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Comparative Characterization of Phenolic Compounds, Fatty Acid Profile, and Antioxidant Activity of Pecan (*Carya illinoinensis*) Oil Obtained Using Conventional and Green Extraction Methods

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Abstract

Pecan oil is a valuable source of unsaturated fatty acids and bioactive compounds whose recovery and stability may be influenced by the extraction process. This study compared Soxhlet extraction (SE), ultrasound-assisted extraction (UAE), and supercritical CO_2 extraction (sCO_2) in terms of extraction yield, fatty acid profile, physicochemical properties, total phenolic content (TPC), antioxidant activity, and oxidative stability. SE achieved the highest extraction yield (69.21%), followed by sCO_2 (52.89%) and UAE (48.68%). The fatty acid profile was relatively stable among extraction methods, although differences were observed in the relative proportions of oleic and linolenic acids. UAE produced oil with the highest TPC (32.55 μg GAE/g oil), whereas sCO_2 showed the highest initial antioxidant activity (2681 μmol TE/100 g oil). After 12 days, UAE oil exhibited the lowest peroxide value (10.49 meq O_2/kg) and the highest TPC retention, whereas sCO_2 oil showed the greatest peroxide accumulation (28.16 meq O_2/kg) and the largest decrease in TPC. These results demonstrate that extraction conditions differently affect oil recovery, antioxidant-related properties, and oxidative stability. Under the conditions evaluated, UAE showed particular advantages in preserving TPC and limiting primary oxidation during accelerated storage, despite its lower extraction yield.

Keywords: pecan oil; ultrasound-assisted extraction; supercritical fluid extraction; antioxidant activity; oxidative stability; bioactive compounds

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1. Introduction

Pecan (*Carya illinoinensis*) is a native nut belonging to the southern United States and northern Mexico, and now it is also grown in Australia, South Africa, Israel, Brazil, and other South American countries [1,2]. Currently, Mexico accounts for 40.1% of global production of this type of nut, with nearly 80% of its domestic production destined for export [3], while in countries such as Brazil, cultivation continues to expand, driven by growing evidence of its health benefits [4]. This nut is characterized by its high lipid content, which, depending on the crop, can reach up to 78% [2,4], and it is also an important source

of edible vegetable oil [5]. The fatty acid profile of pecan oil consists mainly of unsaturated fatty acids, which can account for up to 90% of the total content, with more than 60% being monounsaturated fatty acids, primarily oleic acid [6,7]. This composition contributes to its relatively high oxidative stability compared to other vegetable oils with a higher proportion of polyunsaturated fatty acids, such as walnut, peanut, soybean, and canola oils [7].

Regular consumption of pecans has been associated with various health benefits, particularly improvements in cardiovascular risk factors, such as reduced levels of total cholesterol, low-density lipoprotein cholesterol (LDL-C), and triacylglycerols, as well as favorable changes in lipid profiles [1,2]. These effects have been related to their lipid composition and the presence of bioactive compounds, including phytosterols, tocopherols, squalene, and phenolic compounds like gallic acid, catechins, and proanthocyanidins [2,5,6]. Among these components, phytosterols are notable for their ability to lower LDL cholesterol levels, whereas γ-tocopherol has been associated with protection against oxidative damage mediated by reactive nitrogen species (RNS) [2]. Several of these lipid-soluble compounds are also present in pecan oil, contributing to its nutritional and functional value [6].

In addition to its nutritional value, pecan oil is considered a specialty product, prized for its sensory characteristics and used both in dressings and culinary preparations and in the formulation of cosmetic products, including lotions, soaps, perfumes, and massage oils [8]. Pecan processing also generates agro-industrial by-products, particularly pecan cake, which, despite retaining a considerable amount of residual oil, having a favorable nutritional composition, and possessing pleasant sensory characteristics, is primarily used for animal feed, thereby limiting its commercial value [8].

Pecan shells also constitute another agro-industrial byproduct generated in significant quantities during fruit processing, and have been identified as a potential source of bioactive compounds with antioxidant activity [3]. In this context, various studies have evaluated the recovery of the remaining lipid fraction in these byproducts using more sustainable extraction technologies, such as supercritical fluid extraction (SFE), achieving recoveries of over 98% of the oil present in pecan cake [8]. These findings highlight the need to optimize pecan oil extraction processes, not only to maximize yield and preserve quality when using the whole nut, but also to promote the valorization of processing by-products [3,8].

Various techniques have been used to obtain vegetable oils, and the operating conditions of these techniques can affect both the extraction yield and the quality and stability of the resulting product. Among the conventional methods, extraction with organic solvents stands out; this method is frequently evaluated using the Soxhlet apparatus on a laboratory scale due to its high capacity to recover the lipid fraction from plant matrices. However, this technique requires lengthy processing times and large volumes of solvent, in addition to involving high temperatures during the extraction and evaporation stages. These conditions can promote the degradation of heat-labile compounds and increase the risk of solvent residues in the resulting oil [9].

These limitations have driven the development of alternative extraction technologies aimed at reducing solvent consumption, processing time, and environmental impact. Among these, ultrasound-assisted extraction (UAE) is based on the phenomenon of acoustic cavitation. The formation, growth, and collapse of microbubbles generate shear forces and microturbulence that facilitate the disruption of cellular structures and enhance mass transfer between the plant matrix and the extraction medium. Compared to conventional techniques, this procedure can reduce extraction time and temperature, potentially favoring the preservation of heat-sensitive bioactive compounds [3,10].

Supercritical fluid extraction (SFE), particularly using supercritical carbon dioxide (CO_2), is an attractive alternative for obtaining high-quality plant oils. CO_2 is a non-toxic, non-flammable solvent that can be easily separated from the extract through depressur-

ization, resulting in products free of organic solvent residues. Furthermore, the moderate temperatures used during the process may favor the preservation of bioactive compounds susceptible to thermal degradation [10,11]. However, its implementation on an industrial scale requires equipment capable of operating at high pressure, and its economic viability depends on factors such as extraction pressure, plant configuration and capacity, processing time, and the relationship between the yield obtained and the commercial value of the product [12]. Despite these considerations, the process's selectivity, the reduction in the use of organic solvents, and the quality of the extracts obtained support its application in the production of high-value-added oils and ingredients [9,11].

Although various studies have evaluated the influence of extraction techniques on the physicochemical properties of pecan oil, the available evidence has focused primarily on extraction yield and fatty acid composition [6,8]. In contrast, the effect of these technologies on the evolution of the oil's oxidative stability during storage has received less attention [6]. Studies comparing supercritical CO_2 extraction with subcritical or conventional processes have shown that the extraction method can influence the yield, composition, and oxidative stability of pecan oil [6,8].

In some cases, oils obtained using supercritical CO_2 have shown lower resistance to oxidation, which has been attributed to the fluid's low polarity and, consequently, its reduced ability to recover relatively polar antioxidant compounds, such as certain phospholipids and phenolic compounds [6]. Likewise, it has been observed that parameters such as the acidity index and antioxidant capacity of pecan oil can vary depending on the extraction technique used [13].

However, there are still very few studies that directly compare, using the same raw material and under equivalent experimental conditions, the oxidative stability of pecan oils obtained via Soxhlet extraction, ultrasound-assisted extraction, and supercritical fluid extraction. This comparison is particularly relevant when accelerated oxidation tests, such as the Schaal test, are used to evaluate changes in oil quality during storage, as the selection of an extraction technology may involve trade-offs between lipid fraction recovery, the preservation of bioactive compounds, and the oxidative stability of the final product.

In this context, the objective of the present study was to evaluate the influence of three extraction techniques, Soxhlet, ultrasound-assisted extraction, and supercritical fluid extraction, on the physicochemical characteristics and oxidative stability of pecan oil (*Carya illinoinensis*). Oil stability was evaluated using the Schaal test to compare the oxidative behavior of the oils during accelerated storage and to assess the trade-offs between extraction yield, physicochemical quality, and resistance to oxidation, providing relevant information for the potential application of these extraction technologies in the food industry.

