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Investigaciones en pecán en Sudamérica

Optimization of Process Parameters for Pecan oil Extraction

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Abstract

In recent years, interest in obtaining oils through pressing technologies has intensified. The extraction process affects the nutritional quality, flavors, and oxidative stability of the oils. The influence of process variables –pressing temperature and moisture content of the crushed nut– on oil yield and quality was investigated. To maximize oil yield and the quality of the obtained product, the Response Surface Methodology was used. Analyses were performed of oil extraction yield (OY), peroxide value (PV), acidity, conjugated dienes and trienes, oxidative stability and induction time and color parameters (CieL*a*b* system). The pressing conditions that maximized oil yield and quality were 35 °C and 5.5% moisture. Under these conditions, OY was 23.6% and PV was 0.99 meq O_2/kg oil. According to the results, it was concluded that moisture and temperature of the crushed nut during the pressing stage promoted changes in OY and PV.

Keywords: oil yield, oil quality, peroxide index, response surface methodology

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Optimización de los parámetros de proceso para la extracción de aceite de nuez pecán

Resumen

En los últimos años, se ha intensificado el interés por la obtención de aceites a través de tecnologías de prensado. El proceso utilizado para la extracción afecta la calidad nutricional, los sabores y la estabilidad oxidativa de los aceites. Se investigó la influencia de variables de proceso —temperatura de prensado y humedad de la nuez triturada— sobre el rendimiento y la calidad del aceite. Para maximizar el rendimiento de extracción de aceite y la calidad del producto obtenido, se utilizó la Metodología de Superficie de Respuesta. Se realizaron análisis de rendimiento de extracción de aceite (Ra), índice de peróxidos (IP), acidez, dienos y trienos conjugados, estabilidad oxidativa y tiempo de inducción y parámetros de color (sistema CieL*a*b*). Las condiciones de la etapa de prensado que maximizaron el rendimiento y la calidad del aceite fueron 35 °C y 5,5 % de humedad. En estas condiciones el Ra fue de 23,6 % y el IP de 0,99 meq O_2/Kg de aceite. De acuerdo con los resultados, se concluyó que las variables humedad y temperatura de la nuez triturada durante la etapa de prensado promovieron cambios en el Ra y en el IP.

Palabras clave: rendimiento, calidad de aceite, índice de peróxidos, metodología superficie de respuesta

Otimização de parâmetros de processo para extração de óleo de noz-pecã

Resumo

Nos últimos anos, intensificou-se o interesse na obtenção de óleos através de tecnologias de prensagem. O processo utilizado para extração afeta a qualidade nutricional, os sabores e a estabilidade oxidativa dos óleos. Foi investigada a influência de variáveis do processo –temperatura de prensagem e umidade da noz-pecã triturada– no rendimento de produção e na qualidade do óleo. Para obter a melhor combinação de fatores experimentais que maximizassem o rendimento da produção de óleo e a qualidade do produto obtido, foi utilizada a Metodologia de Superfície de Resposta. Foram realizados análises de rendimento de extração de óleo (REO), índice de peróxidos (IP), acidez, dienos e trienos conjugados, estabilidade oxidativa e tempo de indução e parâmetros de cor (sistema CieL*a*b*). As condições da etapa de prensagem que maximizaram o rendimento e a qualidade do óleo foram 35 °C e 5,5 % de umidade. Nessas condições, o REO foi 23,6 % e o IP foi 0,99 meq O2/Kg. De acordo com os resultados conclui-se que as variáveis de umidade e temperatura da noz-pecã triturada durante a etapa de prensagem promoveram alterações no REO e no IP.

Palavras-chave: rendimento, qualidade do óleo, índice de peróxido, metodologia de superfície de resposta

1. Introduction

Recently, interest in obtaining oils through pressing technologies has intensified. Mechanical pressing is the oldest and simplest method of oil extraction (Pradhan et al., 2011). In the case of non-traditional vegetable oils, pressing, both with a hydraulic and screw press, is a simple method to obtain oil from small batches of seeds (Fantino et al., 2020; Martínez et al., 2017). Pecan nuts are particularly rich in oil; in commercial cultivars, the oil content typically ranges from 58% to 66%, although a wider range can be observed depending on genetic diversity, environmental conditions, and growth factors (Grauke, 2010; Reis Ribeiro et al., 2020). Oil content values as high as 76.5% have been found in trees from central Mexico (Toro-Vazquez et al., 1999). This edible oil has a well-balanced fatty acid composition, with high levels of monounsaturated fatty acids (49.76% to 68.12%), moderate amounts of polyunsaturated fatty acids (23.55% to 40.67%), and low levels of saturated fatty acids (8.3% to 9.7%) (Descalzo et al., 2022; Panozzo et al., 2019; Prabhakar et al., 2023). Furthermore, pecan oil is one of the most interesting sources of biologically active compounds due to its considerable content of tocopherols, phenolic compounds and phytosterols (Alvarez-Parrilla et al., 2018; Atanasov et al., 2018). Several studies have investigated the antioxidant properties of pecan oil and its health-related benefits (Alvarez-Parrilla et al., 2018; Gong et al., 2020).

It is worth noting that, given the growing global interest in foods that promote human health and well-being, the increased demand for novel and healthy oils has focused on oils produced from nuts (Du et al., 2023).

Currently, presses are only used to obtain fats and oils produced in small quantities. The efficiency of small-capacity presses can be increased by optimizing the main process variables, by adjusting the pressure applied to the mass –which depends on the pressing equipment available– and by conditioning the raw material to be processed.

Regarding this last aspect, particle size, pressing temperature and moisture content of the seeds must be considered (Bhuiya et al., 2020; Mendonça et al., 2020). In fact, the moisture content of the seed is a key factor among the process variables and must be studied for each type of nut or oilseed (Fantino et al., 2020). Moisture content is very important since it not only increases the plasticity of the material, but also contributes to pressing due to its lubricating action. However, high moisture contents can negatively affect extraction or alter the chemical quality of the oil (Rombaut et al., 2015).

