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Optimisation Using Response Surface Methodology of Quality, Nutritional and Antioxidant Attributes of 'Wichita' Pecan Nuts Roasted by Microwaves

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Abstract: Pecan (*Carya illinoinensis*) nuts are rich in functional compounds (unsaturated fatty acids, phytosterols, polyphenols, and tocopherols) associated with various health benefits. Commercially, pecan nuts are roasted to enhance their physical, chemical, and sensory properties. In the present study, response surface methodology (RSM) was used to optimise the quality and nutritional and antioxidant attributes of 'Wichita' pecan nuts roasted by using a microwave process with a range of microwave power (96.45–803.55 W) and roasting time (1.37–5.62 min). The microwave-roasted pecan nuts were analysed for hardness, total colour difference (TCD), and radical scavenging activity and modelled using the central composite design. The results showed that microwave power and roasting time significantly ($p < 0.05$) influenced the quality attributes of the pecan. The quadratic model adequately described the changes in TCD and hardness, respectively, while the 2FI model adequately described the changes in DPPH radical scavenging activity. To obtain the desired pecan nuts quality attributes (TCD = 1863.391; hardness = 28.755 N and DPPH radical scavenging activity = 33.877 mmol Trolox/g), the determined conditions were 700 W and 2.24 min, with a desirability of 0.557. The primary unsaturated fatty acids, including cis-oleic, cis-linoleic, α-linolenic, and stearic acids, were not affected ($p < 0.05$) by microwave roasting the pecan nuts at determined conditions. Volatile compounds, such as alcohols, aldehydes, ketones, lactones, hydrocarbons, and carboxylic acids, were identified in both raw and microwave-roasted pecan nuts, with limonene, which possesses various health properties, being the major volatile compound. It can be concluded that microwave roasting may be optimised using response surface methodology to produce quality pecan nuts that can be used as snacks or as an ingredient in other snack products.

Keywords: microwave roasting; pecan nuts; response surface methodology; radical scavenging activity; total colour difference; fatty acids

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1. Introduction

Pecan (*Carya illinoinensis*) nut, a member of the Juglandaceae family [1], is native to North America and primarily cultivated in Mexico, South Africa, Canada, Brazil, and Australia [2]. Mexico and the United States of America are the top two producers of pecan nuts, accounting for approximately 93% of the global production and an annual production of more than 40,000 metric tonnes [3].

Pecan nuts have been part of the human diet for centuries and are consumed either raw or roasted [4]. In addition, they are commonly used as an ingredient in snacks and confectionary products [3]. Pecan nuts contain functional compounds (unsaturated fatty acids, phytosterols, polyphenols, and tocopherols) [5–7] with various health benefits, including the reduction of low-density lipoprotein cholesterol [5] and the prevention of

Type 2 diabetes [8], coronary heart disease [6], and cancer [9]. More so, the consumption of pecan nuts has been linked with weight loss [10]. These health benefits have encouraged the development of value-added products, including roasted pecan nuts.

Roasting is a common thermal processing technique that provides important quality attributes to food products, such as colour, texture, flavour, and sensory acceptability [11,12]. The literature has shown that consumers prefer roasted pecan nuts to raw pecan nuts due to their crunchiness, crispness, better sensory attributes, and desirable nutritional properties [4,9,13]. Among the various roasting techniques, microwave roasting has gained more popularity in the food industry due to its advantages, such as control of the operational speed, uniform energy delivery, high thermal conductivity to the interior of the material, and energy savings [14,15]. Among other factors, temperature and time have been reported to affect the product's quality during the roasting process [15,16]. For this reason, the use of appropriate temperature−time combinations during roasting should be prioritised to minimise quality degradation [17].

If not well controlled, microwave roasting of pecan nuts may induce lipid modification, promoting rancidity [11]. This is because pecan nuts have a high ratio of unsaturated to saturated fatty acids (13.54) and unsaturated fatty acids (approximately 93%) [10], a characteristic that makes them susceptible to lipid oxidation during processing and storage [18,19]. Furthermore, this may negatively affect the sensory properties such as colour, texture, bioactive compounds, and antioxidant properties. Therefore, establishing the optimum microwave roasting conditions (temperature and time), which maximises the physiochemical properties with minimal alteration of the fatty acid profiles of the pecan nuts, is desired by the food industry. One way to optimise the quality of microwave-roasted pecan nuts is through the application of response surface methodology (RSM).

RSM is a set of mathematical and statistical tools mainly used to build models and to determine optimum conditions from multiple experimental runs from numerous variables using polynomial equations [20,21]. The main advantage of RSM is its capability to reduce the number of experimental runs required to evaluate multiple variables [22,23]. This optimisation technique has been successfully employed to carry out the roasting conditions of peanuts, cashews, and almonds to improve their quality and shelf life [15,17,24].

Despite the current advancement in using roasting to enhance the quality of nuts, limited studies are available in the literature on the determination of microwave roasting conditions to maximise the quality of nuts. Given the effect of temperature, microwave power, time, food particle size, and shape on the quality of microwave-roasted food, careful consideration of these parameters during the microwave roasting process is essential [25]. Therefore, the current study aimed to apply RSM with a central composite design (CCD) to carry out the microwave roasting power and time and establish the optimal colour, hardness, and radical scavenging activity of the pecan nuts. In addition, the study compared the fatty acid profiles and volatiles from raw pecan nuts and pecan nuts microwave-roasted at determined conditions. These parameters represent the quality indices that primarily affect consumers' preferences for roasted nuts and their nutritional and health benefits.

2. Materials and Methods

2.1. Materials

Mature, dry (10%, *w*/*w* moisture content), and defect-free pecans (cv. Wichita) (harvest season 2021) were purchased from the Northern Cape province, South Africa. The pecans were manually cracked to separate the kernel from the shell, after which the kernels or nuts were stored at −20 °C until further analyses. The Folin−Ciocalteau reagent and 2,2-diphenyl-1-picrylhydrazyl (DPPH), which were used in the current study, were purchased from Sigma−Aldrich in Germany. The other reagents used in this study, including methanol, sodium carbonate, gallic acid, hexane, heptadecanoic acid, sulfuric acid, and sodium chloride, were of analytical grade and purchased from Sigma−Aldrich in South Africa.

2.2. Experimental Design

The central composite design (CCD) was used to evaluate the effect of microwave power (96.45–803.55 W) and time (1.38–5.62 min) (independent variables) on the pecan samples' hardness (N), total colour differences, and DPPH radical scavenging activity (mmol Trolox/g) (dependent variables). The levels of independent variables were determined according to a preliminary study (unpublished). To facilitate multiple regression analysis, the independent variables were coded at five levels (Table 1).

Table 1. Independent variables and the five levels of each factor used in the central composite design (CCD).

