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Pulsed Electric Fields for the Treatment of Olive Pastes in the Oil Extraction Process

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Abstract: The aim of this study was to evaluate the ability of pulsed electric field (PEF) technology to improve the extractability and enhance the oil quality in an industrial olive oil extraction process. Using a PEF device on olive pastes significantly increased the extractability from 79.5% for the control, up to 85.5%. The PEF system did not modify the primary legal quality parameters or total concentrations of phenols, aldehydes, and esters. On the contrary, the non-thermal treatment slightly enhanced the dialdehydic forms of decarboxymethyl elenolic acid linked to hydroxytyrosol (3,4-DHPEA-EDA) and tyrosol (p-HPEA-EDA), and decreased the total saturated and unsaturated C5 and C6 alcohols of the PEF EVOO (Extra Virgin Olive Oil) compared to the control test. This study confirmed that PEF technology can improve olive oil extraction and quality.

Keywords: pulsed electric field; industrial plant; oil yield; quality; phenols; volatile compounds

1. Introduction

Pulsed electric field (PEF) technology is based on the application of high-intensity pulsed electric fields with durations of microseconds to milliseconds, and on the order of 10–80 kV/cm [1].

Regarding the interaction between a PEF and organic matter, the dielectric rupture theory introduced in Zimmermann [2] known as electroporation, in which exposure of the cell membrane to an electric field causes an increase in transmembrane potential (TMP) leading to the formation of pores in weak areas of the membrane, is generally accepted.

According to the intensity of the field strength, electroporation can be reversible or irreversible [3]. Generally, 0.5–1.5 kV/cm is used for reversible electroporation, and 1.0–3.0 kV/cm is used for irreversible permeabilisation, while values above 15 kV/cm cause microbial inactivation [4].

The main components of a PEF processing system are: (1) a high-voltage power generator, consisting of a high-voltage power supply, an energy storage capacitor bank, a charging-current-limiting resistor, and a switch to discharge energy from the capacitor; (2) a treatment chamber constituted of two electrodes separated by an insulator to confine the product to be treated; (3) a fluid-handling system (pumps, water tanks, pipes, and valves); and (4) a control system for monitoring the process [5]. Based on the electrical circuit composed of power supplies, switches, resistors, capacitors, inductors, and the treatment chambers, which constitutes the pulse-forming network (PFN), it is possible to

generate different pulse forms, such as exponential or square pulses (either monopolar or bipolar). The former can be generated with a very simple PFN, but its tail contributes to produce excess heat, while the latter involves a more complicated PFN, but is more efficient and generates less heat. In all cases, an adjunct component, such as a cooling system, is required to balance the ohmic heating effect and control the process temperature [6].

To suitably manage the PEF system, some typical process parameters should be considered, as reported in Raso et al. [4]: electric pulse amplitude (U), electric field strength (E), treatment time (t), pulse shape, pulse width (τ), number of pulses (n), pulse specific energy (W), and pulse repetition frequency (f). The treatment time is calculated by multiplying the number of pulses (n) by the pulse duration (τ). The electric field strength (E) can be estimated by dividing the voltage across the electrodes (U) by the electrode gap (L). Electric field strength and treatment time are generally considered the primary technological parameters.

The treatment chamber has a major influence on the uniformity of the electric field in the treatment region; therefore, the accuracy of its design is of vital importance. The basic requirement is to contain the product while pulsing the electric field. Several treatment chambers have been designed for batch or continuous operation [7].

In the food sector, although PEF technology has been mainly studied for microorganism inactivation in liquid food, such as milk, fruit juices, and liquid eggs, it has also been proposed to improve solid-liquid extraction (e.g., extraction of ingredients, winemaking, sucrose extraction, and recovery of useful compounds from food waste and by-products), cutting/slicing (e.g., vegetable snack or French fry production), food dehydration (e.g., hot-air drying, osmotic dehydration, and freeze-drying), and mechanical extraction (e.g., juicing and mechanical oil extraction) [8–10].

