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Research Article

Laboratory-Scale Optimization of Hemp Seed Roasting Temperature and Time for Producing a High-Quality Pressed Oil

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Roasting is widely applied in oil processing to improve the extraction yield and desired sensory characteristics. The current study is aimed at optimizing roasting conditions (time and temperature) of hemp (*Cannabis sativa* L.) seeds prior to seed pressing to improve the oil yield and antioxidant capacity, using response surface methodology based on central composite design (CCD). Hemp seeds were roasted at five temperatures (132, 140, 160, 180, and 188°C) and for five duration times (9, 15, 30, 45, and 51 min). Mathematical models have shown that roasting conditions significantly affected response variables ($p < 0.05$), including oil yield, total phenolic content (TPC), radical scavenging activity, and oxidative stability index (OSI). The CCD led to the following optimum roasting conditions: 163°C for 15 min, which improved the extraction yield by 45% and oxidative stability by 80%. Thus, the oil produced under these conditions showed a yield of 23.09%, TPC of 121.21 mg GAE kg^{-1}, and OSI of 21.37 hours. In addition, roasting hemp seeds under optimal conditions did not negatively influence the oil quality. Only chlorophylls, tocopherols, and palmitic acid slightly decreased ($p < 0.05$) after roasting (from 39.10 to 36.54 mg kg^{-1}, 483.06 to 469.77 mg kg^{-1}, and 7.65 to 7.29%, respectively). Furthermore, the concentrations of most volatile compounds identified in unroasted hemp seeds decreased after roasting under optimal conditions, with the formation of new compounds sought for their positive attributes, such as pyrazines and aldehydes derived from the Strecker degradation, responsible for the roasted, nutty, and almond odors. The main volatile compounds in raw and roasted hemp seeds were β-myrcene (3170.30 and 1177.69 ng g^{-1}, respectively) and D-limonene (1347.25 and 470.35 ng g^{-1}, respectively). The results obtained in this study could provide valuable information for the food industry to produce hemp seed oil with high nutritional quality that meets consumer demands.

1. Introduction

Hemp (*Cannabis sativa* L.) seed is a valuable source of edible oil, known for its medicinal and nutritional properties. The hemp seed oil content varies from 28% to 35%, depending on several factors, such as the variety, geographical area, year of cultivation, climatic conditions, and extraction methods [1–3]. Hemp seed oil is considered one of the few vegetable oils containing an ω_6/ω_3 ratio estimated at 3/1, which could be ideal for preventing cardiovascular accidents [4]. It is also a good source of γ-tocopherol [2], which could potentially prevent diseases caused by oxidative damage [5]. Phenolic

compounds are also present in the oil, but only in small quantities. Indeed, most phenolic compounds are found in the cake after oil extraction because most are located in the shell [6].

Mechanical pressing is the most used method to produce oil from hemp seeds. This method is mainly used for simplicity, health, economic, and environmental reasons [7]. Indeed, pressing does not involve the use of chemicals. It consists in separating under pressure the oily phase from the solid phase of the seed. However, only about 65% of the oil could be extracted from hemp seeds [8], which is considered low compared to chemical extraction.

Various methods are proposed in the literature to increase the yield of mechanical pressing extraction. For this purpose, the roasting of seeds before oil extraction has been used to improve the oil yield from different oilseeds such as rapeseed [9], flax [10], and babassu seeds [11]. Roasting could increase the extraction yield and improve the specific organoleptic properties of the final product, such as color and flavor [12]. It also promotes the inactivation of certain enzymes responsible for the deterioration of the product during storage, such as lipoxygenase and lipase [10]. Moreover, the roasting treatment facilitates the release of phenolic compounds bound to the cell wall, enriching the extracted oil with natural antioxidants, strengthening its antioxidant capacity, and improving its nutritional quality [12]. However, inappropriate roasting conditions could decrease the yield and quality of the extracted oil. Uncontrolled heat treatment during the roasting process could also generate substances harmful to human health, such as furan and hydroxymethylfurfural [13].

Although the effect of roasting hemp seeds on the oil composition has been previously studied [14], more information is needed regarding optimizing roasting time and temperature for producing high-quality hemp seed oil. The available data in the literature concern a series of experiments based on the variation of a single factor, which leads to an incomplete optimization of the roasting process. However, those results provide an essential database to guide this research.

The response surface methodology (RSM) is an optimization method using the regression method to highlight the relationship between the response value and the studied factors, evaluating their linear, quadratic, and interaction effects [15]. It is often used to define the optimal levels of factors with a reduced number of experimental tests. RSM has been widely used to optimize the roasting parameters of certain agricultural products, such as argan kernels [16], almonds [17], and babassu [11]. The results obtained from these studies allow us to presage that it is possible to optimize hemp seeds' roasting parameters (temperature and time) to improve the oil yield obtained by the press method and enhance its quality.

The present work proposes an application of the response surface methodology to optimize the roasting process of hemp seeds in order to (i) evaluate the time and temperature effects on the oil yield and antioxidant parameters, (ii) establish predictive models for hemp seed roasting, and (iii) determine the optimal conditions for roasting hemp seeds to produce high-quality oil with the maximum yield. After determining the optimal roasting conditions, a comparative study was conducted between unroasted and roasted seeds by analyzing their oil yield, quality indices, total phenolic content, nonenzymatic browning index, color, pigment content, fatty acid and tocopherol composition, DPPH radical scavenging activity, and oxidative stability index. We also evaluated, for the first time, the changes in volatile compounds during roasting hemp seeds under optimal conditions.

2. Materials and Methods

2.1. Plant Material. Hemp (*Cannabis sativa* L.) seeds of a local ecotype (Beldia) were kindly provided by the National Agency of Medicinal and Aromatic Plants (ANPMA), Taounate, Morocco. The cultivation of *Cannabis* was carried out in the spring of 2020 at the ANPMA experimental station in northern Morocco. The collected seeds were harvested in July at the maturity phase, during which most Moroccan cannabis farmers usually harvest (more than 90% of brown seed appeared). The specimen was deposited in the Herbarium of the University Mohammed I, Oujda, Morocco, under voucher number HUMPOM242. The average 1000-seed weight was 10.16 ± 0.05 g, and their dimensions were 3.51 ± 0.18, 2.76 ± 0.22, and 2.32 ± 0.11 mm in length, width, and thickness, respectively.

2.2. The Roasting Process of Hemp Seeds. Hemp seeds were roasted in a conventional oven using natural convection heating (UN-55 Universal Oven Memmert, Germany). The temperature and time were varied according to the central composite design (CCD) detailed in Table 1. For each experiment, 80 g of hemp seeds was spread in a thin layer of approximately 4 mm thickness on an aluminum tray. Each experiment was performed in triplicate. After elapsed roasting, the seeds were cooled to room temperature in a glass desiccator containing silica gel. All the physicochemical analyses were performed in triplicate for each sample.

2.3. Response Surface Methodology. The response surface methodology was used to determine the optimal roasting time and temperature for producing high-quality hemp seed oil with maximum yield using the software JMP Pro 15 (SAS Institute Inc., USA). A total of 13 experiments were chosen randomly, according to a CCD with two controlled factors, including five repetitions of the central point. The studied factors (independent variables) were roasting temperature (X_1, °C) and time (X_2, min). Each variable had five levels: $-\alpha$ (= -1.41), -1, 0, +1, and $+\alpha$ (= +1.41). The actual values of these levels are presented in Table 1. The low and high roasting temperature and time levels were chosen based on preliminary experiments (data not shown). The selected response parameters (Y_i dependent variables) were oil yield (Y_1), TPC (Y_2), RSA (Y_3), and OSI (Y_4). A second-degree polynomial was used to link the responses to the independent variables according to the following equation:

$$Y_i = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2, \quad (1)$$

Table 1: Fully coded central composite design and experimental data of oil yield, total phenolic compounds (TPC), free radical scavenging activity (RSA), and oxidative stability index (OSI) of hemp seed oils, in relation to roasting temperature (X_1) and time (X_2).

