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Application of Pulsed Electric Fields PEF on Pecan Nuts *Carya illinoinensis* Wangenh. K. Koch: Oil Extraction Yield and Compositional Characteristics of the Oil and Its By-product

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Abstract
Pecan nut oil is conventionally obtained by mechanical extraction characterized by a low oil extraction yield (OEY) compared to solvent extraction. Pulsed electric fields (PEF) have been employed as a pretreatment to enhance OEY from several oilseeds, but no studies have been found regarding tree nut oil. Hence, PEF was applied at different specific energy inputs (0.5–17.6 kJ kg^{-1}) to evaluate its impact on OEY, oil acidity, and antioxidant capacity (AC), along with total phenolics (TP), condensed tannins (CT), and AC of the by-product generated from oil extraction. Kernels treated by PEF were compared against untreated and soaked kernels due to sample water immersion during PEF processing. The water immersion reduced the initial oil content of soaked and PEF-treated kernels (7.3–11.7%), transferring between 3.8 ± 0.0 and 6.2 ± 0.1 g of oil into the soaking water (o_{SW}). OEY_{TOTAL} of soaked and PEF-treated samples was calculated considering o_{SW}. The application of 0.5 kJ kg^{-1} increased OEY_{TOTAL} by 21.4 and 17.6% compared to untreated and soaked kernels, respectively, while oil acidity and AC of PEF-treated kernels were within values reported for pecan nut oil. The highest concentration of TP and CT in the by-product was achieved at 0.8 kJ kg^{-1}, increasing 9.5 and 30.1%, respectively, compared to untreated kernels. Results evidenced that PEF processing might be a suitable technology to increase OEY from pecan nuts, but the oil extracted during kernels water immersion must be recovered. Furthermore, the by-product of PEF-treated kernels displayed an enhanced content of phenolic compounds increasing its potential as food ingredient.

Keywords Pecan nuts · Pulsed electric fields · Oil extraction yield · Antioxidant capacity · Phenolic compounds

Introduction

The pecan nut [*Carya illinoinensis* (Wangenh. K. Koch)] is among the most commonly consumed tree nuts worldwide [24]. At being native from North America, pecan nuts are considered an economically important nut crop to Mexico and the USA, being Mexico responsible for almost 50% of pecan nuts worldwide production [16, 24].

Pecan nuts intake has been associated with positive effects on human health due to their significant concentration of phenolic compounds along with mono- and polyunsaturated fatty acids [2, 6]. Kernels phenolic compounds profile is mostly composed by condensed tannins which have been related to pecan nuts antioxidant capacity [29, 39, 41], while kernels fatty acids profile includes oleic, linoleic, and α-linolenic acids [6, 39]. In comparison to olives, pecan nuts contain a higher concentration of polyunsaturated fatty acids and a lower concentration of saturated fatty acids [51]. Phytosterols and tocopherols have also been identified as minor components of pecan nuts. As a result, pecan nut oil has been recognized as a specialty oil increasing its commercial value [3, 10, 23].

Pecan nut oil is commonly extracted by mechanical processes to preserve its compositional characteristics. In these processes, kernels are pressed using a screw press or

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expeller with the advantages of low cost and simple use [9, 10]. Furthermore, a by-product, usually named cake, rich in carbohydrates, proteins, dietary fiber, and phenolic compounds is obtained from the oil extraction [2, 42]. The cake has been suggested as an ingredient in bakery products with the potential to enhance products' functional properties due to its water and oil absorption capacities along with its phenolic compounds concentration [29, 31]. Nevertheless, the main drawback of oil mechanical extraction from pecan nuts is its low oil extraction yield (OEY) compared to solvent extraction (< 60.0%) [10, 11]. In order to increase OEY, drying or enzymatic processes have been applied as pretreatments to oilseeds, modifying the phytochemical profile of the extracted oil and negatively affecting its physicochemical properties [9, 27]. As an alternative to improve extraction processes and maintain oil quality, food processing technologies such as ultrasonics, high voltage electrical discharges, and pulsed electric fields (PEF) have been applied as assisting processes [25, 37].

PEF is a nonthermal technology consisting in the application of high-voltage pulses (1–80 kV cm^{-1}) from μs to ms duration [26, 32]. Its mechanism is based on the cell membrane disruption caused by the increment in the cell membrane conductance leading to pore formation [7]. The cell membrane disruption might occur as a reversible or irreversible process depending on the electrical conditions. In a reversible disruption, the cell membrane closes pores by phospholipids and proteins rearrangement. In an irreversible disruption, the cell membrane is not able to close pores, causing the loss of cell integrity [19]. Recently, PEF is being applied as a pretreatment to induce the secondary metabolism in fruit and vegetables as well as to enhance different industrial processes such as drying, freezing, and frying. For instance, López-Gámez et al. [28] and González-Casado et al. [15] reported an increment in the concentration of carotenoids in carrots and tomatoes treated by PEF after 24 h of storage. Whereas Traffano-Schiffo et al. [49] observed that PEF increased the dehydration rate of kiwifruit by electrolytes' loss, and Tylewicz et al. [50] reported an improvement in the storage stability of freeze-dried apples pretreated by PEF.