Independent Variable	Levels				
	$-\alpha$ ($-\sqrt{2}$)	Low (-1)	Centre (0)	High (+1)	$+\alpha$ ($+\sqrt{2}$)
Power (W)	96.45	200	450	700	803.55
Time (min)	1.38	2	3.5	5	5.62

In total, 13 experimental tests were generated, including 4 factorial points, 4 axial points, and 5 repetitions (used to calculate the pure error and lack of fit) at the central point (Table 2). Randomisation was performed to minimise the influence of uncontrolled factors. The data obtained were fitted to a second-order equation as a function of the dependent variables (Equation (1)).

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} (X_j)^2 + \sum_{i \neq j} \beta_{ij} X_i X_j + E \quad (1)$$

where Y is the experimental dependent variable; β_0 is the constant coefficient; and β_i, β_{ii}, and β_{ij} are linear, quadratic, and interaction coefficients, respectively. The terms X_i and X_j represent the coded values of independent variables, while *E* represents the error between experimental results and calculated ones. The experimental data were analysed by using multiple regression to fit the second-order polynomial equation (Equation (1)), and the analysis of variance (ANOVA) was performed to evaluate the significance of independent variables ($p < 0.05$). To visualise the relationship between independent and dependent variables, surface responses and fitted polynomial regression equations were plotted using the Design Expert software (Design Expert 13, Minneapolis, MN 55413 USA). In addition, the *p*-value of the regression model, the *p*-value of the lack of fit, the coefficient of determination (R^2), adjusted R^2, and predicted R^2 were used to determine the fitness of the regression models. The optimal conditions were determined by creating three-dimensional response surface plots (3D surface plots) with the fitted model equation, followed by numerical optimisation. The optimisation procedure followed in the current study was (i) targeting 700 W for roasted pecan nuts; (ii) minimising the time, total colour difference, and hardness of the roasted pecan nuts; and (iii) maximising the DPPH radical scavenging activity (Table S1 Supplementary Material). The significance of the model's terms was considered statistically different when the *p*-value was <0.05. The fitted values predicted by the models were compared with experimental data to verify the adequacy of the regression models. In addition, experiments were performed under optimised conditions, and the means of the experimental values were compared with those of the predicted values using an independent t-test. In addition, the percentage relative deviation (%) was calculated.

Table 2. Central composite design and experimental data obtained for microwave-roasted pecan nuts at various powers (96.45–803.55 W) and times (1.38–5.62 min).

Point Type	A: Power (W)	B: Time (min)	TCD	Hardness (N)	DPPH Radical Scavenging Activity (mmol Trolox/g)
Factorial	200	2	2051.78	32.21	34.99
Factorial	700	2	1949.37	27.64	32.48
Factorial	200	5	2017.82	28.38	25.70
Factorial	700	5	1527.44	24.24	37.76
Axial	96.45	3.5	1860.10	31.71	30.30
Axial	803.55	3.5	1430.97	25.38	40.01
Axial	450	1.38	2145.70	33.96	34.46
Axial	450	5.62	1943.60	25.66	31.29
Center	450	3.5	2085.18	29.95	31.82
Center	450	3.5	1976.93	32.63	35.12
Center	450	3.5	1897.27	31.68	32.09
Center	450	3.5	1934.46	33.24	32.92
Center	450	3.5	2003.16	32.93	31.91

TCD—total colour difference, DPPH—(2,2-diphenyl-1-picryl hydrazyl).

2.3. Microwave Roasting of Pecan Nuts

Whole pecan nuts (35 g) were microwave-roasted using a domestic microwave (Model: DMO 351, Defy Appliances, Cape Town, South Africa) at different combinations of power (96.45–803.55 W) and time (1.38–5.62 min). The temperature (25.7–168.8 °C) of the microwave-roasted pecan nuts was immediately measured using an infrared thermometer (GM400, Zhangzhou, Fujian, China). After cooling to room temperature (25 ± 2 °C), the roasted samples were packaged in polyethylene plastic bags and stored at −20 °C until further analysis.

2.4. Measurement of Response Variables

2.4.1. Hardness

The hardness of microwave-roasted pecan nuts was measured at room temperature (25 ± 2 °C) using the texture analyser (Agrosta CE Calib 2018, Forges les Eaux, France). The pecan samples were placed singly on the plate, and then double compression was applied using a 5 mm diameter cylinder probe at a speed of 0.1 mm/s. Samples were compressed at a constant deformation speed of 5 mm/min. Ten measurements were taken for each of the samples. Analyses were carried out in triplicates.

2.4.2. Total Colour Differences

The colour attributes of the microwave-roasted pecan nuts, including lightness (L*), redness (a*), and yellowness (b*), were measured using a calibrated chromometer (CR−10 plus, Konica Minolta, Osaka, Japan). The total colour difference (TCD) was calculated using Equation (2). Raw pecan nuts were used as the control.

$$\mathrm{TCD} = \left(\Delta \mathrm{L}^{*2} + \Delta \mathrm{a}^{*2} + \Delta \mathrm{b}^{*2}\right)^{1/2} \quad (2)$$

2.4.3. DPPH Radical Scavenging Activity

Extracts from the microwave-roasted pecan nuts were prepared according to the method described by Zujko and Witkowska [26], with slight modifications. The samples (35 g) were ground to a fine powder using a coffee grinder, and 0.25 g of the pulverised samples was mixed with hot distilled water (5 mL) and 10 mL of 50% v/v methanol. The samples were vortexed (2 min), sonicated (Separation Scientific, Cape Town, South Africa) at −30 °C for 10 min, and then centrifuged (Thermo Fisher Scientific, Biofuge, Stratos, Horsham, Sussex, UK) at 4000× g and 25 °C for 10 min. The supernatants were used for DPPH radical scavenging activity analysis [27]. In triplicate, pecan extracts (15 µL) were mixed with 100% methanol (735 µL) and DPPH solution (750 µL) before incubation in

darkness for 30 min at room temperature. The samples' absorbances were then measured at 517 nm using a UV visible spectrophotometer (SP−UV 300, Shanghai, China). Trolox was used to develop the standard curve (0–10 mM; R^2 = 0.996), and the final results were reported as millimole Trolox equivalents per 100 g of pecan nuts (mmol Trolox/100 g pecan nuts).

2.5. Scanning Electronic Microscope (SEM)

The microstructures of raw and microwave-roasted nuts (optimum conditions) were studied using a scanning electronic microscope (SEM) (Tescan Vega 3, Borno, Czech Republic). Briefly, individual pecan nuts were cut with a clean razor blade to create either a thick cross-section or trimmed to provide a small section of the interior. The samples were placed on adhesive tape before being coated with a fine layer of gold through the sputter-coating attachment of blazers. The coated samples were then examined at 100× 500×, and 1000× magnifications [28]. The obtained images were processed using ImageJ software (National Institutes of Health, Bethesda, MD, USA) by drawing a line over the scale bar of the image acquired by the SEM and correlating the image dimensions in pixels to physical dimensions.