Run	Variables		Moisture content (%)	Responses							
	X_1 temperature (°C)	X_2 time (min)		Oil yield (%)		TPC (mg kg^{-1})		RSA (%)		OSI (hours)	
				Observed	Predicted	Observed	Predicted	Observed	Predicted	Observed	Predicted
1	188 (+α)	30 (0)	4.61	19.74	19.83	229.03	244.94	88.56	91.09	20.76	23.42
2	140 (-1)	15 (-1)	7.02	21.64	21.44	65.29	73.04	45.06	45.74	19.38	19.95
3	160 (0)	30 (0)	5.39	20.37	21.24	148.80	150.03	79.10	79.10	31.05	29.78
4	160 (0)	30 (0)	5.45	21.10	21.24	150.01	150.03	77.81	79.10	28.61	29.78
5	160 (0)	9 (−α)	6.07	22.70	22.75	102.81	94.19	60.94	62.08	23.00	23.94
6	160 (0)	30 (0)	5.42	21.71	21.24	150.01	150.03	78.94	79.10	29.71	29.78
7	160 (0)	51 (+α)	4.54	20.13	20.54	141.71	169.56	81.25	80.70	34.36	34.73
8	160 (0)	30 (0)	5.32	21.50	21.24	150.87	150.03	81.25	79.10	29.05	29.78
9	160 (0)	30 (0)	5.29	21.55	21.24	151.22	150.03	78.44	79.10	30.54	29.78
10	140 (-1)	45 (+1)	5.33	21.19	20.73	110.59	92.81	68.25	70.11	22.28	23.25
11	180 (+1)	45 (+1)	4.21	19.70	19.45	277.44	250.85	87.56	86.30	34.99	33.15
12	132 (−α)	30 (0)	6.89	20.05	20.42	68.05	71.37	54.88	52.94	17.08	15.73
13	180 (+1)	15 (-1)	5.70	21.89	21.89	164.02	162.95	86.50	84.06	23.28	21.04

with β_0 representing the constant, β_1 and β_2 representing the linear coefficients, β_{12} representing the interaction coefficient, and β_{11} and β_{22} representing the quadratic coefficients.

The surface responses of the obtained results were analyzed by ANOVA and presented by a standardized Pareto diagram. The experimental and predicted values of the responses were compared to determine the model's validity at a 5% significance level. The results were confirmed by triplicate trials under optimal roasting conditions that could maximize oil yield, thus producing high-quality hemp seed oil. Student's t-test was used to assess the differences between unroasted and roasted hemp seeds after optimizing roasting conditions at a threshold of 5%.

2.4. Measurement of Dependent Variables

2.4.1. Hemp Seed Oil Extraction. The oil was extracted from hemp seeds using a mechanical screw press (Les Ateliers Afyach SARL., Agadir, Morocco) powered by an electric motor with a nominal power of 0.75 kW. Before oil pressing, the temperature inside the press head was set at $60 \pm 5 \circ$ C. The extraction was then carried out at a constant speed (32 rpm), with an outflowing oil temperature of $45 \pm 3 \circ$ C. The extracted oil was centrifuged at 6°C (5778 × g for 10 min), transferred directly into dark bottles, and stored under nitrogen at 4°C until use. The oil yield (%) was calculated by dividing the oil content obtained after centrifugation by the weight of the pressed seeds. All the physicochemical parameters were determined in triplicate.

2.4.2. Total Phenolic Content. The phenolic extracts of hemp seed oil samples were prepared according to the protocol described by Ben Moumen et al. [18]. Thus, 2.5 g of oil was extracted twice with 80% (v/v) methanol/water. The combined extracts were filtered through a 0.45 μm membrane filter. The total phenol content (TPC) of hemp seed extracts was quantified using Folin-Ciocalteu's reagent. One hundred microliters of the extract was mixed with 900 μL of water and 100 μL of the Folin-Ciocalteu reagent. The mixture was incubated for 10 min before adding 1 mL of Na_2CO_3 solution (10%). After incubation for 1 h and 20 min in the dark at room temperature, the absorbance was read at 765 nm using a UV-visible spectrophotometer (Jenway 7305, France). The TPC was expressed in mg equivalent of gallic acid per kg of hemp seed oil (mg GAE kg^{-1}), based on a standard curve of gallic acid (Sigma-Aldrich, St. Louis, USA) established from a range of linearity concentrations of 15-250 μg mL^{-1} ($r^2 > 0.99$). The quantification (LOQ) and detection (LOD) limits were 2.96 and 0.98 μg mL^{-1}, respectively.

2.4.3. Radical Scavenging Activity. The DPPH (1,1-diphenyl-2-picrylhydrazyl) test was used to measure the free radical scavenging ability of the hydromethanolic extract. One hundred microliters of the extract was added to 2 mL of DPPH solution prepared in methanol (1.2×10^{-4} M). After stirring, the mixture was kept in the dark for 30 min. The absorbance was measured against methanol at 517 nm. The free radical scavenging activity was expressed as the DPPH percentage reduced by the extract (% RSA) using the following equation:

$$\text{RSA}\,(\%) = \frac{A_0 - A}{A_0} \times 100, \quad (2)$$

where A represents the absorbance of the DPPH solution after reaction with the extract and A_0 represents the absorbance of the DPPH solution without extract.

2.4.4. Oxidative Stability Index. The Rancimat method was used to evaluate hemp seed oil's oxidative stability index

(OSI), expressed as the induction time of lipid oxidation (hours). The measurement was carried out using a Metrohm 743 apparatus (Metrohm Co., Basel, Switzerland) at 100 ± 1.6 ∘ C with an airflow of 20 L h^{-1} using 3 ± 0.01 g of oil [19].

2.5. Physicochemical Analyses of Hemp Seed

2.5.1. Seed Moisture Content. The moisture content was assessed using the AOAC International Standard Method 925.40 [20] by drying ground hemp seeds at 100 ± 3 ∘ C.

2.5.2. Oil Quality Indices. The free acidity (mg KOH g^{-1}), peroxide value (meq O_2 kg^{-1}), and UV absorbance (K_{232} and K_{274}) of oil samples were evaluated according to the official European methods used for virgin olive oil EEC/2568/91 [21].

2.5.3. Oil Pigment Content. The carotenoid and chlorophyll contents of oil samples were determined according to the protocol described in the literature [22]. Hemp seed oil was prepared in cyclohexane (30%; *w*/*v*). The mixture's absorbance (A_{670} and A_{470}) was measured at 670 and 470 nm for the chlorophylls and carotenoids, respectively, and the pigment contents were calculated using the following equations:

$$\text{Total chlorophylls}\left(\text{mg kg}^{-1}\right) = \frac{A_{670} \times 10^6}{613 \times 100 \times \text{density}},$$
$$\text{Total carotenoids}\left(\text{mg kg}^{-1}\right) = \frac{A_{470} \times 10^6}{2000 \times 100 \times \text{density}}. \quad (3)$$

2.5.4. Measurement of Oil Color. The oil color was measured by a spectrocolorimeter (MiniScan XE chromameter, HunterLab, Reston, VA) using the International Commission on Illumination's $L^*a^*b^*$ color scale. The L^* values range from 0 (black) to 100 (white), while a^* expresses the variation between green (-60) and red (+60), and b^* represents the variation between yellow (-60) and blue (+60).

2.5.5. Nonenzymatic Browning Index. The browning index (BI) of oil samples was measured according to the protocol described in the literature [23]. Briefly, the absorbance of the oil dissolved in chloroform (1/40; *w*/*v*) was measured at 420 nm. BI was expressed in arbitrary units.

2.5.6. Tocopherol Analysis. The tocopherol analysis was performed using a Shimadzu LC-6AD system (Shimadzu, Japan) equipped with a photodiode array detector (PDA). The chromatographic separation was carried out on a Uptisphere NH_2 column (internal diameter of 4.6 mm × 250 mm length and the particle size of 5 μm) according to the procedure described by Ben Moumen et al. [24], using a mobile phase composed of hexane and isopropanol (99/1; *v*/*v*). Identification and quantification were carried out at 292, 296, and 298 nm, using commercial tocopherol standards (Sigma-Aldrich, St. Louis, USA) with linear concentrations ($r^2 > 0.99$) of 10–360, 1–36, 50–1400, and 20–540 μg g^{-1} for α, β, γ, and δ tocopherols, respectively. LOQ and LOD were, respectively, 43.16 and 14.24 μg g^{-1} for α, 1.56 and 0.51 μg g^{-1} for β, 84.73 and 27.96 μg g^{-1} for γ, and 50.42 and 16.64 μg g^{-1} for δ.

2.5.7. Fatty Acid Analysis. Fatty acids were analyzed using gas chromatography (GC HP 6890) equipped with a flame ionization detector (FID). Before GC analysis, fatty acids were transformed into fatty acid methyl esters (FAMEs) according to the AOCS Ce 1K-07 method [25]. FAME separation was performed on a capillary column (omega wax: 30 m × 0.25 mm and 0.25 μm film thickness; Agilent Technologies, CA., USA) as described in the literature [26], using helium as carrier gas (flow rate: 1 mL min^{-1}). The fatty acids were identified by comparing their retention times with a FAME standard (Sigma-Aldrich, St. Louis, USA). The identified fatty acids were expressed as the relative percentage of total fatty acids (%).

2.5.8. Volatile Profile of Hemp Seed. Volatile compounds of hemp seeds were extracted and concentrated using solid-phase microextraction with DVB/carboxen/PDMS (50/30 μm) SPME fiber (Supelco Co., Bellefonte, PA), which was previously used for roasted almonds [27] and sunflower seeds [28]. The fiber was conditioned at 270°C for 30 min used to extract volatile compounds from 3 g of ground hemp seeds. An internal standard solution of 1,2,3-trichloropropane (Sigma-Aldrich, St. Louis, USA) in ethanol was prepared in a cold room (5°C) and then diluted in ultrapure water. Two hundred microliters of the internal standard solution was added to the sample in a 20 mL headspace extraction vial to give a final concentration of 25 ng g^{-1}. The flasks were then thermally equilibrated at 40°C for 20 min. After equilibration, the SPME fiber was exposed to the headspace for 30 min at 40°C. The volatile compounds absorbed by the fiber were thermally desorbed in the injection port of the GC for 10 min at 260°C in splitless mode.

