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Influence of mechanical comminution of raw materials and PEF treatment on the aqueous extraction of phenolic compounds from artichoke wastes

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ABSTRACT

In this study the combined effect of mechanical comminution and pulsed electric fields (PEF) treatments on both cell disintegration and extractability of phenolic compounds during aqueous extraction from artichoke external bracts, was investigated. Different-sized bract discs were treated with varying PEF conditions, namely 0.5–5 kV/cm of electric field strength (E), and 1–20 kJ/kg of total specific energy input (W_T). The cell disintegration index (Z_p) of bract tissues, as well as the total phenolic content (TPC) and antioxidant activity (FRAP) of the extracts, were assessed.

The results showed that increasing the comminution process intensity led to greater cell disintegration, resulting in a peak extraction yield of phenolic compounds (17.61 ± 1.24 mg_{GAE}/100 g FW) achieved with the smallest sample size. Moreover, the application of PEF treatment further increased the Z_p value of the bract tissues in a size-dependent manner. The greater the sample size, the stronger the PEF efficiency. Coherently, under optimized PEF conditions (E = 3 kV/cm, W_T = 5 kJ/kg), the extracts exhibited higher TPC (+112–361%) and FRAP values (+83–836 %) as compared to the control samples after 120 min of diffusion. The extraction rate of phenolic compounds increased when the comminution degree was increased for both untreated and PEF-treated samples, and this was successfully predicted using Peleg's model.

These findings suggest that PEF can be a viable alternative to energy-intensive comminution pretreatment, thus enhancing the extraction of phenolic compounds without requiring finely ground raw material handling.

1. Introduction

The concept of recycling and valorizing biomass, in particular agro-food residues and food processing by-products, has garnered significant attention in recent decades. This approach offers a promising solution to reduce waste whilst obtaining valuable, high-priced products such as polyphenols (Francavilla et al., 2021), which possess the potential to serve as active ingredients in various food, pharmaceutical, and cosmetic sectors.

Within this framework, the production and processing of globe artichoke stands as an intriguing case study. Globe artichoke is an ancient herbaceous perennial plant belonging to the Asteraceae family, originally found in the Mediterranean region (Francavilla et al., 2021; Gominho et al., 2018). In 2019, global artichoke production exceeded 1.6 million tons, with Italy being the leading producer, followed by Spain, and France (Ilahy et al., 2022). The edible part of the plant consists of the flower heads (capitula), commonly known as "artichoke heads," and the part of the stems just below them. This portion of the plant possesses high nutritional value and health-promoting compounds such as phenols, inulin, fibers, and minerals (Carpentieri et al., 2022a; Francavilla et al., 2021; Guida et al., 2013; Lattanzio et al., 2009).

During the harvesting and industrial processing of artichokes, a substantial amount of biomass waste is generated, comprising approximately 60–80 % of the total raw material weight (Francavilla et al., 2021). This waste mainly consists of stems and leaves left after harvesting, and outer bracts of the flowers removed during processing, which are unsuitable for human consumption and typically discarded. Consequently, processors face huge environmental and disposal costs (Christaki et al., 2012; Lavecchia et al., 2019; López-Salas et al., 2021; Zuorro et al., 2014).

However, previous research has unveiled that artichoke wastes are still a rich source of polyphenols (Zuorro et al., 2016), including

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chlorogenic acid, mono- and dicaffeoylquinic acids, apigenin, and luteolin (Francavilla et al., 2021), with proven anti-inflammatory, antioxidant, and anticancer properties (Carpentieri et al., 2022a; Lattanzio et al., 2009; Llorach et al., 2002). Therefore, the development of effective and sustainable methods for recovering these bioactive compounds, which are found within the intracellular space of artichoke tissues, is of utmost importance.

Traditional techniques commonly used in industrial-scale extraction of bioactive compounds from plant-based biomass, such as Soxhlet extraction, percolation, and maceration extraction, rely on solid-liquid extraction (SLE) methods involving solvent application and leaching (Ameer et al., 2017). However, while these methods offer a straightforward approach, they often suffer from drawbacks related to the presence of the envelope (membranes and wall) surrounding the plant cell and internal organelles (vacuoles), which hinder solvent penetration and the mass transfer of intracellular compounds, thus resulting in slow extraction processes (Carpentieri et al., 2021a).

To overcome these limitations, conventional solvent extraction processes often require pre-treatments like size reduction (cutting/grinding) of the raw material. These pre-treatments aim to enhance mass transfer rates not only by increasing the surface-volume ratio and promoting solid-liquid contact but also by causing cell disintegration by mechanical destruction of cells at the cutting area (Ayala-Zavala et al., 2011; Galanakis, 2012; Jaeger et al., 2012). However, the size reduction of the raw material poses its own set of challenges. Excessively small particle sizes can boost extraction yield by increasing the proportion of disrupted cells, but this improvement comes at the expense of energy-intensive pre-treatments, typically quantified at approximately 100–400 kJ/kg, particularly when dealing with wet products (Cao and Rosentrater, 2015; Galanakis, 2015). On the other hand, finely ground raw material exposes a larger surface area, potentially compromising extract quality through oxidation (Turk et al., 2010), while making downstream separation and purification processes more complicated (Bouras et al., 2016; Galanakis, 2015; Moreira et al., 2019). Applying mild mechanical pre-treatments may result in excessively large particle sizes, retaining a significant number of intact cells. This hinders the recovery of intracellular compounds during extraction, necessitating longer extraction times, higher temperatures, and increased solvent volumes (Gerschenson et al., 2015). Furthermore, the extraction process is often conducted upon drying of plant biomass, which requires a significant amount of energy (3000–8000 kJ/kg) and may cause thermal degradation of valuable compounds (Singh et al., 2012).

To address these issues, researchers have explored the use of mild cell disruption pre-treatments of wet plant-based biomass intending to intensify the extractability of target intracellular compounds. The goal is to enhance the extractability of target intracellular compounds, prevent degradation, reduce energy costs, and ensure the effectiveness of subsequent separation/purification steps (Carpentieri et al., 2021a).

In this frame, pulsed electric fields (PEF) treatment has been shown to be promising as an efficient alternative to conventional cell disintegration techniques (Arshad et al., 2021; Carpentieri et al., 2021a). During PEF treatment, the exposure of plant tissue to an external electric field in the form of microsecond pulses of moderate intensity (0.5–10 kV/cm) and relatively low specific energy (1–10 kJ/kg), leads to electroporation of cell membranes and promotes extraction of intracellular compounds with maintained particle size of the raw material (Raso et al., 2016). This has shown great potential to intensify the selective recovery of target intracellular compounds including polyphenols from a wide range of food processing wastes and by-products (Arshad et al., 2021), while reducing energy costs, solvent consumption, and treatment time (Carpentieri et al., 2021b, 2022a,b; Frontuto et al., 2019; Martín-García et al., 2020; Pataro et al., 2017).

Despite these potential advantages, there is currently limited research on the feasibility of using PEF technology to enhance the extraction efficiency of phenolic compounds from artichoke wastes. Furthermore, the existing research focused exclusively on the outer bracts and stems, which were prepared by either cutting the bracts into fixed-size strips measuring 2 cm in length or dicing the stems into 1 cm^3 cubes (Battipaglia et al., 2009; Carpentieri et al., 2022a). Additionally, no or very few systematic studies account for the complex interactions between mechanical cell damages of plant tissues occurring during the size reduction of raw material, cell disintegration caused by PEF treatment, and the subsequent SLE step (Bouras et al., 2016; Jaeger et al., 2012). This information will be essential for the integration of PEF technology in the industrial scale extraction process since it may require adjustment of operating parameters of the related conventional processing steps, in order to maximize the beneficial effects of PEF on extraction yield and purification.