2.6. Volatile Compounds Analysis

Volatile compounds of raw pecan nuts and microwave-roasted pecan nuts (optimised conditions) were analysed using HS−SPME−GC−MS following a method described by Kaseke et al. [29]. Ground pecan nuts (5 g) were transferred to 20 mL pyrex bottles and capped with open-top caps coupled to polytetrafluoroethylene-faced silicone septa. Before the volatiles were measured, the samples were held in a temperature-controlled water bath at 50 °C for 60 min. A gas chromatograph (6890 N, Agilent Technologies Network) was coupled to an Agilent Technologies inert XL EI/CI Mass Selective Detector (MSD) (5975 B, Agilent Technologies Inc., Palo Alto, CA). The GC−MS system was coupled to a CTC Analytics PAL autosampler and used for separation on a ZBWaxPlus (30 m, 0.25 mm ID, 0.25 µm film thickness) capillary column. The carrier gas, helium, was used at a flow rate of 1 mL/min. The injection mode was splitless, and the injector temperature was maintained at 250 °C. The oven temperature was programmed as follows: 35 °C for 5 min, followed by a ramping rate of 5 °C/min until 50 °C and 5 °C/min until 120 °C and held at both temperatures for 3 min. The temperature was finally ramped up to 240 °C at a rate of 10 °C/min for 3 min. The mass spectrometer was operated in electron impact (EI) mode at an ionisation energy of 70 eV, scanning from 25 to 650 m/z. Volatile compounds were identified using mass spectral data from NIST and the Wiley Library and retention indices. The relative content (%) of each volatile compound was calculated by dividing the peak area of each component by the total peak area of all of the compounds identified.

2.7. Analysis of Fatty Acid Composition

Oil extraction from the raw pecan nuts and microwave-roasted pecan nuts (optimised conditions) was performed according to the method described by Kaseke et al. [25]. The pecan's oil fatty acid composition was determined using the gas chromatography–mass spectrometry (GC–MS) method [25]. Briefly, pecan oil (0.1 g) was weighed into 15 mL glass vials, followed by the addition of 2.0 mL hexane, 50 µL heptadecanoic acid (1000 ppm, internal standard), and 1.0 mL of 20% (v/v) H_2SO_4 in methanol. The mixture was then vortexed and incubated in an oven at 80 °C for 1 h. The samples were cooled before saturated NaCl (3 mL) was added, and the mixtures were vortexed and centrifuged (4000 rpm for 3 min) (Thermo Fisher Scientific, Biofuge, Stratos, Horsham, Sussex, UK). The supernatant (with hexane phase) was transferred into vials for GC−MS analysis using a 6890 N, Agilent technologies network coupled to an Agilent technologies inert XL EI/CI Mass Selective Detector (MSD) (5975 B, Agilent Technologies Inc., Palo Alto, CA, USA). The carrier gas (helium) was operated at a flow rate of 0.017 mL/s. The pecan oil extracts (1 L) were injected in a 10:1 split ratio, and the oven temperatures were set to 100 °C/min, 180 °C at

25 °C/min (held for 3 min), 200 °C at 4 °C/min (held for 5 min), 280 °C at 8 °C/min, and 310 °C at 10 °C/min (held for 5 min). The fatty acid profiles were identified using the NIST library. The results were expressed as mg/g and mean area (%) relative abundance.

2.8. Statistical Analysis

The Design Expert Software version 13 (Stat−Ease, Inc., Minneapolis, USA) was used for the experimental design, the regression analysis of the experimental data, and plotting the response surface plots. The analyses of variance (ANOVA) tables were carried out, and the effects and the regression coefficients of individual linear, quadratic, and interaction terms were determined. The STATISTICA software (STATISTICA v13, TIBC, Palo Alto, CA 94304, USA) was used to perform a paired *t*-test, evaluate the model significance, and determine the variation in fatty acids and volatile compounds between raw and microwave-roasted pecan nuts under optimised conditions ($p < 0.05$).

3. Results and Discussion

3.1. Total Colour Difference (TCD)

The colour of roasted nuts can be a key indicator of their flavour, quality, and effectiveness of the roasting process. The colour difference (TCD), based on L*, a* and b* colour values, represented the pecan nuts' overall colour change due to microwave roasting compared to raw pecan nuts. The very highly significant (F-value = 24.60; $p < 0.001$) predicted model obtained for TCD from regression analysis is given in Equation (3). The calculated "Lack of Fit" *p*-value of 0.73 indicated that the ''Lack of Fit" was insignificant relative to the pure error. In addition, the predicted R^2 value of 0.8392 agreed with the adjusted R^2 value of 0.9077 (difference < 0.2). "Adeq Precision" measures the signal-to-noise ratio, and the obtained ''Adeq Precision" ratio of 17.397 suggested an adequate signal to navigate the design space. A CV of 3.29 indicated that the model has good reproducibility.

$$TCD = 1979.4 - 149.96\ A - 92.71\ B - 96.99\ AB - 156.55\ A^2 + 43.00\ B^2 \quad (3)$$

Equation (3) depicts a significant negative linear effect of power (A) and time (B), a significant negative interactive effect of power and time (AB), a significant quadratic effect of power (A^2), and an insignificant positive quadratic effect of time (B^2).

The 3D surface image further illustrated the negative effect of both microwave power and time on TCD (Figure 1a). The lowest TCD was observed when pecan nuts were microwaved above 600 W for a time between 3 and 4 min (Figure 1b). The observation suggests minimal variation in the colour of roasted pecan nuts compared to raw pecan nuts under these conditions. In the literature, different results have been reported. Bagheri et al. [30] reported that peanuts' TCD increased (7.45–23.37) with an increase in infrared power (250–450 W) and time (20–30 min). Microwave roasting of cashew nuts showed that increasing microwave power (240–480 W) and time (30–240 s) had a positive effect on TCD (6.11–12.96) [31]. Increased TCD in the roasted nuts could be attributed to the development of brown pigments during roasting through caramelisation and Maillard reactions [17,31–35]. However, the TCD results in the present study could have been affected by the natural brown colour of pecan nuts, which could have masked the brown pigments formed during microwave roasting.