The application of heat treatment before or during pressing generally improves oil extraction because of the breakdown of oil cells, coagulation of proteins and decreased oil viscosity, allowing the oil to flow more easily (Bhuiya et al., 2020; Rombaut et al., 2015). Softening of seed tissues tends to weaken and break down the cellular structure of oil under pressure, while low viscosity increases oil flow and therefore increases oil recovery (Indrasari et al., 2001).

On the other hand, the variables that affect oil extraction yield may also influence the quality of the oils obtained; therefore, their evaluation is essential. The main chemical quality indicators for edible vegetable oils include acid value, peroxide value, extinction coefficients at 232 and 270 nm (K_{232}, K_{270}), and oxidative stability (Martínez & Maestri, 2015). Free acidity is one of the key chemical characteristics that best defines the quality of an oil or fat, as it reflects the degradation of triglycerides due to chemical or enzymatic hydrolysis, leading to the formation of free fatty acids (García Martínez et al., 2014). One of the main causes of oil quality deterioration is lipid autoxidation reactions. These reactions can be quantified through the determination of the peroxide value (PV). Lipid peroxides or hydroperoxides are the primary initial products of lipid oxidation; thus, their quantification provides a measure of the degree of oxidation in oils (Martínez & Maestri, 2015). As a consequence of the migration of double bonds from their original positions, hydroperoxides derived from polyunsaturated fatty acids exhibit a conjugated diene structure. Oil oxidation can also be assessed optically via ultraviolet light absorption by degradation compounds at wavelengths of 232 and 270 nm (De Oliveira et al., 2017). Absorbance at 232 nm is a simple method to evaluate the early stages of lipid autoxidation. As a complementary measure, absorbance at 270 nm is also used to indicate the presence of conjugated trienes (K_{270}) (Martínez & Maestri, 2015; Shahidi & Zhong, 2005). Moreover, oxidative stability refers to the oil's ability to resist or delay oxidation and is a significant parameter for determining oil quality (Karrar et al., 2020). Oxidative stability is commonly assessed through accelerated tests that rapidly induce oxidation, such as the Rancimat method (Descalzo et al., 2021; García-Moreno et al., 2013; Rodríguez et al., 2015). Additionally, color is one of the quality attributes that influences the acceptance of vegetable oils and food products in general (Garitta et al., 2013; Karrar et al., 2020). According to Czwartkowski et al. (2022), a high-quality niche oil, such as pecan nut oil, is characterized by a very light, uniform, and clear color.

Considering that several factors can influence extraction efficiency and oil quality during pressing extraction, the Response Surface Methodology (RSM) offers an effective approach for determining the optimal combination of experimental variables to maximize oil yield (OY) and quality. RSM is a robust statistical design method that identifies optimal conditions with a minimal number of experiments, avoiding the inefficiency of testing one variable at a time (Sevda & Singh, 2020). This reduces the cost of optimizing a specific process (Nwabueze, 2010).

The objective of this study was to investigate the effect of process variables on the yield and quality of pecan oil. The experiments aimed to maximize both oil yield and quality by focusing on two variables during the pressing stage: the moisture content of the crushed nuts and the pressing temperature, using an experimental design approach.

2. Materials and Methods

2.1 Samples

Cheyenne pecans were used to carry out tests corresponding to the experimental design, while Success was used to verify the best experimental conditions. The nuts were purchased shelled and conditioned to a commercial moisture content (4.0-5.5%) on a farm in the town of San José (Entre Ríos, Argentina).

2.2 Procedures

The nuts were ground in an industrial stainless steel meat grinder (Pagani). Next, the particle sizes were separated using a sieve (Zonytest) with a 1 mm mesh opening, as established in previous tests. Larger particles were ground again to achieve a smaller size.

To reach the moisture content defined by the experimental design, in some cases, the sample from the initial batch needed to be humidified, while in others, it needed to be dried. Humidification was carried out according to the methodology proposed by Singh and Bargale (2000), in which water was added to the samples by spraying. The crushed and moist nuts were placed in heat-sealed high-density polyethylene bags and stored for 48 h until equilibrium was reached. The bags were shaken at regular time intervals to ensure uniform moisture distribution in the material. Drying was carried out at 32 ± 1 °C in an oven with forced air circulation (Instrumentalia DHG-9240). Afterward, the crushed nuts were wrapped in a filter cloth in batches of 2.0 kg and brought to the temperature specified in the experimental design. Pressing temperatures were achieved by heating in an oven (Instrumentalia DHG-9240). The temperature of the samples was controlled with a digital thermometer (Luft).

The pressing stage was then carried out in an open hydraulic press (Morano) to which a plate system provided by the company IDEM Alimentos S.H. was adapted. The following scheme was followed:

1- Compression for 5 min at 100 kgf/m^2

2- Compression for 10 min at 200 kgf/m^2

3- Compression for 5 min at 300 kgf/m^2

After each compression stage, the sample was decompressed before moving on to the next stage.

At the end of the pressing, the residual cake was crushed to achieve homogenization of the oil distribution for the subsequent quantification of the residual oil content in the cake.

To eliminate the turbidity oils obtained, they were packaged in amber-colored bottles and left to decant for 20 days in a refrigerator (Gafa) at 7 ± 1 °C. The oils cleared were then racked, and subsequently frozen at -30 °C in a freezer (Gafa) until the physicochemical analyses were performed.

2.3 Experimental Design of the Pressing Stage of the Oil Extraction Process

RSM was used as an experimental and analytical strategy to determine the optimal values of the variables in the pressing stage based on the response, or dependent, variables (Gutiérrez-Pulido & de la Vara Salazar, 2008). A rotatable central composite design was developed using STATGRAPHICS Centurion XV software (Version 15.2.06), with two experimental factors: moisture content and pressing temperature of the crushed nuts. Each factor was tested at three levels, and the central point was repeated in triplicate. The experimental design is shown in Table 1.