The analysis of volatile compounds was carried out on a GC-7890A (Agilent Technologies, Palo Alto, CA, USA) coupled with a selective mass detector (HP 5975C; Agilent Technologies, Palo Alto, CA, USA). The chromatographic separation was performed on an HP-5MS column (30 m × 0.25 mm and 0.25 μm film thickness; Agilent Technologies, CA, USA), using helium at 1 mL min^{-1} as carrier gas. The column oven was initially set at 40°C for 5 min. The temperature was then programmed at 5°C min^{-1} to 250°C and maintained for 5 min until the end of the analysis. Mass spectra were recorded with a 4.27 scans/s speed in the m/z range of 35 to 500 using an electron impact ionization mode of 70 eV (quadrupole temperature: 150°C and source temperature: 230°C). Compound identification was based on comparison with NIST database spectra and Kovats retention index, determined by injecting a mixture of C7-C30 alkanes (1 μg mL^{-1} in hexane; Supelco Co., Bellefonte, PA, USA) under the same conditions described above. Authentic standards from Sigma-Aldrich (St. Louis, USA) were also used to identify benzaldehyde, caryophyllene, 2,5-dimethylpyrazine, 2-ethyl-3,5-dimethylpyrazine, furfural, hexanal, hexan-1-ol, D-limonene, myrcene, 2-methylbutanal,

Table 2: Regression equation coefficients and model fitting for oil yield, total phenolic compounds (TPC), free radical scavenging activity (RSA), and oxidative stability index (OSI) of hemp seed oils.

Responses (Y)	β_0	β_1	β_2	β_{12}	β_{11}	β_{22}	R^2	Adjusted R^2	p value	Lack of fit
Oil (%)	-18.045	0.488	0.124	-0.001	-0.001	0.001	0.83	0.71	0.0126	0.5924
TPC (mg kg^{-1})	101.077	-1.920	-4.819	0.057	0.010	-0.041	0.95	0.91	0.0003	<0.0001
RSA (%)	-378.857	4.127	4.443	-0.018	-0.009	-0.017	0.99	0.98	<0.0001	0.0874
OSI (h)	-298.951	4.084	-0.857	0.007	-0.013	-0.001	0.94	0.90	0.0004	0.0527

$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2$, with X_1: temperature (°C) and X_2: time (min).

3-methylbutanal, nonanal, α-pinene, phenylacetaldehyde, and α-terpinene.

The whole spectrum was scanned with the total ion chromatogram (TIC) mode. The relative concentrations of each identified volatile compound in raw and roasted hemp seeds were determined using the extracted ion peak area of 1,2,3-trichloropropane and expressed as ng equivalent of 1,2,3-trichloropropane per gram of hemp seed. The relative concentrations were used to evaluate the differences in volatile profiles between raw and roasted hemp seeds.

3. Results and Discussion

3.1. Optimal Roasting Conditions for Hemp Seeds. The RSM was used to optimize hemp seeds' roasting temperature and time. Thirteen experiments were carried out randomly according to the CCD. The responses of these experiments, including oil yield (Y_1), TPC (Y_2), RSA (Y_3), and OSI (Y_4), are shown in Table 1. The prediction models of these responses as a function of roasting temperature (X_1) and time (X_2) can be described by the second-order polynomial equations presented in Table 2. The analysis of variance showed that the four prediction models were significant ($p < 0.05$), with a high correlation coefficient (R^2) varied from 0.83 to 0.99, ensuring a good fit of the regression equations to the experimental data. The obtained R^2 coefficients indicate that the regression models can explain 83, 94, 95, and 99% of the variation in the response of oil yield, OSI, TPC, and RSA, respectively. Furthermore, the lack fit test showed nonsignificant results ($p > 0.05$), indicating the validity of the models, except for the TPC model, which exhibited a lack of fit ($p < 0.05$) but with a high R^2 value.

The Pareto diagram represents the different effects of roasting temperature and time (linear, interaction, and quadratic effects) on the studied responses in a standardized manner. Each effect was represented on the diagram by a bar, whose length is proportional to the absolute magnitude of its estimated coefficient (Supplementary Table S1). It can be seen from the Pareto chart corresponding to oil yield (Figure 1(a)) that the linear effect of roasting time is the variable with the most significant influence, followed by the quadratic effect of temperature. These two effects negatively influence the oil yield. As shown in Table 1, the experimental results vary between 19.70 (roasted seeds at 180°C for 45 min) and 22.70% (roasted seeds at 160°C for 9 min). A remarkable increase in oil yield can be observed on the response surface with the increased roasting temperature up to 160°C (Figure 2(a)). However, a further increase in temperature causes, on the contrary, a decrease in oil yield. These results also showed that prolonged roasting of seeds at the same temperature causes a significant decrease in oil yield, especially at high temperatures. These variations could be related to changes in the microstructure of hemp seeds during roasting. In fact, the roasting process vaporizes water from the microstructure of the seed, which is accompanied by cell membrane rupture, thus leading to a more significant rupture of tissues [29]. This makes the plant material more brittle, facilitating the extractability of the oil during pressing. In addition, roasting could decrease the oil's absolute viscosity while increasing the effective diffusion coefficients of the components constituting the extracted oil [12]. However, roasting at high temperatures and/or for a long duration causes a substantial decrease in the seeds' water content, reducing the plasticity and elasticity of the plant material [29, 30]. The seeds would thus be reduced to powder during the screw press extraction, which would not allow the pressure to be maintained during pressing. Therefore, the low oil extraction rates observed in this work after roasting hemp seeds at high temperatures and long duration could be due to a significant decrease in the hemp seeds' water content. As shown in Table 1, the moisture content decreases significantly with the roasting temperature and time, and the maximum oil yield is observed for roasted seeds at 160°C for 9 min with a moisture content of 6.07%. The response surface of oil yield (Figure 2(a)) showed that moderate roasting conditions of hemp seeds (160°C for 9 to 30 min) make it possible to increase the extraction yield. Ferchau [31] thus reported in his book that a low water content of 6.5% in rapeseed and canola seeds could lead to a low oil yield. In contrast, Wroniak et al. [9] reported that a moisture content of 9% was ideal for obtaining a maximum yield of rapeseed oil by cold pressing.

Therefore, it was interesting to see if the heat treatment influenced the quality of these oils obtained after roasting. First, the TPC of the analyzed oil samples was compared after different roasting times and temperatures. Table 1 shows that the TPC of the oil samples fluctuates between 65.29 (roasted seeds at 140°C for 15 min) and 277.44 GAE mg kg^{-1} (roasted seeds at 140°C for 15 min). The ANOVA results (Figure 1(b)) showed that the TPC is positively influenced by roasting temperature and time ($p < 0.05$). As shown by the response surface presented in Figure 2(b), a significant increase in TPC was recorded at a fixed roasting time with increased temperature. A similar TPC trend was