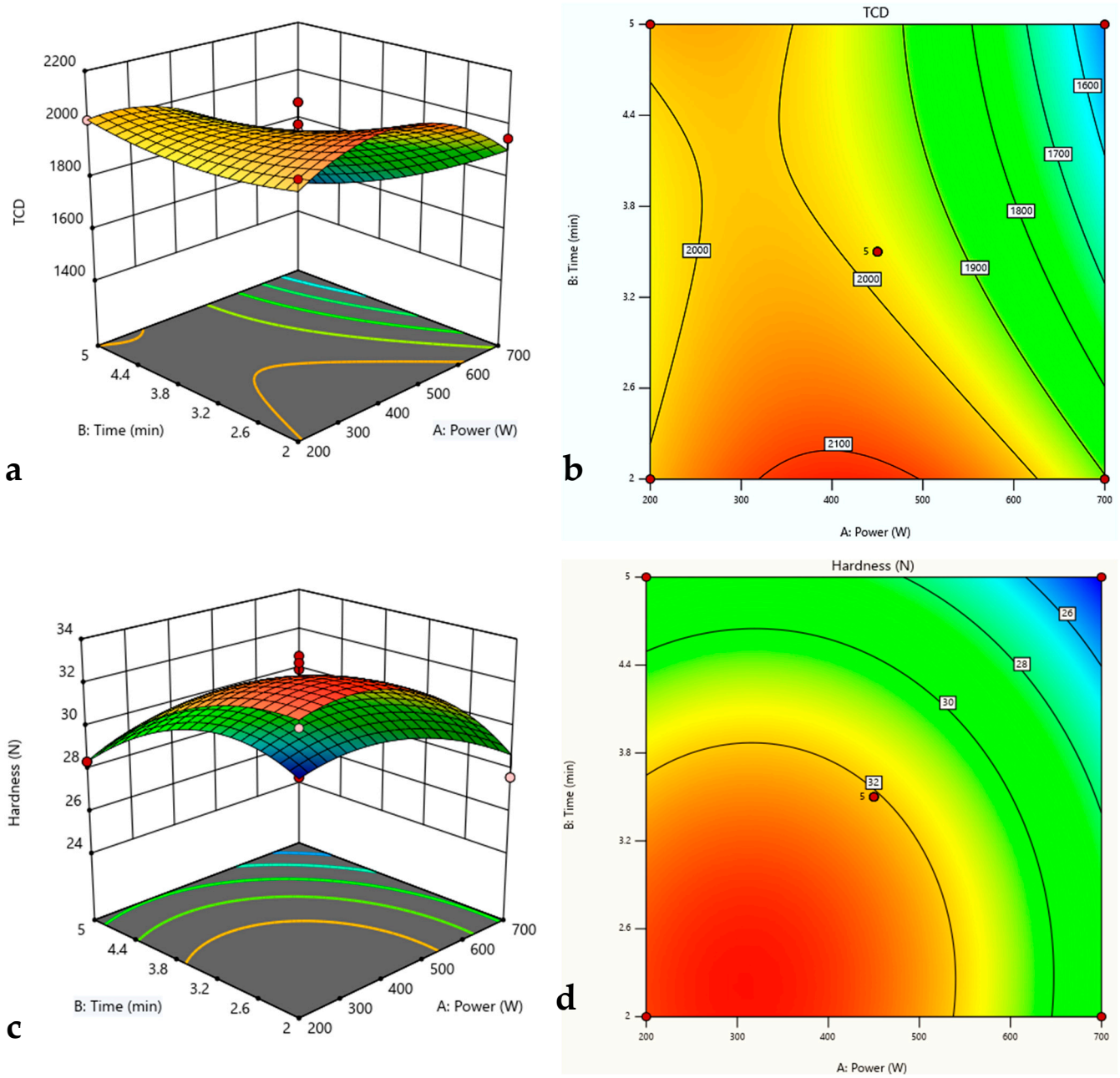


Figure 1. *Cont.*

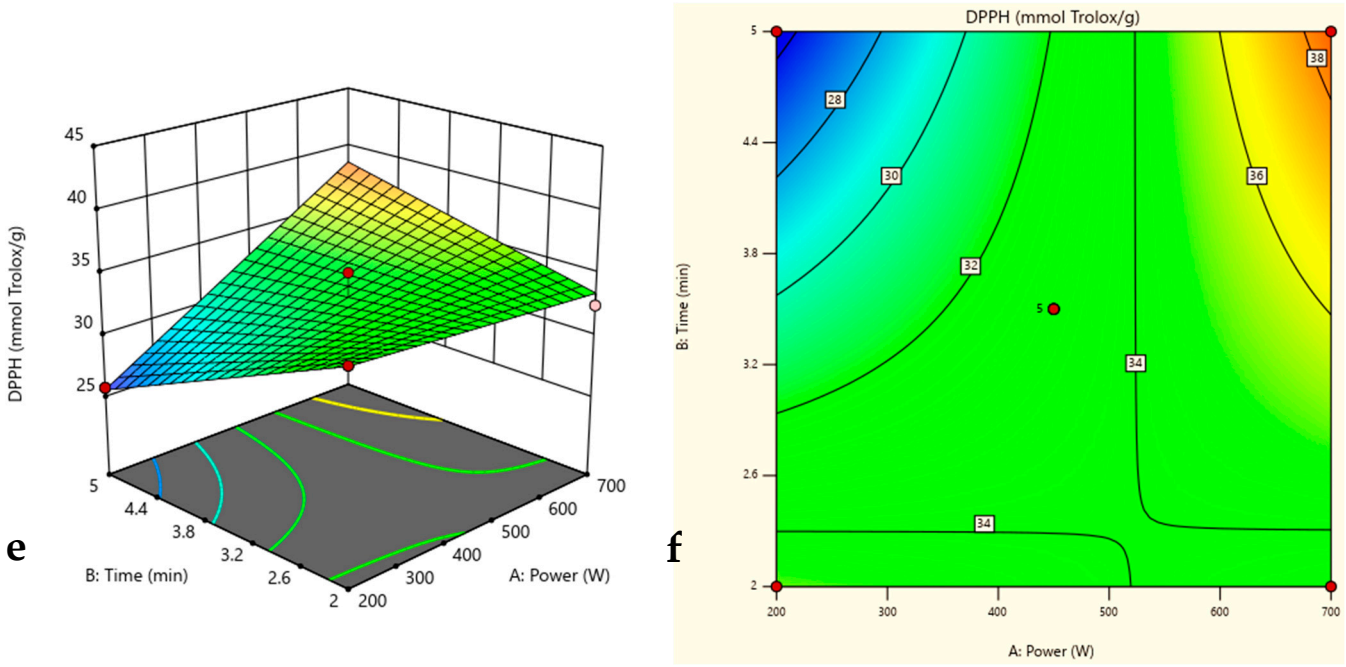


Figure 1. Interactive effect of microwave power (W) and time (min) on total colour differences (TCD) (**a**,**b**), hardness (**c**,**d**), and DPPH radical scavenging activity (**e**,**f**).