Table 1. Experimental design of the pressing stage of the oil extraction process

Symbols	Independent variables	Coded levels				
		-1.4142	**-1**	**0**	**1**	**1.4142**
x_1	Temperature of the pressing process (°C)	18.8	25	40	55	61.2
x_2	Moisture content of crushed pecans (%)	4	5	7.5	10	11

2.4 Effect of Experimental Factors on Physicochemical Parameters

Once the experimental assays were conducted according to the design outlined above, physicochemical analyses were performed on each of the oils obtained. Next, the analysis of the experimental design was carried out. The influence of the experimental factors –temperature and moisture content of the crushed nuts during pressing– on the physicochemical parameters was determined using ANOVA ($p < 0.05$). When temperature, moisture content or their interactions significantly affected a parameter, that parameter was considered a response variable in optimizing the experimental design.

The optimal combination of levels of the experimental factors that resulted in the highest or lowest level (as appropriate) of the response variables was obtained following the RSM. The experimental results were fitted to a polynomial equation describing the relationships between each response variable and the factors studied through multiple regression analysis. In order to have a clearer idea of the experimental data, response surface and contour graphs were obtained.

For the optimization of multiple responses, the desirability function (Montgomery, 2013) was used. For this, each response yi was converted into an individual desirability function di, which takes values between 0 and 1. The closer the response was to the ideal value, the closer the desirability function was to 1.

The goodness of fit the model was evaluated using the coefficient of determination (R^2), the mean absolute error (MAE) and the Durbin-Watson (DW) test.

Once the combination of experimental factors indicated by the RSM was obtained, pecan oil was obtained under these conditions to verify that the predicted results for each response variable corresponded to the experimental results. For this purpose, pecan nuts of the Success cultivar were used, as it was the only cultivar available at the time of the experiments. It was not possible to work with the Cheyenne cultivar due to its low yield, which was caused by scab-related issues.

Model validation was performed by comparing the experimental data with the predicted values within a 95% confidence interval. The absence of significant differences was assumed when the experimental values fell within this interval.

2.5 Oil Yield

The oil yield (OY) was calculated considering the initial oil content of the pecans and the residual oil content in the pressed cake. It was expressed as g extracted oil/g oil present in the nuts × 100 (g/100 g oil). Oil contents

from the nuts and pressed cakes were determined by extraction (6 h) using Soxhlet devices (Torbell), with n-hexane as solvent, and expressed as weight percent on dry base.

2.6 Oil Analysis

Chemical quality parameters peroxide value (PV), specific extinction coefficients (K_{232} and K_{270}) and free fatty acid content (FFAC) were quantified according to standard methods of AOCS (American Oil Chemists' Society, 2009).

2.7 Oxidative Stability and Induction Time

The oxidative stability (OS) was determined by the Rancimat (Metrohm 893) method using 3 g oil aliquots. The air flow rate was set at 20 L/h while the temperature of the heating block was maintained at 110 °C. Results corresponded to the break points in the plotted curves and were expressed as induction time (in hours) (IT) and oxidative stability (OS). Oxidative stability is the time (in hours) that passes from the beginning of the test until the maximum value of electrical conductivity is obtained.

2.8 Color

Color was evaluated by measuring the parameters of the CIE color system: L*, a*, and b*. The determinations of lightness (L*), a*, and b* values were carried out using a Konica Minolta colorimeter (CR-400, Japan) equipped with a CR-A33d measuring head. Oil samples were placed in an optical glass cuvette with two translucent faces and a light path of 50.8 mm. The cuvette had two front translucent faces and two lateral opaque faces, with external dimensions of 40 × 20 × 54.8 mm, an internal slit width of 16 mm, and a volume of 28.4 mL. The measuring head of the colorimeter was positioned against the 20 mm face of the cuvette so that the aperture was fully covered. Measurements were performed in triplicate for each oil sample, with the back end of the cuvette covered using a white ceramic tile.

2.9 Statistical Analysis

The comparison of means for physicochemical parameters and acceptability was performed using ANOVA and the Tukey Multiple Range Test with the STATGRAPHICS Centurion XV software Version 15.2.06. Significant differences between means were considered when $p < 0.05$.

3. Results

3.1 Oil Yield

The initial oil content of the pecans was 70.52 ± 0.39%; while the moisture content was 4.92 ± 0.07%.

The OY in each assay of the experimental design is shown in Table 2, whose range of variation was between 12.73 ± 2.40% and 30.18 ± 1.11%. For the same moisture content of the crushed nut during pressing, OY tended to increase with rising temperature. The highest yield was achieved at 61.2 °C and 7.5% moisture.

3.2 Physicochemical Parameters

The peroxide value (PV) ranged from 1.00 ± 0.14 to 2.45 ± 0.61 meq O_2/kg oil (Table 2). In general terms, it can be noted that, for the same temperature value, the PV increased the higher the moisture content of the crushed nut; while this variable also increased with the rise in temperature for a given moisture content.

The K_{232} coefficient varied between 0.97 ± 0.00% and 1.31 ± 0.04% and the K_{270} coefficient ranged from 0.04 ± 0.00% to 0.08 ± 0.00% (Table 2). For both coefficients, lower moisture content of the crushed nuts resulted in lower values at a given temperature. However, no clear pattern was observed when the temperature changed while the moisture content remained constant.