2. Materials and Methods

2.1. Raw Material

The pecans (*Carya illinoinensis*) used in this study were obtained in the town of Los Tambos, Alto del Carmen commune, Huasco province, Atacama Region, Chile, and were purchased directly from local producers. They were then visually inspected to remove any that contained foreign matter, visible damage, or signs of fungal contamination. The selected nuts were packaged in polyethylene bags and stored in a cool, dry place until processing. Before oil extraction, the nuts were shelled, and the kernels were crushed manually in a porcelain mortar. The sample was then passed through a sieve with a 500 µm aperture to obtain a more uniform particle size and facilitate the extraction process.

2.2. Proximate Composition and Water Activity

The proximate composition of pecans was determined using Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC), 15th edition [14]. Crude protein content was quantified using the Kjeldahl method, with a nitrogen-to-protein conversion factor of 6.25 (AOAC Official Method 960.52). Crude fat was determined via Soxhlet extraction with petroleum ether (AOAC Official Method 960.39), crude fiber by sequential acid and alkaline digestion (AOAC Official Method 962.09), and total ash by incineration at 550 °C until constant weight (AOAC Official Method 923.03). No modifications were made to the cited AOAC procedures. Total carbohydrates were calculated by difference. All determinations were performed in triplicate.

Water activity (a_w) was determined at 25 °C using a dew point water activity analyzer (AQUALAB, model 4TE, Pullman, WA, USA). Three measurements were taken per sample, and the reported value corresponded to the average of the determinations.

2.3. Mineral Content

The content of copper (Cu), iron (Fe), calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na) was determined via atomic absorption spectrophotometry (AAS) (PinAAcle 900F, PerkinElmer, Shelton, CT, USA). Prior to analysis, the samples were incinerated at 550 °C. To determine Cu, Fe, Ca, Mg, and K, the resulting ash was digested with 2 mol/L HCl, and the mixture was boiled until its volume was reduced to approximately half. Subsequently, the solutions were filtered, and 1 mL of lanthanum solution (10 g/L) was added as an interference suppressant. To quantify Na, the ashes were treated with nitric acid and heated until completely dissolved. Finally, the solutions were transferred to volumetric flasks of 25 mL and brought to volume with deionized water. All determinations were performed in triplicate, and the results were expressed in mg/100 g of dry matter (d.m.).

2.4. Oil Extraction Methods

Three methods were evaluated for extracting oil from crushed and sieved pecans: conventional Soxhlet extraction (SE), ultrasound-assisted extraction (UAE), and supercritical carbon dioxide (sCO_2) extraction.

2.4.1. Conventional Soxhlet Extraction

A portion of the dried and ground sample was extracted using a Soxhlet apparatus (Gerhardt, Königswinter, Germany), with petroleum ether as the solvent. The extraction was carried out for 6 h under continuous solvent reflux. After extraction, the solvent was removed by evaporation, and the round-bottom flask with a ground-glass joint containing the lipid extract was allowed to cool to room temperature. The resulting oil was filtered and stored in a sealed container protected from light until analysis. All extractions were performed in triplicate.

2.4.2. Ultrasound-Assisted Extraction (UAE)

The crushed sample was placed in a 150 mL beaker, and absolute ethanol was added at a solid-to-solvent ratio of 1:1.63 (g/mL). The mixture was homogenized using an Ultra-Turrax T25 homogenizer (IKA, Staufen, Germany) at 13,000 rpm for 5 min until a visually homogeneous suspension was obtained. The beaker was then placed in an ultrasonic bath (Dentsply Ney®, Yucaipa, CA, USA) for 40 min. During sonication, the distilled water level in the bath was maintained above the level of the sample–solvent mixture to ensure uniform transmission of ultrasonic energy. After extraction, the mixture was centrifuged at 5000 rpm and 30 °C for 30 min, resulting in three distinct phases: a solid phase, an oily phase, and a supernatant. The oily phase was carefully collected using a

micropipette, filtered, and stored in sealed containers protected from light until analysis. All extractions were performed in triplicate. These operating conditions were selected to promote adequate contact between the ground grains and ethanol while maintaining a moderate sonic duration. The initial high-shear homogenization was used to improve matrix dispersion and facilitate mass transfer, while ethanol was selected because it was suitable for the recovery of both lipid and relatively polar components and its lower toxicity compared to conventional petroleum solvents. These conditions were used as operational parameters for the method comparison and did not derive from an optimization study.

2.4.3. Supercritical Carbon Dioxide (sCO_2) Extraction

The extraction was performed using a Spe-ed SFE supercritical fluid system (Applied Separations, model 7070, Allentown, PA, USA), employing carbon dioxide as the extraction fluid. The dried, ground, and sieved sample was placed in the extraction vessel between layers of polypropylene wool, forming a fixed bed inside the vessel. Extraction was carried out at a pressure of 300 bar and a temperature of 40 °C. The CO_2 flow was maintained at a constant rate of 1.0 L/min and regulated by a micrometric valve. Initially, the system was kept under static conditions for 60 min to promote contact between the supercritical CO_2 and the matrix. Subsequently, dynamic extraction was performed for 180 min, maintaining a continuous flow of CO_2. The oil was collected in pre-weighed glass vials protected from light. The mass of the extracted oil was determined gravimetrically by calculating the difference between the mass of the empty vial and that of the vial containing the extract. Extractions were performed in triplicate, and the oils were stored away from light until subsequent analysis. Operating conditions of 300 bar and 40 °C were chosen because this pressure provides sufficient density and solvent resistance for CO_2 for lipid extraction, while moderate temperature limits thermal exposure of oxidation-sensitive oil components. The static period was included to facilitate CO_2 penetration and equilibrium within the fixed bed, followed by a dynamic phase to promote the continuous removal of the solvent. These conditions were selected on the basis of previously reported applications of sCO_2 for plant oil extraction [6,8,11] and were not intended to represent optimized conditions for this pecan material.

2.4.4. Determination of Extraction Yield and Efficiency

The extraction yield was calculated as the ratio between the mass of oil recovered after extraction and solvent removal and the initial mass of pecan kernels used in the extraction, according to Equation (1):

$$\textbf{\textit{Extraction yield}}\ (\%) = \frac{\textbf{\textit{weight of extracted oil}}\ (\mathbf{g})}{\textbf{\textit{weight of sample used}}\ (\mathbf{g})} \times \mathbf{100} \quad (1)$$

Extraction efficiency was calculated relative to the total lipid content of the pecan kernels determined using the AOAC reference method (Section 2.2 and Table 1). The experimentally determined lipid content (73.90 g/100 g) was used to estimate the initial lipid mass present in the quantity of kernels subjected to each extraction.

$$\textbf{\textit{Extraction efficiency}}\ (\%) = \frac{\textbf{\textit{extraction yield}}\ (\%)}{\textbf{\textit{total lipid content}}\ (\%)} \times \mathbf{100} \quad (2)$$

Table 1. Water activity, proximate composition, and mineral profile of pecan nut.

Parameter	Value
Water activity [1]	0.5327 ± 0.002
Moisture content (g water/100 g sample)	4.31 ± 0.01
Crude protein (N × 6.25) (g/100 g w.b.)	10.25 ± 0.02
Fat (g/100 g w.b.)	73.90 ± 0.16
Crude Fiber (g/100 g w.b.)	2.50 ± 0.16
Ash (g/100 g w.b.)	1.20 ± 0.02
Total carbohydrates (by difference) (g/100 g w.b.)	10.34 ± 0.30
Minerals (mg/100 g d.m.)	
Copper	1.2 ± 0.23
Iron	2.47 ± 0.58
Calcium	43.0 ± 2.51
Magnesium	21.0 ± 2.28
Potassium	126.3 ± 0.26
Sodium	0.8 ± 0.48

[1] Dimensionless. Values are expressed as mean ± standard deviation (n = 3).

2.5. Fatty Acid Profile

The fatty acid profile of pecan oil was determined via gas chromatography. Fatty acid methyl esters (FAME) were prepared according to UNE-EN ISO 5509:2000 [15]. For this purpose, a 0.5 M NaOH solution was used during the saponification step, isooctane as the solvent, and a solution of boron trifluoride in methanol (BF_3–methanol) as the methylation agent. Chromatographic analysis was performed on a PerkinElmer gas chromatograph equipped with an FID detector and a 60 m long SGE BPX70 capillary column with an internal diameter of 0.25 mm and a film thickness of 0.25 µm. Helium was used as the carrier gas at a constant flow rate of 1.0 mL/min, and the detector temperature was maintained at 250 °C. Fatty acids were identified by comparing their retention times with those of a standard FAME mixture. The results were expressed as a relative percentage of the total identified fatty acids.