The objective of this study was to investigate the effects of size reduction (cutting), applied alone or in combination with PEF treatment, on the degree of cell disintegration of external bracts of artichoke tissues and the recovery yield of phenolic compounds during subsequent aqueous extraction. Specifically, different PEF treatments of variable electric field strength (E, kV/cm) and total specific energy input (W_T, kJ/kg) were applied to samples of different sizes, in order to define the optimal conditions that maximize the cell disintegration degree of the tissue with the minimum energy consumption. The latter was then used to investigate the effect of the PEF-assisted extraction on the recovery yields of phenolic compounds, as well as on the antioxidant power of the obtained extracts.

2. Materials and methods

2.1. Raw materials

Fresh artichoke heads of the "*Cavaliere*" variety were supplied by a local producer and kept refrigerated at 4 °C until use, within 3 days from harvesting. The moisture content on wet basis of artichoke bracts, evaluated on arrival at the laboratory, was found to be 84.0 ± 0.9 %. Before the experiments, the outer bracts, constituting the non-edible part of the artichoke head, were manually removed, and subsequently cut into discs of variable diameter (D = 0.5, 0.8, 1.0, 1.5, 2.0, and 3.0 cm) utilizing cork borers (Fig. 1). To prevent enzymatic oxidative phenomena of polyphenols, the discs of artichoke bracts were immediately immersed in a 1% (w/v) citric acid solution in water, before carrying out impedance analyses and PEF-assisted extraction experiments.

2.2. Chemicals

Folin-Ciocalteu reagents, sodium carbonate, citric acid, as well as iron chloride, and 2,4,6-tripyridyl-S-triazine (TPTZ), were purchased from Sigma-Aldrich (Sigma-Aldrich, Steinheim, Germany). Ascorbic acid for FRAP assay was obtained from Acros Organics (Geel, Belgium), while sodium acetate and acetic acid were purchased, respectively, from Panreac (Panreac Quimica, Barcelona, Spain) and Fisher (Fisher Scientific, Rodano, Italy).

2.3. PEF apparatus

The PEF treatments of artichoke bract discs were conducted using a laboratory-scale batch system as described in a previous study (Pataro et al., 2017). Briefly, the setup included a high voltage pulsed power generator (Modulator PG, ScandiNova, Uppsala, Sweden) capable of delivering monopolar square waveform pulses with a wide range of pulse widths (3–25 μs) and repetition frequencies (1–450 Hz). A batch treatment chamber was employed, consisting of two cylindrical stainless-steel electrodes (3 cm in diameter) separated to a distance of up to 5 cm by a polycarbonate tube containing the sample. The actual voltage and current signals at the treatment chamber were measured using a high voltage probe (Tektronix, P6015A, Wilsonville, OR, USA) and a Rogowsky coil (2–0.1 Stangenes, Inc., USA), respectively, and displayed on a 300 MHz digital oscilloscope (Tektronix, TDS 3034 B,

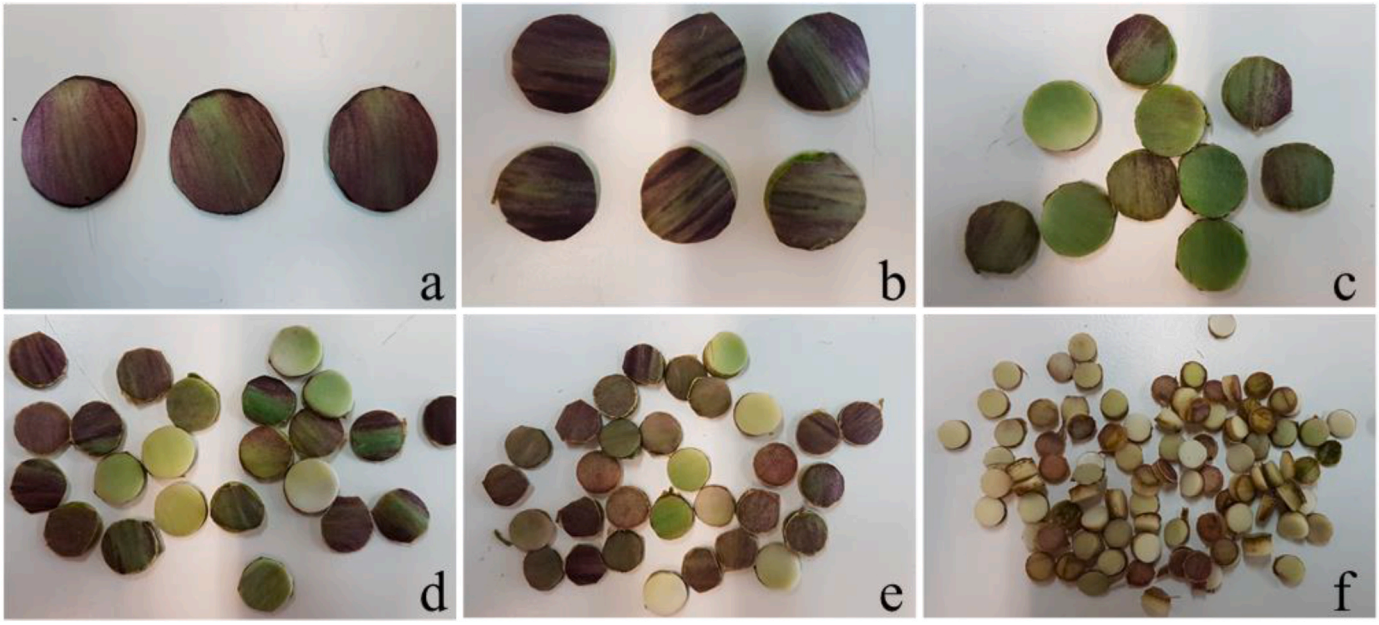

Fig. 1. Representative photographs of artichoke external bracts discs of variable diameter: (a) 3 cm, (b) 2 cm, (c) 1.5 cm, (d) 1 cm, (e) 0.8 cm, and (f) 0.5 cm.

Wilsonville, OR, USA). The maximum electric field intensity (E, in kV/cm) was determined by calculating the ratio of the peak voltage to the electrode gap. The total specific energy input (W_T, in kJ/kg of fresh-weight artichoke bracts) was calculated according to Eq. (1) (Carpentieri et al., 2021b):

$$W_T = \frac{n}{m}\int_0^{\infty} U(t) \bullet I(t)dt \quad (1)$$

where *U(t)* and *I(t)* represent the voltage across the electrodes and the current intensity through the treated product at time *t*, respectively, *n* is the number of pulses applied, and *m* is the mass of the treated plant material.

2.4. Quantification of cell disintegration degree via impedance analyses

Measurements of electrical complex impedance and the calculation of cell disintegration index (Z_p) of the artichoke bract tissues were used to characterize the electrical behavior and the degree of cell membrane disruption induced by mechanical comminution (cutting) operation in discs of different sizes as well as by subsequent PEF treatment, according to the method described by Donsì et al. (2010a).