Table 2. Effect of process variables on pecan oil yield (OY) and quality parameters

Factors			**OY** (g/100g g oil)	**PV** (meq/kg oil)	K_{232} (%)	K_{270} (%)	**OS** (h)	**IT** (h)	**FFAC** (g oleic acid / g oil)
Assay	**T (°C)**	**MC (%)**							
1	55	5	24.55 ± 1.71 bc	1.90 ± 0.15 abc	0.97 ± 0.00 d	0.04 ± 0.00 ab	5.56 ± 0.13 e	4.71 ± 0.13 cd	0.19 ± 0.00 e
2	18.8	7.5	12.73 ± 2.40 e	1.10 ± 0.15 c	1.03 ± 0.01 bcd	0.05 ± 0.00 ab	6.68 ± 0.07 c	5.89 ± 0.06 b	0.24 ± 0.00 d
3	40	11	24.20 ± 1.28 bcd	2.06 ± 0.22 abc	1.03 ± 0.01 bcd	0.05 ± 0.00 a	8.06 ± 0.01 ab	7.25 ± 0.06 a	0.36 ± 0.01 b
4	40	7.5	25.99 ± 0.15 b	1.27 ± 0.30 c	1.04 ± 0.02 bcd	0.04 ± 0.00 ab	5.80 ± 0.04 de	4.75 ± 0.16 cd	0.27 ± 0.00 c
5	25	10	20.53 ± 1.57 cd	2.45 ± 0.61 a	1.31 ± 0.04 a	0.05 ± 0.00 ab	7.90 ± 0.20 b	7.15 ± 0.04 a	0.33 ± 0.01 b
6	40	7.5	24.60 ± 1.34 bc	1.10 ± 0.14 c	1.10 ± 0.01 b	0.05 ± 0.00 ab	6.86 ± 0.42 c	6.05 ± 0.24 b	0.26 ± 0.02 cd
7	40	4	20.19 ± 1.15 d	1.20 ± 0.00 c	0.98 ± 0.01	0.08 ± 0.00	7.96± 0.13 b	7.16 ± 0.01 a	0.16 ± 0.01 f
8	61.2	7.5	30.18 ± 1.11 a	2.37 ± 0.02 ab	1.07 ± 0.02 bc	0.04 ± 0.00 ab	5.53 ± 0.01 e	4.51 ± 0.07 d	0.28 ± 0.01 c
9	40	7.5	25.66 ± 1.45 b	1.53 ± 0.23 bc	1.23 ± 0.00 a	0.05 ± 0.00 ab	6.11 ± 0.06 d	5.38 ± 0.04 bc	0.28 ± 0.01 c
10	25	5	25.90 ± 0.27 b	1.00 ± 0.14 c	1.23 ± 0.02 a	0.04 ± 0.00 b	8.45 ± 0.34 a	7.66 ± 0.04 a	0.15 ± 0.01 f
11	55	10	27.28 ± 1.53 ab	1.46 ± 0.11 bc	1.26 ± 0.05 a	0.05 ± 0.00 a	8.11 ± 0.08 ab	7.41 ± 0.11 a	0.52 ± 0.02 a

Values expressed as average of 3 determinations ± standard deviation. Different letters indicate significant differences between the means ($p < 0.05$). For the k_{270} statistical test, the oil obtained in assay 7 was not taken into account since a turbid oil was obtained. T, temperature; MC, moisture content; PV, peroxide value; OS, oxidative stability; IT, induction time; FFAC, free fatty acid content.

In assay 7 (T= 40.0 °C - MC= 4.0%) the K_{270} value was 0.08 ± 0.00%, which was higher than that of the other oils. This value was attributed to the high turbidity of the sample due to the presence of fine suspended solids, so it was not included in the statistical analyses nor as a response to evaluate the effectiveness of the treatments.

Table 2 shows that when one of the experimental factors was kept constant, FFAC was directly proportional to both temperature and moisture content. The variation range was between 0.15 ± 0.01% and 0.52 ± 0.02% of oleic acid.

3.3 Oxidative Stability and Induction Time

The oxidative stability of the different oils ranged from 5.53 ± 0.01 h to 8.45 ± 0.34 h; while the induction time varied between 4.51 ± 0.07 h and 7.66 ± 0.04 h (Table 2).

Regarding the results of OS and IT for the oils obtained in the experimental design, it can be observed that, in general, for a given moisture content, oil stability was inversely proportional to the pressing temperature. Greater oxidative stability in the oil implies higher OS and IT values.

3.4 Color

The luminosity ranged from 25.14 ± 0.44 to 32.13 ± 0.12 and the parameters a* and b* ranged from 0.27 ± 0.03 to 2.47 ± 0.04 and 11.68 ± 0.49 to 26.29 ± 0.20, respectively (Table 3). For a given moisture content, an increase in temperature resulted in the oils darkening. This is evidenced by lower L* and b* values and higher a* values. However, when moisture content increased while temperature remained constant, no clear pattern in behavior was observed.

Table 3. Effect of process variables on pecan oil color

Assay	Factors		Color		
	T (°C)	MC*	L*	a*	b*
1	55	5	30.47 ± 0.13 c	0.96 ± 0.05 e	24.35 ± 0.38 b
2	18.8	7.5	31.36 ± 0.05 b	1.16 ± 0.03 d	26.29 ± 0.20 a
3	40	11	29.77 ± 0.12 de	0.95 ± 0.04 e	23.48 ± 0.03 bcd
4	40	7.5	30.04 ± 0.03 cd	1.36 ± 0.02 c	24.07 ± 0.11 bc
5	25	10	32.13 ± 0.12 a	0.72 ± 0.04 f	24.54 ± 0.17 b
6	40	7.5	32.03 ± 0.09 a	0.90 ± 0.03 e	24.28 ± 0.49 b
7	40	4	25.14 ± 0.44 f	1.07 ± 0.07 d	11.68 ± 0.49 f
8	61.2	7.5	29.34 ± 0.13 e	1.84 ± 0.05 b	22.56 ± 0.27 de
9	40	7.5	29.98 ± 0.37 d	1.08 ± 0.06 d	22.95 ± 1.44 cde
10	25	5	32.07 ± 0.10 a	0.27 ± 0.03 g	26.29 ± 0.19 a
11	55	10	29.37 ± 0.08 e	2.47 ± 0.04 a	22.11 ± 0.13 e

Values expressed as average of 3 determinations ± standard deviation. Different letters indicate significant differences between the means ($p < 0.05$). The L* parameter measures lightness, ranging from white (L* = 100) to black (L* = 0). The a* value indicates the degree of red or green color components (a* > 0: red; a* < 0: green), while the b* value reflects the degree of yellow or blue components (b* > 0: yellow; b* < 0: blue).