Solid-liquid extraction is a process for recovering desired compounds under the influence of a concentration gradient and by using solvents. Luengo et al. [11] investigated the influence of different PEF intensities (3–7 kV/cm and 0–300 ms) on carotenoid extraction from tomato peel and pulp. They found that PEF treatment at 5 kV/cm improved carotenoid extraction by 39% compared to control samples and reduced the solvents percentage from 30% to 45% without affecting the carotenoid extraction yield. Puértolas et al. [12] studied the effect of PEF treatment on anthocyanin extraction yield from purple-fleshed potatoes with different extraction time and temperature ranging from 10 °C to 40 °C. A PEF treatment of 3.4 kV/cm for 105 μ s significantly increased the extraction yield and enabled the use of water as a solvent. PEF was also used to stimulate the carotenoid accumulation in plant tissues by changing the respiratory activity and primary quality attributes of tomatoes. Total carotenoid and lycopene concentrations were enhanced by about 50% after applying 30 pulses at 200 kV/m (2.31 kJ/kg) [13].

The turgor of fruits and vegetables is one of the greatest concerns in mechanical operations, such as cutting or slicing. PEF treatment could reduce the turgor pressure and soften plant tissue with no modification to its fresh appearance [14,15], and thus reduce the energy consumption required for mechanical operations. PEF is also effectively used for drying and freezing food by enhancing heat and mass transfer processes [9,16,17].

An extraction process is used to produce juices from fruits and vegetables. This generally consists of a mechanical extraction (e.g., pressing or centrifugation). PEF technology can improve the extraction efficiency by increasing the yield, decreasing the processing time, and reducing the energy requirements of the process and subsequent processes [10]. Kantar et al. [18] evaluated the impact of PEF treatment on oranges, pomelos, and lemons in aqueous media. They observed juice extraction yield increases, after pressing, of 25% for oranges, 37% for pomelos, and 59% for lemons.

Recently, different, new technologies in the olive oil extraction process have been introduced and studied to support the malaxation step with the aim of improving the oil extraction yield and final quality, such as low-frequency ultrasound [19–22], microwave [23,24], high-frequency ultrasound [25–27], and PEF. Andreou et al. [28] used a PEF (1.6–70.0 kJ/kg) process before malaxation (30 min at 30 °C) with an extraction yield increase up to 18%. Another study found that the extraction yield improved

54% when olive paste was treated with a PEF (2 kV/cm) without malaxation, and at 15 °C (malaxation temperature), the extraction yield improved by 14.1% [29]. This indicates a reduction of malaxation temperature without negative effect on extraction yield. Also, Puértolas et al. [30] observed a 13.3% extraction increase with the application of a PEF treatment (2 kV/cm) to olive paste. Moreover, olive oil at the end of the process exhibited significantly higher total phenolic content, total phytosterols, and total tocopherols than control samples.

The aim of this study was to evaluate the effects of PEF technology applied in an industrial olive oil extraction process to improve extractability and enhance oil quality. In order to achieve this aim, PEF technology installed downstream of the crushing stage and upstream of malaxation was applied to olive paste by means of a specific PEF device.

2. Materials and Methods

2.1. Pulsed Electric Field System

PEF system (Figure 1) was used to facilitate the olive oil extraction process. The device generated monopolar exponential-decay electric pulses of 200 μ s with a frequency up to 75 Hz. The peak voltage and current were 16 kV and 145 A, respectively. The pulse-forming network (PFN) consisted of a high-voltage transformer, which doubled the mains voltage in a special circuit before rectifying it. This rectified voltage (+850 V) charged and discharged the capacitors (600 μ F) via semiconductor switches to the pulse transformer. On the secondary side of the transformer, a sphere opposite the ground potential chamber was connected. A pump propelled the olive paste at 2300 kg/h such that the average residence time in the chamber was 0.19 s.