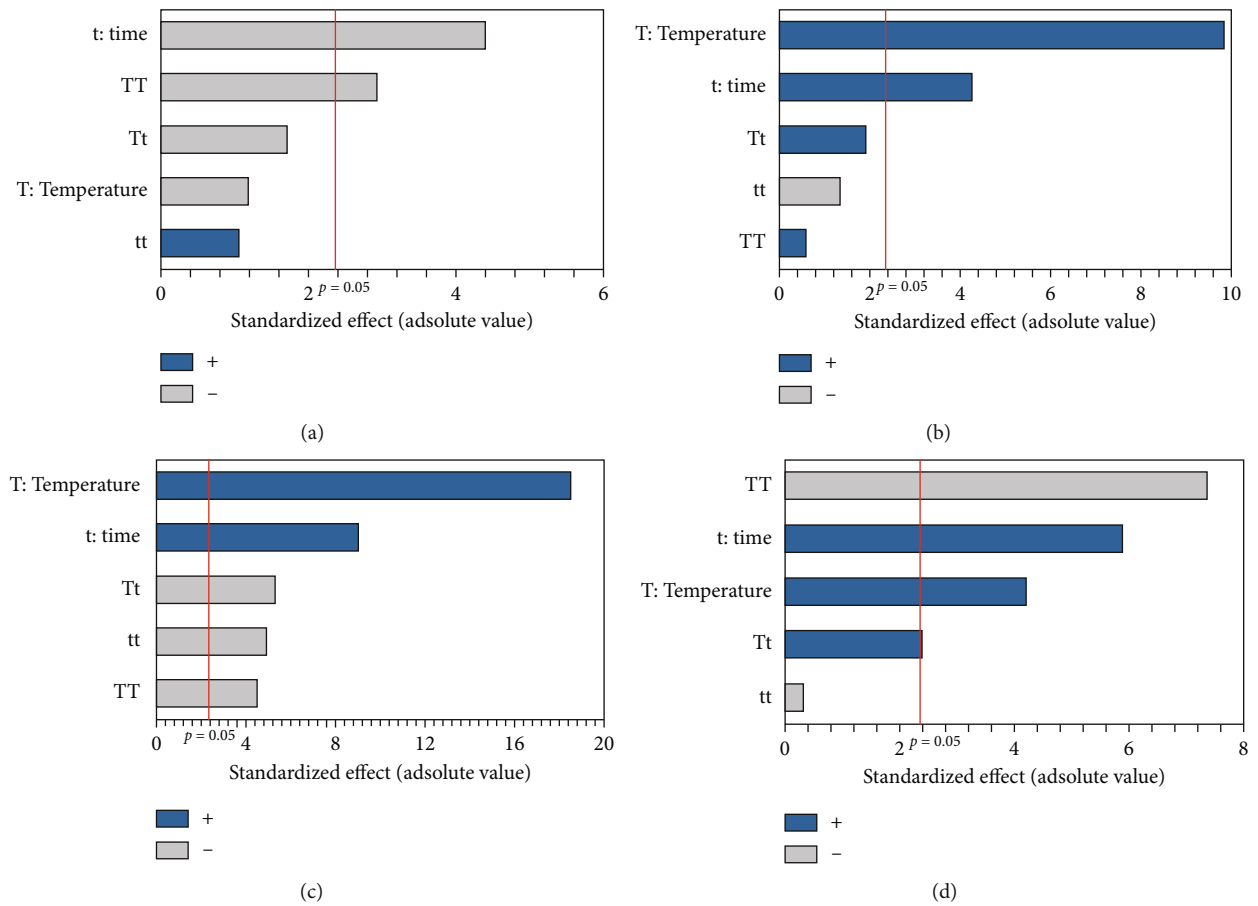

Figure 1: Standardized Pareto charts obtained from the optimization study of the roasting variables (temperature and time) of hemp seeds for (a) oil yield, (b) total phenolic content, (c) DPPH radical scavenging activity, and (d) oxidative stability index.

observed for roasting time. At a fixed temperature, prolonged roasting significantly improved the TPC. These results suggest that roasting facilitates the transfer of phenolic compounds into the oil during extraction. This could be due to the release of phenols from their bonds with other cellular constituents, mainly in the hull [32, 33]. Roasting at high temperatures could also cause a chemical alteration of phenolic compounds, facilitating their transfer into the oil. In addition, the Maillard reaction, which can occur in the seed during roasting, would increase the proportion of fat-soluble phenols via the interaction of carbonyls with amines [34]. This reaction also generates compounds, such as furans and pyrroles, which can react with Folin's reagent, thus leading to an overestimation of the TPC [35].

In addition to the increase in TPC, the roasting process enhanced the antioxidant capacity of oils extracted from roasted hemp seeds. The DPPH radical scavenging activity (RSA) increased from 45.06% for the oil extracted from roasted seeds at 140°C for 15 min to 88.56% for roasted seeds at 188°C for 30 min (Table 1). The antioxidant activity of the hydromethanolic extract improved linearly with the increase in temperature and roasting time ($p < 0.05$) to reach a maximum plateau within 10 to 40 min after roasting between 160 and 180°C (Figure 2(c)). On the contrary, an additional increase in temperature and/or time significantly ($p < 0.05$) decreased antioxidant activity (Figure 1(c)). The increased RSA could be mainly linked to the phenolic compounds in the analyzed oils. Several studies have shown that the improvement in the antioxidant capacity of oils is closely related to the increase in phenolic compounds after roasting oilseeds [10, 33]. In addition, the Maillard reaction could form other melanoidin-like compounds with antioxidant power during the roasting process. These products could improve the radical scavenging power, increasing antioxidant capacity [36]. Following an excessive increase or extension of the roasting time, the decrease in RSA is probably due to the loss of certain thermolabile antioxidant compounds during prolonged roasting and at high temperatures.

Oxidative stability is one of the essential qualities of edible oil. It determines the shelf life and potential use of the oil. In this work, the oxidative stability of the oil samples was evaluated by determining the oxidation induction time using the accelerated aging method of the Rancimat test. Although this method has the disadvantage of accelerating oxidation reactions in conditions far from natural storage conditions, it allows for a simple and easy comparison between several oil samples subjected to the same conditions. The OSI results of the analyzed roasted samples fluctuated between 17.08 (roasted at 132°C for 30 min) and 34.99 h (roasted at 180°C for 45 min). Therefore, the results show that the roasting of

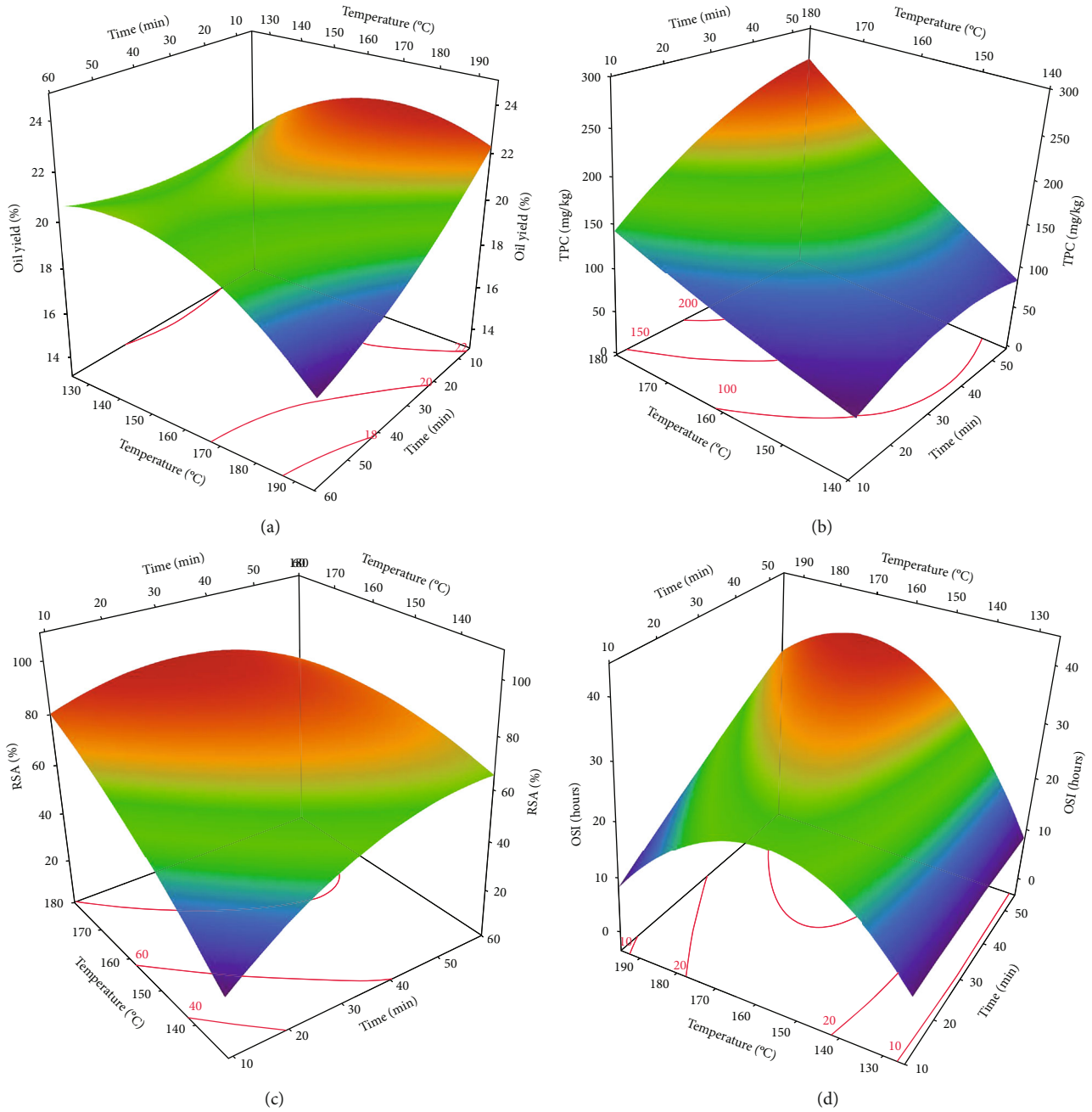

FIGURE 2: The 3D surface responses of roasting temperature and time effects on (a) oil yield, (b) total phenolic content (TPC), (c) radical scavenging activity (RSA), and (d) oxidative stability index (OSI) of hempseeds.

hemp seeds influenced the oxidative stability of the extracted oil. Except for the quadratic effect of roasting time, all effects of roasting conditions significantly ($p < 0.05$) influenced OSI (Figure 1(d)). The linear effects of temperature and time and their interaction caused an improvement in OSI. However, the quadratic effect of temperature negatively influenced the OSI, indicating that roasting at high temperatures causes a decrease in OSI. As mentioned above for RSA, this decrease in OSI could be linked to the destruction of certain thermolabile antioxidant compounds. Thus, high temperatures could accelerate oil oxidation, resulting in the generation of oxidation products.