3.2. Hardness

Tables 2 and 3 present the samples' hardness experimental data and regression coefficients, respectively. The negative coefficient values (−2.21 and −2.37) for hardness indicate that increasing microwave power (A) and time (B) reduced the pecan nuts' hardness. Meanwhile, positive interactive effects of power and time (AB) and negative quadratic effects of power (A^2) and time (B^2) were also observed. Therefore, the predictive model in terms of coded factors for hardness is shown in Equation (4).

$$\text{Hardness (N)} = 32.09 - 2.21\ A - 2.37\ B + 0.1075\ AB - 2.04\ A^2 - 1.40\ B^2 \quad (4)$$

Table 3. Regression coefficients, mean, R^2, F and *p*-values for dependent variables of microwave-roasted pecan nuts at various powers (96.45–803.55 W) and times (1.38–5.62 min).

	Responses		
Regression Coefficients	**TCD**	**Hardness (N)**	**DPPH Radical Scavenging Activity (mmol Trolox/g)**
A—Power	−149.96 *	−2.21 *	2.91 *
B—Time	−92.71 *	−2.37 *	−1.06 *
AB	−96.99 *	0.11	3.64 *
A^2	−156.55 *	−2.04 *	–
B^2	43.00	−1.40 *	–
Mean	1909.52	29.94	33.01
R^2	0.9462	0.9113	0.8556
Adjusted R^2	0.9077	0.8480	0.8075
Predicted R^2	0.8392	0.6622	0.6853
Model (F-value)	24.60	14.39	20.01
Model (*p*-value)	0.0003 *	0.0014 *	0.0003 *
Lack of Fit (*p*-value)	0.73 ^{ns}	0.51 ^{ns}	0.43 ^{ns}
CV	3.29	4.35	4.44

* Significant at $p < 0.05$. ns—Not significant at $p < 0.05$. TCD—total colour difference, R^2—Coefficient of determination, CV—Coefficient of variation, DPPH—(2,2-diphenyl-1-picryl hydrazyl).

The coefficient of variation (CV) was 4.35, implying good reproducibility of the model. The model F value of 14.39 ($p < 0.01$) indicated that there is only a 0.14% chance that this F-value occurred due to noise. The "Lack of Fit" p-value was 0.51, suggesting that the "Lack of Fit" was insignificant relative to the pure error. The predicted and adjusted R^2 values were 0.6622 and 0.8480, respectively, and showed good agreement between each other (difference < 0.2). The "Adeq Precision" ratio of 10.352 suggested an adequate signal for navigating the design space. The 3D surface image in Figure 1c illustrated that microwave power and time both decreased the pecan nuts' hardness. Microwave roasting of the pecan samples beyond 500 W and 3 min decreased their hardness (Figure 1d). This observation could be attributed to increased brittleness due to moisture loss during roasting [15]. Furthermore, the decrease in hardness could be associated with starch gelatinisation, kernel expansion, and fissure development during roasting [36]. This is a desirable result given that consumers and food manufacturers prefer soft to hard pecan nuts. A decrease in hardness with increases in roasting temperature and time was also reported on peanuts, pistachio nuts, cashews, and almonds [17,24,30,37].

3.3. DPPH Radical Scavenging Activity

Scavenging free radicals is vital to preventing the damage of biologically important molecules in the human body, which include deoxyribonucleic acid, proteins, carbohydrates, and lipids. Therefore, the maximum release of antioxidant compounds from plant matrices through processes such as microwave roasting is desired. The F value of 20.01 ($p < 0.01$) showed that the model terms A, B, and AB were significant (Table 3). The observation indicates that microwave power and time had a significant positive and negative effect, respectively, while their interaction had a significant positive influence on the pecan samples' DPPH radical scavenging activity (Equation (5)). The effect of microwave power and time on DPPH radical scavenging activity is illustrated in Equation (5).

$$\text{DPPH radical scavenging activity (mmol Trolox/g)} = 33.14 + 2.91\ A - 1.06\ B + 3.64\ AB \quad (5)$$

The calculated "Lack of Fit" p-value of 0.43 indicated that the "Lack of Fit" was insignificant compared to the pure error. In addition, the predicted and adjusted R^2 values were 0.6853 and 0.8075, respectively (difference < 0.2). The "Adeq Precision" ratio was 16.065, showing an adequate signal for the model to navigate the design space. A CV of 4.44 indicated that the model has good reproducibility (Table 3). As illustrated in Figure 1e,f, the interactive effect of power, time, and DPPH radical scavenging activity microwave showed that power had a more positive impact on DPPH radical scavenging activity than time. Enhanced DPPH radical scavenging activity could be attributed to the increased extraction of antioxidant compounds such as polyphenols and tocopherols due to the increased disintegration of the pecan cell walls and loosening of the cellulose and pectin networks [25]. On the other hand, increased microwave power could have increased the development of Maillard reaction products with antioxidant properties [3]. Enhanced antioxidant activity with increased roasting power was also reported on cashews, fox nuts, and almonds [35,38,39].

3.4. Optimisation and Validation

The determined microwave roasting conditions for improved TCD, hardness, and DPPH radical scavenging activity with the highest desirability of 0.557 was given as 700 W for 2.24 min (Table S2 Supplementary Material). The predicted values of response variables for the roasted pecan nuts, as calculated from the formulas, were TCD = 1863.391, hardness (N) =28.755 N, and DPPH radical scavenging activity (mmol Trolox/g) = 33.877 (Table 4). For validation, an independent t-test (by variables) was employed to compare the predicted mean for each response variable and experimental values and test the adequacy of the final mathematical models. No significant ($p > 0.05$) difference was observed between the experimental and predicted values, indicating the adequacy of the fitted mathematical

models. Furthermore, the percentage relative deviation for the parameters TCD, hardness, and DPPH radical scavenging activity were 1.50%, 0.83%, and 2.90%, respectively (Table 4).

Table 4. The predicted and experimental values of responses of microwave-roasted pecan nuts under optimised conditions (700 W for 2.46 min).

Response Variable	Predicted Value	Experimental Value (Mean ± SD)	*p*-Value	Relative Deviation (%)
TCD	1863.391	1835.496 ± 37.39	0.3762	1.50
Hardness (N)	28.755	28.515 ± 0.50	0.2247	0.83
DPPH radical scavenging activity (mmol Trolox/g)	33.877	34.859 ± 2.94	0.8678	2.90

DPPH—(2,2-diphenyl-1-picryl hydrazyl), TCD—total colour difference.