Furthermore, the application of PEF to improve mechanical extraction processes of juices, oils, and other products have been reported to enhance the content of the bioactive compounds and preserve the sensory characteristics of extracted products [12, 46, 48]. Veneziani et al. [52] reported an increment between 2.3 and 6.0% in OEY from olives treated by PEF, producing an oil with a higher concentration of phenolic compounds and no significant changes in its sensory properties. Han et al. [19] used PEF as a pretreatment to oil extraction from microalgae *Chlorella pyrenoidosa*, reporting an increase of 12.0% in OEY compared to an ultrasound pretreatment. Sarkis et al. [43] reported a higher OEY in sesame seeds treated by PEF in comparison with untreated seeds. In another research, Abenoza et al. [1] evaluated the application of PEF to increase OEY from olive paste reporting an improvement of 13.9%. Guderjan et al. [17] observed an increment of 39.1% in OEY from rapeseeds treated by PEF. Concerning research relating tree nuts and PEF, Manzoor et al. [30] combined PEF and ultrasound technologies to improve phenolic compounds extraction from defatted almonds, increasing their extraction and antioxidant capacity by 33.3 and 41.7%, respectively. However, no studies concerning the impact of PEF technology on OEY and tree nuts oil composition have been found. Therefore, the objective of this work was to apply PEF as a pretreatment to improve oil extraction from pecan nut kernels evaluating the effect of the specific energy input (*W*) on OEY, oil characteristics (acidity and antioxidant capacity), and cake phenolic compounds (total phenolics, condensed tannins, and antioxidant capacity).

Materials and Methods

Chemicals

Acetone, ethyl acetate, hexane, methanol (MeOH), 2,2-diphenyl-1-picrylhydrazyl (DPPH), 6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (trolox), catechin, Folin-Ciocalteu reagent, gallic acid, hydrochloric acid (HCl), potassium hydroxide (KOH) solution (0.1 M), sodium carbonate (Na_2CO_3), acetic acid (CH_3COOH), and vanillin were purchased from Sigma-Aldrich (USA). Solutions were protected from light and stored at 4 °C.

Pecan Nuts

Fresh pecan nuts (*Carya illinoinensis*, Western variety) were harvested in autumn 2018 directly from the orchard [Sonora, Mexico (27° 29′ 38″ N, 109° 56′ 20″ W)]. In-shell nuts were vacuum-packaged (EVD 4, TORREY, Mexico) and immediately delivered by air to the University of Lleida in Spain where experiments were conducted. In the laboratory, in-shell nuts were shelled, placed in polyethylene bags (Cryovac Europe, Spain), vacuum-sealed (Egarvac® Basic 9, Egarvac S. C. P., Spain), and stored at 4 °C until experiments. Kernel halves were manually sectioned in half and divided into three different groups: (i) a reference that consisted of kernels without soaking nor PEF treatment, (ii) a control of kernels soaked in tap water (1:3 w/w) for 20 min, and (iii) pecan nuts treated by PEF. Experiments were performed at room temperature, and the conductivity of tap water was 463 μS cm^{-1}. Among PEF treatments, the temperature of the soaking water was randomly measured, not being higher than 30 °C. Control

and PEF-treated kernels were drained for 10 min; then, samples were taken to moisture determination. Before oil extraction, reference, control, and PEF-treated kernels were frozen at − 16 °C for 24 h and freeze-dried (− 50 °C, 1 mbar) for 72 h (Cryodos 50, Telstar Cryodos, Spain).

Pulsed Electric Field Application

PEF treatments were conducted in a batch equipment with a 0.1-μF capacitor (Physics International, USA) delivering monopolar exponential-wave pulses (pulse width, 4 μs) using a TG-70 gas control unit and a pulse generator (PT-55, Pacific Atlantic Electronics Inc., USA). Kernel halves were manually sectioned in half, immersed in tap water with a conductivity of 463 μS cm^{-1} (1:3 w/w), and placed in parallelepiped methacrylate containers (20 × 8 cm) equipped with stainless steel parallel electrodes. Treatments were performed at different electric field strengths (E, 2.0, 5.0, 7.5 kV cm^{-1}) and pulse number (n, 10, 55, 100) (Table 1). The specific energy input (W), expressed as kJ per kg of kernels in wet basis (kJ kg^{-1} wb), was calculated according to Eqs. 1 and 2:

$$V = E \times d \tag{1}$$

$$W = \frac{V^2 \bullet C \bullet n}{2 \bullet m} \tag{2}$$

where V is the input voltage (kJ $Coulomb^{-1}$), E is the electric field strength (kV cm^{-1}), d is the distance between electrodes (cm), C is the capacitance of the energy storage capacitor ($Coulomb^2$ kJ^{-1}), n is the number of pulses, and m is the initial mass of kernels (kg wb).