2.6. Physicochemical Characterization of the Oils

2.6.1. Refractive Index

The refractive index of the oils was determined in accordance with the AOCS Official Method Cc 7–25 [16], using a previously calibrated Atago benchtop refractometer (Atago, Model 1T, Tokyo, Japan). Before analysis, the refractometer was calibrated with distilled water, and the prism temperature was maintained at 25 ± 0.2 °C using a circulating thermostatic water bath. The oil samples were homogenized and equilibrated to the measurement temperature before analysis. A sufficient amount of oil was placed directly on the clean and dry prism surface to ensure complete coverage without air bubbles. After thermal equilibration, the refractive index was recorded at 25 °C. The prism was cleaned and dried between measurements. Three independent measurements were performed for each oil sample, and the results were expressed as the mean ± standard deviation.

2.6.2. Specific Gravity

The relative density of the oils was determined via pycnometry at 25 °C. First, the clean, dry pycnometer was weighed empty. It was then filled with distilled water previously equilibrated in a thermostatic water bath to 25 ± 0.2 °C, and its mass was recorded. The

procedure was repeated by replacing the water with the oil sample. The relative density was calculated using the following equation:

$$\textbf{Specific gravity} = \frac{\mathbf{w_2 - w_0}}{\mathbf{w_1 - w_0}} \quad (3)$$

where w_0 is the mass of the empty pycnometer, w_1 is the mass of the pycnometer filled with distilled water, and w_2 is the mass of the pycnometer filled with the oil sample.

2.6.3. Acid Value (AV)

Acidity was determined using the official AOCS method Cd 3d-63 [17], which measures the milligrams of potassium hydroxide (KOH) required to neutralize the free fatty acids in 1 g of oil. Between 2 and 5 g of oil were weighed and dissolved in a mixture of denatured ethanol and ethyl ether. The solution was titrated with 0.02 N ethanolic KOH, using phenolphthalein as an indicator. The acid value (AV) was expressed as mg of potassium hydroxide required to neutralize the fatty acids in 1 g of fatty matter and calculated using the following equation:

$$\boldsymbol{AV} = \frac{\boldsymbol{V \times N \times 56.1}}{\boldsymbol{m}} \quad (4)$$

where V is the volume of KOH solution used in the titration (mL), N is the normality of the KOH solution, and m is the mass of oil analyzed (g).

2.6.4. Estimation of Iodine Value (IV)

The iodine value was estimated based on the composition of unsaturated fatty acids, according to the equation proposed by Martínez and Maestri [18]. The percentages of oleic (C18:1), linoleic (C18:2), and linolenic (C18:3) acids obtained via GC-FID, as described in Section 2.5, were used in the following equation:

$$\boldsymbol{IV} = (\boldsymbol{\%\ oleic\ acid} \times \mathbf{0.899}) + (\boldsymbol{\%\ linoleic\ acid} \times \mathbf{1.814}) + (\boldsymbol{\%\ linolenic\ acid} \times \mathbf{2.737}) \quad (5)$$

where 0.899, 1.814, and 2.737 are the respective iodine contribution coefficients for oleic, linoleic, and linolenic acids, reflecting the number of carbon–carbon double bonds in each fatty acid. Fatty acid contents were expressed as relative percentages of the total identified fatty acids. The results were expressed as grams of iodine absorbed per 100 g of oil (g I_2/100 g).

2.6.5. Peroxide Value (PV)

The peroxide value was determined according to the official AOCS method Cd 8b-90 [19]. To carry out this, 2 to 5 g of oil were weighed into a 250-mL Erlenmeyer flask and dissolved in a mixture of glacial acetic acid and chloroform. Next, a saturated solution of KI was added, and the released I_2 was titrated with a standardized solution of sodium thiosulfate ($Na_2S_2O_3$), using a starch solution as an indicator. The endpoint was determined by the disappearance of the blue color. The results were expressed as milliequivalents of active oxygen per kilogram of oil (meq O_2/kg). Under the analytical conditions used, the detection limit was 0.5 meq O_2/kg oil; therefore, values below this limit were reported as not detected (n.d.).

2.7. Total Polyphenol Content

The total phenolic content (TPC) of the oils was determined via the Folin–Ciocalteu assay following Yang et al. [20], with minor modifications. Briefly, 250 mg of oil was weighed into a centrifuge tube, and 0.5 mL of Folin–Ciocalteu reagent (Sigma-Aldrich,

St. Louis, MO, USA) was added. The mixture was allowed to react for 5 min, after which 2 mL of methanol was added, and the mixture was vortexed to facilitate the transfer of reducing compounds from the lipid matrix to the methanol phase. After the addition of 1.5 mL of 15% (*w*/*v*) Na_2CO_3, the mixture was kept at room temperature for 10 min and vortexed again. Distilled water (7 mL) was subsequently incorporated, and the samples were maintained at 50 °C for 20 min. The mixtures were centrifuged at 5000 rpm for 10 min, and the recovered supernatant was filtered before measurement. Absorbance was read at 750 nm using a spectrophotometer (Spectronic Genesys 20, New York, NY, USA). TPC was calculated from a gallic acid calibration curve and expressed as micrograms of gallic acid equivalents per gram of oil (μg GAE/g oil). All determinations were performed in triplicate.

2.8. Antioxidant Activity by the ORAC Assay

The antioxidant activity of the oils was determined using the oxygen radical absorbance capacity (ORAC) assay, according to the method described by Marineli et al. [21], with modifications. The analysis was performed on a multilabel plate reader (Victor^3, PerkinElmer, Turku, Finland), using 96-well polystyrene microplates (OptiPlate™-96HB, PerkinElmer, Turku, Finland). To each well, 40 μL of the oil extract and 200 μL of a 100 μmol/L fluorescein solution were added. The mixture was incubated at 37 °C for 20 min. Subsequently, the reaction was initiated by adding 35 μL of a 0.36 mol/L solution of 2,2′-azobis(2-amidinopropane) dihydrochloride (AAPH, Sigma-Aldrich, St. Louis, MO, USA). Fluorescence was recorded kinetically using excitation and emission wavelengths of 485 and 535 nm, respectively, until the signal decreased by approximately 95% from the initial reading.

Antioxidant activity was calculated from the net area under the fluorescence decay curve (AUC), obtained by subtracting the area corresponding to the blank from the area corresponding to the sample. Trolox (Sigma-Aldrich, St. Louis, MO, USA), a water-soluble vitamin E analogue, was used as the antioxidant standard. The blank contained the same reagents and extraction solvent as the samples but without the oil extract. Quantification was performed using a Trolox calibration curve over a concentration range of 6.25 to 200 μmol/L. The results were expressed as micromoles of Trolox equivalent per 100 g of oil (μmol TE/100 g oil).

2.9. Schaal Test for Evaluating Oxidative Stability

The oxidative stability of the oils was evaluated using the Schaal test. For this purpose, oil samples without added antioxidants were kept at 60 °C in a natural air convection oven (Memmert, model UN110, Schwabach, Germany) for 12 days. Aliquots were collected at the start of the test and after 6 and 12 days of storage. At each time point, the peroxide value and total phenolic content were determined according to the methods described in Sections 2.6.5 and 2.7, respectively.

2.10. Statistical Analysis

The analyses were performed in triplicate, and the results are expressed as mean ± standard deviation. For variables determined immediately after extraction, differences between extraction methods were evaluated using a one-way analysis of variance (ANOVA). For the accelerated storage experiment, peroxide value and total phenolic content were analyzed separately using two-way ANOVA, considering extraction method and storage time (days 6 and 12) as fixed factors, including their interaction. When significant effects were detected ($p < 0.05$), means were compared using Tukey's multiple comparison test. Statistical processing was performed using Statgraphics Centurion XV software (version 19.5.01, StatPoint Technologies Inc., The Plains, VA, USA).