3.5. Scanning Electron Microscope

Scanning electron microscopy (SEM) was applied to compare the microstructures of raw pecan nuts and pecan nuts microwave-roasted under optimised conditions (700 W for 2.24 min). As shown in Figure 2d, microwave-heated pecan samples were characterised by conspicuous perforations on the cell walls. According to Tu et al. [13] the observed perforations could be due to electromagnetic waves that were converted into heat energy and caused a rapid increase in the nuts' temperature and created intracellular pressure that ruptured the pecan nuts' cell walls. Meanwhile, the parenchymal cells from raw pecan nuts had intact cell walls and a smaller number of pores or void spaces (Figure 2b,c). This could be attributed to the moisture content in raw nuts, which maintained and supported their structural integrity [24,40]. Figure 2e,f show disintegrated and damaged parenchymal cells due to microwave treatment. Greater porosity and void spaces were also observed in the cells of microwave-roasted pecan nuts compared to raw pecan nuts [41]. Similar results were reported from microwave-roasted almonds [40] and microwave-treated hazelnuts [41]. These microstructural changes could be responsible for the enhanced DPPH radical activity in the current study (Figure 1e,f) due to increased porosity and dissociation of antioxidant compounds bound in the seed matrices that led to their improved extraction with methanol (Figure 1e,f; [25]).

3.6. Fatty Acid Composition

Table 5 compares the fatty acid composition of raw and microwave-roasted pecan nuts (optimised roasting conditions: 700 W for 2.24 min). The dominant fatty acids identified in pecan nuts oil were cis-oleic, cis-linoleic, palmitic, and stearic acids, which accounted for 757.68–770.56 mg/g, 304.19–305.23 mg/g, 77.06–77.65 mg/g, and 18.40–20.19 mg/g, respectively. Other fatty acids identified but in minor quantities were lauric acid, myristic acid, pentadecyclic acid, heptadecanoic acid, arachidic acid, docosanoic acid, tricosanoic acid, tetracosanoic acid, palmitoleic acid, dihomo-γ-Linolenic acid, eicosatetraenoic acid, and eiocosapentaenoic acid, with concentrations varying from 0.09 to 1.31 mg/g. The fatty acid composition and profiles were comparable to the findings from previous studies [42].

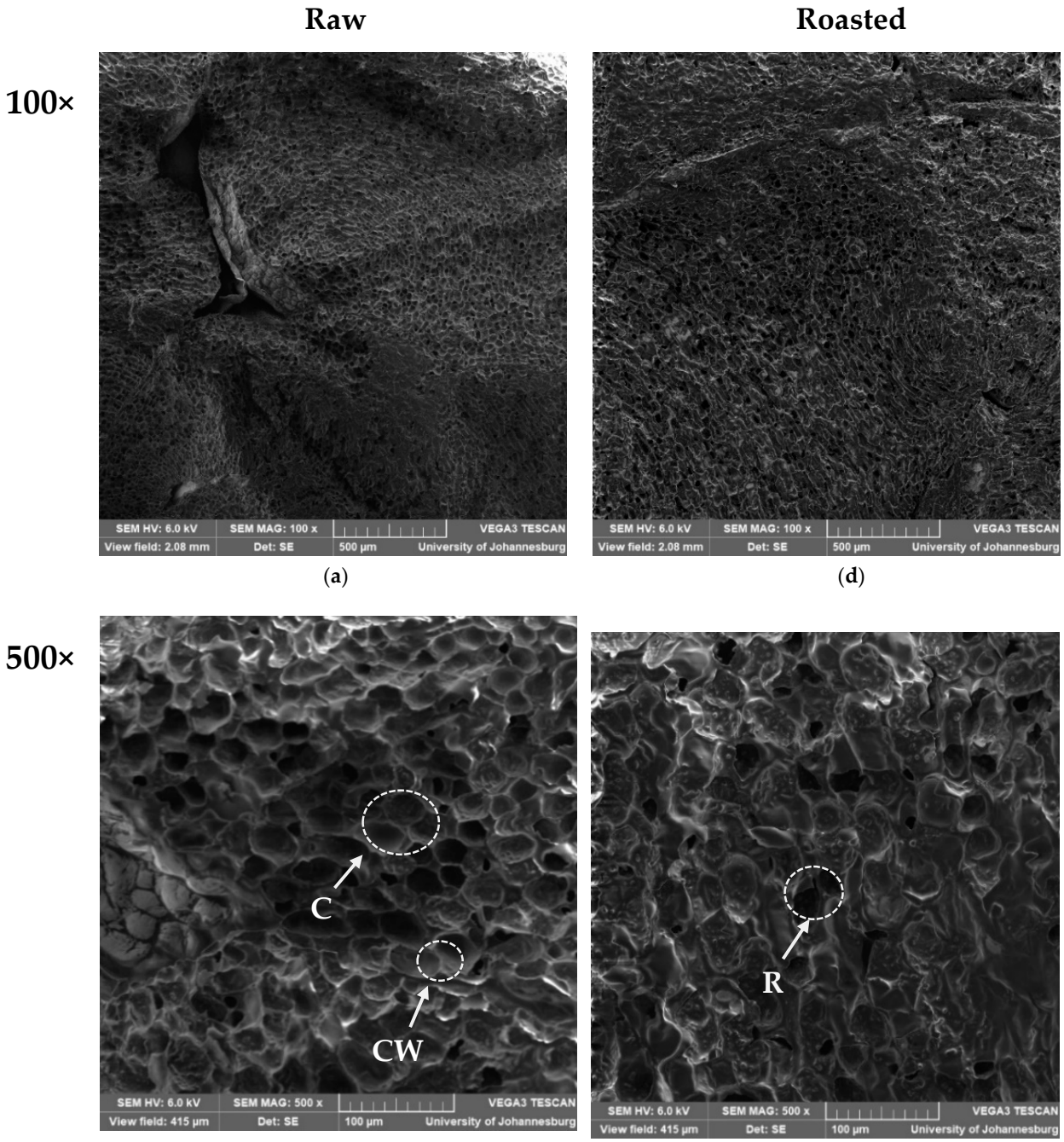
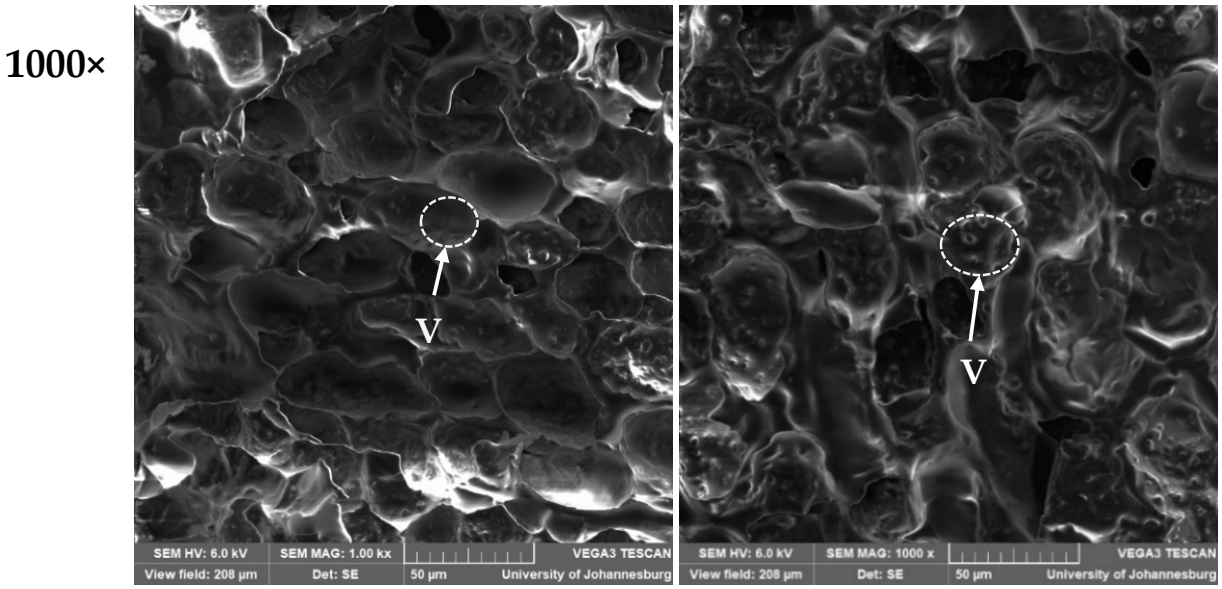