Table 1 Electric field strength (E), pulse number (n), and specific energy input (W) employed to investigate PEF effect on oil extraction from pecan nut kernels

E (kV cm^{-1})	n	W (kJ kg^{-1})
2.0	10	0.5
5.0	10	0.8
7.5	10	1.8
2.0	55	2.8
5.0	55	4.3
2.0	100	5.0
5.0	100	7.8
7.5	55	9.7
7.5	100	17.6

Mechanical Extraction of Pecan Nut Oil

Freeze-dried reference, control, and PEF-treated kernels (85.0 g) were placed in an expeller type screw press (YD-ZY-02A, Yoda Europe, China) for oil mechanical extraction. All samples were submitted to the same preset conditions while kernels feeding along with oil and cake recovery were standardized to prevent oil and cake loss. The extracted oil was stored at − 40 °C in 50-mL centrifuge tubes avoiding oil oxidation by flushing N_2 in the head-space. The tubes were sealed with parafilm until analyses. The cakes generated from the oil extraction were placed in 12 × 15 cm polyethylene bags, vacuum-sealed, and stored at − 40 °C.

Oil Extraction Yield

The oil extraction yield (OEY, %) of reference, control, and PEF-treated kernels was calculated as follows:

$$OEY = \frac{(m_K \times L_K) - (m_C \times L_C)}{(m_K \times L_K)} \times 100 \tag{3}$$

where m_K and m_C are the mass (g) of freeze-dried kernels and cakes, respectively, while L_K and L_C are the oil content expressed as g of oil per 100 g of freeze-dried kernels and cakes, respectively, all in dry basis (g 100 g^{-1} db).

The oil extracted into the soaking water (o_{SW}) was calculated to determine the total OEY (OEY_{TOTAL}) of control and PEF-treated kernels using Eqs. 4 and 5:

$$o_{SW} = \left(m_K \times L_{Reference}\right) - (m_K \times L_K) \tag{4}$$

where o_{SW} is the mass (g) of oil retained into the soaking water and $L_{Reference}$ is the oil content of reference kernels expressed as g·100 g^{-1} db.

$$OEY_{TOTAL} = \frac{[(m_K \times L_K) - (m_C \times L_C)] + o_{SW}}{(m_K \times L_K)} \times 100 \tag{5}$$

Moisture

The AOAC 920.151 method was employed to moisture determination of reference, control, and PEF-treated kernels [5]. Results were expressed as g·100 g^{-1} db.

Oil content

Oil content of freeze-dried kernels and cakes were determined by solvent extraction as reported by Villarreal-Lozoya et al. [53] with modifications. Freeze-dried kernels were ground in a laboratory mortar (2.5 g) while cakes were directly weighed (2.5 g). Samples were mixed with hexane

(1:10 w/v) for 1.5 min at 6000 rpm (IKA® T25 Ultra-turrax, IKA, Germany) then centrifuged (8500 rpm, 15 min, 20 °C) (Beckman Avanti™ J-25, Beckman Instruments Inc., USA) and supernatants collected. This procedure was repeated three times. Pooled supernatants were concentrated using a rotary evaporator (25 rpm, 45 °C) (BÜCHI Rotavapor R-3000, BÜCHI Labortechnik AG, Spain), and the extracted oil was used to determine oil content gravimetrically based on the AOAC 960.39 procedure [5]. Oil content of freeze-dried kernels and cakes was expressed as g 100 g^{-1} db.

Oil Analysis

Acidity

Oil acidity was determined following the AOAC 940.28 method [5], and results were expressed as mg KOH per 100 g of pecan nut oil (mg KOH·100 g^{-1}).

Antioxidant Capacity

Antioxidant capacity (AC) was evaluated using the DPPH radical scavenging capacity method reported by Gao et al. [14] with modifications. A DPPH solution was prepared by dissolving 0.05 g of DPPH in 250 mL of MeOH. Pecan nut oil (200 µL) diluted in ethyl acetate (2 mL) was mixed with the DPPH solution (2 mL). The reaction was left 15 min in darkness and absorbance measured at 515 nm using a UV-VIS spectrophotometer (Cecil CE 1010, Cecil Instruments Ltd., England). Trolox was used for the standard curve (0.003–0.030 mg mL^{-1}) to express results as mg trolox equivalents per 100 g of pecan nut oil (mg trolox EQ·100 g^{-1}).

Cake Analysis

A defatted cake was obtained after oil content determination by allowing to evaporate overnight the remaining hexane. Defatted cakes were sieved, placed in 6 × 15 cm polyethylene bags, vacuum sealed, and stored at − 40 °C. Aqueous and methanolic extractions were performed as described by Rábago-Panduro et al. [39].