On the other hand, the increased oxidative stability of oils from samples roasted between 160 and 180°C for 30 to 50 min (Figure 2(d)) could be attributed to the better extraction of natural antioxidants, such as phenolic compounds, and the generation of Maillard reaction products (MRPs). Subsequent studies have indeed reported that roasting improves the oxidative stability of oils produced from rapeseed [30], walnuts [34], and argan kernels [16] by increasing the extractability of antioxidants such as phenolic compounds. Shrestha et al. [37] also pointed out that MRPs can enhance the oxidative stability of roasted seed oils.

In order to obtain oil with better oxidative stability, hemp seeds should be roasted at 180°C for 45 min. Although the oil yield was not maximum (19.70%) at these conditions, the oil obtained had better quality with a high TPC (277.44 mg kg^{-1}), RSA of 87.56%, and OSI of 34.99 h. Therefore, the oil yield (Y_1) was used as the principal response. The hemp seed pretreatment parameters, allowing for maximum oil yield, were obtained by a first derivation of the second-degree polynomial equation of the Y_1 response. Through processing the data, the optimal roasting conditions predicted by the model were 163°C and 15 min. A repeatable study was conducted using these optimal conditions to assess the model's predictive capacity. Under the optimized conditions, the experimental results obtained were 23.09 ± 0.43% for the oil yield, with TPC of 121.21 ± 5.36 mg kg^{-1}, RSA of 72.06 ± 1.37%, and OSI of 21.37 ± 1.51 h (Table 3). These experimental results highly correlated with the predicted values ($R^2 = 0.82$ and adjusted $R^2 = 0.73$). Therefore, the RSM procedure could be effectively applied to predict the oil quality of roasted hemp seeds. In addition, optimizing the hemp seed roasting process has improved the yield and quality of the pressed oil, enhancing its antioxidant capacity, as has been shown for argan [16] and almond kernels [17]. The oil yield obtained in our study (23.09%) was improved significantly by about 45% compared to that observed for unroasted seeds (15.85%). The moisture content of hemp seeds roasted at 163°C for 15 min, corresponding to this yield, was 5.98 ± 0.19% (Table 3).

The TPC of the oil extracted from the roasted hemp seeds at 163°C for 15 min recorded a very significant increase of about two times compared to the content observed for the oil extracted from unroasted seeds (64.42 mg kg^{-1}). This significantly improved the oil's RSA and OSI, from 43.67 to 72.06% and 11.83 to 21.37 h, respectively. Therefore, the oil from the seeds roasted under optimum conditions has a good oxidation resistance and would allow for a better storability of about 80% compared to the oil extracted from unroasted hemp seeds.

3.2. Comparison of Unroasted and Roasted Hemp Seeds after Optimization by RSM. The quality of the oil produced from hemp seeds roasted under optimal conditions (163°C for 15 min) was compared to that of unroasted seeds. The oil parameters evaluated were the quality indices, fatty acid and tocopherol composition, color, pigment content (chlorophylls and carotenoids), and nonenzymatic browning index. Thus, changes in volatile compounds after roasting were assessed at the whole-seed level.

3.2.1. Oil Quality Indices. The quality of vegetable oils is generally assessed by measuring different chemical parameters. Thus, the level of deterioration of the oil is determined by measuring the level of hydrolytic degradation of fat (free acidity: FA) and its degree of primary oxidation by the dosage of the primary oxidation products (peroxide value: PV) and conjugated dienes and trienes (CD and CT). The results of these parameters in the analyzed samples are shown in Table 3. The oil samples from roasted and unroasted hemp seeds had FA values of 2.97 and 2.95 mg KOH g^{-1}, PV of 2.99 and 2.98 meq O_2 kg^{-1}, CD of 0.26 and 0.28, and CT of 0.11 and 0.12. These results are lower than the standard recommended for crude vegetable oils [38]. Moreover, roasting hemp seeds at 163°C for 15 min did not influence the extracted oil's FA, PV, and CT ($p > 0.05$). However, roasting slightly increased the CD ($p < 0.05$).

Subsequent research findings from several studies have indicated that the quality indices of vegetable oils can vary during the roasting process, depending on the roasting

Table 3: Seed moisture and oil characteristics of raw and roasted hemp seeds at 163°C for 15 min.

Parameters	Raw hemp seeds	Roasted hemp seeds[a]
Moisture (%)	8.25 ± 0.18	5.98 ± 0.11*
Oil (%)	16.18 ± 0.76	23.09 ± 0.43*
Free acidity (mg KOH g^{-1})	2.97 ± 0.32	2.95 ± 0.31
Peroxide value (mg O_2 kg^{-1})	2.99 ± 0.06	2.98 ± 0.10
Conjugated diene (λ232 nm)	0.26 ± 0.01	0.28 ± 0.01*
Conjugated triene (λ270 nm)	0.11 ± 0.01	0.12 ± 0.01
Total chlorophylls (mg kg^{-1})	39.10 ± 0.22	36.54 ± 0.36*
Total carotenoids (mg kg^{-1})	10.55 ± 0.77	10.05 ± 0.79
Color parameters		
L^*	19.94 ± 0.20	20.10 ± 0.21
a^*	2.32 ± 0.09	2.33 ± 0.10
b^*	1.25 ± 0.08	1.61 ± 0.12*
Browning index	0.33 ± 0.01	0.37 ± 0.02*
Total phenol content (mg GAE kg^{-1})	64.42 ± 4.46	121.21 ± 5.36*
Free radical scavenging activity (% DPPH)	43.67 ± 0.59	72.06 ± 1.37*
Oxidative stability index (hours)	11.83 ± 0.75	21.37 ± 1.51*
Tocopherols (mg kg^{-1})		
γ-Tocopherol	426.91 ± 5.00	417.02 ± 4.28*
α-Tocopherol	31.83 ± 0.94	35.82 ± 1.17*
β-Tocopherol	11.27 ± 0.40	6.86 ± 0.41*
δ-Tocopherol	13.05 ± 0.46	10.07 ± 0.52*
Total tocopherols	483.06 ± 6.06	469.77 ± 4.80*
Fatty acids (%)		
Palmitic acid	7.65 ± 0.37	7.29 ± 0.16*
Stearic acid	2.48 ± 0.10	2.45 ± 0.06
Oleic acid	19.34 ± 0.26	19.73 ± 0.11*
Linoleic acid	52.50 ± 0.31	52.59 ± 0.48
γ-Linolenic acid	1.94 ± 0.34	1.71 ± 0.35
α-Linolenic acid	16.09 ± 0.21	16.22 ± 0.44
SFA	10.13 ± 0.40	9.74 ± 0.21*
MUFA	19.34 ± 0.26	19.73 ± 0.11*
PUFA	70.53 ± 0.45	70.53 ± 0.25

[a]Hemp seeds were roasted at 163°C for 15 min. *Significant at $p < 0.05$ for unroasted hemp seeds vs. roasted hemp seeds.

temperature and time [12]. Anjum et al. [39] reported that moderate microwave roasting of sunflower seeds increases the PV and CD of the oil over time. These variations can be attributed to the oxidation of fatty acids during roasting, which initially results in the accumulation of primary oxidation products (increased PV) followed by a decrease in PV due to their decomposition into secondary products. Consequently, oils obtained from roasted seeds for a long duration tend to have low PV values [40]. Our results indicate that the oil would be relatively thermally stable when hemp seeds are roasted at 163°C for 15 min. The richness of hemp seeds in natural antioxidants, particularly in phenolic compounds, could contribute to the oil's antioxidant protection, thereby limiting the production of primary oxidation products.

On the other hand, the absence of a significant difference in the FA value after roasting hemp seeds can be attributed to the formation of aromas during the primary phase of the Maillard reaction, which involves several compounds like free amino acids, peptides, nucleotides, and lipids [41]. Additionally, volatile-free acids in hemp seed evaporate during roasting at 163°C for 15 min.

3.2.2. Pigment Content and Oil Color. The oil color is mainly related to its pigment content (mainly chlorophylls and carotenoids). The carotenoid and chlorophyll contents of the analyzed samples are presented in Table 3. The oil obtained from unroasted hemp seeds had total carotenoid and chlorophyll contents of 10.55 and 39.10 mg kg^{-1}, respectively. The oil from hemp seeds roasted at 163°C for 15 min contained 10.55 and 36.54 mg kg^{-1}, respectively. The chlorophyll content decreased slightly after roasting ($p < 0.05$). This could be due to the thermal degradation of chlorophylls when roasting hemp seeds under optimal conditions. However, the total carotenoid content did not differ significantly ($p > 0.05$) between the analyzed samples. Similar results were reported by Vaidya and Eun [42], pointing out that roasting mustard seeds at 165°C for 30 min does not affect the lutein content, the main component of carotenoids in mustard oil. Conversely, other studies have shown that roasting improves the pigment content of the oil [23].