The lowest values of L* and b* were observed in the oil from assay 7, which was attributed to the presence of fine solids in suspension. The oil obtained under the conditions of assay 10 (T= 25.0 °C - MC= 5.0%) was the one that showed lighter tones, as indicated by higher L* and b* values and a lower a*. In contrast, the oil from assay 11 (T= 55.0 °C - MC= 10.0%) had lower L* and b* values and a higher a*, signifying that it exhibited darker tones.

3.5 Effect of Experimental Factors on Physicochemical Parameters

The influence of experimental factors –temperature and moisture content of crushed pecans during pressing– on the previously analyzed variables was determined by Analysis of Variance. Table 4 shows the p-values for temperature and moisture content in their linear and quadratic components, as well as for the temperature-moisture content interaction. The quadratic component of temperature (T^2) and the temperature-moisture

content interaction (T×MC) had a $p < 0.05$ for OY. Additionally, the linear component of temperature (T) and T×MC showed a p-value < 0.05 for the peroxide value (PV). The experimental factors did not significantly influence the other dependent variables measured. Therefore, OY and PV were selected as the response variables for the experimental design. The response surface equation obtained for OY was as follows:

$$OY = 12.89 - 0.006 \times T^2 + 0.05 \times T \times MC \quad \text{Equation 1}$$

And the equation for PV was:

$$PV = 0.77 + 0.03 \times T - 0.01 \times T \times MC \quad \text{Equation 2}$$

Where:

T= Temperature of the crushed nut during pressing (°C).

MC= Moisture content of the crushed pecans during pressing (%).

Table 4. Analysis of Variance table of the experimental factors on the physicochemical variables

Dependent variables	p-Temperature (T)	p-Moisture content (MC)	p-T^2	p-T × MC	p-MC^2	Coefficient of determination (R^2) (%)	Mean absolute error (MAE)	p-Durbin-Watson (DW)
OY	0.17	0.08	**0.04**	**0.03**	0.09	67	1.91	0.84
PV	**0.04**	0.58	0.13	**0.04**	0.2	81	0.19	0.8
k_{232}	0.31	0.44	0.91	0.4	0.68	32	0.05	0.3
k_{270}	0.18	0.18	0.62	0.31	0.49	78	0.01	0.08
OS	0.06	0.07	0.89	0.1	0.05	88	0.32	0.06
IT	0.08	0.08	0.9	0.13	0.06	86	0.35	0.09
FFAC	0.8	0.21	0.8	0.23	0.87	87	0.03	0.88
L*	0.73	0.18	0.68	0.78	0.24	52	1.08	0.88
a*	0.82	0.42	0.44	0.29	0.66	71	0.26	0.99
b*	0.73	0.16	0.51	0.95	0.23	52	2.05	0.63

The p values of the experimental factors that had a significant influence on the response variables are shown in bold.

It should be noted that in the above equations the terms of the experimental factors were rejected, either in their linear or quadratic components, which had no significant influence on the OY or the PV.

The R^2 statistic, the MAE and the p-Durbin-Watson shown in Table 4 determine the quality of the adjusted models. The R^2 for OY indicated that the model explained 67% of the variability in this response variable. Additionally, OY satisfied the other two criteria for model fit quality: a low MAE value and a DW p-value greater than 0.05.

For PV, the same criteria were met, and the R^2 value of 81% indicated that the model fit was satisfactory.

From each equation, the influence of the experimental factors on the OY and the PV was studied. Response surface graphs and contours were generated to illustrate these influences. Figure 1 shows the estimates for OY as a function of temperature and moisture content of the crushed nuts during pressing. In the figure it can be observed that, for the experimental region studied, the highest OY were obtained at high values of temperature and moisture content.

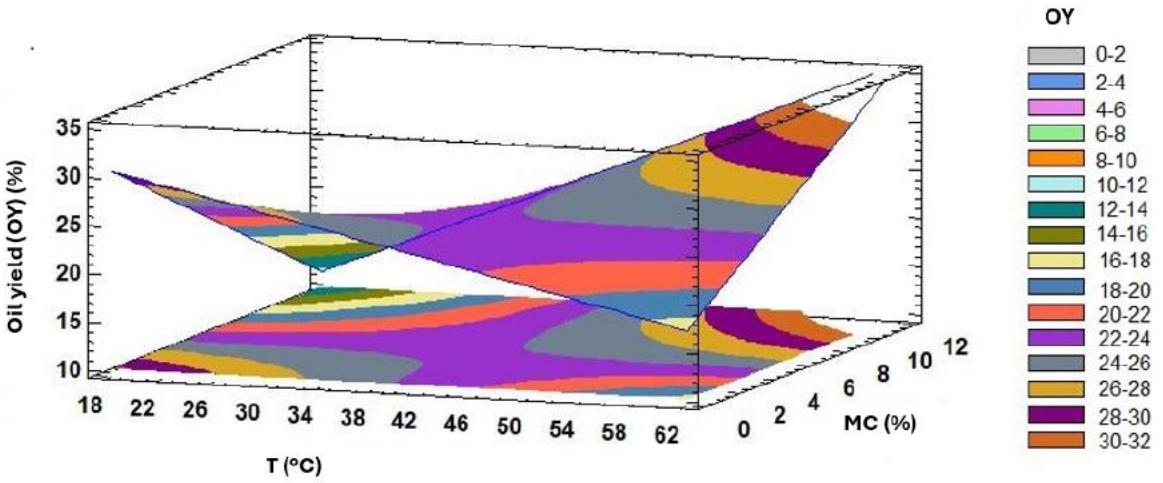


Figure 1. Response surface (upper graph) and response surface contours (lower graph) for oil yield as a function of temperature and moisture content of the crushed nuts during the pressing stage

The levels of the experimental factors that maximized the OY were then obtained (Table 5).