Before each measurement, approximately 6 g of artichoke bract discs of a given size (0.5, 0.8, 1.0, 1.5, 2.0, or 3.0 cm in diameter) were loaded into the measuring cell, which was the same treatment chamber used for application of the PEF treatments with the electrodes separated at a distance of 1 ± 0.1 cm. Distilled water was added to the bract discs until achieving a solid-liquid ratio of 1:1 (g/mL). This ratio was found to be the minimal water volume required to ensure electrical continuity between the electrodes. PEF treatments were carried out at different field strength (E = 0.5, 1, 3, and 5 kV/cm) and total specific energy input (W_T = 1, 3, 5, 10, and 20 kJ/kg) at a constant pulse width (20 μs) and frequency (10 Hz). The PEF treatment time ranged between 20 μs (E = 5 kV/cm, W_T = 1 kJ/kg) and 45.6 ms (E = 0.5 kV/cm, W_T = 20 kJ/kg). The initial temperature of the samples was 20 ± 2 °C and no appreciable temperature increases were detected due to the relatively low energy input delivered during the treatment.

For the impedance measurements, the electrodes of the measuring cell were connected to an impedance analyzer (1260, Solartron, UK), which provides a frequency response of the complex impedance of the sample in the range of 10^2–10^6 Hz. The results were plotted as the absolute complex impedance |*Z*| as a function of the frequency, as well as for the different sizes of the artichoke discs and PEF treatment conditions.

The Z_p value was calculated on the basis of the measurement of the absolute value of the complex impedance of untreated 3 cm in diameter artichoke bract tissues ($|Z_{untr}|$), which served as the reference (control), and the absolute value of the complex impedance of bract tissues following both comminution (0.5–3 cm in diameter) and subsequent PEF treatment ($|Z_{tr}|$) in the low (0.1 kHz) and high (1 MHz) frequency ranges, using Eq. (2) (Frontuto et al., 2019):

$$Z_p = \frac{\left|Z_{untr\ (0,1\ kHz)}\right| - \left|Z_{tr\ (0,1\ kHz)}\right|}{\left|Z_{untr\ (0,1\ kHz)}\right| - \left|Z_{tr\ (1\ MHz)}\right|} \quad (2)$$

The calculated Z_p values range between 0 (for intact tissue) and 1 (for fully permeabilized tissue). All the measurements were carried out in triplicate.

The achieved Z_p values were used to elucidate the interaction between size reduction of the artichoke bracts and cell membrane disintegration induced by PEF treatment. Additionally, these values were instrumental in establishing the optimal PEF treatment conditions, in terms of electric field strength (E_{opt}) and total specific energy input ($W_{T,opt}$), to be used as an alternative or to complement mechanical comminution to boost the extractability of intracellular compounds with minimum energy consumption and without compromising the subsequent purification steps.

2.5. PEF-assisted aqueous extraction of phenolic compounds

For each PEF-assisted extraction experiment, three replicates of 6 g of artichoke bract discs of different sizes (D = 0.5–3 cm) were loaded into the treatment chamber, together with distilled water (solid-to-liquid ratio = 1:1 g/mL), and subjected to electropermeabilization treatment at the optimal treatment conditions ($E_{OPT.}$, $W_{T,OPT.}$) previously defined according to the results of impedance analyses. During the PEF treatment, the pulse repetition frequency and the pulse duration were kept constant at 10 Hz and 20 μs, respectively.

After each triplicated electro-permeabilization treatment, the discs of artichoke bracts (18 g) were immediately subjected to SLE by placing them into 250 mL Erlenmeyer flasks, where distilled water was added as extracting solvent at a constant solid-to-liquid ratio (1:10 g/mL)

according to previous findings (Battipaglia et al., 2009; Francavilla et al., 2021). The flasks were then introduced in an orbital incubator S150 (PBI International, Milan, Italy) where the extraction process was carried out under constant shaking at 160 rpm for different times (up to 360 min) at room temperatures (25 °C). For the sake of comparison, the conventional SLE process was carried out using the same protocol but without any PEF pre-treatment.

To examine the effect of extraction time on the release of phenolic compounds, three replicates of 1 mL extract of both untreated (only comminuted, $W_T = 0$ kJ/kg) and PEF-treated samples were withdrawn from the flasks at different diffusion times (10, 20, 30, 45, 60, 120, 240, and 360 min). The extracts were immediately centrifuged at 5700×*g* (PK121R model, ALC International, Cologno Monzese, IT) for 10 min at 4 °C to separate the supernatant, which was subsequently filtered through 0.45 μm syringe filters. The final extracts were then stored at −20 °C until further analysis.

The extraction kinetics data of total phenolic content (TPC) in water were fitted using the empirical equation (Eq. 3) proposed by Peleg (1988). This equation is widely employed to describe the extraction kinetics of intracellular compounds from various food matrices (Bucic-Kojic et al., 2007; Odriozola-Serrano et al., 2008; Pataro et al., 2020; Poojary and Passamonti, 2015). The equation is expressed as follows:

$$TPC = \frac{t}{\frac{1}{v_0} + \frac{t}{TPC_\infty}} \quad (3)$$

Here, TPC is expressed as milligrams of gallic acid equivalents (GAE) per 100 g of fresh weight (FW) artichoke samples (mg_{GAE}/100 g FW). It signifies the total phenolic content at time t (in min). v_0 (in mg_{GAE}/100 g FW·min) denotes the extraction rate at the very beginning ($t = t_0$), while TPC_∞ (in mg_{GAE}/100 g FW) indicates the maximum concentration of phenolic compounds in the extracts, representing the equilibrium concentration of the total extracted analyte as time approaches infinity (Pataro et al., 2020).

2.6. Determination of total phenolic content (TPC) and ferric reducing antioxidant power (FRAP)

The total phenolic content of extracts from both untreated and PEF-treated artichoke bract discs of different sizes was determined using the Folin-Ciocalteau method as described by Bobinaitė et al. (2015), with slight modifications. Briefly, 1 mL of undiluted extract was mixed with 5 mL of 10% (v/v) Folin-Ciocalteau reagent and allowed to stand for 5 min at room temperature. Afterward, 4 mL of sodium carbonate (7.5% w/v in water) was added to the mixture, which was incubated for 60 min at room temperature in a dark place. The absorbance of the obtained mixture was then measured at 765 nm using a V-650 spectrophotometer (Jasco Inc. Easton, MD, USA). Gallic acid dissolved in distilled water was used to generate a five-point external standard calibration curve in a concentration range comprised between 10 and 100 mg/L. TPC of extracts was then expressed as milligrams of gallic acid equivalents (GAE) per 100 g of fresh weight (FW) artichoke bracts (mg_{GAE}/100 g FW).

FRAP assay of extracts from both untreated and PEF-treated samples was carried out according to the method described by Benzie and Strain (1996), with slight modifications. Briefly, 2.5 mL of freshly prepared FRAP working solution and 0.5 mL of undiluted water extract were mixed and incubated for 10 min at ambient temperature. The absorbance of the reacting mixture was then spectrophotometrically measured at 593 nm. Ascorbic acid dissolved in distilled water was used as the standard for obtaining a calibration curve in a concentration range comprised between 0 and 2 mmol/L. The FRAP values were expressed as mmol of ascorbic acid equivalents (AAE) per 100 g of fresh-weight artichoke bracts ($mmol_{AAE}$/100 g FW).