Figure 2. *Cont.*



(**c**) (**f**)

Figure 2. SEM images for raw and roasted pecan nuts at optimised microwave conditions (700 W and 2.24 min). (**a**–**c**) raw pecan nuts, (**d**–**f**) microwave-roasted pecan nuts, (**a**,**d**) at 100×, (**b**,**e**) at 500×, and (**c**,**f**) at 1000× magnification. C—parenchyma cell, CW—Cell wall, V—void spaces, and R—ruptured cells.

Table 5. Fatty acid composition and content (mg/g; %) in oil from raw and pecan nuts roasted under optimised conditions (700 W for 2.24 min).

Fatty Acids	Raw (mg/g; %)	Roasted (700 W/2.24 min) (mg/g; %)
SFA		
Lauric acid (C12:0)	0.21 ± 0.00 ^{a} (0.018%)	0.19 ± 0.01 ^{b} (0.016%)
Myristic acid (C14:0)	0.75 ± 0.01 ^{a} (0.064%)	0.71 ± 0.01 ^{b} (0.059%)
Pentadecyclic acid (C15:0)	0.22± 0.00 ^{a} (0.019%)	0.21 ± 0.00 ^{a} (0.018%)
Palmitic acid (C16:0)	77.06 ± 0.98 ^{a} (6.56%)	77.65 ± 1.32 ^{a} (6.55%)
Heptadecanoic acid (C17:0)	0.85 ± 0.03 ^{a} (0.072%)	0.90 ± 0.03 ^{a} (0.076%)
Stearic acid (C18:0)	20.19 ± 0.48 ^{a} (1.72%)	18.40 ± 0.55 ^{b} (1.55%)
Arachidic acid (C20:0)	1.31 ± 0.05 ^{a} (0.11%)	1.33 ± 0.03 ^{a} (0.11%)
Docosanoic acid (C22:0)	0.60 ±0.02 ^{a} (0.051%)	0.59 ±0.04 ^{a} (0.050%)
Tricosanoic acid (C23:0)	0.13 ± 0.01 ^{a} (0.011%)	0.14 ± 0.00 ^{a} (0.012%)
Tetracosanoic acid (C24:0)	0.17 ± 0.00 ^{a} (0.014%)	0.16 ± 0.00 ^{a} (0.014%)
ΣSFA	101.49 ± 1.58 ^{a} (8.64%)	100.28 ± 1.99 ^{a} (8.46%)
MUFA		
Palmitoleic acid (C16:1)	0.95 ± 0.02 ^{a} (0.081%)	0.89 ± 0.02 ^{a} (0.075%)
Oleic acid (C18:1 *cis*)	757.68 ± 18.50 ^{a} (64.53%)	770.56 ± 25.51 ^{a} (65.03%)
ΣMUFA	758.63 ± 18.52 ^{a} (64.61%)	771.45 ± 25.53 ^{a} (65.11%)
PUFA		
Linoleic acid (C18:2 *cis*)	305.23 ± 5.26 ^{a} (26.00%)	304.19 ± 7.52 ^{a} (25.67%)
α-Linolenic acid (C18:3 *n*-3)	8.20 ± 0.14 ^{a} (0.70%)	8.65 ± 0.26 ^{a} (0.73%)
Dihomo-γ-Linolenic acid (C20:3 *n*-6)	0.090 ± 0.00 ^{a} (0.0077%)	0.081 ± 0.00 ^{b} (0.0068%)
Eicosatetraenoic acid (C20:4 *n*-3)	0.13 ± 0.00 ^{a} (0.011%)	0.11 ± 0.01 ^{b} (0.0093%)
Eiocosapentaenoic acid (C20:5 *n*-3)	0.37 ± 0.10 ^{a} (0.032%)	0.11 ± 0.01 ^{b} (0.0093%)
ΣPUFA	314.02 ± 5.50 ^{a} (26.74%)	313.14 ± 7.80 ^{a} (26.42%)
ΣUFA	1072.65 ± 24.02 ^{a} (91.36%)	1084.59 ± 33.33 ^{a} (91.54%)
ΣMUFA/ΣPUFA index	2.42 ± 3.37 ^{a} (0.21%)	2.46 ± 3.27 ^{a} (0.21%)
ΣUFA/ΣSFA index	10.57 ± 15.20 ^{a} (0.90%)	10.82 ± 16.75 ^{a} (0.91%)
ΣPUFA/ΣSFA index	3.09 ± 3.48 ^{a} (0.26%)	3.12 ± 3.92 ^{a} (0.27%)

Values (means ±SE of triplicate determinations) in the same row and followed by different superscript letters are significantly different ($p < 0.05$) according to Duncan's multiple range test. SFA—saturated fatty acid, MUFA—monounsaturated fatty acid, PUFA—polyunsaturated fatty acid, UFA—unsaturated fatty acid, Σ—sums of SFA, MUFA, or PUFA.

As shown in Table 5, microwave roasting of pecan nuts under optimised conditions did not significantly ($p > 0.05$) affect the primary unsaturated fatty acids, including oleic acid, linoleic acid, and α-linolenic acid. These fatty acids are responsible for the pecan nuts' functional properties, including reducing low-density lipoprotein cholesterol and preventing type 2 diabetes and coronary heart disease [8,43]. Since the human body cannot synthesise linoleic acid or α-linolenic acid due to a lack of suitable enzymes, it is crucial to preserve these fatty acids during pecan nut roasting [12,44]. The present study, therefore, demonstrated that microwave roasting of pecan nuts under optimised conditions did not negatively affect their health properties. Similar results were reported in the literature. For example, Olatidoye et al. [12] and Li et al. [45] observed that roasting cashews and walnuts did not affect their fatty acid composition or content. Roasted and fried pistachios (175 °C for 2.5 min) showed no significant effect on the fatty acid composition [44]. However, in the present study, a significant decrease in stearic acid (9% decrease) was observed after microwave roasting. This was not desired since stearic acid has unique properties and has been associated with a decrease in LDL cholesterol, cancer, and atherosclerosis risk [25,46].