In the aqueous extraction, defatted cake samples (0.3 g) were mixed with an extraction solution consisting on acetone:H_2O:CH_3COOH (70:29.5:0.5) in 1:10 w/v proportion. The mixture was sonicated in an ultrasonic bath for 30 min, centrifuged (8500 rpm, 15 min, 20 °C), and supernatants collected. The extraction process was performed twice. Next, the extraction solution was evaporated using N_2 and the concentrated was diluted to 5 mL with distilled water. Aqueous extracts were stored in 15-mL centrifuge tubes at 4 °C until total phenolics (TP) and AC analysis. The methanolic extraction was performed by mixing defatted cakes (0.1 g) with a 1% MeOH:HCl solution (1:30 w/v). The mixture was placed in a water bath (20 min, 30 °C). After this time, supernatants were collected by centrifugation (8,500 rpm, 15 min, 20 °C) and diluted to 5 mL with 1% MeOH:HCl solution. Methanolic extracts were stored in 15-mL centrifuge tubes at 4 °C until condensed tannins (CT) analysis.

Total Phenolics

Folin-Ciocalteu method reported by Singleton and Rossi [47] and adapted by Villarreal-Lozoya et al. [53] was followed to TP determination. Aqueous extracts (13 µL) were pipetted into a 96-well flat-bottom plate (Costar® Assay Plate #9017, Corning, USA) followed by Folin-Ciocalteu solution (221 µL) and led to react for 3 min in the dark. Next, 0.50 M Na_2CO_3 solution (26 µL) was added and the plate was incubated for 2.5 h in darkness. A microplate reader (Multiskan™ GO, Thermo Scientific™, Finland) was used to absorbance measurement at 765 nm employing a curve of gallic acid (0.1–1.0 mg mL^{-1}) as standard. Results were expressed as mmol gallic acid equivalents per 100 g of defatted cake db (mmol gallic acid EQ·100 g^{-1} db).

Antioxidant Capacity

DPPH radical scavenging capacity was employed to evaluate cakes antioxidant capacity [53]. A DPPH stock solution (1.3 mM) was diluted (1.5:10 v/v) in MeOH. Aqueous extracts (26 µL) were loaded into a 96-well flat bottom plate (Costar® Assay Plate #9017, Corning, USA) along with 234 µL of diluted DPPH. Absorbance measurements were made in the microplate reader at 515 nm and registered every minute until 15 min of reaction. Trolox was used for the standard curve (0.02–0.10 mg mL^{-1}) to express the results as mmol trolox equivalents per 100 g of defatted cake db (mmol trolox EQ·100 g^{-1} db).

Condensed Tannins

The HCl-vanillin method reported by Price et al. [36] and modified by Herald et al. [22] was employed to analyze CT. From a vanillin stock solution (0.065 M), a dilution (1:1 v/v) was made with 8% MeOH:HCl. Methanolic extracts (30 µL) were pipetted in a 96-well flat-bottom plate (Costar® Assay Plate #9017, Corning, USA) followed by the vanillin dilution (150 µL) and led to react for 20 min at 30 °C. The blank was 1% MeOH:HCl and absorbance was measured at 500 nm using the microplate reader. A curve of catechin (1.0–3.5 mg mL^{-1}) was utilized as standard and results expressed as mmol catechin equivalents per 100 g of defatted cake db (mmol catechin EQ·100 g^{-1} db).

Soaking Water Analysis

Given the low water solubility of condensed tannins, TP was selected to follow the release of the water-soluble phenolic compounds present in pecan nuts into the soaking water [21]. Aliquots of the soaking water (500 µL) were centrifuged (8000 rpm, 15 min, 20 °C) (Hettich® Universal 320R, Hettich, Germany), and supernatants were employed to measure TP as described in the "Total Phenolics" section. Results were expressed as mmol gallic acid equivalents per 100 g of soaking water (mmol gallic acid EQ·100 g^{-1}).

Statistical Analysis

Reference, control, and PEF processing along with oil and cake analytical determination were performed by duplicate. Results were analyzed through a one-way ANOVA followed by the Dunnett test and calculation of correlation coefficients using Minitab 18 software (Minitab® 18.1, USA). Pearson (r) and Spearman (ρ) correlation coefficients were determined based on data distribution; r for data normally distributed, and ρ for not normally distributed data or data with outliers [45].