3. Results and Discussion

3.1. Proximate Composition, Water Activity, and Mineral Content of Pecans

Pecan kernels had a water activity of 0.5327 ± 0.002 and a moisture content of 4.31 ± 0.01 g/100 g, reflecting low water availability in the matrix. Lipids constituted the predominant fraction with 73.90 ± 0.16 g/100 g, followed by total carbohydrates, crude protein, and crude fibre, with contents of 10.34 ± 0.30, 10.25 ± 0.02, and 2.50 ± 0.16 g/100 g, respectively (Table 1). Meanwhile, the ash content was 1.20 ± 0.2 g/100 g. Taken together, these results confirm the high oil content of pecans and support their application as a raw material for oil production.

The determined nutritional composition was consistent with that previously described for various pecan cultivars. Venkatachalam et al. [22] reported moisture, protein, lipid, and ash contents ranging from 2.1–6.4%, 6.0–11.3%, 65.9–78.0%, and 1.2–1.8%, respectively. Similarly, Ferrari et al. [23], when evaluating 16 cultivars produced in Uruguay, recorded mean values of 3.99% moisture, 69.28% oil, 6.82% protein, and 1.71% ash.

The reported lipid content (73.8%) confirms that the analyzed sample corresponds to a fruit with a compositional profile typical of the range reported for the species. Ferrari et al. [23] reported lipid content values for varieties such as Apache and Pawnee, which reached 73.54% and 74.69%, respectively. Siebeneichler et al. [2,4] report fat contents of up to 78% depending on the cultivar.

Regarding the mineral profile (Table 1), potassium (126.3 ± 0.26 mg/100 g), calcium (43.0 ± 2.51 mg/100 g) and magnesium (21.0 ± 2.28 mg/100 g) were the most abundant elements, followed by iron (2.47 ± 0.58 mg/100 g) and copper (1.2 ± 0.23 mg/100 g), while sodium remained at low levels (0.8 ± 0.48 mg/100 g). These results demonstrate a contribution from macrominerals such as calcium and magnesium, along with trace elements such as iron, copper, and sodium. Siebeneichler et al. [2] studied 27 pecan cultivars grown in China and also reported that magnesium and calcium were the predominant elements, and that iron and copper were present at lower levels than those determined here. These differences are to be expected, considering that the mineral content of tree nuts is strongly influenced by the cultivar, the soil and climate conditions of the orchard, and agronomic management practices, rather than by a fixed pattern associated with the species.

3.2. Oil Extraction Yield

Figure 1 shows the results for oil extraction yield from pecans, which varied significantly depending on the extraction technique used ($p < 0.05$). Soxhlet extraction achieved the highest yield (69.21 ± 1.30%), followed by supercritical CO_2 extraction (52.89 ± 0.39%) and, finally, ultrasound-assisted extraction (48.68 ± 0.62%). Considering the total lipid content of the pecan kernels (73.90 g/100 g), the extraction efficiencies were 93.7% for Soxhlet extraction, 71.6% for sCO_2 extraction, and 65.9% for UAE. Thus, Soxhlet recovered most of the lipid fraction present in the kernels, whereas the two alternative extraction protocols achieved partial lipid recoveries under the operating conditions evaluated.

The higher yield obtained using the Soxhlet method can be attributed to the exhaustive nature of the process, since the solvent continuously recirculates over the sample for 6 h, repeatedly carrying away the lipid fraction until the matrix is virtually depleted. These conditions promote prolonged, virtually exhaustive extraction of compounds soluble in petroleum ether. Furthermore, the low polarity of this solvent facilitates the recovery of triacylglycerides and other nonpolar lipid constituents present in the pecan kernel. But high yield does not necessarily imply higher oil quality, since prolonged exposure to heat and the intensive use of organic solvents constitute significant limitations from an environmental standpoint and in terms of the preservation of sensitive compounds. This behavior has been described in various oil-bearing matrices and is consistent with

the findings of Kumar et al. [9] regarding the high lipid recovery capacity of the Soxhlet method compared to gentler methods, even though this entails longer processing times and greater thermal exposure.

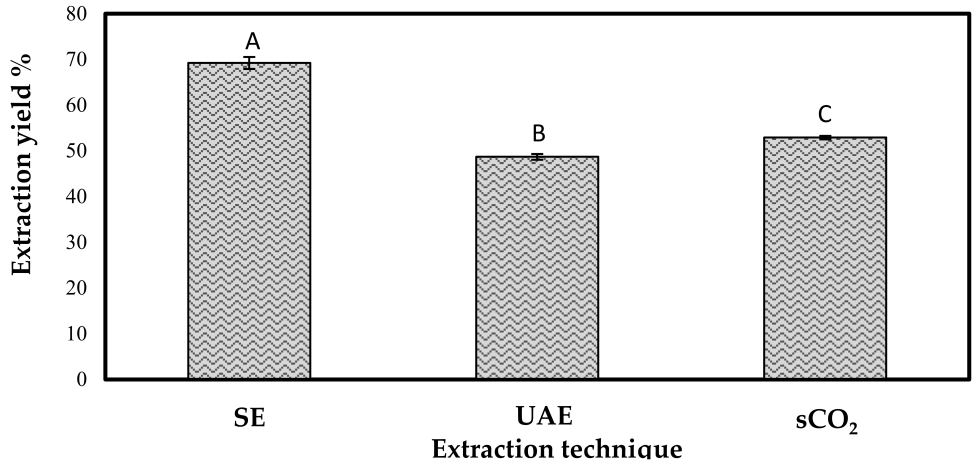

Figure 1. Extraction yield of pecan nut obtained using different extraction techniques: Soxhlet extraction (SE), ultrasound-assisted extraction (UAE), and supercritical carbon dioxide extraction (sCO_2). Different uppercase letters indicate significant differences among extraction techniques ($p < 0.05$). Values are expressed as mean ± standard deviation ($n = 3$).

Petroleum ether was selected because it is a conventional nonpolar solvent used for the gravimetric extraction of lipids, with a high affinity for triacylglycerols and a relatively low boiling range that facilitates its removal after extraction. Its use is consistent with standardized Soxhlet procedures and previous studies on pecan oil extraction [14,22]. Other nonpolar solvents were not evaluated because the objective of this study was to compare three complete extraction protocols rather than to perform solvent screening or optimization within the Soxhlet method.

The lower yield obtained from ultrasound-assisted extraction may reflect that, under the power and time conditions used in this study, the acoustic cavitation generated was not sufficient to extensively disrupt the cellular structure of the matrix; a factor that Salinas-Maldonado et al. [10] identified as a key determinant of this technique's efficiency when compared to more aggressive processes, since during sonication, the cavitation phenomenon can promote the disruption of cellular structures and enhance mass transfer between the matrix and the solvent. However, its efficiency depends on the solvent, the solid-to-liquid ratio, the ultrasonic intensity, and the procedure used to recover the oil from the liquid phase.

Supercritical CO_2 extraction produced an extraction yield of 52.89%, corresponding to the recovery of approximately 71.6% of the total lipid content of the kernels. This was significantly higher than that obtained via ultrasound-assisted extraction, although lower than that observed via the Soxhlet method ($p < 0.05$). The extractive capacity of supercritical CO_2 is related to its high affinity for nonpolar lipid compounds and to the operating conditions used. In this study, extraction was performed at 300 bar and 40 °C, with a 60 min static phase followed by 180 min of dynamic extraction. Under these conditions, the density and solvating power of CO_2 allowed for the recovery of a considerable proportion of the oil contained in the matrix, without the use of conventional organic solvents.

On the other hand, Salvador et al. [8] reported a yield of 58.4% when extracting residual oil from pecan cake at 300 bar and 40 °C, pressure and temperature conditions similar to those used in the present study. Similarly, Jamila dos Santos et al. [24] demonstrated that the recovery of oil from pecan cake using compressed CO_2 is strongly influenced by the

process pressure and temperature. However, these comparisons should be interpreted with caution, since both studies used cake derived from nut pressing, whereas the present study used the kernel directly.

Differences in initial composition, matrix structure, and available oil content can significantly affect extraction yield. The intermediate yield obtained with supercritical CO_2 is consistent with that reported by Salvador et al. [8], who achieved recoveries of over 98% of the oil present in pecan by-products using this technology, although in matrices with a larger contact surface area or under pressure and time conditions different from those applied here.