Table 5. Combination of experimental factors that maximized oil extraction yield using response surface methodology

Experimental factors	Optimal value	OY value maximized with RSM
Temperature of the pressing process (°C)	61.2	30.9%
Moisture content of crushed pecans (%)	11	

Figure 2 shows the estimates for PV as a function of temperature and moisture content. In contrast to what was observed for OY, lower PV values are observed at lower temperature and moisture content levels.

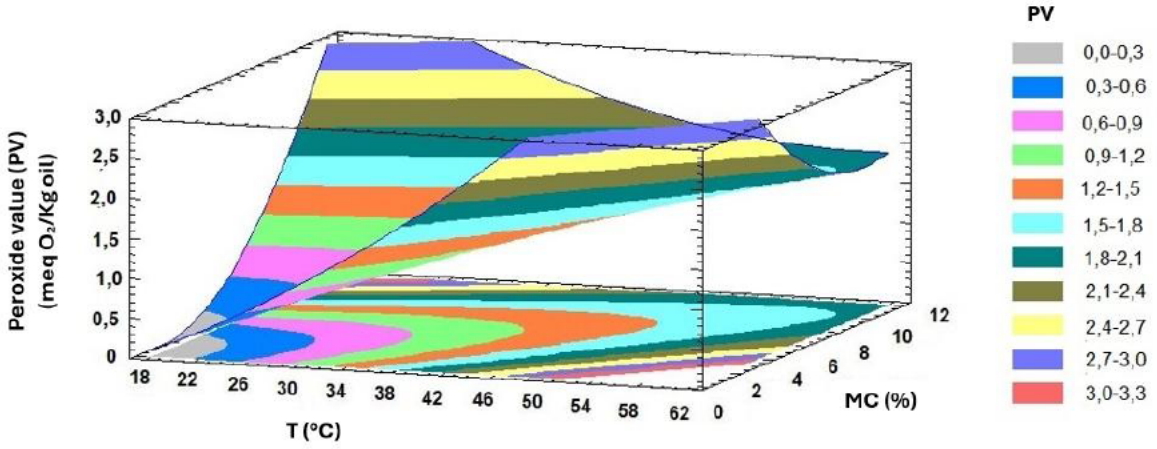


Figure 2. Response surface (top graph) and response surface contours (bottom graph) for peroxide value as a function of temperature and moisture content of crushed nuts during the pressing stage

The combination of experimental factors that minimized the PV is shown in Table 6.

Table 6. Combination of experimental factors that minimized peroxide value using response surface methodology

Experimental factors	Optimal value	PV minimized with RSM
Temperature of the pressing process (°C)	18.8	0.44 meq O_2/kg oil
Moisture content of crushed pecans (%)	4	

From the above, it can be observed that the experimental conditions that maximized OY and those that minimized PV were in contrast. Therefore, the desirability function was used to find the combination of experimental factors that maximized OY and minimized PV simultaneously. The desirability function showed higher values for intermediate values of temperature and moisture content. Figure 3 shows the estimates of this function versus the temperature and moisture content of the nuts during the pressing stage.

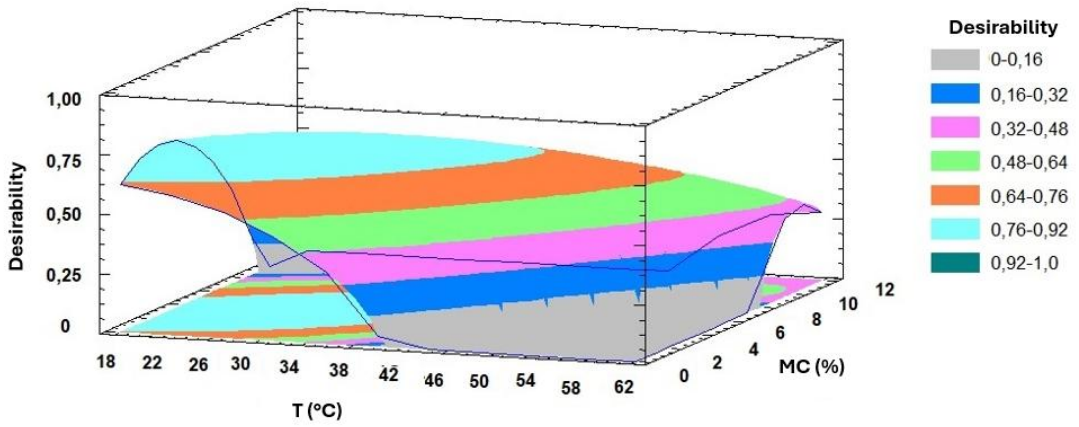


Figure 3. Desirability as a function of temperature and moisture content of crushed nuts during the pressing stage

Using the desirability optimization tool of the STATGRAPHICS Centurion XV software Version 15.2.06, it was found that the optimal temperature and moisture content for the pressing stage are 34.7 °C and 5.4%, respectively (Table 7). Under these conditions, the desirability function achieved a value of 0.79, indicating that the combination of experimental factors was suitable (Montgomery, 2013). The predicted values for OY and PV under these conditions were 23.6% and 0.99 meq O2/kg, respectively.

Table 7. Combination of experimental factors that maximized the desirability function

Experimental factors	Optimal value	Desirability
Temperature of the pressing process (°C)	34.7	0.79
Moisture content of crushed pecans (%)	5.4	

3.6 Model Validation

As mentioned in the methodology, to validate the model obtained through the RSM, oil was produced to compare the predicted values with the experimental results for OY and PV. In order to simplify the temperature control in the pressing stage, the process was carried out with crushed nuts at 35 °C and 5.5% moisture content.

The initial moisture content of the pecans was 8.21 ± 0.5%, so it was necessary to dry them until reaching 5.56 ± 0.39% moisture content.