Minor fatty acids, including dihomo-γ-Linolenic acid, eicosatetraenoic acid, and eiocosapentaenoic acid, also significantly ($p < 0.05$) decreased after microwave roasting pecan nuts under optimised conditions. The different components of fatty acids, including saturated fatty acids (SFAs), monounsaturated fatty acids (MUFAs), and polyunsaturated fatty acids (PUFAs), as well as the MUFA/PUFA, UFA/SFA, and PUFA/SFA indices, were not significantly affected after microwave roasting. Therefore, microwave roasting of the pecan nuts under optimised conditions may not alter the balance between these different pecan nuts fatty acid components.

3.7. Volatile Compounds

The sensory qualities, in particular flavour and aroma, are closely related to the quality of the nuts. The flavour and aroma are closely linked to the volatile compounds either produced during fruit growth or nut processing [42]. The results of the volatile compounds identified in the pecan nut samples are shown in Table 6. In total, 23 well-known volatile compounds, including alcohols, aldehydes, ketones, lactones, hydrocarbons, and acids, were detected and identified in the pecan nuts. The groups of volatile compounds were comparable to those observed in roasted cashews and almonds [12,47].

Many of the aliphatic alcohols are produced by the decomposition of hydroperoxides of fatty acids or ketone and aldehyde reduction [48]. Alcohols, including 1-pentanol, 3-octanol, and 1-vinylhexanol, were only observed in raw pecan nuts, ranging from 0.47 to 0.99%. Meanwhile, isopentanol (0.77%) was only identified in pecan nuts roasted under optimised conditions. Aldehydes have been reported to contribute to the pungent, vegetal, and oily odours of nuts [42,45]. These volatile compounds may be produced through the lipoxygenase pathway during oil cell fragmentation or automatic oxidation during thermal processing [42]. Aldehydes (2-hexanal, furfuralaldehyde, nonanal, and 5-methylfurfural) were found in both pecan samples but at a significantly higher level in raw pecan nuts. Hexanal is a typical oxidation volatile and has been commonly used as a quality indicator for lipid oxidation in seed oils [29]. This volatile compound was significantly higher in raw pecan nuts, suggesting minimum oxidation of fatty acids during roasting.

Hydrazoic acid, the only acid identified, is only found in roasted pecan nuts. Lasekan et al. [47] reported carboxylic acids as the most abundant volatile compounds in roasted (200 °C, 50 min) almonds. However, factors such as the type of nut, roasting temperature, and time play a significant role in the amounts and types of volatile compounds. The hydrocarbons formed the majority of the volatile compounds and included *p*-xylene, m-xylene, myrcene, limonene, *p*-cymene, ethenyl benzene, phenylethane, bromobenzene, and chlorobenzene (Table 6). Limonene, a terpenoid characterised by a fresh citrus-like flavour, was the major volatile compound (84.50–85.42%) and was significantly higher in pecan nuts roasted under optimised conditions. Limonene was also observed as a major volatile compound in pistachios [49]. Limonene possesses a number of health-promoting

properties, including gastroprotective, anti-inflammatory, antioxidant, antinociceptive, anticancer, antidiabetic, antihyperalgesic, and antiviral activities [50]. Ketoisophorone (a ketone) is a key intermediate in the synthesis of carotenoids and flavouring agents formed during the fruit ripening stage [49]. This compound was only identified in raw pecan nuts (Table 6).

Table 6. Main volatile compounds identified in raw and microwave-roasted pecan nuts (optimised conditions: 700 W/2.24 min).

Volatiles Compounds (%)	Raw Pecan Nuts	Roasted Pecan Nuts (700 W/2.24 min)
Alcohol		
1-Pentanol	0.75 ± 0.01	ND
3-Octanol	0.47 ± 0.01	ND
1-vinylhexanol	0.99 ± 0.01	ND
Isopentanol	ND	0.77 ± 0.01
Aldehyde		
2 Hexanal	1.99 ± 0.02 ^{a}	1.01 ± 0.01 ^{b}
Furfuralaldehyde	2.31 ± 0.03 ^{a}	0.89 ± 0.01 ^{b}
Nonanal	0.66 ± 0.01 ^{a}	0.36 ± 0.00 ^{b}
Phenylacetaldehyde	0.40 ± 0.00	ND
5-Methylfurfural	0.54 ± 0.01 ^{a}	0.44 ± 0.01 ^{b}
Acids		
Hydrazoic acid	ND	2.73 ± 0.02
Hydrocarbons		
ρ-Xylene	0.41 ± 0.01 ^{b}	0.98 ± 0.01 ^{a}
m-Xylene	ND	1.02 ± 0.01
Myrcene	4.01 ± 0.04 ^{a}	3.30 ± 0.02 ^{b}
Limonene	84.50 ± 0.84 ^{b}	85.42 ± 0.85 ^{a}
ρ-cymene	0.53 ± 0.00	ND
Ethenylbenzene	1.16 ± 0.01	ND
Phenylethane	0.70 ± 0.01 ^{b}	0.90 ± 0.01 ^{a}
Bromobenzene	0.31 ± 0.01	ND
Chlorobenzene	ND	1.02 ± 0.01
Ketone		
4-Ketoisophorone	0.28 ± 0.00	ND
Lactones		
4,4-Dimethylbutyrolactone	ND	0.90 ± 0.01

Values (mean ± SE, n = 3) in the same row with different superscript letters are significant ($p < 0.05$). ND—none detected.

Esters are derived from the esterification of alcohols and free fatty acids, occur naturally in fruits, and enhance their flavours. The 4.4-dimethylbutyrolactone, the only cyclic ester (lactone) identified, was only observed in pecan nuts microwave-roasted under optimised conditions. However, in the present study, 4.4-dimethylbutyrolactone could have been formed through the thermal oxidation of higher fatty acids during microwave roasting [29].

4. Conclusions

The study successfully utilised response surface methodology (RSM) to enhance the sensory attributes, nutritional qualities, and antioxidant activity of microwave-roasted pecan nuts. The process variables, power and time, significantly influenced the total colour difference (TCD), hardness, and DPPH radical scavenging activity. Optimal roasting conditions were identified as 700 W power for 2.24 min, yielding desirable quality attributes. The primary unsaturated fatty acids remained unaffected, indicating preserved nutritional quality. Thus, microwave roasting may be optimised using RSM to produce pecan nuts with desired quality attributes and for various uses.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/pr11082503/s1, Table S1: Constraints and Table S2: Solutions.

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