It should be noted that the extraction methods evaluated in this study involve different solvents: petroleum ether for Soxhlet extraction, ethanol for ultrasound-assisted extraction, and supercritical CO_2 for supercritical fluid extraction. Therefore, the effects of extraction technology and solvent polarity cannot be separated, and observed differences should be interpreted as the combined result of the extraction system used, rather than exclusively attributed to the extraction technology. However, this comparison reflects the performance of the complete extraction protocols under the operating conditions chosen for each method.

3.3. Fatty Acid Profile

The fatty acid profile of the oils obtained using the three extraction methods is presented in Table 2. Regardless of the technique used, the predominant fatty acids were oleic acid (C18:1) and linoleic acid (C18:2), followed by palmitic acid (C16:0), stearic acid (C18:0), and linolenic acid (C18:3). Collectively, oleic, linoleic, and linolenic acids accounted for between 76.8% and 78.3% of the total identified fatty acids, confirming the predominance of the unsaturated fraction in pecan oil. This predominance, with oleic acid as the major component, is consistent with the findings of Cui et al. [6], who report that monounsaturated fatty acids account for more than 60% of the total fatty acid content in pecan oil. Oleic acid, as the predominant fatty acid, was also reported by Wu et al. [25] in 27 pecan cultivars grown in China (62.50–73.97%). Similarly, Zhang et al. [7], evaluating ten grafted pecan varieties cultivated in southeastern China, reported oleic acid contents ranging from 66.85 to 76.33%, while linoleic acid ranged from 14.57 to 23.65%. Prado et al. [26] reported oleic acid contents ranging from 69.2 to 75.9% and linoleic acid contents from 16.7 to 23.1% in pecan oils obtained from different cultivars and harvest years. Jamila dos Santos et al. [24], evaluating pecan nut cake oils obtained using compressed fluids, reported oleic and linoleic acid contents of 58.81–64.70% and 28.31–31.20%, respectively. These values show a higher proportion of oleic acid and a lower proportion of linoleic acid than those observed in the present study. The lower oleic acid content cannot be attributed exclusively to the extraction method because similarly low values were obtained using Soxhlet, ultrasound-assisted, and supercritical CO_2 extraction. Instead, it may reflect the intrinsic composition of the raw material obtained from Los Tambos, Atacama Region, Chile. The fatty acid composition of pecans can vary among cultivars and production years because of genetic and seasonal influences [26,27]. Moreover, oleic and linoleic acids generally exhibit an inverse relationship during pecan kernel development, with oleic acid increasing and linoleic acid decreasing as maturation progresses. Consequently, differences in cultivar, environmental adaptation, kernel maturity, and harvest conditions may have contributed to the composition observed in the Chilean samples. Nevertheless, because the specific cultivar, maturity stage, and detailed agronomic history of the pecans were unavailable, the individual contribution of these factors cannot be established and should be considered a limitation of the study.

Table 2. Fatty acid profile of pecan oil obtained using different extraction techniques.

Fatty Acid (% of Total Fatty Acids)	Extraction Technique		
	SE	**UAE**	**sCO_2**
Saturated Fatty Acids			
Palmitic acid (C16:0)	14.0 ± 0.20 ^{A}	13.8 ± 0.20 ^{A}	13.6 ± 0.10 ^{A}
Stearic acid (C18:0)	8.1 ± 0.40 ^{A}	7.2 ± 0.30 ^{A}	7.3 ± 0.40 ^{A}
Unsaturated Fatty Acids			
Oleic acid (C18:1)	44.60 ± 0.10 ^{B}	44.50 ± 0.10 ^{B}	47.30 ± 0.20 ^{A}
Linoleic acid (C18:2)	29.1 ± 0.30 ^{A}	28.5 ± 0.30 ^{A}	27.8 ± 0.40 ^{A}
Linolenic acid (C18:3)	3.10 ± 0.10 ^{B}	4.20 ± 0.30 ^{A}	3.20 ± 0.30 ^{B}
Minor fatty acids (C20:0; C20:1; C22:0)	1.10 ± 0.10 ^{B}	1.80 ± 0.10 ^{A}	0.80 ± 0.10 ^{C}

Values are expressed as mean ± standard deviation and as a percentage of total fatty acids. Different superscript letters within the same row indicate significant differences among extraction techniques ($p < 0.05$). SE: Soxhlet extraction; UAE: ultrasound-assisted extraction; sCO_2: supercritical carbon dioxide extraction. Minor fatty acids include arachidic acid (C20:0), eicosenoic acid (C20:1), and behenic acid (C22:0), which together account for less than 2% of the total fatty acid profile and were reported as a combined value.

The relative content of oleic acid varied significantly depending on the extraction method ($p < 0.05$). The oil obtained using supercritical CO_2 had the highest proportion of this fatty acid, accounting for 47.30 ± 0.20% of total fatty acids. In contrast, the oils obtained via Soxhlet and ultrasound-assisted extraction had values of 44.60 ± 0.10% and 44.50 ± 0.10%, respectively, with no significant differences between the two treatments. Furthermore, the linoleic acid (C18:2) content ranged from 27.8 to 29.1%, with no significant differences attributable to the extraction method. Taken together, these results show that supercritical CO_2 extraction yielded an oil with a slightly higher relative proportion of oleic acid, while the relative abundance of linoleic acid remained virtually constant across the three methods evaluated. The higher proportion of oleic acid observed in the oil obtained with supercritical CO_2 could be related to the fluid's selectivity toward low-polarity lipid compounds. Under certain pressure and temperature conditions, supercritical CO_2 can modify the solubility and relative recovery of the various triglycerides present in the matrix. A similar behavior was described by Cui et al. [6] when comparing supercritical extraction with conventional methods in oil-bearing matrices of comparable composition.

Linolenic acid, on the other hand, was significantly higher in the oil obtained via ultrasonic extraction (4.20 ± 0.30%) compared to Soxhlet and sCO_2 (3.10% and 3.20%, respectively). Given its higher degree of unsaturation, linolenic acid is particularly susceptible to oxidative degradation. However, because fatty acid composition was expressed as a percentage of total identified fatty acids, the higher relative proportion observed in the UAE oil should not be interpreted as greater retention or recovery of this fatty acid. Rather, it may reflect differences in the relative distribution of fatty acids among the oils obtained via the different extraction methods. Palmitic acid (13.6–14.0%) and stearic acid (7.2–8.1%) showed no significant differences between the techniques, suggesting that the fraction of saturated fatty acids is not affected by the extraction method applied.

Overall, the predominance of oleic and linoleic acids confirms that the unsaturated fraction constitutes an important component of pecan oil. The relative proportions of monounsaturated and polyunsaturated fatty acids are particularly relevant from both nutritional and technological perspectives, since these influence oil quality and susceptibility to oxidation. Zhang et al. [7] highlighted the high proportion of unsaturated fatty acids, particularly oleic acid, as an important quality attribute of pecan oil and noted that oils with higher proportions of monounsaturated fatty acids may exhibit greater oxidative stability than those richer in polyunsaturated fatty acids. Similarly, Prado et al. [28] showed that the

oxidative behavior of pecan oil is associated not only with its fatty acid composition but also with endogenous antioxidants, particularly tocopherols and other minor components. Therefore, the oxidative stability of pecan oil cannot be predicted solely from its fatty acid profile.

The limited influence of extraction methods on the major fatty acids is also consistent with other studies on various nuts and oilseeds. Liu et al. [29] observed similar fatty acid compositions in pecan oils obtained via mechanical pressing and ultrasonic-assisted enzymatic aqueous extraction. Similarly, Gharibzahedi et al. [30] noted that the overall fatty acid composition of Persian walnut oil was not substantially altered by maceration, the modified Bligh–Dyer method, or cold pressing. Similarly, in baru almond oil, the profiles obtained using Soxhlet and supercritical CO_2 extraction showed similar proportions of the major fatty acids [31], while in pequi almond oil, the concentrations remained stable as supercritical extraction conditions were varied [32]. These findings indicate that extraction methods tend to have a greater influence on oil yield and minor components than on the distribution of its major fatty acids. In the present study, the small but significant differences detected for oleic and linolenic acids could be related to the selectivity of each extraction system and to the composition of the recovered fractions, without representing a general alteration of the characteristic lipid profile of pecan oil.