2.7. Statistical analysis

All PEF treatments and analyses, unless otherwise specified, were performed in triplicate. The mean values and standard deviations (SD) of experimental data were calculated. Statistically significant differences ($p \leq 0.05$) among the averages were evaluated using a one-way analysis of variance (ANOVA) and Tukey's test. The Pearson product-moment correlation coefficient was used to measure the strength of the linear relationship between two variables. Statistical analyses were carried out using the SPSS 20 (SPSS Inc., Chicago, USA) statistical package. SigmaPlot 12.0 (Systat Software, Inc) was used for nonlinear regression analysis by Eq. (3) to assess the effects of comminution degree and PEF processing conditions on the kinetic parameters v_0 and TPC_∞. The goodness of model fitting was evaluated by calculating the determination coefficient (R^2).

3. Results and discussions

3.1. Effect of comminution and PEF treatment on the electrical behavior of artichoke bract tissues

Measurements of complex electrical impedance were utilized to characterize the electrical behavior and the extent of cell membrane disruption of artichoke bract tissues resulting from either mechanical comminution (cutting) operation, or its combination with PEF treatment.

Fig. 2a depicts the frequency impedance spectra of artichoke bracts after single cutting operation in discs of different sizes (0.5–3 cm in diameter). The results show that the absolute impedance value of the largest discs of artichoke bract tissue is strongly frequency-dependent showing a characteristic sigmoidal behavior as a result of β dispersion (i.e. the phenomenon associated with the polarization of the cell membrane) (Genovese et al., 2021). This is because, in the low-frequency field (0.1–1 kHz), the large proportion of intact cell membranes likely remained in the larger bract discs acts as a capacitor preventing the flow of the electric current in the intracellular medium (ohmic-capacitive behavior). With increasing frequency, the cell membrane becomes less and less resistant to the current flow. At very high-frequency values (0.1–1 MHz), the membrane is shorted out and the absolute value of the complex impedance is representative of the contribution of both the extra- and intracellular medium (pure ohmic behavior). Hence, the tissue permeabilization induced by external stresses such as mechanical comminution or PEF treatment is detectable in the low frequency range. In the high-frequency range, because the cell membranes exhibit no or negligible resistance to the current flow, there is practically no difference between the impedance of intact cells and cells with ruptured membranes (Adekanmbi and Srivastava, 2019; Battipaglia et al., 2009; Carpentieri et al., 2022b, 2023; Donsì et al., 2010a, 2010b; Pataro et al., 2009; Sui et al., 2020).

The findings presented in Fig. 2a also show the transition from an almost intact to a ruptured state as the size of the artichoke discs diminishes. This is because the size reduction of the artichoke discs below 3 cm in diameter likely reduces the number of intact cells at the cutting area (Jaeger et al., 2012). Consequently, a gradual reduction in the impedance modulus of the samples is observed at low-frequency values (0.1–1 kHz), indicating a diminishing capacitive behavior as the degree of comminution increases. This trend persists until reaching an almost flat profile for the smallest investigated disc sizes (D = 0.5–0.8 cm). Consequently, in such conditions, the electrical characteristics of the vegetable tissue undergo a transition from an ohmic-capacitive behavior to a purely ohmic one, signifying a substantial disruption in cell membrane integrity.

Fig. 2b–g shows the frequency-impedance spectra achieved after PEF treatments of artichoke discs of different sizes (D = 0.5–3 cm) at a fixed field strength (3 kV/cm) and for different total specific energy inputs (1–20 kJ/kg). Similar trends were also obtained for the other field

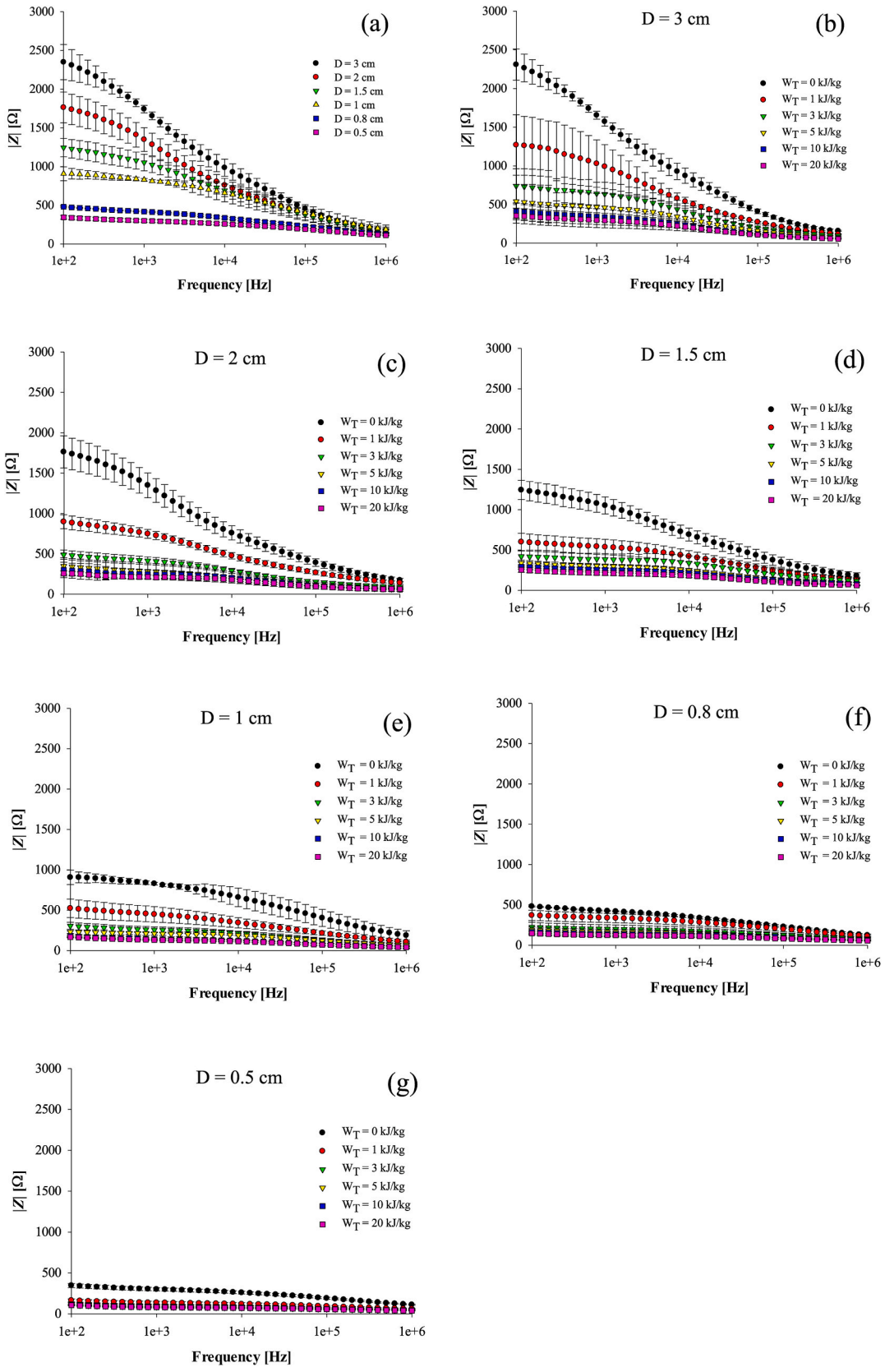

Fig. 2. Frequency dependency of the absolute value of complex impedance (|Z|) for artichoke bract discs achieved after (a) single comminution operation at different discs diameter, and (b–g) subsequent PEF treatment at constant field strength (E = 3 kV/cm) and different total specific energy inputs (W_T = 1–20 kJ/kg) with discs diameter set at (b) 3 cm, (c) 2 cm, (d) 1.5, (e) 1 cm, (f) 0.8 cm, (g) 0.5 cm. n = 9.