Results and Discussion

Moisture and Oil Content of Pecan Nut Kernels

Moisture and oil content of reference, control, and PEF-treated kernels are shown in Table 2. Reference samples contained a moisture and oil content of 3.2 ± 0.1 and 61.2 ± 3.0 g 100 g^{-1} db, respectively. Moisture increased up to 24.9 ± 0.7 g 100 g^{-1} db in control kernels while the moisture content of PEF-treated kernels ranged from 18.7 ± 2.3 to 21.7 ± 2.3 g 100 g^{-1} db (Table 2). According to the Dunnett test, no significant differences between moisture of control and PEF-treated samples were observed ($\alpha = 0.05$). The oil content of reference kernels was 61.2 ± 3.0 g 100 g^{-1} db decreasing to 54.9 ± 0.8 g 100 g^{-1} db in control kernels, whereas in PEF-treated kernels reduced between 54.0 ± 2.9 and 56.7 ± 1.1 g 100 g^{-1} db. Regarding the o_{SW}, control and PEF-treated samples displayed comparable values ranging from 3.8 ± 0.0 to 6.2 ± 0.1 g with the lowest o_{SW} observed in kernels pretreated at 5.0, 7.8, and 17.6 kJ kg^{-1} (Table 2). Moisture and oil content of control and PEF-treated samples were similar, suggesting that these changes might be related to the soaking process. The oil retained into the soaking water could be related to kernels grinding and water immersion, modifying their microstructure. Fatty acids are located in small and spherical structures called oleosomes; these organelles are constituted by a core of triacylglycerols stabilized by a monolayer of phospholipids and proteins found in the cotyledon tissue of pecan nut kernels [20, 39, 54]. According to Zhang et al. [54], it is possible to extract oleosomes from the cotyledon tissue of pecan nuts by grinding and water immersion. In this line, it is suggested that the exposure of cotyledon tissue due to kernels sectioning and the moisture gained during water immersion might facilitate oleosomes transfer to the soaking water.

Effect of PEF Processing on OEY, Acidity, and AC

OEY

The effect of PEF processing on OEY of pecan nut kernels along with those of reference and control kernels are displayed in Fig. 1. The OEY of reference samples was 63.8 ± 1.5% being comparable to OEY reported for the mechanical extraction of pecan nut oil [11, 35, 44]. Water immersion of control kernels decreased OEY to 54.2 ± 2.0%, representing a loss 14.9% compared to reference samples. The reduction of OEY due to water immersion was also reported by Polmann et al. [35] and Sarkis et al. [43] for pecan nuts and sesame seeds, respectively. Concerning the application of PEF, kernels treated at 0.5, 1.8, 4.3, 5.0, and 17.6 kJ kg^{-1} equaled OEY of reference kernels (Fig. 1). No linear relationship between W and OEY

Table 2 Effect of soaking on moisture and oil content of control kernels and kernels pretreated by PEF at different specific energy inputs (W)

	Reference	Control	W (kJ kg^{-1})		
			0.5, 0.8, 1.8	2.8, 4.3, 9.7	5.0, 7.8, 17.6
Moisture (g 100 g^{-1} db)	3.2 ± 0.1*	24.9 ± 0.7	21.3 ± 2.0	21.7 ± 2.3	18.7 ± 2.3
Lipid content (g 100 g^{-1} db)	61.2 ± 3.0*	54.9 ± 0.8	54.3 ± 3.7	54.0 ± 2.9	56.7 ± 1.1

Reference, kernels without soaking nor PEF processing. Control, kernels soaked in tap water (1:3 w/w) for 20 min. PEF-treated kernels were categorized according to the pulse number applied: 0.5, 0.8, and 1.8 kJ kg^{-1} corresponded to 10 pulses; 2.8, 4.3, and 9.7 kJ kg^{-1} corresponded to 55 pulses; 5.0, 7.8, and 17.6 kJ kg^{-1} corresponded to 100 pulses. Moisture and lipid content were expressed as g per 100 g of kernels in dry basis (db). Means with an asterisk within rows were significantly different from control kernels according to the Dunnett test ($\alpha = 0.05$)

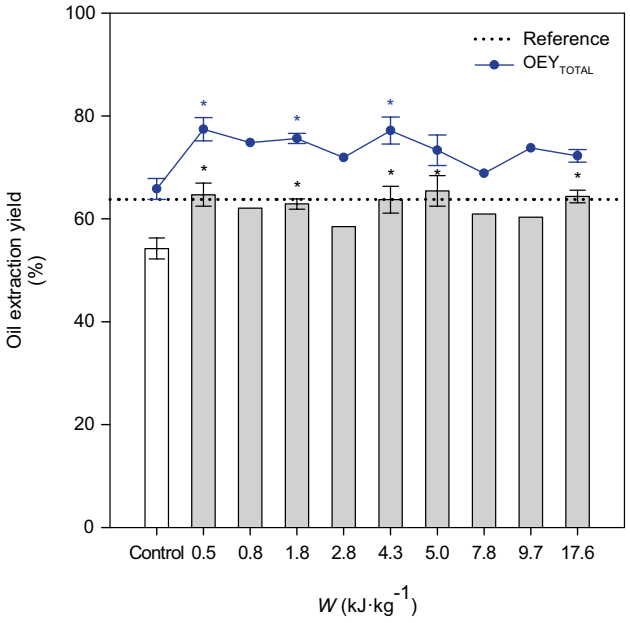

Fig. 1 Effect of PEF pretreatments on oil extraction yield (OEY) of pecan nut kernels. OEY_{TOTAL} is the extraction yield considering oil extracted during the soaking process. Means with an asterisk were significantly different from the control according to the Dunnett test ($\alpha = 0.05$)