3.4. Physicochemical Properties of the Oil

The physicochemical properties of pecan oils extracted using the Soxhlet, ultrasonic, and supercritical CO_2 methods are presented in Table 3. The refractive index ranged from 1.464 to 1.471, with no significant differences between the extraction methods ($p > 0.05$). The oils obtained using the Soxhlet and supercritical CO_2 methods had values of 1.471 ± 0.001 and 1.470 ± 0.001, respectively, while the oil extracted using ultrasound had a value of 1.464 ± 0.012. The similarity among the treatments is consistent with the general fatty acid composition previously observed, as all three oils showed a predominance of oleic and linoleic acids and comparable levels of unsaturation. These results are similar to those reported by Liu et al. [29], who recorded a refractive index of 1.46 for both pecan oil obtained via mechanical pressing and that produced via ultrasonic-assisted enzymatic aqueous extraction. Furthermore, they found no significant differences in refractive index, iodine value, and specific gravity, which is consistent with a similar overall composition in terms of fatty acid chain length and degree of unsaturation.

Table 3. Physicochemical properties of pecan nut oil obtained using different extraction techniques.

Extraction Technique	Properties				
	Refractive Index (25 °C)	**Specific Gravity**	**Acid Value (mg KOH/g)**	**Iodine Value (g I_2/100 g)**	**Peroxide Value (mEq O_2/kg)**
SE	1.471 ± 0.001 ^{A}	0.912 ± 0.002 ^{A}	1.77 ± 0.095 ^{A}	101.31 ± 0.46 ^{A}	n.d.
UAE	1.464 ± 0.012 ^{A}	0.905 ± 0.002 ^{B}	1.07 ± 0.076 ^{B}	100.70 ± 0.67 ^{A}	n.d.
sCO_2	1.470 ± 0.001 ^{A}	0.907 ± 0.001 ^{B}	1.01 ± 0.146 ^{B}	101.26 ± 0.56 ^{A}	n.d.

Values are expressed as mean ± standard deviation ($n = 3$). Different superscript uppercase letters within the same column indicate significant differences among extraction techniques ($p < 0.05$). SE: Soxhlet extraction; UAE: ultrasound-assisted extraction; sCO_2: supercritical carbon dioxide extraction; n.d.: not detected (peroxide value < 0.5 meq O_2/kg oil).

The specific gravity ranged from 0.905 to 0.912 (Table 3). The oil obtained via Soxhlet extraction showed the highest value, at 0.912 ± 0.002, and was significantly different from the oils obtained via ultrasound and supercritical CO_2 extraction. Although the difference was significant, its magnitude was small, so it does not appear to represent a substantial change in the overall composition of the oils. These variations could be related to small

differences in the relative proportions of triacylglycerides and minor components recovered by each extraction method. Salvador et al. [8] reported similar values, ranging from 0.9121 to 0.9156, for commercial pecan oils and pecan waste oils.

Regarding the acid value, the oil extracted using the Soxhlet method had the highest value with 1.77 ± 0.10 mg KOH/g, and showed a significant difference compared to the oils extracted using ultrasound and supercritical CO_2, whose values were 1.07 ± 0.08 and 1.01 ± 0.15 mg KOH/g, respectively (Table 3). The acid value represents the amount of free fatty acids present in the oil and is used as an indicator of the hydrolysis of triacylglycerides and the initial quality of the lipid fraction [6]. The acid values determined in this study fell within the range reported by Salvador et al. [8], who obtained values of 0.93–2.18 mg KOH/g in various pecan oils.

The highest value observed in the oil obtained via the Soxhlet method could be related to the prolonged exposure of the matrix to heat during the 6 h extraction period, a condition that promotes the hydrolysis of the ester bonds in triacylglycerides through the action of heat [13], thereby releasing free fatty acids.

The iodine value ranged from 100.70 to 101.31 g I_2/100 g of oil, with no significant differences among the treatments ($p > 0.05$) (Table 3). This indicates that, although some extraction techniques altered the relative proportions of individual fatty acids, the overall degree of unsaturation of the oil remained stable, reflecting the robustness of the pecan's lipid profile under different processing conditions. The values obtained were slightly lower than those reported by Liu et al. [29], who recorded iodine values of 103.34 and 103.84 g I_2/100 g for pecan oils obtained via ultrasonic-assisted enzymatic aqueous extraction and mechanical pressing, respectively, with no significant differences between the methods.

Finally, the peroxide values of the three freshly extracted oils were below the analytical detection limit (<0.5 meq O_2/kg oil) and were therefore reported as not detected (n.d.) in Table 3. This result indicates that, under the analytical conditions used, the primary oxidation products were below the detection limit, demonstrating the samples' good initial oxidative status. This result is consistent with the findings of Cui et al. [6], who reported peroxide values well below the limit recommended by the Codex Alimentarius for edible vegetable oils (<10 meq O_2/kg). The absence of detectable peroxides at this stage also serves as the starting point (t = 0) for evaluating the oxidative evolution of the oils during accelerated storage, which is presented later in the Schaal test results (Figure 2). This finding suggests that the initial oxidative state does not depend exclusively on the extraction method, but also on the quality and conditioning of the raw material, exposure to oxygen, harvesting conditions, and the time elapsed until analysis.

3.5. Total Phenol Content and Antioxidant Activity

The total phenolic content (TPC) and the antioxidant activity (as determined via ORAC) of the pecan oils obtained using the three extraction methods are presented in Table 4. The oil obtained via ultrasonic extraction had the highest total phenolic content (32.55 ± 0.01 µg GAE/g), which was significantly higher ($p < 0.05$) than the values obtained via SE (21.22 ± 0.02 µg GAE/g) and sCO_2 (20.97 ± 0.01 µg GAE/g), which did not differ significantly from each other. The higher total phenolic content obtained in the oil extracted via ultrasound may be associated with the combined effect of acoustic cavitation and the polarity of the solvent used. The formation and collapse of cavitation bubbles generate shear forces, microcurrents, and turbulence, which promote the disruption of cell structures, increase solvent penetration, and reduce the diffusion distance of intracellular compounds [33]. Furthermore, the ethanol used in this procedure has a greater affinity for phenolic compounds of intermediate polarity than the petroleum ether used in the Soxhlet method or supercritical CO_2, which is predominantly nonpolar. Consequently,

the ultrasound–ethanol system likely facilitated a more efficient release and transfer of phenolic compounds from the pecan matrix into the oil extract.

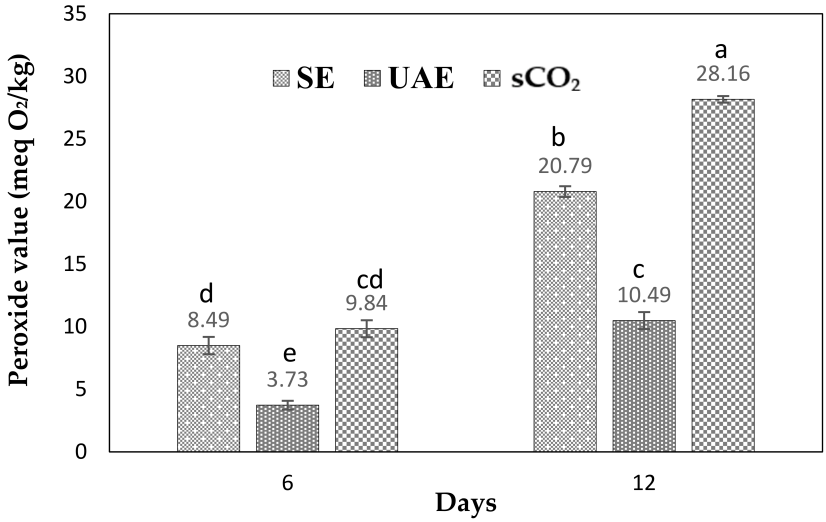

Figure 2. Peroxide value of pecan oils obtained using different extraction techniques after 6 and 12 days of accelerated storage at 60 °C. Values are expressed as mean ± standard deviation (n = 3). Different lowercase letters indicate significant differences among extraction method × storage time combinations according to Tukey's multiple comparison test ($p < 0.05$). SE: Soxhlet extraction; UAE: ultrasound-assisted extraction; sCO_2: supercritical carbon dioxide extraction.

Table 4. Total phenolic content and antioxidant activity of pecan nut oil obtained using different extraction techniques.