The color parameters (expressed as L^*, a^*, and b^*) of the oil samples extracted from unroasted and roasted hemp seeds are summarized in Table 3. Except for the yellow index (b^*), which significantly increased ($p < 0.05$), the roasting of hemp seeds at 163°C for 15 min did not influence the oil color parameters ($p > 0.05$). The oil extracted from the unroasted seeds had L^*, a^*, and b^* of 19.94, 2.32, and 1.25, respectively, while the oil obtained from the roasted seeds had values of 20.10, 2.33, and 1.61, respectively. The increase in the b^* value may be due to the decrease in the content of chlorophylls compared to carotenoids in oils after roasting the hemp seeds (Table 3). In fact, the oil color depends on the concentration of chlorophylls (responsible for the green color) and carotenoids (responsible for the yellow color). Several studies have shown that roasting significantly affects oil color [23, 43]. These studies have reported that the oils obtained after roasting seeds are darker than those extracted from unroasted seeds. This change could be related to the production of colored MRPs and the variation in pigment content, particularly the ratio between chlorophylls and carotenoids. The latter would be more resistant than chlorophylls to heat treatment.

3.2.3. Browning Index. Evaluating the nonenzymatic browning index (BI) makes it possible to determine the generation of MRPs and their passage in the oil after extraction. After roasting, the BI recorded a slight significant increase ($p < 0.05$), from 0.33 in the oil obtained from unroasted seeds to 0.37 in the oil extracted from roasted seeds (Table 3). These results suggest that roasting conditions of 163°C for 15 min caused the formation of browning products in hemp seeds, such as melanoidins, via the Maillard reaction [44]. The slight difference observed in this work would result in a low production of MRPs during the roasting process. As already indicated, roasting could initiate the formation of melanoidins characterized by a brown color [44], thus leading to a variation in the color of the oil, which changes to a darker color due to brown color development. This change in BI was not reflected in the color of hemp seed oil after roasting, evaluated by spectrocolorimeter, particularly the L^* value, which did not show a significant difference (Table 3).

3.2.4. Tocopherols. Tocopherols are lipophilic antioxidants that play an essential role in protecting oil against oxidation [45]. Therefore, assessing the variation in tocopherols after roasting hemp seeds is necessary. The content of α-, β-, γ-, and δ-tocopherol isomers and their total content in the oils of roasted and unroasted seeds are presented in Table 3. Compared to the oil extracted from the unroasted seeds, a slight significant decrease ($p < 0.05$) was observed in the total tocopherol content (from 483.06 to 469.77 mg kg^{-1}). The b, g, and d isomers showed a decrease in their contents, ranging from 426.91, 11.27, and 13.05 mg kg^{-1} in the unroasted seed oil to 417.02, 6.86, and 10.07 mg kg^{-1} in the oil extracted from roasted seeds at 163°C for 15 min, respectively. However, hemp seeds roasted under these conditions significantly increased the α-tocopherol content from 31.83 to 35.82 mg kg^{-1}. A recent study by Karrar et al. [46] found similar results and reported that α-tocopherol content increased in gurum seed oil after 2-4 min of microwave roasting and then decreased after 6 minutes. The study also observed a significant reduction for β, γ, and δ isomers during all roasting periods. Several studies have reported that moderate roasting of certain nuts and seeds improves the total tocopherol content of oils, improving their extractability and releasing certain tocopherols bound to proteins and phospholipids [34, 43]. However, excessive roasting decreases the tocopherol content of oils [47, 48]. In addition, Vaidya and Eun [42] suggest that MRPs formed during roasting serve as the preferred antioxidants before tocopherols, which likely contributes to the protection of tocopherols and their higher content in the extracted oil after roasting of seeds. In our study, roasting at 163°C for 15 min leads to a slight decrease in tocopherol content. This decrease could be due to the thermolability of tocopherols, particularly γ isomer. The decrease in γ-tocopherol could be explained by the fact that it would be more fragile and could participate through its oxidation

in protecting other tocopherols against possible oxidation favored by the roasting process [49].

3.2.5. Fatty Acids. As shown in Table 3, polyunsaturated fatty acids (PUFA) constitute 70.53% of fatty acids in the oil samples extracted from unroasted and roasted hemp seeds, followed by monounsaturated fatty acids (19.34 and 19.73%, respectively) and saturated fatty acids (10.13 and 9.74%, respectively). The unroasted hemp seed oil had 52.50 linoleic, 19.34 oleic, 16.09 α-linolenic, 7.65 palmitic, 2.48 stearic, and 1.94% γ-linolenic acids. The oil extracted after roasting consisted of 52.59 linoleic, 19.73 oleic, 16.22 α-linolenic, 7.29 palmitic, 2.45 stearic, and 1.94% γ-linolenic acids (Supplementary Figure S1). Roasting the hemp seeds at 163°C for 15 min had no negative impact on the fatty acid composition of the oil ($p > 0.05$), particularly on the PUFA fraction. The only exception was observed for oleic and palmitic acids, which showed slightly significant variations ($p < 0.05$). Under optimal conditions, the roasting process would not cause fatty acid oxidation or isomerization. This could be due to the participation of antioxidants in protecting fatty acids against oxidation during the roasting process, such as γ-tocopherol and chlorophylls, which showed a decrease in their levels after roasting hemp seeds. These results align with previous studies that showed little or no effect of proper roasting on the fatty acid composition of oil from flaxseeds [48] and apricot kernels [50]. Meanwhile, it has been reported that in walnut oil [51], the SFA content slightly increased after roasting, which is consistent with our results.

3.2.6. Volatile Compounds of Hemp Seeds. Table 4 shows 57 volatile compounds identified in hemp seeds, including 36 in the unroasted sample and 56 in the roasted sample. These compounds belong to the chemical classes of terpenes, aldehydes, ketones, alcohols, *N*-heterocycles (pyrazines, pyrimidines, and pyridines), furan derivatives, and hydrocarbons (alkanes and alkenes).

The volatile compounds of unroasted hemp seeds are predominated by terpenes (7352 ng g^{-1}), constituting more than 86%, followed by aldehydes (580.67 ng g^{-1}), alcohols (366.80 ng g^{-1}), hydrocarbons (194.28 ng g^{-1}), and ketones (7.55 ng g^{-1}). The identified terpenes consisted of twenty-four compounds. β-Myrcene, D-limonene, α-pinene, caryophyllene, and β-pinene were the most abundant terpenes identified in unroasted hemp seeds (3170.30, 1347.25, 1139.42, 495.26, and 436.08 ng g^{-1}, respectively). Several studies have identified these compounds as the most representative of the volatile compounds in hemp and its products [56–58]. They are responsible for the pleasant and characteristic flavor of hemp. Their levels in unroasted hemp seeds were above their olfactory thresholds reported in the literature (Table 4). It is accepted that aroma compounds are only perceived when their concentration is above the olfactory threshold. β-Myrcene has a balsamic and spicy aroma, and a pleasant lemony odor characterizes D-limonene. α- and β-pinenes and caryophyllene contribute to the green pine and woody-spicy odors, respectively. In addition, four aldehydes and four alcohols were identified in unroasted hemp seeds. Among them, hexanal and hexen-1-ol represented the most abundant compounds in these two chemical classes, with contents of 511.16 and 261.70 ng g^{-1}, respectively, above their olfactory thresholds. Several studies have reported the presence of hexanal and hexen-1-ol in various unroasted oilseeds, such as almonds [27] and sunflower seeds [28]. These compounds have been linked to the oxidation of PUFAs, contributing to green, oily, and fruity odors. Their presence in unroasted hemp seeds suggests that they were produced by lipoxygenase in the seed's lipid compartment, as has been reported for the biosynthesis of volatile compounds in olive oil [59].

The roasting process of hemp seeds at 163°C for 15 min caused the formation of 21 new compounds and a decrease in the levels of most identified volatile compounds in the unroasted seeds (Figure 3). After roasting, the content of terpenes decreased by 63.93%, from 7352 to 2651.60 ng g^{-1}, with the total disappearance of α-thujene. However, despite this decrease, terpenes still constitute the major compounds, constituting more than 69% of the compounds identified in the roasted hemp seeds, with a dominance of β-myrcene (1177.69 ng g^{-1}), D-limonene (470.35 ng g^{-1}), α-pinene (405.11 ng g^{-1}), caryophyllene (165.21 ng g^{-1}), and β-pinene (153.28 ng g^{-1}). The concentrations of these compounds remained above their olfactory thresholds. In addition, the levels of hexanal and hexen-1-ol decreased (57.62 and 53.99%, respectively) after roasting the hemp seeds to levels below their olfactory thresholds.

