}) = 415.29474\text{-}135{,}2122X_1\text{-}107.6177X_2\ \text{-}68.64034X_1{*}X_1 \quad \text{(Eq 6)}$$

Torrefaction time (X_1) and torrefaction temperature (X_2) had a significant negative linear effect ($p < 0.05$). Also, the quadratic effect of the torrefaction temperature X_1 *X_1 had a significant effect (p-value = 0.0022), and the interaction effect between the two parameters was not significant (Table 7).

3.5.2. Response Surface Methodology (RSM) analysis

Fig. 2e shows the RSM plot of the ABTS inhibition assay, where we observed that the antioxidant power increased significantly when the torrefaction temperature X_1 and torrefaction time X_2 increased. The more IC_{50} decreased, the more the antioxidant power increased. The optimum of the antioxidant power by ABTS assay was at torrefaction temperature of 200 °C and torrefaction time of 50 min with 96.75 % inhibition (predicted IC_{50} = 130.581 μg/ml, d = 0.89) (Fig. 3e). These results are consistent with those of Gao et al. [12] who mentioned that the ABTS capacity was increased significantly during the torrefaction at160 °C for 10 min compared to the raw sample. Also, Yin et al. [39] reported that the ABTS scavenging capacity increased during the

heating at 130–140 °C after 60 min. Moreover, these results can depend on several conditions; for instance, plants have bound antioxidant phenols and bound polymeric compounds that, during the thermal treatment, can undergo degradation and releasing leading to an increase in the antioxidant activity [50]. Additionally, the characteristics of antioxidants can be improved through the degradation of heat-labile antioxidant compounds or the formation of new compounds through the Maillard reaction [37]. Also, the solubility of non-phenolic molecules was improved by torrefaction [51].

3.6. Comparisons of predicted (models) and experimental results

The verification experiments for five responses, such as DPPH IC_{50} (μg/ml), ABTS•+ IC_{50} (μg/ml), TPC, TFC, and TTC were reported in Table 8. These experiments were done at the optimal conditions of responses and in the experimental range. These results showed that the values of experimental responses are close to the predicted ones.

3.7. Correlation matrix

Table 1S (Supplementary Materials) shows the correlation coefficient data between all responses studied. Moreover, Table 2S (Supplementary Materials) presents the p-value of these correlation coefficients. Additionally, the DPPH (1/DPPH IC_{50}) and ABTS (1/ABTS IC_{50}) represent the power to inhibit DPPH free radical and $ABTS^{\cdot+}$radical, respectively.

According to Tables 1S and 2 S (Supplementary Materials), we observed that the TPC had high positive correlations (p-value <0.05) with the antioxidant power. The correlation coefficients of TPC were 0.949 and 0.966 with free radical scavenging effect on DPPH and $ABTS^{\cdot+}$ respectively. These positive correlations were justified by the fact that the antioxidant capacity depends on the presence of phenolic compounds in the extracts; these results are similar to those reported in literature [52,53]; [54]. It was found that the TFC had positive correlations with the DPPH and ABTS inhibition effects, with correlation coefficients of 0.784 and 0.727, respectively. Moreover, we observed a strong positive correlation significance (p-value <0.05) between TFC and TFC ($r2 = 0.652$). We observed that the p-values of TTC with the antioxidants power were not significant (p-value >0.05), which indicates that tannins contribute slightly to this bioactivity. Furthermore, the strong and statistically significant positive correlation ($r^2 = 0.920$) between the two antioxidant assays (DPPH and ABTS) employed in this study indicates that the same bioactive molecules in our extracts are responsible for the scavenging activity against both DPPH and ABTS radicals.

3.8. Principal Component Analysis (PCA)

According to Fig. 5, the projections of the responses studied and the experimental assays (extracts) were done by the factorial plan reported in Fig. 5. The cumulative percentage was 95.14 %, which indicates that it was representative of the variables because it was more than 50 %. Moreover, the two axes were suitable for explaing all information, with the first (F1) and second (F2) components explaining 70.63 % and 24.51 of total variability, respectively. The correlations between all variables studied were explained by a plan formed by the F1 and F2 axes. Besides, the F1 axis was formed by the positive correlation between TPC, TFC, ABTS ($1/IC_{50}$), and DPPH ($1/IC_{50}$) on the contrary, the F2 axis was constructed by TTC (Fig. 4). Our 11 extracts studied from biomass (*Opuntia ficus indica* seeds), were distributed in three groups according to the responses (Fig. 5).

Group I: This group was formed by four extracts (2, 3, 5, and 6), these extracts had strong values of the TPC and TFC, as well as high power antioxidants by DPPH and ABTS assays.

Group II: it contains four extracts (1, 4, 10, and 11), these extracts are characterized by a strong value of TTC, and by lower values of TPC and TTC. Therefore, their antioxidant activity is lower compared to group I.