Extraction Technique	TPC	ORAC
	µg GAE/g Oil	µmol TE/100 g Oil
SE	21.22 ± 0.02 ^{B}	1610 ± 14.41 ^{C}
UAE	32.55 ± 0.01 ^{A}	1883 ± 10.81 ^{B}
sCO_2	20.97 ± 0.01 ^{B}	2681 ± 21.54 ^{A}

Values are expressed as mean ± standard deviation (n = 3). Different superscript letters within the same column indicate significant differences among extraction techniques ($p < 0.05$). SE: Soxhlet extraction; UAE: ultrasound-assisted extraction; sCO_2: supercritical carbon dioxide extraction; TPC: total phenolic content; ORAC: oxygen radical absorbance capacity; GAE: gallic acid equivalents; TE: Trolox equivalents.

Nevertheless, the Folin–Ciocalteu results should be interpreted considering the limitations of the method. This assay is based on the overall reducing capacity of the sample and is not specific to phenolic compounds, because other reducing substances present in the oil may also react with the reagent. Furthermore, in lipid matrices, the lipophilic nature of the medium, the extraction conditions, and the presence of matrix components may influence the analytical response of the Folin–Ciocalteu assay [34]. Because spike-recovery experiments and chromatographic validation of individual compounds were not performed in this study, the reported values should be regarded as operational estimates of Folin–Ciocalteu-reactive substances rather than as an exhaustive quantification of the phenolic compounds present in the oils.

In pecan matrices, Salvador et al. [8] demonstrated that the effect of ultrasound also depends on the solvent and the characteristics of the raw material. In one of the cake samples evaluated, the extracts obtained using ultrasound with ethanol or acetone had a higher phenolic content than their respective Soxhlet extracts; however, this trend was not observed in the other cake sample. In contrast, the extracts obtained using supercritical

fluids did not contain detectable amounts of phenolic compounds, which is consistent with the lower content observed in the present study for the oil obtained using sCO_2 and may be related to the low polarity of this fluid. Similarly, Liu et al. [29] directly determined the total phenol content in pecan oil. They observed significantly higher values in the oil obtained via ultrasound-assisted enzymatic aqueous extraction than in that obtained via mechanical pressing. The authors attributed this result to the combined effect of ultrasound and enzymatic hydrolysis, which facilitated the release of bioactive compounds present in the matrix. Taken together, these findings support the notion that ultrasound-assisted extraction can enhance the recovery of phenolic compounds from pecans. However, its efficiency depends on the extraction conditions, the solvent used, and the characteristics of the matrix.

Conversely, the lower phenolic content of the oil obtained via the Soxhlet method could be related to the low polarity of petroleum ether and to the prolonged exposure of the sample to high temperatures—conditions that may limit extraction or promote the degradation of certain thermolabile phenolic compounds. Similarly, the low phenolic content observed in the oil obtained using supercritical CO_2 can be explained by the low polarity of this fluid, which primarily promotes the solubilization of triglycerides and other lipophilic components. This selectivity of supercritical CO_2 toward low-polarity compounds has been noted by Cui et al. [6] when comparing conventional and unconventional techniques for extracting pecan oil.

The antioxidant activity determined via ORAC assay showed significant differences among the three extraction techniques ($p < 0.05$) (Table 4). However, unlike what was observed for total phenolic content, the oil obtained using supercritical CO_2 had the highest value, with 2681 $\pm$ 21.54 µmol TE/100 g of oil, followed by the oils obtained using ultrasound and the Soxhlet method, with 1883 $\pm$ 10.81 and 1610 $\pm$ 14.41 µmol TE/100 g of oil, respectively. The three treatments were significantly different from one another.

The absence of a direct relationship between total phenol content and antioxidant activity indicates that the antioxidant activity of pecan oil was not determined exclusively by the total amount of phenolic compounds. Extraction with supercritical CO_2 may have favored the recovery or preservation of lipophilic antioxidant compounds, potentially including tocopherols, phytosterols, squalene, or other minor components with a high capacity to neutralize peroxyl radicals. Furthermore, the Folin–Ciocalteu method quantifies substances with reducing capacity overall but does not provide information on the identity or individual antioxidant activity of the compounds present. Therefore, differences in antioxidant activity among pecan oils may reflect differences in the composition and activity of individual antioxidant compounds and/or potential synergistic interactions among them. In this regard, Cordero-Clavijo et al. [35] reported that supercritical CO_2 extraction more effectively preserves other bioactive compounds, such as tocopherols, sterols, and terpenoids, compared to conventional extraction methods; these compounds could contribute significantly to the oil's total antioxidant activity without necessarily being reflected in the Folin-Ciocalteu assay. The presence of tocopherols in pecan lipids may be relevant to the ORAC response observed in the present study. Robbins et al. [36] identified γ-tocopherol as the predominant tocopherol homologue in pecan kernels and noted that these lipid-soluble compounds contribute to the lipophilic antioxidant activity of pecans. Therefore, differences in the recovery or preservation of tocopherols among extraction methods could potentially contribute to the differences in ORAC values observed in the present study, including the higher value obtained for the oil extracted using supercritical CO_2.

3.6. Oxidative Stability During Accelerated Storage

The evolution of the peroxide value of pecan oils during accelerated storage at 60 °C is shown in Figure 2. On day 0, no peroxides were detected in any of the oils. Two-way ANOVA, performed using the results obtained on days 6 and 12, revealed significant effects of extraction method, storage time, and their interaction on the peroxide value ($p < 0.001$). The significant extraction method × storage time interaction indicated that the increase in peroxide value during accelerated storage depended on the extraction protocol.

After 6 days, primary oxidation products were detected in all three oils. The oil obtained using supercritical CO_2 showed a peroxide value of 9.84 ± 0.68 meq O_2/kg, followed by the Soxhlet-extracted oil at 8.49 ± 0.69 meq O_2/kg, with no significant difference between these two treatments ($p > 0.05$). In contrast, the UAE oil exhibited the lowest peroxide value, at 3.73 ± 0.35 meq O_2/kg, and differed significantly from both SE and sCO_2 oils ($p < 0.05$).

After 12 days of storage, the peroxide value increased significantly in all oils. The oil obtained using supercritical CO_2 reached the highest value, at 28.16 ± 0.26 meq O_2/kg, followed by the Soxhlet oil, at 20.79 ± 0.43 meq O_2/kg, and the UAE oil, at 10.49 ± 0.68 meq O_2/kg. At this storage time, the three oils differed significantly from one another ($p < 0.05$). These results indicate that peroxide formation during storage was influenced by the extraction protocol. The UAE oil consistently showed the lowest hydroperoxide formation and, therefore, greater resistance to primary oxidation than the oils obtained using Soxhlet and supercritical CO_2 extraction.

The lower oxidative stability observed in the oil obtained with sCO_2 is consistent with the findings reported by Cui et al. [6] for pecan oil, who found that extracts obtained using supercritical fluids exhibited significantly lower oxidative stability than those obtained using conventional methods. This finding is also consistent with the study of Cordero-Clavijo et al. [35] on sacha inchi (*Plukenetia volubilis*) oil, who reported that the oxidative stability index of oils extracted with supercritical fluids was notably lower than that of oils obtained using subcritical fluids and n-hexane. The authors attribute this behavior to the nonpolar nature of compressed CO_2, which makes it less efficient at extracting antioxidants with higher polarity, such as phospholipids and phenolic compounds. A similar trend could explain the behavior observed in the present study, where the oil obtained using sCO_2 exhibited lower resistance to oxidation than that obtained using UAE, despite both methods being considered non-conventional extraction technologies. This behavior is particularly relevant given that the oil obtained with supercritical CO_2 initially exhibited the highest antioxidant activity as determined via the ORAC assay (Table 4); however, this higher initial antioxidant activity did not translate into greater stability against oxidation during storage, as this oil showed the greatest accumulation of peroxides by day 12. This suggests that the antioxidant activity detected via ORAC could be primarily associated with nonpolar lipophilic compounds, distinct from the phenols responsible for the retention observed in the oil obtained via UAE, and consistent with the characteristic nonpolar selectivity of supercritical CO_2 [6,35]. This apparent discrepancy highlights that antioxidant activity measured via ORAC cannot be directly extrapolated to the oxidative stability of a lipid matrix. Oxidative stability depends not only on the presence of antioxidant compounds but also on factors such as fatty acid composition, the concentration and distribution of endogenous antioxidants and pro-oxidants, and their interactions within the oil matrix.