strengths (0.5, 1, and 5 kV/cm) investigated (data not shown). The application of a PEF treatment led to a significant lowering of the impedance value due to the further loss of the capacitive properties of the cell membrane after electropermeabilization (Carpentieri et al., 2022b; Genovese et al., 2021). However, the impedance value experiences a more significant decrease after PEF treatment in the case of samples with larger sizes. For example, the application of the PEF treatment at 3 kV/cm and 5 kJ/kg reduced the absolute value of the complex impedance detected at 1 kHz from 1655 Ω to 497 Ω for bract discs of 3 cm in diameter (Fig. 3b), and from 299 Ω to 88 Ω for samples of 0.5 cm in diameter (Fig. 3g). This effect can be attributed to the presence of a higher number of intact cells within the larger bract discs available for electroporation. Moreover, for any field strength applied, the absolute value of the complex impedance decreased as long as the energy input increased, especially for values greater than 1 kJ/kg. On the other hand, data not shown revealed that for the same energy input, the higher the field strength applied, the greater the membrane permeabilization effect, which was more effective for field strength above 0.5 kV/cm and when the PEF treatment was applied to the larger particles.

Overall, the outcomes presented here provide clear evidence of the potential of single mechanical comminution or its combination with PEF treatment in altering the electrical behavior of artichoke bract tissues by inducing the disruption of the cell membrane to an extent that depends on the treatment intensity.

3.2. *Effect of comminution and PEF treatment on the cell disintegration index of artichoke bract tissues*

The impedance measurements (Fig. 2) were used for the evaluation of the cell disintegration index (Z_p) according to Eq. (2), which has been widely demonstrated to be a reliable indicator to quantify the proportion of damaged cells in terms of cell membrane disruption in diverse agri-food by-products tissues (Bobinaitė et al., 2015; Carpentieri et al., 2022b, 2023; Frontuto et al., 2019; Pataro et al., 2019, 2020). For the Z_p calculation, the almost intact tissue associated with the untreated (W_T = 0 kJ/kg) artichoke bract discs of 3 cm in diameter was used as the reference (control) sample (Z_p = 0).

Results are plotted in Fig. 3 as cumulative cell disintegration index achieved after the comminution operation of artichoke bracts in discs of different sizes (D = 3–0.5 cm), and subsequent PEF treatment at different field strength and energy input. A Z_p value of only 0.18 ± 0.08 was found for untreated artichoke bracts cut in discs of 2 cm in diameter, indicating that only a minor part of the plant cells (18%) was destroyed upon mild cutting operation applied alone (Fig. 3b). Increasing the

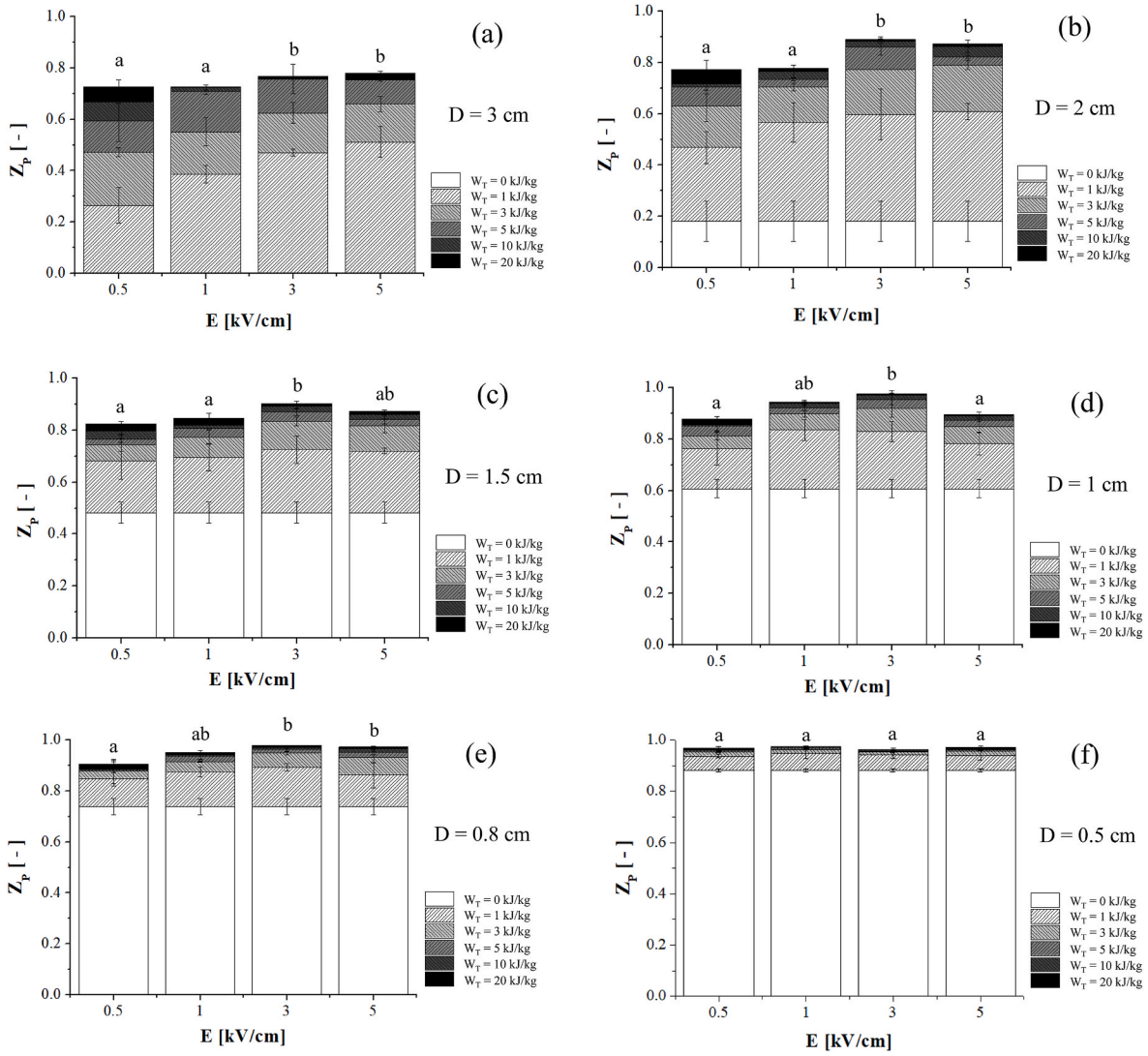

Fig. 3. Cumulative cell disintegration index (Z_P) of artichoke bract tissues after comminution to discs of (a) 3 cm, (b) 2 cm, (c) 1.5 cm, (d) 1 cm, (e) 0.8 cm, and (f) 0.5 cm in diameter and subsequent PEF treatment at different intensity (E = 0.5–5 kV/cm; W_T = 1–20 kJ/kg). Different letters above the bars indicate significant differences among the mean values ($p \leq 0.05$). n = 9.

degree of comminution intensity resulted in bract discs of smaller sizes and therefore in a higher degree of mechanical cell disruption. As a result, the Z_p value of untreated artichoke bracts cut in discs of 1.5, 1.0, 0.8, and 0.5 cm in diameter, increased up to 0.48 ± 0.04 (Fig. 3c), 0.61 ± 0.04 (Fig. 3d), 0.74 ± 0.03 (Fig. 3e), and 0.88 ± 0.01 (Fig. 3f), respectively.