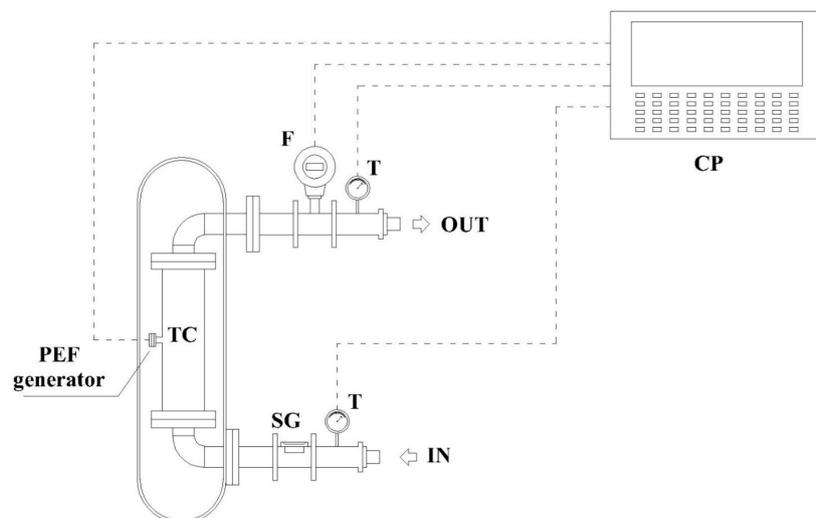


Figure 1. The Pulsed Electric Field (PEF) device. IN—paste flow Input; OUT—paste flow Output; TC—Treatment Chamber, T—Temperature probe; F—Flowmeter; SG—Sight Glass; and CP—Control Panel.

The actual current, before the pulse transformer, was measured using a Rogowski coil, while the actual voltage, at the treatment chamber, was measured using a multilevel voltage driver. Both devices were connected to an oscilloscope.

2.2. Plant for Olive Oil Extraction and Experimental Plan

Experimental tests were performed in a commercial olive mill (Pietro Leone & figli, s.n.c., Puglia, Italy), described in Leone et al. [31]. Once cleaned and washed, olives were crushed using a hammer crusher at a rotor rotation speed of 2900 rpm and with 7 mm grid holes (mod. Hammer Mill Crusher; Alfa Laval Corporate AB, Lund, Sweden). Olive paste was then continuously pumped to a malaxer

machine and malaxated for 30 min at 27 ± 0.5 °C. The obtained olive paste was continuously pumped at 2300 kg/h using a progressive cavity pump to the three-phase decanter (mod. NX X32; Alfa Laval Corporate AB) and two following vertical plate centrifuges (mod. UVPX 507; Alfa Laval Corporate AB) for liquid–liquid separation.

For PEF treatment of olive paste, the oil extraction line described above was implemented with a PEF device placed between the hammer crusher and the malaxer machine.

PEF treatment of 2 kV/cm was applied to the olive paste at a frequency of 25 Hz and with a pulse width of 50 μ s. The mass flow rate used in the experiments was 2300 kg/h for the olive paste treatment, and considering that the energy delivered per pulse was 210 J (at 25 Hz), the specific energy is 7.83 kJ/kg.

Turning the PEF devices on or off enabled two different olive oil extraction system configurations: the control (PEF devices off) and treatment of olive pastes (PEF devices on).

A homogeneous lot of cultivar Nocellara del Belice (*Olea europaea* L.) olives was used to test the effects of the PEF device on olive pastes.

Fruits were mechanically harvested, transported on the same day to the mill, and processed within six hours.

An experimental plan of the comparative tests was performed with five comparative tests for each treatment (five control runs and five PEF runs).

2.3. Extractability and Oil Content in the Olives, Pomace, and Wastewater

A plant extractability parameter and the oil contained in pomace and wastewater were used to evaluate the quantitative performance of the oil extraction plant.

Oil extractability (Ex) is the percentage ratio between the percentage of oil extracted from the olives (Poe) by the plant with respect to the percentage of oil content in the olives (Pe).

Ex was calculated using the following equation:

$$Ex = \frac{Poe}{Pe} \times 100 \quad (1)$$

The oil content in olives, pomace, and wastewater was evaluated according to Leone et al. [19]. The total oil content was determined using a 25 g of sample for olives and pomace, and 80 g for wastewater. The samples were previously dehydrated until reaching a constant weight, and the moisture removed from the sample was recorded. The oil in the dried sample was extracted using hexane in an automatic extractor (Randall 148, Velp Scientifica, Milan, Italy) by following the analytical technique described by Cherubini et al. [32]. Each sample was analyzed three times. The results were expressed as percentages of oil in wet and dry matter.

2.4. Olive Oil Quality

2.4.1. Legal Quality Parameters

The primary legal quality parameters of an EVOO, such as peroxide values, free acidity, and UV spectrophotometric indices (K232, K270, and Δ K), were evaluated as described by the European Official Methods [33].