The new compounds formed after roasting belong mainly to the chemical classes of aldehydes, alcohols, *N*-heterocycles (pyrazines, pyridines, and pyrimidines), furan derivatives, ketones, and hydrocarbons. Most of these compounds were produced from a series of complex reactions responsible for the characteristic taste properties of roasted products, such as the Maillard reaction, fatty acid oxidation, and amino acid degradation [12]. Pyrazines are associated with the attractive odor of heat-treated foods, contributing to the typical roasted and nutty aromas [60]. However, they have a high odor threshold, particularly pyrazines with methyl groups. Their threshold decreases significantly by replacing methyl groups with ethyl groups [60]. Table 4 shows 2,5-dimethylpyrazine, 2,3-dimethylpyrazine, 2-ethyl-3,5-dimethylpyrazine, and 3-ethyl-2,5-dimethylpyrazine which were detected in roasted hemp seeds with concentrations of 19.67, 6.31, 13.91, and 19.78 ng g^{-1}, respectively. These compounds were formed via the Maillard reaction during the roasting process of hemp seeds, involving the carbonyl group of reducing sugars and the free amine function of amino acids [60]. Pyrazines are, according to several authors, the compounds responsible for the typical roasted, hazelnut, and cocoa aromas of certain roasted seeds, such as sunflower seeds [28], pumpkin seeds [61], and almonds [27]. However, only 2-ethyl-3,5-dimethylpyrazine was present above its olfactory threshold, suggesting that this compound is considered active and could be detected in roasted hemp seeds, giving a roasted odor.

The compounds containing furan are also known as typical MRPs. They could be produced from the thermal degradation of glucose and fructose [62]. The formation of these compounds contributes to the caramel, sweet, fruity, and nutty sensory characteristics of roasted foods [63]. Three

Table 4: Identification of volatile compounds in raw and roasted (at 163°C for 15 min) hemp seeds and their relative concentrations ($ng\,g^{-1}$).

Peak N°	Volatiles compounds	KI[a]	ID[b]	Odor description[c]	OT ($ng\,g^{-1}$)[d]	Relative concentration ($ng\,g^{-1}$) Unroasted	Roasted	Increase (%)[e]
	Aldehydes							
1	Acetaldehyde	<800	MS	Pungent	17	41.65 ± 5.04	2.91 ± 0.37*	-93.01
2	2-Methylpropanal	<800	MS	Pungent, malt, green	3.4	nd[f]	55.31 ± 9.38*	New
3	3-Methylbutanal	<800	MS, S	Malt	23	nd	108.17 ± 15.85*	New
4	2-Methylbutanal	<800	MS, S	Cocoa, almond	10.8	nd	250.77 ± 28.34*	New
10	Hexanal	801	MS, KI, S	Grass, tallow, fat	479	511.16 ± 52.85	216.61 ± 50.54*	-57.62
18	Heptanal	898	MS, KI	Fat, citrus, rancid	50	12.43 ± 1.18	9.99 ± 1.63*	-19.63
25	Benzaldehyde	958	MS, KI, S	Almond, burnt sugar	3600	nd	17.66 ± 1.05*	New
38	2-Phenylacetaldehyde	1043	MS, KI, S	Hawthorne, honey, sweet	22	nd	10.71 ± 1.88*	New
45	Nonanal	1104	MS, KI, S	Fat, citrus, green	260	15.43 ± 1.26	7.10 ± 2.53*	-53.99
	Total aldehydes					580.67 ± 51.91	679.23 ± 88.75*	16.97
	Alcohols							
5	Cyclopentanol	<800	MS	—	—	nd	20.89 ± 3.84*	New
9	Pentan-1-ol	<800	MS	Balsamic	470	nd	25.70 ± 5.78*	New
14	Hexan-1-ol	864	MS, KI, S	Resin, flower, green	400	261.70 ± 30.61	105.51 ± 20.77*	-59.68
27	1-Octen-3-ol	979	MS, KI	Mushroom	36	nd	9.41 ± 1.18*	New
44	Linalool	1099	MS, KI	Flower, lavender	6	46.00 ± 4.99	16.27 ± 3.49*	-64.63
46	α-Fenchol	1113	MS, KI	Camphor	—	53.15 ± 4.88	16.63 ± 4.46*	-68.70
47	Borneol	1166	MS, KI	Camphor	140	5.95 ± 0.38	1.42 ± 0.52*	-76.20
	Total alcohols					366.80 ± 36.44	195.83 ± 32.89*	-46.61
	Ketones							
17	Heptan-2-one	887	MS, KI	Soap	1500	nd	42.49 ± 5.89*	New
28	6-Methylhept-5-en-2-one	987	MS	Pepper, mushroom, rubber	50	7.55 ± 1.17	8.98 ± 5.07	18.95
	Total ketones					7.55 ± 1.17	51.47 ± 8.38*	581.43
	Terpenes							
—	α-Thujene	924	MS, KI	Wood, green, herb	980	5.56 ± 1.48	nd*	nd
22	α-Pinene	930	MS, KI, S	Pine, turpentine	6	1139.42 ± 63.11	405.11 ± 71.28*	-64.45
24	Camphene	944	MS, KI	Camphor	—	50.80 ± 1.81	18.51 ± 3.16*	-63.57
26	β-Pinene	973	MS, KI	Pine, resin, turpentine	140	436.08 ± 9.04	153.28 ± 26.51*	-64.85
29	β-Myrcene	990	MS, KI, S	Balsamic, must, spice	15	3170.30 ± 78.91	1177.69 ± 193.30*	-62.85
31	δ-3-Carene	1007	MS, KI	Lemon, resin	770	39.95 ± 0.65	19.12 ± 2.10*	-52.15
32	α-Terpinene	1014	MS, KI, S	Lemon	85	13.25 ± 0.57	6.60 ± 1.85*	-50.15
33	Ocimene	1018	MS	Balsamic	—	9.85 ± 1.18	4.42 ± 1.54*	-55.08
34	*p*-Cymene	1023	MS, KI	Solvent, gasoline, citrus	11.4	32.10 ± 0.91	15.62 ± 2.28*	-51.32
35	D-limonene	1027	MS, KI, S	Lemon, orange	34	1347.25 ± 33.75	470.35 ± 81.44*	-65.09
36	L-limonene	1030	MS, KI	Citrus, mint	200	110.99 ± 9.67	31.95 ± 5.13*	-71.22
37	*cis*-β-Ocimene	1037	MS, KI	Sweet, herb	—	35.49 ± 1.86	11.55 ± 2.47*	-67.45
39	*trans*-β-Ocimene	1048	MS, KI	Citrus, herb, flower	34	140.95 ± 1.73	52.66 ± 8.40*	-62.64
40	γ-Terpinene	1058	MS, KI	Gasoline, turpentine	1000	31.30 ± 2.01	13.79 ± 3.02*	-55.95

Table 4: Continued.

Peak N°	Volatiles compounds	KI[a]	ID[b]	Odor description[c]	OT ($ng\,g^{-1}$)[d]	Relative concentration ($ng\,g^{-1}$) Unroasted	Roasted	Increase (%)[e]
43	α-Terpinolene	1087	MS, KI	—	200	134.19 ± 5.73	55.29 ± 8.31*	-58.80
48	Isocaryophyllene	1411	MS, KI	Wood	—	6.49 ± 0.47	2.08 ± 0.36*	-68.04
49	α-Gurjunene	1413	MS	Wood, balsamic	—	3.13 ± 0.04	0.82 ± 0.18*	-73.66
50	α-Longipinene	1418	MS	—	—	3.60 ± 0.40	0.98 ± 0.24*	-72.79
51	β-Caryophyllene	1424	MS, KI, S	Wood, spice	64	495.26 ± 3.67	165.21 ± 42.74*	-66.64
52	Aromadendrene	1432	MS	Wood	—	8.97 ± 0.31	2.59 ± 0.70*	-71.16
53	α-Bergamotene	1439	MS, KI	Wood, warm, tea	—	14.64 ± 0.43	4.53 ± 1.15*	-69.03
54	α-Humulene	1458	MS, KI	Wood	160	99.59 ± 0.89	32.10 ± 8.04*	-67.77
55	*allo*-Aromadendrene	1466	MS, KI	—	—	16.59 ± 0.72	5.61 ± 1.50*	-66.15
56	β-Selinene	1492	MS	Herb	—	6.26 ± 0.94	1.75 ± 0.59*	-72.12
	Total terpenes					7352.00 ± 139.96	2651.60 ± 453.64*	-63.93
	Pyrazines							
19	2,5-Dimethylpyrazine	908	MS, KI, S	Cocoa, roasted nut	800	nd	19.67 ± 4.11*	New
22	2,3-Dimethylpyrazine	923	MS	Nut, peanut butter, cocoa	100	nd	6.31 ± 0.80*	New
41	3-Ethyl-2,5-dimethylpyrazine	1079	MS, KI	Potato, roast	79	nd	19.78 ± 4.12*	New
42	2-Ethyl-3,5-dimethylpyrazine	1082	MS, KI, S	Roast	7.5	nd	13.91 ± 0.55*	New
	Total pyrazines						59.67 ± 7.43*	New
	Furan derivatives							
7	2-Ethylfuran	<800	MS	Butter, caramel-like, burnt	8000	nd	29.69 ± 6.10*	New
11	Furfural	829	MS, KI, S	Bread, almond, sweet	3000	nd	16.86 ± 4.94*	New
15	2-Methyl-5-propylfuran	882	MS	—	—	nd	3.12 ± 0.77*	New
	Total furan derivatives						32.81 ± 6.68*	New
	Other compounds							
6	Heptane	<800	MS	Alkane	—	nd	52.10 ± 8.82*	New
8	Toluene	<800	MS	Paint	527	nd	8.44 ± 2.01*	New
12	Ethylbenzene	854	MS, KI	—	2205.25	32.00 ± 1.93	10.63 ± 2.01*	-66.77
13	*p*-Xylene	862	MS, KI	—	1000	nd	1.26 ± 0.24*	New
16	Styrene	885	MS, KI	Balsamic, gasoline	—	91.93 ± 22.76	9.41 ± 4.08*	-89.77
20	4,6-Dimethylpyrimidine	912	MS	—	—	nd	6.53 ± 1.04*	New
21	3-Ethylidene-1-methylcyclopentene	914	MS	—	—	70.35 ± 3.44	38.46 ± 6.80*	-45.32
20	Pyridin-2-amine	997	MS	—	—	nd	19.38 ± 5.95*	New