By applying PEF treatment at different field strengths (0.5–5 kV/cm) and energy inputs (1–20 kJ/kg), additional cell disintegration could be achieved in a sample size-dependent manner (Fig. 3a–f). For instance, when subjecting artichoke bract tissues cut into 3 cm diameter discs ($Z_p = 0$) to PEF treatment at 3 kV/cm and 20 kJ/kg, a Z_p value of 0.77 was obtained (Fig. 3a). Upon reducing the diameter of the bracts to 2.0, 1.5, 1.0, 0.8, and 0.5 cm, in addition to the cell damage incurred from mechanical comminution (Fig. 3b–f), further membrane disruption was observed following PEF treatment. However, the incremental increase in Z_p values diminished to 0.71 (Fig. 3b), 0.42 (Fig. 3c), 0.37 (Fig. 3d), 0.24 (Fig. 3e), and 0.08 (Fig. 3f), respectively. The more pronounced increase in cell disruption observed in mildly comminuted samples might be ascribed to the greater number of intact cells available for electroporation in the larger bract discs (Jaeger et al., 2012). However, as the comminution process becomes more intense, a greater proportion of cells undergoes mechanical damage. Consequently, the contribution of the subsequent PEF treatment to Z_p becomes progressively less significant, regardless of the applied treatment intensity. This is corroborated by the findings of Fig. 3f, which shows that the majority of the plant cells (88% on average) were destroyed when the highest comminution intensity was applied, and applying the PEF treatment yielded either no or only marginal (9% on average) additional cell disintegration ($p > 0.05$).

Additionally, results reveal that the extent of cell membrane permeabilization increased with increasing the field strength and energy input, even though the effect of energy input appeared slightly more pronounced than that of the field strength. In particular, according to previous findings (Barba et al., 2015; Carpentieri et al., 2021b, 202; Fincan, 2015; Frontuto et al., 2019; Luengo et al., 2013; Pataro et al., 2019), regardless of the field strength applied, the extent of cell membrane permeabilization significantly ($p \leq 0.05$) increased with the augmentation of energy input, reaching a saturation point at approximately 5 kJ/kg. Beyond this threshold, no additional significant ($p > 0.05$) cellular damages could be detected. The only exception was observed in mild comminuted samples with diameters of 3 cm, 2 cm, and 1.5 cm when exposed to the lowest field strength (0.5 kV/cm). In these cases, a significant ($p \leq 0.05$) increase in Z_p value was noted when the energy input was raised from 5 to 20 kJ/kg (Fig. 3a–c). On the other hand, results of ANOVA revealed that, at any given energy input and for samples ranging in size from 2 cm to 0.8 cm in diameter (Fig. 3a–e), significant differences ($p \leq 0.05$) in the Z_p value were detected only when the field strength increased from 0.5 to 3 kV/cm for bract discs of 3, 2 and 1.5 cm in diameter, and from 1 to 3 kV/cm for bract discs of 1 and 0.8 cm in diameter. However, applying PEF treatment at the highest field strength (5 kV/cm) did not result in additional increases ($p > 0.05$) in the extent of cellular damages. For the smallest artichoke discs (D = 0.5 cm), no significant differences ($p > 0.05$) in Z_p values were detected within the explored spectrum of field strengths, regardless of the specific energy input delivered to the sample (Fig. 3f). It is also worth noting that artichoke discs of 2 cm in diameter subjected to PEF treatment at 3 kV/cm and 5 kJ/kg (Fig. 3b) had a similar level of cell disintegration ($Z_p = 0.88$) as the smallest discs (0.5 cm in diameter) after single comminution pre-treatment (Fig. 3f). These findings emphasize the potential of PEF as a gentle alternative or complementary approach to traditional mechanical cell disintegration methods that rely on reducing the size of raw material. PEF offers the advantage of eliminating the necessity to manage fine-grinded samples, thereby preventing potential complications in subsequent separation and purification processes.

Based on the results of Fig. 3, 3 kV/cm and 5 kJ/kg were selected as the optimal PEF treatment conditions to be used as a pre-treatment of artichoke bract discs before the subsequent extraction process. Interestingly, the energy consumption needed for the effective electropermeabilization of the studied bract tissues represents a negligible fraction, approximately 2.5%, compared to the energy typically used in the comminution operations of wet matrices, as highlighted by Galanakis (2015).

The general trend of the influence of electric field strength and total specific energy input on Z_p values observed in this research is somehow consistent with previously reported data for other plant tissues, where a dependency on the electric field strength and the energy input was identified (Carpentieri et al., 2022b, 2023; Frontuto et al., 2019; Pataro et al., 2019). However, through a comprehensive literature survey, it was found that only Battipaglia et al. (2009) investigated the impact of PEF treatment on the degree of cell disintegration in artichoke bract tissues. The authors found that the application of PEF treatment, carried out under milder (0.8 kV/cm and 1 kJ/kg) or more intense (1.6 kV/cm and 5 kJ/kg) conditions, increased the permeabilization degree (Z_p) of 2 cm-thick strips of artichoke bracts tissues up to 0.5 and 0.9, respectively, which is consistent with results achieved in this work.

Furthermore, data are very limited regarding the level of cell disintegration in fruits or vegetables upon cutting and grinding pre-treatment applied alone or in combination with PEF treatment to intensify the subsequent extraction processes of juice or valuable compounds from plant tissues. As an example, Angersbach et al. (1999) found a Z_p value of 0.55 for coarsely ground potato (strips with a cross-section area of 2.5 × 0.6 mm) and 0.84 for fine-grained potato (particle size 0.3–0.7 mm), thus corroborating the remarkable influence of the size reduction of plant tissue on the degree of cell disruption detected in this work. However, only in the work of Jaeger et al. (2012) the mutual interaction between mechanical grinding and PEF treatment on cell disruption was studied, with the aim to intensify the recovery yield of juice during PEF-assisted pressing from apple mash of different size (2, 5, and 9 mm). Results showed that mash of 2 mm in size (fine grinding) had already experienced significant cell destruction (86%) through grinding, making PEF treatment unnecessary. On the other hand, mash with larger particles (5 and 9 mm), and more intact cells, led to a Z_p value of 0.48 and 0.51, respectively, thus showing potential for further cell disintegration by PEF treatment.

3.3. Effect of comminution degree and PEF pre-treatment on TPC extraction kinetic and antioxidant activity

Fig. 4 illustrates the impact of comminution degree alone (Fig. 4a) or in combination with PEF treatment at fixed electric field strength (3 kV/cm) and total specific energy input (5 kJ/kg) (Fig. 4b) on the extraction kinetics of total phenolic content (TPC) from artichoke bract discs (D = 0.5–3 cm) in water. The kinetic experimental data from untreated and PEF-treated artichoke bract discs were fitted by Peleg's model (Eq. (3)). The calculated parameters of this model, namely v_0 and TPC_∞, and values of determination coefficients R^2 are presented in Table 1. A higher v_0 value in Eq. (3) indicates a faster extraction rate, while a greater TPC_∞ value suggests a higher extraction yield (Pataro et al., 2020). The determination coefficients ranged from 0.904 to 0.993, thus indicating that Peleg's model could be reasonably applied to predict the extraction rate of TPC in water. This is in agreement with previous findings on the extraction of intracellular compounds like polyphenols or carotenoids from different plant tissues (Bucic-Kojic et al., 2007; Carpentieri et al., 2021b; Odriozola-Serrano et al., 2008; Pataro et al., 2020; Poojary and Passamonti, 2015).