was observed (Table 3).The OEY_{TOTAL} estimated by o_{SW} determination was used to analyze the PEF effect on oil extraction yields. OEY_{TOTAL} of control kernels was 65.8% being comparable to OEY of reference kernels. On the other hand, PEF-treated samples displayed OEY_{TOTAL} that ranged from 68.9 to 77.4% improving oil extraction between 8.0 and 21.4% compared to reference samples (Fig. 1). The application of PEF as a pretreatment to increase oil extraction from pecan nut kernels displayed higher OEY than the enzymatic pretreatment reported by Polmann et al. [35] (65.2%). Furthermore, the OEY_{TOTAL} of PEF-treated kernels was comparable to OEY achieved in the extraction of pecan nut oil using pressurized CO_2 and *n*-butane (65.3–70.5%) [3, 44].

The improvement of oil extraction processes after PEF has been reported for maize, olives along with sunflower and sesame seeds, being associated with irreversible cell disruption due to the electroporation mechanism [18, 34, 43]. However, it is proposed that rather than irreversible cell disruption, pecan nut kernels pretreated by PEF might undergo reversible electroporation, producing changes in the cell structure that facilitates oil extraction. Han et al. [19] suggested that the improvement of OEY by PEF application could be related to the fusion of oil bodies within the cell and the release of intracellular water-soluble compounds. Furthermore, kernels water immersion reduced OEY, demonstrating that not only PEF parameters (*W*, *E*, *n*, pulse shape, and width) and food characteristics contribute to the OEY but also processing steps such as soaking, drying, and grinding. Andreou et al. [4] observed that more intense PEF pretreatments (≥ 20 kJ kg^{-1}) lead to higher OEY, attributing this effect to a combination of cell disruption and demulsification of oil-in-water emulsions formed at the malaxation step during olive oil extraction. In contrast, Guderjan et al. [18] reported higher OEY of maize germ by combining a PEF processing of 0.6 kJ kg^{-1} with incubation and drying previous to oil extraction, and Sarkis et al. [43] reported

Table 3 Probability values (*p* value) of one-way analysis of variance ($\alpha = 0.05$) and Pearson correlation coefficients (*r*) of oil and cake from pecan nut kernels pretreated by PEF

Response variables	*W* (kJ kg^{-1})	Pearson correlation test	
	p value	*p* value	*r*
Kernels			
Oil extraction yield (%)	0.006	0.530	− 0.158
Total oil extraction yield[a] (%)	0.008	0.058	− 0.455
Oil			
Acidity[b] (mg KOH 100 g^{-1})	0.000	0.789	− 0.054
Antioxidant capacity (mg trolox EQ 100 g^{-1})	0.003	0.033	− 0.356
Cake			
Total phenolics (mmol gallic acid EQ 100 g^{-1})	0.000	0.014	− 0.289
Condensed tannins (mmol catechin EQ 100 g^{-1})	0.000	0.037	0.285
Antioxidant capacity (mmol trolox EQ 100 g^{-1})	0.080	n.s.	n.s.
Soaking water			
Total phenolics (mmol gallic acid EQ 100 g^{-1})	0.000	0.000	0.866

Antioxidant capacity was determined by the DPPH radical scavenging capacity method

W specific energy input, *n.s.* not significant

[a]Total oil extraction yield (OEY_{TOTAL}) is the extraction yield considering oil extracted during the soaking process

[b]The Spearman correlation coefficient was employed to determine the relationship between *W* and acidity

Table 4 Acidity and antioxidant capacity (AC) of the oil extracted from reference, control, and PEF-treated pecan nut kernels

	Acidity (mg KOH 100 g^{-1})	AC (mg trolox EQ 100 g^{-1})
Reference	29.0 ± 2.1	55.2 ± 2.3
Control	28.4 ± 1.0	56.4 ± 1.4
W (kJ kg^{-1})		
0.5	27.8 ± 1.9	51.8 ± 2.5
0.8	38.3 ± 1.4*	61.4 ± 6.1
1.8	21.3 ± 1.3*	56.7 ± 3.2
2.8	31.7 ± 0.1	55.4 ± 0.8
4.3	22.2 ± 1.9*	55.5 ± 4.1
5.0	30.8 ± 1.1	54.1 ± 1.7
7.8	26.1 ± 0.1	61.2 ± 0.4
9.7	27.9 ± 0.1	55.7 ± 2.1
17.6	21.8 ± 0.9*	49.4 ± 2.6*

Acidity was expressed per 100 g of pecan nut oil. AC was determined by the DPPH radical scavenging capacity method and concentrations were expressed as mg equivalents (EQ) per 100 g of pecan nut oil. Reference, kernels without soaking nor PEF processing. Control, kernels soaked in tap water (1:3 w/w) for 20 min. *W*, specific energy input. Means with an asterisk within rows were significantly different from control kernels according to the Dunnett test (α = 0.05)

higher OEY from sesame seeds pretreated at 40 kJ kg^{-1} followed by drying.