Group III is formed by three extracts (7, 8, and 9) these extracts are characterized by the low values of TPC and TFC, and their antioxidant activity is low compared to extracts of the other groups.

The extracts from Group I are characterized by a high torrefaction temperature that varies between 130 °C and 200 °C, and a high time of torrefaction(50 min) for the extracts torrefied at 130 °C, which shows that their antioxidant capacity is higher than the extracts from Groups II and III obtained at a low torrefaction temperature. Therefore, the torrefaction makes it possible to increase the extraction of bioactive compounds responsible for antioxidant power.

3.9. Hierarchical clustering analysis HCA

Extracts were categorized using the squared Euclidean and Wards techniques to determine consistency measures, according to HCA. As shown in Fig. 6, HCA was employed to examine the relationship and similarity among the eleven extracts based on antioxidant activity results and their bioactive compounds. Eleven extracts were split into three clusters based on this data.

According to bioactive molecules, the 11 extracts were clustered into three Clusters. Cluster I contains four extracts (2, 3, 5, and 6) representing for 36.36 % of the total extracts, it characterized by a high range mean of TPC, 63.15 mg GAE/g extract, and TFC 64.23 mg QE/g extract respectively, and a medium range mean of TTC2.05 mg CE/g extract, as well, having a stronger antioxidant capacity by DPPH and ABTS assays.

Cluster II, the largest cluster formed using 6 four extracts (1, 4, 10, and 11), accounted for 36.36 % of total extracts; these extracts had the medium mean value of TPC and TFC, 22.33 mg GAE/g extract, 58.02 mg QE/g extract, and a high range mean of TTC 5.37 mg CE/g extract.

Cluster III contained three extracts (7, 8, and 9) representing 27.27 % of the total extracts, with low mean values of antioxidant compounds TPC, TFC, and TTC, 19.28 mg GAE/g extract, 31.24 mg QE/g extract, and 1.43 mg CE/g extract, respectively, as well, a lower antioxidant power. These findings are consistent with the PCA data, showing similar trends in the distribution of all extracts on the score plots. Moreover, the PCA results were corroborated with the HCA results.

3.10. Chi-squared automatic interaction detector (CHAID)

CHAID is a very useful technique for finding out which variables are most helpful in separating the samples under study into known sets. Here, the CHAID method was used to analyze eleven extracts using antioxidant activity, providing a model to differentiate these extracts (Fig. 7).

Using antioxidant activity, CHAID achieved a correct classification rate of 27.27 % for these extracts. The process uses a basic algorithm to build a non-binary tree that relies on a chi-square test to find the best split. Regarding the findings, it should be emphasized that the extracts 2, 3, 5, and 6 have significant antioxidant capacity compared to the other extracts. The great interest in the CHAID analysis stems from the fact that the correct classification was made according to the adjacent classification of samples based on antioxidant activity.

4. Conclusion

In this study, the influence of roasting on the bioactive components and antioxidant activity of prickly pear seeds was investigated using a central composite design (CCD) method.In conclusion, based on the results, the bioactive components (TPC, TFC, and TTC) were strongly affected by roasting temperature and duration.Additionally, the response surface model (RSM) offered a globally optimized green

method for all the tested variables to achieve the maximum response in TPC, TFC, TTC, ABTS, and DPPH of the extracts. Compared to the experimental and predicted results, their values were close. The PCA highlighted tree extracts (extracts 3,5, and 6) with high values of TPC and TFC and strong antioxidant activity in the DPPH and ABTS assays. These extracts had the longest roasting time of 30 and 50 min and a temperature of 130 and 200 °C. Additionally, the antioxidant activities were increased at higher roasting temperatures and times. Consequently, this study demonstrated that torrefaction can be employed as a pre-treatment method to enhance the antioxidant capacity of prickly pear seeds.

CRediT authorship contribution statement

Chakir El Guezzane: Writing – original draft, Visualization, Validation. **Hamza El Moudden:** Methodology, Investigation, Formal analysis, Data curation. **Hicham Harhar:** Visualization, Validation, Supervision, Software, Methodology. **Abdelkader Zarrouk:** Investigation, Formal analysis, Data curation, Conceptualization. **Learn-Han Lee:** Visualization, Validation, Supervision, Resources. **Waleed Al Abdulmonem:** Funding acquisition. **Abdelhakim Bouyahya:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration. **Filippo Maggi:** Writing – review & editing. **Giovanni Caprioli:** Writing – review & editing. **Mohamed Tabyaoui:** Writing – review & editing, Visualization, Validation, Supervision, Resources.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jafr.2024.101465.

Data availability

The authors are unable or have chosen not to specify which data has been used.

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