Furthermore, Fig. 4 demonstrates that, regardless of the cell disintegration pre-treatment application, the TPC extraction was highly dependent on the extraction time. Initially, TPC increased rapidly as the solvent penetrated the solid matrix, driven by the high concentration gradient between the solid and liquid phases (Pataro et al., 2020). However, the extraction rate gradually decreased over time due to the diminishing driving force and the decrease in analyte concentration in the solid phase (Poojary and Passamonti, 2015), eventually reaching an

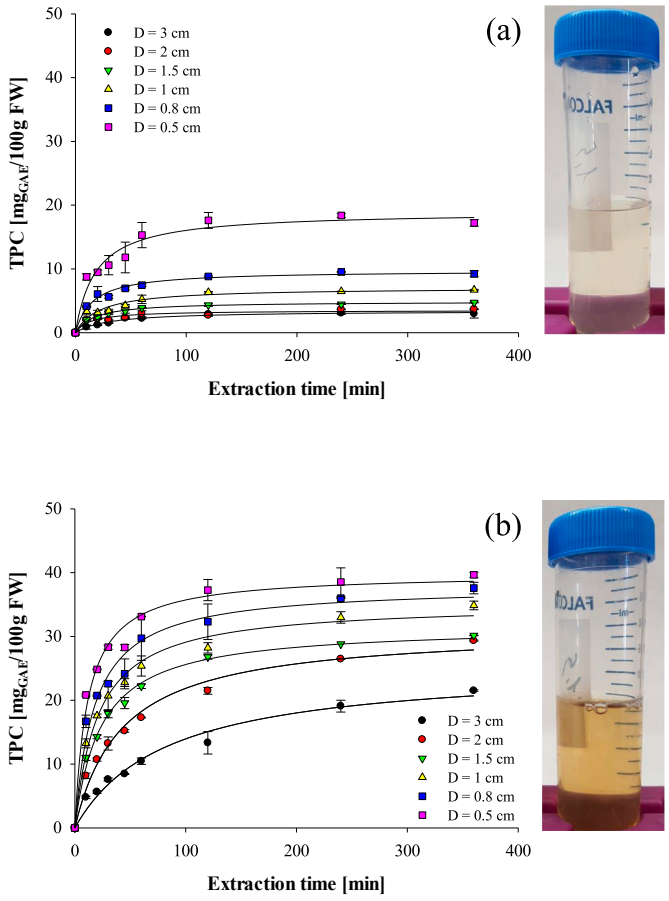

Fig. 4. Extraction kinetics of total phenolic content (TPC) in water from (a) untreated and (b) PEF (3 kV/cm and 5 kJ/kg)-treated artichoke bract discs of variable size (D = 0.5–3 cm). The extraction temperature was set at 25 °C. The symbols represent experimental data, whereas the curves arise from fitting using Peleg's model (Eq. (3)). n = 9. The inserts depict the pictures of the extracts after 120 min of extraction from untreated (Fig. 4a) and PEF (3 kV/cm, 5 kJ/kg)-treated (Fig. 4b) artichoke bract discs (D = 3 cm).

almost equilibrium condition. Most of the phenolic compounds were recovered in approximately 120 min of extraction, with longer diffusion times yielding no substantial increment in the total phenolics' amount, regardless of the bract disc size and cell disintegration method applied.

Interestingly, from the results of Fig. 4, it appears that water is a good extraction solvent because it can dissolve a substantial amount of phenolic compounds. This is corroborated by the findings of Battipaglia et al. (2009), who found that the aqueous extraction of phenolic compounds from artichoke bracts tissue led to similar recovery yields as those detected when hydro-alcoholic mixtures were used, thus avoiding the need for organic solvents, and contributing to a more sustainable and "green" process. However, the extraction kinetic in water of phenolic compounds, originally located in the intracellular space of artichoke cell tissues, is significantly dependent on the bract disc size and on the applied PEF treatment. Specifically, regardless of the extraction time and PEF application, a gradual and significant ($p \leq 0.05$) enhancement in the diffusion rates of phenolic compounds in the extracts could be detected with increasing the intensity of the comminution process, due to the consequent increase in the surface–volume ratio of the samples and partial cell disintegration induced at the cutting area (Fig. 4a). Notably, the artichoke bract discs of 2 and 3 cm in diameter exhibited a relatively low surface–volume ratio and contained a substantial proportion of intact cells. Consequently, the extraction of phenolic compounds in water was quite slow, with a limited recovery yield even after long diffusion times (Fig. 4a). In contrast, higher comminution

Table 1
Initial extraction rate (v_0) and maximum total phenolic content (TPC_∞) of extracts from untreated and PEF (E = 3 kV/cm, W_T = 5 kJ/kg)-treated artichoke bract discs of variable size (D = 0.5–3 cm), obtained by fitting the experimental data of TPC extraction kinetics (Fig. 4) with Peleg's model (Eq. (3)).

Sample	E (kV/cm)	W_T (kJ/kg)	Size (cm)	v_0 (mg_{GAE}/100 g FW min)	TPC_∞ (mg_{GAE}/100 g FW)	R^2
Untreated	0	0	0.5	1.04 ± 0.07^{d}	18.97 ± 0.44^{e}	0.957
			0.8	0.62 ± 0.05^{c}	9.73 ± 0.37^{d}	0.980
			1	0.32 ± 0.01^{b}	7.05 ± 0.23^{c}	0.941
			1.5	0.28 ± 0.01^{ab}	4.88 ± 0.21^{b}	0.990
			2	0.25 ± 0.01^{ab}	3.51 ± 0.17^{a}	0.904
			3	0.11 ± 0.01^{a}	3.42 ± 0.08^{a}	0.981
PEF-treated	3	5	0.5	3.40 ± 0.14^{h}	39.91 ± 0.69^{l}	0.984
			0.8	2.15 ± 0.08^{g}	37.32 ± 0.54^{i}	0.978
			1	1.68 ± 0.12^{f}	35.18 ± 0.14^{h}	0.987
			1.5	1.34 ± 0.04^{e}	31.63 ± 0.27^{g}	0.993
			2	0.73 ± 0.03^{c}	31.20 ± 0.38^{g}	0.983
			3	0.31 ± 0.01^{b}	25.42 ± 0.11^{f}	0.975

Data are means ± standard deviations (n = 9). Different lowercase letters in the same column indicate significant differences among the mean values ($p \leq 0.05$).

intensities led to the generation of smaller bract discs, resulting in a more pronounced mechanical disruption of cells (Fig. 3). As a result, the extraction rate of phenolic compounds exhibited a gradual increment, culminating in a TPC value of 17.6 ± 1.2 mg_{GAE}/100 g FW in extracts derived from the smallest bract discs (0.5 cm in diameter). This level was approximately six times higher compared to the observed TPC value for the largest bract discs (3 cm in diameter), which measured 2.89 ± 0.06 mg_{GAE}/100 g FW after 120 min of water diffusion.