Oil Acidity and Antioxidant Capacity

Acidity and AC of oil extracted from reference, control, and PEF-treated kernels are shown in Table 4. Oil acidity of PEF-treated kernels varied from 21.3 ± 1.3 to 38.3 ± 1.4 mg KOH 100 g^{-1} being within values reported for cold-pressed and virgin oils of the Codex Standards for Fats and Oils from Vegetable Sources (≤ 40.0 mg KOH 100 g^{-1} oil) [13]. Similar results were described by Guderjan et al. [17], Puértolas and Martínez de Marañón [38], Andreou et al. [4], Moradi and Rahimi [34], and Veneziani et al. [52] for the acidity of oil extracted from rapeseeds, olives, and sunflower seeds pretreated by PEF. Guderjan et al. [17] reported that increments in oil acidity of rapeseeds pretreated by PEF might be due to the degradation of triacylglycerols by lipase activity. Likewise, Mohseni et al. [33] suggested that changes of intracellular materials and cell membrane rupture, as a consequence of PEF application followed by mechanical extraction, might favor the lipid-water interface changes necessary to lipase activation. Concerning oil antioxidant capacity, no significant differences were observed in AC of oil extracted from reference, control, and PEF-treated samples, except at 17.6 kJ kg^{-1} which produced the lowest AC (49.4 ± 2.6 mg trolox EQ 100 g^{-1}) (Table 3). AC reduction at the most intense PEF treatment could be related to the loss of phenolic compounds into the soaking water evidenced by its increment in TP, as discussed below.

Effect of PEF Processing on TP, CT, and AC of Cakes and TP of Soaking Water

Total phenolics and condensed tannins of the cake generated from oil extraction of reference, control, and PEF-treated samples along with TP of the soaking water are shown in Fig. 2. TP and CT values of the cake of reference

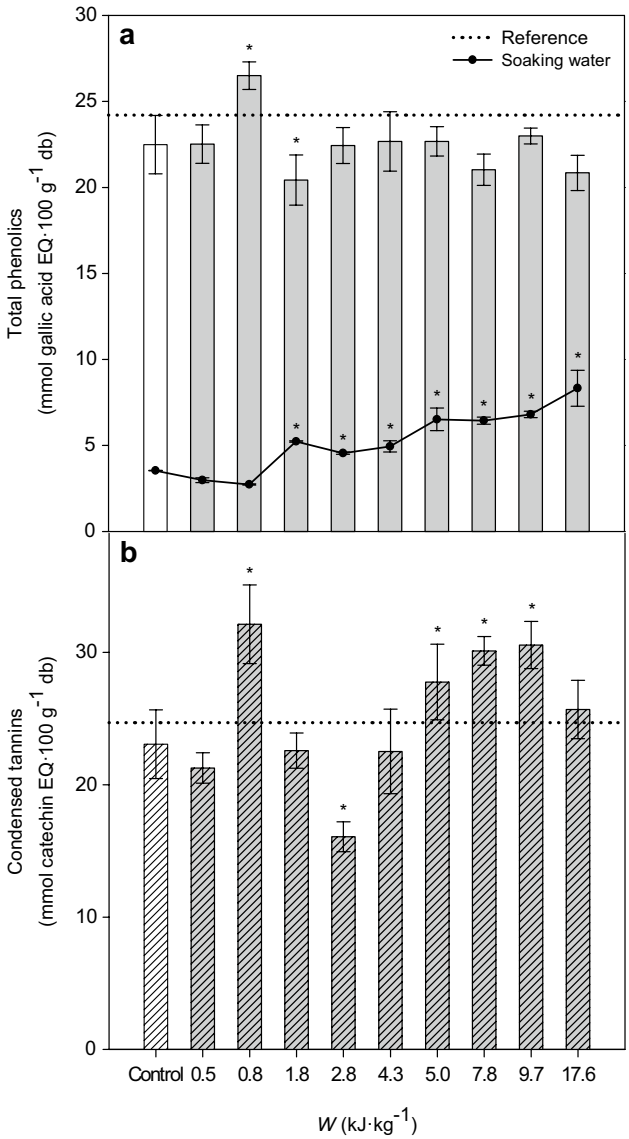

Fig. 2 Effect of PEF pretreatments on total phenolics (TP) (a) and condensed tannins (CT) (b) of the cakes generated from pecan nut kernels and TP of the soaking water. Concentrations were expressed as mmol equivalents (EQ) per 100 g of defatted cakes in dry basis (db) and 100 g of soaking water, respectively. Means with an asterisk were significantly different from the control according to the Dunnett test (α = 0.05)