2.4.2. Phenolic Compounds

Phenolic compounds were extracted using the liquid–liquid extraction procedure described in Selvaggini et al. [34] by mixing the oil samples with a methanol/water solution (80/20 *v/v*) using an Ultra-Turrax T 25 homogeniser (IKA Labortechnik, Staufen, Germany). The phenolic extracts, filtered with 25 mm polyvinylidene fluoride (PVDF) filters with a pore size of 0.2 μ m (Agilent Technologies, Palo Alto, CA, USA), were directly injected into an Agilent Technologies model 1100 controlled using ChemStation (Agilent Technologies, Palo Alto, CA, USA) for the high-performance liquid

chromatography (HPLC) analyses. The detection and quantification of phenolic molecules were performed using the chromatographic conditions described in Selvaggini et al. [35], and the data were expressed as milligrams of phenols per kilogram of oil.

2.4.3. Volatile Compounds

The evaluation of the volatile molecules present in the headspace (HS) of EVOO samples was performed with a solid-space microextraction (SPME) using a 50/30 μm divinylbenzene/Carboxen/poly(dimethylsiloxane) (DVB/CAR/PDMS) 1-cm-long fiber (Stableflex; Supelco, Inc., Bellefonte, PA, USA) and controlled using a Varian CP 8410 autoinjector (Varian, Walnut Creek, CA, USA). The gas chromatography-mass spectrometry analyses (GC-MS) were performed using a Varian 4000 GC-MS equipped with a 1079 split/splitless injector with a fused-silica capillary column (DB-Wax-ETR, 50 m, 0.32-mm i.d., 1 μm film thickness; J&W Scientific, Folsom, CA, USA) using the method described in Veneziani et al. [36], and the data of the main C5 and C6 saturated and unsaturated aldehydes and alcohols and of esters were expressed as micrograms of volatile compound per kilogram of oil.

2.5. Data Processing

The collected data were processed to calculate quantitative and qualitative parameters. MatlabTM high-level programming software (R2016b, The Mathworks Inc., Natick, MA, USA, 2016) was used to read, elaborate, and statistically analyze the experimental data. The Matlab statistical toolbox was used to analyze the data series to assess the significance between the treatments, using the ANOVA test and Tukey's test for means separation at p -values < 0.05 .

3. Results and Discussion

3.1. Effect of the Pulsed Electric Field Treatment of Olive Paste Versus Extractability

Table 1 includes the extractability and pomace characterization values. Only trace amounts of oil were found in all the analyzed wastewater samples, and therefore, the data are not shown.

Table 1. Quantitative results and process parameters.

Test Conditions	Variety	Pomace		Extractability
		Moisture (%)	Oil (% Dry Basis)	(%)
Control	Nocellara del Belice	60.6 \pm 1.1 <i>a</i>	10.4 \pm 0.7 <i>a</i>	79.5 \pm 0.8 <i>b</i>
PEF	Nocellara del Belice	60.2 \pm 0.5 <i>a</i>	7.4 \pm 0.7 <i>b</i>	85.5 \pm 1.5 <i>a</i>

Different letters in columns denote statistically significant differences (Tukey's test, $p < 0.05$).

PEF treatments exhibited different behaviors. A positive effect on extractability was observed when olive paste was treated with a PEF device (2 kV/cm, 7.83 kJ/kg). In fact, the extractability value was 79.5% for the control and 85.5% when a PEF device was used. The resulting difference was statistically significant. The PEF treatment of olive paste delivered a significant reduction in pomace oil loss of about 40.5%. This confirmed the results reported in Puértolas and Martínez de Marañón [30], where a PEF treatment (2 kV/cm; 11.25 kJ/kg) was applied to Arroniz-variety olive paste. The authors assert that the PEF treatment significantly increased the extraction yield by 13.3% with respect to a control. The results of the present study are also in accordance with Abenoza et al. [29], where the authors reported an increase of 13.85% in Arbequina oil yield by combining a PEF treatment (2 kV/cm; 5.22 kJ/kg) with a subsequent, gentle malaxation step of 30 min at 15 °C. In addition, the authors assert that no significant improvement was observed when the malaxation temperature was increased to 26 °C.

The positive effects of PEF technology on olive oil yield could be explained by a double mechanism: the improvement of oil extraction from olive tissue, and the release of olive oil trapped in oil–water emulsions [30,37,38].

3.2. Olive Oil Quality

In accordance with the previous studies concerning the recent thermal and non-thermal technologies applied to the olive oil mechanical extraction process and based on the alteration of cell membrane permeability and structure [22,26,29,30,39,40], the prototype industrial PEF model used for the treatment of Nocellara del Belice cv. olive paste did not impact values of the EVOO primary legal quality parameters, as shown in Table 2.