[a]Kovats index calculated for HP-5MS capillary column (30 m × 0.25 mm and 0.25 μm). [b]Identification method: MS, identification based on the NIST mass spectral database; KI, Kovats index on HP-5MS capillary column [52–54]; S, identification was performed using authentic standard compounds. [c]Odor description was obtained from http://flavornet.org/flavornet.html. [d]Odor threshold values were obtained from the literature [28, 53, 55]. [e]Increase% = [(concentration in roasted hemp seeds at 163 ∘ C for 15 min) − (concentration in unroasted hemp seeds)] ∗ 100/(concentration in unroasted hemp seeds). [f]Not detected. *Significant at $p < 0.05$ for unroasted hemp seeds vs. roasted hemp seeds.

furan compounds were identified in roasted hemp seeds: 2-ethylfuran, furfural, and 2-methyl-5-propylfuran. Their levels were 29.69, 16.86, and 3.12 $ng\,g^{-1}$, respectively. Furfural is widely found in roasted food products. It is the most common compound of the Maillard reaction. However, its content was below the olfactory threshold for its almond, bread, and sweet odors (Table 4).

The aldehydes formed during roasting can positively or negatively contribute to the aroma of roasted food. In fact, aldehydes derived from fatty acid oxidation have often been

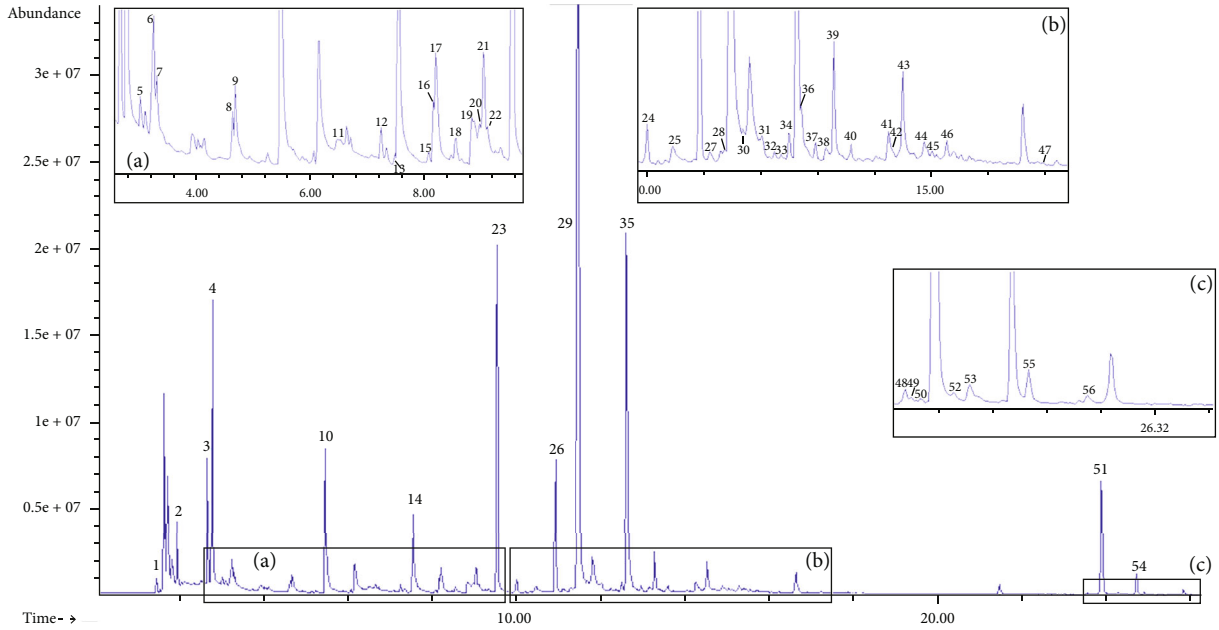

FIGURE 3: Typical total ion chromatogram (TIC) of headspace from ground roasted hemp seeds at 163°C for 15 min. Peak numbers correspond to those of Table 4.

correlated with the dominance of undesirable flavors [64], while aldehydes produced from the Strecker degradation are often related to the development of roasted aroma [65]. The results obtained in this work show that only aldehydes derived from the Strecker degradation were formed during roasting. These compounds are 2-methylpropanal, 2-methylbutanal, 3-methylbutanal, 2-phenylacetaldehyde, and benzaldehyde. They possess aroma characteristics such as sweet, green, almond, cocoa, bread, and malt. Their formation begins during roasting from amino acids, such as methionine, phenylalanine, leucine, and isoleucine, followed by the reaction with compounds, such as 2-oxopropanal or deoxyosones, formed by carbohydrate degradation [66]. 2-Methylpropanal, 2-methylbutanal, and 3-methylbutanal were detected in remarkable quantities (55.31, 250.77, and 108.17 ng g^{-1}, respectively) and above their olfactory thresholds (3.4, 10.8, and 23 ng g^{-1}, respectively), thus showing the development of cocoa, almond, and malt aromas in roasted hemp seeds. Benzaldehyde and 2-phenylacetaldehyde had low levels (17.66 and 10.71 ng g^{-1}, respectively) at their olfactory thresholds.

The alcohols formed after roasting hemp seeds are composed of 1-octen-3-ol (9.41 ng g^{-1}), propan-1-ol (25.70 ng g^{-1}), and cyclopentanol (20.89 ng g^{-1}). These compounds would be generated mainly from the oxidation of fatty acids, as is the case for 1-octen-3-ol produced from linoleic acid oxidation [27]. These compounds could contribute to the mushroom and balsamic aromas. However, their levels in hemp seeds were low compared to the olfactory thresholds reported in the literature (Table 4). These low levels follow our results which show that the polyunsaturated fatty acid content does not vary significantly after roasting and would, therefore, not be altered during this process (Table 3).

Heptane, *p*-xylene, and toluene were also identified in roasted hemp seeds. These hydrocarbons have often been correlated with undesirable aromas in roasted foods. Smith et al. [67] reported that toluene negatively affects roasted peanuts' flavor profile. Its roasted hemp seed content (8.44 ng g^{-1}) was below its olfactory threshold. Another compound, 2-heptanone, which can give a soapy odor, also appears after roasting, but fortunately with a content (42.49 ng g^{-1}) below its olfactory threshold. This compound would mainly develop from the oxidation of PUFAs [62].

4. Conclusion

We investigated in this work the temperature and time effects on the pressed hemp seed oil yield and antioxidant parameters using the RSM. Based on response surface plots and mathematical models, roasting temperature and time significantly affected response variables ($p < 0.05$). Optimal roasting conditions included a temperature of 163°C for 15 min, which improved the oil yield and oxidative stability. The model of optimized conditions showed a good correlation between the predicted and experimental values, indicating that the CCD was satisfactory when optimizing the roasting conditions. Moreover, roasting the hemp seeds under optimal conditions did not negatively influence the oil quality. In addition, the results of volatile compounds showed the formation of the typical aroma compounds from the Maillard reaction and the Strecker degradation sought for their positive attributes, such as pyrazines and aldehydes, responsible for the roasted, nutty, and almond odors. Thus, the optimum roasting conditions established in this work can be explored commercially in hemp seed oil extraction.

Data Availability

All data supporting the findings of this study are included within the article.

Conflicts of Interest

The authors declare no conflict of interest.

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Supplementary Materials

Supplementary 1. Table S1: ANOVA for oil yield, total phenolic compounds, free radical scavenging activity, and oxidative stability index obtained from the optimization study of the roasting variables (temperature and time) of hemp seeds in the central composite design. The bold values show significant effects ($p < 0.05$).

Supplementary 2. Figure S1: GC-FID chromatogram of fatty acids identified in oil extracted from roasted hemp seeds at 163°C for 15 min.

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