Table 5 Antioxidant capacity (AC) of cakes from reference, control, and PEF-treated pecan nut kernels

	AC (mmol trolox EQ 100 g^{-1} db)
Reference	19.5 ± 0.3
Control	19.6 ± 0.4
W (kJ kg^{-1})	
0.5	19.5 ± 0.2
0.8	19.7 ± 0.4
1.8	19.3 ± 0.2
2.8	19.7 ± 0.0
4.3	19.5 ± 0.3
5.0	19.6 ± 0.2
7.8	19.1 ± 0.1
9.7	19.5 ± 0.2
17.6	19.3 ± 0.4

AC was evaluated by the DPPH radical scavenging capacity method and expressed as mmol equivalents (EQ) per 100 g of defatted cake in dry basis (db). No significant differences were determined according to one-way ANOVA ($\alpha = 0.05$)

kernels were 24.2 ± 1.8 mmol gallic acid EQ 100 g^{-1} db and 24.7 ± 2.9 mmol catechin EQ 100 g^{-1} db, respectively, being comparable to values reported by Maciel et al. [29] for pecan nut cakes. PEF-treated samples resulted in cakes with similar TP values to those from control samples but below to the reference, except at 0.8 kJ kg^{-1}. The application of 0.8 kJ kg^{-1} increased TP by 17.8 and 9.5% compared to control and reference cakes, respectively. Whereas PEF pretreatments greater than 0.8 kJ kg^{-1} led to an increment in TP in the soaking water directly proportional to the W applied ($r = 0.866$) (Fig. 2a). CT concentration of pecan nut cakes also increased with the specific energy input applied ($W \geq 5.0$ kJ kg^{-1}) except at 0.8 kJ kg^{-1}, where the highest CT value was achieved (32.1 ± 3.0 mmol catechin EQ 100 g^{-1} db) (Fig. 2b). No significant differences were found between AC of cakes from kernels pretreated by PEF and those from control and reference samples (Tables 3, 5).

Extraction of phenolic compounds by PEF processing has also been described on sesame seeds [43], grape seeds [8], and defatted almonds [30], attributing it to electroporation of the cell membrane improving the release of hydrophilic compounds. Based on the changes of TP and CT concentration of cakes obtained after PEF pretreatment, it is suggested that a rearrangement of intracellular materials (ions and small molecules movement, vacuoles rupture, and enzyme activation) might occur at less-intense PEF processing conditions ($W < 1.8$ kJ kg^{-1}), not being enough the intensity to initiate phenolic compounds release evidenced by TP of the soaking water, and also by the fact that the highest TP and CT values were observed at 0.8 kJ kg^{-1}. Contrarily, at higher W (≥ 1.8 kJ kg^{-1}), the release of phenolic compounds starts increasing along with the specific energy input applied, promoting the interaction between CT and cell wall materials [40] and retaining condensed tannins in the cake.

Conclusion

In this study, pecan nut kernels were immersed into water in order to apply PEF processing, which led to an increment of moisture (18.7–24.9 g 100 g^{-1} db) and a decrement of oil content (54.0–56.7 g 100 g^{-1} db). After considering oil extracted into the soaking water, OEY_{TOTAL} of PEF-treated samples increased up to 68.9 and 77.4%. The highest OEY_{TOTAL} was achieved at 0.5 kJ kg^{-1}, being 21.4 and 17.6% higher than the values of reference and control samples, respectively. The acidity and antioxidant capacity of extracted oils were not affected by PEF processing. Moreover, an increase of TP and CT of 17.8 and 39.3%, respectively, was observed in the cake produced from the oil extraction of kernels pretreated at 0.8 kJ kg^{-1}. This is probably due to the rupture of condensed tannins vacuoles. The increment of the specific energy input applied (≥ 1.8 kJ kg^{-1}) increased phenolic compounds release into the soaking water. These data demonstrate that PEF technology might be an appropriate pretreatment to enhance mechanical extraction of pecan nut oil with no effect in neither its acidity nor AC, leading to a cake that is a valuable by-product with potential functional properties due to its enhanced content of phenolic compounds. However, oil recovery from the soaking water might be a necessary step to achieve higher OEY employing PEF technology, affecting the feasibility of PEF as an assisting process in pecan nut oil extraction. Further research related to microscopy, enzymatic, and compositional analysis is also needed to corroborate the mechanism of PEF and understand kernels microstructural changes involved with the application of PEF to improve oil extraction from pecan nuts.

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Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

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