The oil was then obtained as described in the procedures and the OY and PV were quantified. The results are shown in Table 8.

Table 8. Comparison between predicted values and experimental results for oil extraction yield and peroxide value

Experimental factors	Predicted values	Experimental values
Oil yield (%)	23.60	22.27 ± 1.18
Peroxide value (meq O_2/Kg oil)	0.99	0.95 ± 0.10

The experimental values obtained for oil yield (22.27 ± 1.18%) and peroxide value (0.95 ± 0.10 meq O_2/kg of oil) were within the 95% confidence interval of the values predicted by the central composite design (23.60% and 0.99 meq O_2/kg, respectively). Therefore, the experimental validation conducted under the optimal conditions predicted by the model confirmed its adequacy.

4. Discussion

In this research, an increase in OY was observed as the temperature and moisture content of the crushed pecan nut were higher during the pressing stage, which was attributed to the application of thermal and humidification treatments prior to pressing, which promoted the expansion and rupture of the cellular structures and increased the plasticity and permeability of the material, facilitating the release of oil and the consequent increase in OY (Martínez et al., 2017). For oilseeds with high protein content, protein coagulation induced by heat treatment can significantly enhance oil recovery (Pradhan et al., 2011). However, Rombaut et al. (2015) found that, as moisture content increased, oil yield decreased in three types of grape seeds. Similar effect was found by Khan and Hanna (1983) for soybeans and by Singh et al. (2002) for crambe seeds. On the other hand, Pradhan et al. (2011) found an increase in OY from jatropha seeds as moisture content increased up to a certain optimal value, after which further increases in seed moisture led to a rapid decline in oil recovery. Similar results were reported by Singh and Bargale (2000) and Martínez et al. (2013) in the case of almond pressing.

In general, the PV increased with increasing pressing temperature and moisture content of crushed nuts. As in the present research, reports were found that refer to the increase in PV when working at high temperatures. Thus, when oil was extracted from pistachios with a hydraulic press, previously dried at 70 °C, PVs slightly higher than 2 meq O_2/kg of oil were reached, compared to 0.5 meq O_2/kg of oil when the temperature was less than or equal to 50 °C (Sena-Moreno et al., 2015). According to Godson & Bassey (2015) and Lafont et al. (2019), when PV values are higher than 2 meq O_2/kg, the oils are prone to present rancidity. In the present investigation, only two of the oils had PV higher than the value indicated by these researchers and they were the oils which conditions corresponded to the highest moisture content (Assay 3: T= 40.0 °C - MC= 11.0%) and the highest temperature (Assay 8: T= 61.2 °C - MC= 7.5%). However, none of the results exceeded the maximum value set by the Codex Alimentarius (Joint FAO/WHO Codex Alimentarius Commission, 1992) for unrefined special oils (15 meq O_2/kg of oil).

Regarding the higher oil extraction observed in the first stages of assay 8 (T= 61.2 °C - MC= 7.5%), it was attributed to the high temperature used during pressing. As mentioned above, the application of heat treatment before or during pressing improves oil extraction (Bhuiya et al., 2020; Rombaut et al., 2015).

As with the PV, higher moisture content values resulted in higher K_{232} and K_{270} values. The values obtained in this research were slightly lower than those reported for oils from walnut grown in Catamarca (Argentina) and obtained under similar conditions (T= 50.0 °C - MC= 7.5%), whose K_{232} and K_{270} were 1.18 ± 0.01% and 0.06 ± 0.00%, respectively (Martínez et al., 2013). Additionally, the results were comparable to those found by Oro et al. (2009) for pecan oil (K_{232} of 0.83 ± 0.00% and K_{270} of 0.03 ± 0.02%); and lower than those reported by Fantino et al. (2020), who found K_{232} and K_{270} values within the ranges of 1.6-1.8% and 0.14-0.16%,

respectively. These latter authors concluded that the different extraction treatments had minimal effects on the physicochemical quality parameters of pistachio oil.

The OS and IT parameters showed that the oils were better preserved at lower temperatures, suggesting that heat treatments reduced oil quality. This is attributed to the fact that oxidation products tend to increase with increasing temperature, as pointed out by Sena-Moreno et al. (2015) during the extraction of pistachio oil when temperatures were raised from room temperature to 30, 50 and 70 °C. In this sense, the values obtained in the present work were lower than those reported by other authors (Oro et al., 2009; Rábago Panduro et al., 2020) for pecan oil obtained with a screw press. Oro et al. (2009) reported an OS value of 9.8 h, while Rábago Panduro et al. (2020) had values that fluctuated from 10.4 ± 0.4 h to 10.9 ± 0.3 h, depending on the treatment applied.

On the other hand, the FFAC of the oils decreased as the temperature and moisture content of the nuts decreased. The FFAC is one of the chemical characteristics that best define the quality of an oil or fat, as it indicates the alteration of triglycerides due to chemical or enzymatic hydrolysis (García Martínez et al., 2014). Martínez et al. (2017) reported FFAC values ranging from 0.06% to 0.17% oleic acid for walnut oil, while almond oils showed values between 0.33% and 0.47%. These authors concluded that the different extraction treatments had minimal effects on the chemical quality parameters of oils, which might be due to the relatively small variations in moisture content (5.5 to 10.0% for walnut and 9.0 to 11.0% for almond), compared to the broader range assessed in this study. In comparison, pecan oils from the Barton cultivar, extracted using a hydraulic press without heating, showed FFAC values of 0.13 ± 0.01% oleic acid (Polmann et al., 2022), which are similar to the values observed in this study at lower pressing temperatures.