Table 2. Standard virgin olive oil parameters specified by the International Olive Council (IOC, 2017). EVOO: extra virgin olive oil.

	Test Conditions	Variety	Free Acidity (%)	Peroxide Value (meq O ₂ /kg)	K232	K270	ΔK
Legal limit EVOO			<0.8	≤20	≤2.50	≤0.22	≤0.01
Treatment on paste	Control	Nocellara del Belice	0.59 ± 0.02 <i>a</i>	6.78 ± 0.39 <i>a</i>	1.51 ± 0.20 <i>a</i>	0.17 ± 0.01 <i>a</i>	0.001 ± 0.001 <i>a</i>
	PEF	Nocellara del Belice	0.57 ± 0.02 <i>a</i>	7.13 ± 0.24 <i>a</i>	1.54 ± 0.20 <i>a</i>	0.17 ± 0.01 <i>a</i>	0.001 ± 0.002 <i>a</i>

Different letters in columns denote statistically significant differences (Tukey's test, $p < 0.05$).

The concentration of primary EVOO hydrophilic phenols was not negatively affected by the PEF system applied to the olive paste (Table 3). On the contrary, a slight increase in the amount of dialdehydic forms of decarboxymethyl elenolic acid linked to hydroxytyrosol (3,4-DHPEA-EDA or oleacein) and to tyrosol (p-HPEA-EDA or oleocanthal) was detected in the EVOO extracted with PEF technology compared to the control test. However, the total phenols data contrasted with the previous studies [30,41], that showed a general increase of the phenolic fraction of EVOOs treated with a PEF. The positive effect on EVOO quality, mainly connected to the phenolic composition, is probably due to the position of the PEF system during the mechanical extraction process, which in this study was downstream of the crusher instead of after the malaxation phase, as described by Veneziani et al. [41]. This statement is also confirmed by the results of Abenoza et al. [29]; applying the PEF chamber in the same position after the crushing step did not measurably enhance the phenolic fraction due to non-thermal treatment of the olive paste. This result could be related to a limited effect on the release and solubilisation phenomena of phenolic compounds into the oily phase and to a putative, minor impact on the inhibition of the degradative enzyme (peroxidase and polyphenoloxidase) activity compared to the tests in which the PEF system operated after the malaxation step.

The volatile fraction of the PEF EVOO (Table 4) did not show significant differences in the sum of the saturated and unsaturated C5 and C6 aldehydes and of total esters (freshly cut grass and floral), whereas the concentration of total alcohols (fruity and ripe fruits) decreased 25% compared to the control trial [42]. These results, which were not confirmed by Veneziani et al. [41] for the other three cultivars, could be due to an impact of a different olive genetic origin and/or a different effect of PEF technology on the different enzymes of the lipoxygenase pathway when applied after the crushing phase.

Table 3. Phenolic composition of EVOOs (mg/kg).

Phenolic Compounds	Nocellara del Belice	
	Control	PEF-Treated
3,4-DHPEA	2.6 ± 2.6 <i>a</i>	1.5 ± 0.3 <i>a</i>
<i>p</i> -HPEA	2.1 ± 2.1 <i>a</i>	1.3 ± 0.3 <i>a</i>
Vanillic acid	0.0 ± 0.0 <i>a</i>	0.0 ± 0.0 <i>a</i>
3,4-DHPEA-EDA	97.8 ± 9.8 <i>b</i>	114.8 ± 2.2 <i>a</i>
<i>p</i> -HPEA-EDA	27.8 ± 0.5 <i>b</i>	30.1 ± 1.5 <i>a</i>
(+)-1-acetoxypinoresinol	3.1 ± 0.2 <i>a</i>	3.0 ± 0.3 <i>a</i>
(+)-pinoresinol	5.7 ± 1.7 <i>a</i>	4.4 ± 0.4 <i>a</i>
3,4-DHPEA-EA	40.0 ± 4.4 <i>a</i>	41.8 ± 9.5 <i>a</i>
Ligstroside aglycone	4.8 ± 0.7 <i>a</i>	5.4 ± 0.3 <i>a</i>
Total phenols	184.0 ± 11.4 <i>a</i>	202.3 ± 9.9 <i>a</i>

Different letters in rows denote significant statistical differences at $p < 0.05$ (Tuckey's test).