The total phenol content also decreased significantly during storage (Figure 3). Two-way ANOVA revealed significant effects of extraction method, storage time, and their interaction on total phenolic content ($p < 0.05$). The significant extraction method × storage time interaction indicated that the magnitude of the decrease during storage depended on the extraction protocol.

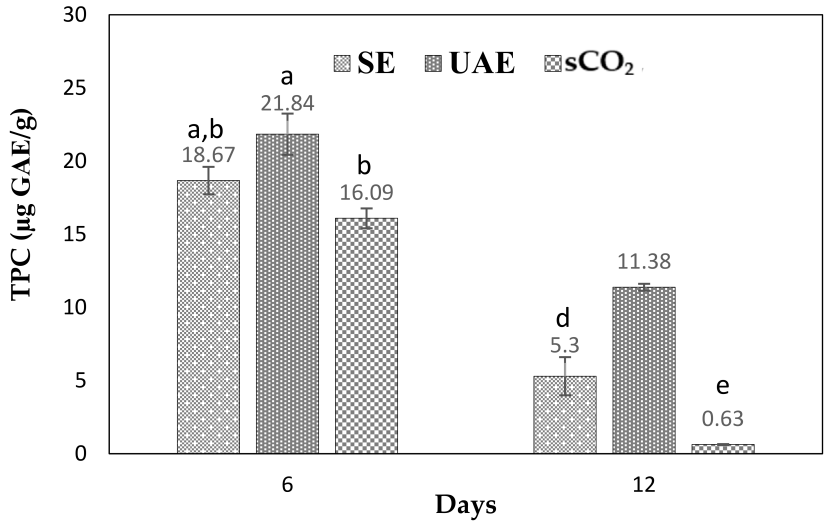

Figure 3. Total phenolic content (TPC) of pecan oils obtained using different extraction techniques after 6 and 12 days of accelerated storage at 60 °C. Values are expressed as mean ± standard deviation (n = 3). Different lowercase letters indicate significant differences among extraction method × storage time combinations according to Tukey's multiple comparison test ($p < 0.05$). SE: Soxhlet extraction; UAE: ultrasound-assisted extraction; sCO_2: supercritical carbon dioxide extraction; GAE: gallic acid equivalents.

After 6 days, the oil obtained using ultrasound had the highest content, of 21.84 ± 1.41 µg GAE/g, while the oil obtained using supercritical CO_2 had the lowest value, of 16.09 ± 0.68 µg GAE/g. The Soxhlet oil had an intermediate content of 18.67 ± 0.94 µg GAE/g and did not differ significantly from the other two treatments (Figure 3). After 12 days of storage, the differences between the extraction methods were more pronounced. The oil obtained using ultrasound retained the highest total phenol content, of 11.38 ± 0.24 µg GAE/g, followed by the Soxhlet oil, of 5.30 ± 1.3 µg GAE/g, and the oil obtained using supercritical CO_2, with only 0.63 ± 0.03 µg GAE/g. During this period, the three treatments differed significantly from one another ($p < 0.05$). Compared to the initial values, the oil obtained using ultrasound retained approximately 35% of its phenolic content after 12 days, while the Soxhlet oil retained about 25%.

In contrast, the oil obtained using supercritical CO_2 retained only about 3% of its initial content. The higher retention of phenols in the ultrasonically extracted oil coincided with its lower peroxide value, while a higher accumulation of primary oxidation products accompanied the marked loss of these compounds in the supercritical CO_2-extracted oil. Taken together, these results suggest that the retention of phenolic compounds may have contributed to the oxidative stability of pecan oil during accelerated storage, although this response may also be influenced by other minor antioxidants and by the specific composition of each extracted fraction, as has been described for other oils obtained using supercritical fluids [6,35].

The higher resistance to oxidation shown by the oil obtained via UAE is consistent with the milder conditions characteristic of this method, namely, lower temperatures and shorter extraction times, which favor the preservation of heat-labile bioactive compounds such as phenolic compounds. This is consistent with the findings of García et al. [3], who note that ultrasound-assisted extraction operates at lower temperatures and for shorter durations than conventional methods, thereby preserving the integrity of heat-sensitive compounds.

The increase in the peroxide value observed during accelerated storage is consistent with the behavior described for other nut oils evaluated using the Schaal test. Xie et al. [37] observed a progressive formation of primary oxidation products in walnut oil stored at 60 °C and demonstrated that the presence of lycopene helped limit the increase in the

peroxide value and better preserve the oil's phenolic content and reducing capacity. Similarly, Li et al. [38] recorded a progressive accumulation of peroxides during the accelerated oxidation of hickory nut oil, with a rate of deterioration accelerating as the storage period progressed. These results support the use of the peroxide value as an indicator of the formation of primary oxidation products during the Schaal test.

The differing stability of the oils may also be related to their content and composition of endogenous antioxidants. Ma et al. [39], when evaluating ten vegetable oils using the Schaal test, noted that the interaction between fatty acid composition and minor components such as tocopherols, phenolic compounds, sterols, and squalene determined oxidative stability at moderate temperatures. In the present study, the higher preservation of total phenols in the oil obtained via ultrasound coincided with a lower accumulation of peroxides, while the marked decrease in these compounds in the oil extracted with supercritical CO_2 was accompanied by a higher formation of primary oxidation products.

4. Conclusions

The results of this study demonstrated that the extraction method significantly influenced both the yield and certain physicochemical characteristics, such as the content of bioactive compounds, and the oxidative stability of pecan oil. Soxhlet extraction yielded the highest oil yield (69.21%), although it also resulted in the highest acid value. In contrast, ultrasound-assisted extraction and supercritical CO_2 extraction yielded oils with lower acidity values. Overall, the fatty acid profile remained relatively stable across the methods, with a predominance of oleic and linoleic acids, although the oil obtained via supercritical CO_2 extraction had a slightly higher proportion of oleic acid.

Ultrasound-assisted extraction yielded the oil with the highest initial content of phenolic compounds, while the oil obtained with supercritical CO_2 exhibited the highest antioxidant activity as determined via ORAC assay. However, this higher initial antioxidant activity did not translate into greater stability during accelerated storage. After 12 days at 60 °C, the oil obtained via ultrasound showed the lowest formation of peroxides and the highest retention of phenolic compounds, while the oil extracted with supercritical CO_2 showed the highest accumulation of primary oxidation products and a marked decrease in its phenolic content.

Taken together, these results show that no single extraction method was superior across all the evaluated parameters, highlighting a trade-off between yield, recovery of bioactive compounds, and oxidative stability. Under the conditions studied, ultrasound-assisted extraction showed particular advantages when preservation of phenolic compounds and oxidative stability were prioritized. For its part, supercritical CO_2 extraction remains an attractive alternative due to its lack of conventional organic solvents and the high initial antioxidant activity of the resulting oil, although its operating conditions should be optimized to improve stability during storage. Future studies should complement these results by identifying and quantifying lipophilic antioxidants, particularly tocopherols, phytosterols, and squalene, to better understand their contribution to the oil's stability. These findings provide useful criteria for selecting the extraction method based on the prioritized quality attribute, yield, phytochemical profile, or oxidative stability, in the value-added processing of pecan oil.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

RNS	Reactive nitrogen species
AAS	Atomic absorption spectrophotometry
SFE	Supercritical fluid extraction
AAPH	2,2′-Azobis(2-amidinopropane) dihydrochloride
FDA	Food and Drug Administration
SE	Soxhlet extraction
UAE	Ultrasound-assisted extraction
sCO_2	Supercritical carbon dioxide extraction
AOAC	Association of Official Analytical Chemists
AOCS	American Oil Chemists' Society
FAME	Fatty acid methyl esters
FID	Flame ionization detector
ISO	International Organization for Standardization
GC	Gas chromatography
TPC	Total phenolic content
LDL-C	Low-density lipoprotein cholesterol
UV–Vis	Ultraviolet–visible spectroscopy
ORAC	Oxygen radical absorbance capacity
GAE	Gallic acid equivalents
TE	Trolox equivalents
KOH	Potassium hydroxide
ANOVA	Analysis of variance

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