It should be noted that none of the parameters indicating oxidative deterioration of the oils, regardless of the treatment, exceeded the limits specified in Chapter VII of the Código Alimentario Argentino (CAA) (Argentina, 2012) for extra virgin olive oil, which stipulates maximum values of free acidity expressed as oleic acid of 2 g per 100 g, K_{232} of 2.50%, K_{270} of 0.22%, and PV of 20 meq O_2/kg of oil (Art. 535 CAA). As for sunflower oil, the CAA in article 528 indicates for virgin sunflower oil a maximum PV of 15 meq O_2/kg of oil and FFAC of 2% expressed as oleic acid; while for refined sunflower oil the maximums established are 10 meq O_2/kg of oil and 0.2%, respectively. Therefore, none of the oils obtained, according to the treatments of the experimental design, exceeded the PV values indicated by the CAA for either virgin or refined sunflower oil, nor the FFAC for the case of virgin sunflower oil. However, only the oils whose raw material had low moisture content (between 4.0% and 5.0%) did not exceed the FFAC indicated by the CAA for the refined sunflower oil category.

The results of the color parameters measured in the CIEL*a*b* space showed that the increase in the pressing temperature affected the color of the final product, causing a darkening, while at low temperatures the oils were lighter. However, as previously noted, changes in moisture content did not result in a clear trend in these parameters. In this sense, Prabhakar et al. (2022) indicated that at higher temperatures, the decomposition rate of flavonoids and Maillard reaction products increases, contributing to a higher a* component (redness) in the oils. On the other hand, Abenoza et al. (2013) indicated that the higher temperature used during the production of olive oil could facilitate the extraction of pigments. Oils pressed at lower temperatures exhibit higher L* values (luminosity), as pigments absorb light rather than transmitting it. Therefore, higher pressing temperatures lead to oils with a higher a* component and lower L* and b* components, resulting in darker colors.

On the other hand, the presence of fine suspended solids in assay 7 (T= 40.0 °C - MC= 4.0%) was attributed to the low moisture content of the crushed nut at the time of pressing. Fantino et al. (2020) also obtained high values of fines in oils in which pistachio seeds had a low moisture content (3%); while at higher moisture contents (7-11%) the fine content was lower. The authors attributed this behavior to the fact that the pressed cake becomes more compact at higher moisture content, reducing the amount of sediment to pass through the holes in the canvas during pressing.

Regarding the physicochemical analysis, oils produced from crushed nuts with low moisture content and moderate pressing temperatures (T= 40.0 °C - MC= 4.0% and T= 25.0 °C - MC= 5.0%) had high OS and IT values and low PV, K_{232} and K_{270}, and FFAC values. These oils were the ones that presented lighter shades.

Oils extracted at high pressing temperatures (T= 55.0 °C, MC= 5.0% and T= 55.0 °C, MC= 10.0%) displayed intermediate PV. On the other hand, the oil produced from crushed nuts with a moisture content of 5.0% had low values of K_{232} and K_{270}, OS, IT, and FFAC. In contrast, the oil obtained from crushed nuts with a moisture content of 10.0% showed elevated values for K_{232}, K_{270}, OS, IT, and FFAC. In terms of color parameters, both oils showed dark tones.

In the analysis of the experimental design, through ANOVA, it was found that the experimental factors had a significant influence on the OY and the PV, so these attributes were considered as response variables in the optimization of the experimental design. With the RSM, using the desirability function, the temperature and moisture content values that simultaneously maximized the OY and minimized the PV were obtained. The validation tests indicated correspondence between the OY and PV values predicted with the RSM and the experimental values. Therefore, in the experimental region studied, the temperature and moisture content of the crushed nuts during pressing that maximized the OY and minimized the PV were 35 °C and 5.5%, respectively.

Several publications have explored experimental conditions that maximize oil production. For instance, Martínez et al. (2017) investigated walnut oil extraction at temperatures between 35 and 40 °C using an industrial-scale press. They reported an OY of 84.5 ± 2.3% at a moisture content of 7.21%, with PV ranging from 0.65 to 1.14 meq O_2/kg of oil. On the other hand, Preziuso et al. (2010) obtained olive pastes using three different methods, reporting yields ranging from 80.1 to 85.2%. The authors conducted their work at 27 °C using an industrial olive mill equipped with a three-phase centrifugal decanter for oil recovery. The peroxide value (PV) ranged from 4.9 to 6.2 meq O_2/kg of oil. The moisture content of the olives ranged from 50.5 to 52.6%. This OY was significantly higher than the values obtained in the present research, which can be attributed to the types of press utilized and the industrial-scale processing. While Fantino et al. (2020) concluded that it is possible to obtain oils without generating oxidative and/or hydrolytic damage in the optimization of pistachio oil extraction with a screw press, with a PV of 0.05 to 0.28 meq O_2/kg of oil in the experimental region between MC= 8-10% and T= 40-75 °C. Under these conditions, they reported OY values between 65.97% and 79.61%. Other researchers (Nader et al., 2016) used response surface methodology (RSM) to optimize the oil extraction process from peanuts using a hydraulic press. Nader et al. (2016) achieved an efficient oil yield of 70.6%, with a seed moisture content of 5%. These yields were also higher than those observed in the present study, likely due to differences in the raw material matrix and the type of press used.

5. Conclusions

Consumers are increasingly demanding nutritious foods with health-promoting properties. Therefore, maintaining pecan oil nutritional quality during the manufacturing process is crucial, as the fatty acids oil are susceptible to oxidation. This study demonstrated that the moisture content and temperature of the crushed nuts during the pressing stage significantly influenced both the oil yield and peroxide value. The experimental results indicated that, under the tested conditions, the optimal values for maximizing oil yield and minimizing peroxide value were achieved at a temperature of 35 °C and a moisture content of 5.5%. In this work, pecan oil quality was optimized to meet consumer demands, while the manufacturing process yield was maximized, which is of interest to the industry.

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Transparency of Data

Available data: The entire data set that supports the results of this study was published in the article itself.

Author Contribution Statement

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Conceptualization	■	■					
Data curation	■	■					
Formal analysis		■					
Funding acquisition	■		■				
Investigation	■			■	■	■	■
Methodology		■					
Writing – original draft	■						
Writing – review and editing		■	■				

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