Table 4. Volatile compounds detected in EVOOs ($\mu\text{g}/\text{kg}$).

Heading	Nocellara Del Belice	
	Control	PEF-Treated
Aldehydes		
Pentanal	51.8 ± 16.6 <i>b</i>	206.5 ± 13.4 <i>a</i>
(<i>E</i>)-2-Pentenal	40.0 ± 2.8 <i>a</i>	19.0 ± 2.1 <i>b</i>
Hexanal	468.0 ± 53.7 <i>b</i>	871.5 ± 24.0 <i>a</i>
(<i>E</i>)-2-Hexenal	5647.0 ± 618.0 <i>a</i>	5878.3 ± 1258.3 <i>a</i>
(<i>E,E</i>)-2,4-Hexadienal	274.3 ± 23.0 <i>a</i>	199.3 ± 19.4 <i>b</i>
2,4-Hexadienal (<i>i</i>)	575.8 ± 54.1 <i>a</i>	426.3 ± 37.8 <i>b</i>
Σ Aldehydes	7056.8 ± 623.3 <i>a</i>	7600.8 ± 1259.3 <i>a</i>
Alcohols		
1-Pentanol	30.8 ± 0.4 <i>b</i>	71.8 ± 9.5 <i>a</i>
1-Penten-3-ol	223.0 ± 30.4 <i>a</i>	198.3 ± 11.0 <i>a</i>
(<i>E</i>)-2-Penten-1-ol	110.8 ± 8.1 <i>a</i>	91.5 ± 9.9 <i>b</i>
(<i>Z</i>)-2-Penten-1-ol	211.8 ± 30.8 <i>a</i>	127.8 ± 7.4 <i>b</i>
1-Hexanol	1052.8 ± 188.4 <i>a</i>	792.3 ± 148.1 <i>a</i>
(<i>E</i>)-2-Hexen-1-ol	1000.5 ± 127.3 <i>a</i>	961.8 ± 117.0 <i>a</i>
(<i>Z</i>)-2-Hexen-1-ol	0.0 ± 0.0 <i>a</i>	0.0 ± 0.0 <i>a</i>
(<i>E</i>)-3-Hexen-1-ol	106.8 ± 26.5 <i>a</i>	55.0 ± 8.5 <i>b</i>
(<i>Z</i>)-3-Hexen-1-ol	3735.8 ± 22.3 <i>a</i>	2558.5 ± 383.3 <i>b</i>
Σ Alcohols	6472.0 ± 305.2 <i>a</i>	4856.8 ± 437.2 <i>b</i>
Esters		
Hexyl acetate	921.5 ± 106.8 <i>a</i>	850.3 ± 151.0 <i>a</i>
(<i>Z</i>)-3-Hexenyl acetate	3423.3 ± 308.7 <i>a</i>	3282.8 ± 264.8 <i>b</i>
(<i>E</i>)-2-Hexenyl acetate	35.8 ± 1.1 <i>a</i>	17.0 ± 2.1 <i>b</i>
Σ Esters	4380.5 ± 326.6 <i>a</i>	4150.0 ± 304.8 <i>a</i>

Different letters in rows denote significant statistical differences at $p < 0.05$ (Tukey's test).

4. Conclusions

Regarding the evaluation of extractability, the PEF device has proved to be very efficient on olive pastes, and significantly increased the quantity of oil extracted compared to the control.

The PEF system, when applied downstream of the crushing stage, did not alter the concentrations of the primary legal quality parameters and did not increase the quantitative variability of total phenols and of the main volatile compounds responsible for freshly cut grass and floral sensory

notes. Qualitatively, significant differences were detected in the concentrations of some oleuropein and ligstroside derivatives, and of the sum of alcohols.

From a plant engineering perspective, a PEF device used during experimental tests demonstrated easy assembly and implementation inside the mill without substantially modifying the basic layout.

A PEF device could be a useful tool when inserted in an oil extraction plant to improve its efficiency, both from quantitative and qualitative perspectives, by contributing to the conditioning phase of olive pastes (the malaxation step).

However, as a new technology with insufficient industrial testing, further investigations are necessary to assess the PEF effects using olives of different cultivars and maturity indexes, and varying the machines and process parameters of the extraction plant.

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