Furthermore, in agreement with the results shown in Fig. 3, the additional cell membrane permeabilization induced by the application of PEF (3 kV/cm - 5 kJ/kg) treatment positively influenced both the extraction rates and yields of phenolic compounds, which resulted significantly ($p \leq 0.05$) higher compared to the untreated samples, regardless of the bract size (Fig. 4b and Table 1). For instance, in the case of 3 cm diameter bract discs, the amount of phenolic compounds recovered after 120 min of extraction was 2.89 ± 0.06 mg_{GAE}/100 g FW for untreated samples, whereas it increased to 13.33 ± 1.76 mg_{GAE}/100 g FW for PEF-treated samples. This significant difference is visually corroborated by the inserts of Fig. 4, where the appearance of the extracts after 120 min of extraction for untreated and PEF-treated artichoke bract discs (D = 3 cm) is shown. The color intensity of the extract is closely related to the content of phenolic compounds.

However, in accordance with previous research (Jaeger et al., 2012; Turk et al., 2010), the effect of PEF treatment was more pronounced for larger bract discs due to the higher number of intact cells remained in the artichoke tissue, which are susceptible to cell disintegration during PEF treatment (Figs. 3–4).

As per the literature survey, the impact of a PEF pre-treatment on polyphenol extractability from artichoke wastes has been investigated only by Battipaglia et al. (2009). In this preliminary work, the authors observed that PEF (0.8–1.6 kV/cm, 1–5 kJ/kg)-induced permeabilization of the cell membranes significantly accelerated the release of polyphenolic compounds from the outer bracts of artichokes, leading to higher extractability of TPC (+30–150%) as compared with SLE extraction of untreated samples. These results perfectly align with the outcomes obtained in the present work (Fig. 4 and Table 1).

However, only a few works investigated the combination of size reduction and PEF pre-treatment for extracting valuable compounds from plant tissues, but none of them dealt with artichoke wastes. Nevertheless, in line with our findings, Bouras et al. (2016) found that PEF treatment of Norway spruce bark slices (10 mm in length) significantly enhanced the aqueous SLE of phenolic compounds compared to control extraction, yielding results comparable to sawdust diffusion (1.2 mm in diameter). Other researchers have demonstrated that the application of PEF treatment before pressing lemon residues (Peiró et al., 2019) or apple mash (Turk et al., 2010) of various sizes significantly increased the release of phenolic compounds. However, partially in contrast with our results, the impact of size reduction on phenolic compound extraction yield was observed only in untreated samples, with no significant differences detected among PEF-treated samples of different sizes. This suggests that PEF pre-treatment could eliminate the need for grinding or cutting procedures in intensifying industrial processes for plant-based materials.

Table 2 displays the antioxidant activity (FRAP) of extracts obtained from both untreated and PEF-treated (3 kV/cm - 5 kJ/kg) artichoke bract discs after 120 min of water diffusion, as a function of the sample size (D = 0.5–3 cm) and the corresponding percentage increase upon the application of PEF pre-treatment. Additionally, the table includes the TPC values previously presented in Fig. 4 to emphasize the relationship between phenolic content and the antioxidant activity of the extracts. The results reveal that, in alignment with the respective TPC values, the FRAP values for both untreated and electropermeabilized samples exhibit a significant increase ($p \leq 0.05$) as the degree of comminution increases. Specifically, significant differences ($p \leq 0.05$) in the FRAP values were observed when the size of the bract discs decreased from 1 cm to 0.5 cm for untreated samples, and from 3 cm to 1.5 cm and from 1.5 cm to 0.5 cm for PEF-treated samples.

Furthermore, as reported in Table 2, extracts obtained from PEF-treated samples exhibited significantly ($p \leq 0.05$) higher TPC (+112–361%) and antioxidant activity (+83–836%) as compared to extracts from untreated samples. However, the difference in FRAP between extracts from untreated and electrically permeabilized bract discs tended to decrease at smaller sizes, likely due to the reduced difference in TPC at smaller sizes between untreated and PEF-treated samples (Fig. 4 and Table 2). The maximum FRAP values were detected when the smallest discs were considered, with (2.28 ± 0.41 $mmol_{AAE}$/100 g FW) or without (1.25 ± 0.04 $mmol_{AAE}$/100 g FW) the application of PEF treatments.

It is worth noting that, coherently with the findings presented in Fig. 4b, the antioxidant activity of the extracts obtained after PEF treatment of the largest bract discs (1.34 ± 0.02 $mmol_{AAE}$/100 g FW) appeared to be comparable to that achieved from untreated samples of the smallest size (1.25 ± 0.04 $mmol_{AAE}$/100 g FW). These observations align with the study conducted by Bouras et al. (2016). The authors observed that the antioxidant activity of aqueous extracts from Norway spruce bark slices (10 mm in length) was approximately 30 times higher than that of the untreated samples but was nearly the same for PEF treatment and smallest particles (i.e., sawdust).

3.4. Correlation analysis

The correlation between the Z_p of artichoke bract tissues, TPC, and FRAP of extracts from untreated and PEF-treated samples of various sizes is summarized in Table 3. Notably, the results indicate a strong correlation between the degree of size reduction and cell membrane disintegration caused by mechanical comminution with the TPC in the corresponding extracts (Fig. 4a–Table 3). However, a more robust correlation was observed between TPC and Z_p values when PEF was applied in combination with the comminution step (Fig. 4b–Table 3). This finding suggests that the combined treatment has the potential to further enhance the degree of cell membrane permeabilization in artichoke tissues, thereby facilitating solvent penetration and the recovery of phenolic compounds.

Additionally, in line with previous findings (Bouras et al., 2016), a significantly positive correlation was found between TPC and FRAP values of artichoke bract extracts (Table 3). This observation implies that the phenolic compounds present in artichoke bract tissues play a crucial role in contributing to the overall antioxidant activity of the extracts.

4. Conclusions

This study demonstrated the effectiveness of combining PEF technology with gentle mechanical size reduction (cutting) to disrupt cell membranes in artichoke bract tissues without requiring finely ground raw material handling. The degree of cell disruption depended on the treatment intensity and sample size, offering the potential to optimize PEF's impact on the extractability of phenolic compounds.

Enhancing the intensity of the comminution process resulted in improved diffusion rates by inducing partial cell disintegration. This, in turn, led to higher yields in TPC and increased antioxidant activity in the extracts. However, PEF application further accelerated the release of phenolic compounds, resulting in substantially higher yields. Interestingly, the effect of PEF on the extraction rate was mitigated by the increase in cutting pre-treatment intensity, as smaller-sized samples had fewer intact cells susceptible to PEF-induced disruption. The optimal PEF treatment conditions were found at 3 kV/cm and 5 kJ/kg for artichoke bract discs with a diameter ranging from 3 to 0.8 cm, while for the smallest-sized bract discs, PEF appeared ineffective. Operating under these optimal conditions significantly increased TPC (5-fold on average) and antioxidant activity (6-fold on average) as compared to the untreated samples. For larger bract discs (2–3 cm), PEF treatment yielded similar TPC and FRAP values as extracts from smaller discs (0.5 cm) after a single comminution pre-treatment.

In conclusion, PEF application reduces the need for energy-intensive mechanical size reduction of plant biomass, enabling the processing of larger-sized samples while preserving the integrity of valuable compounds and facilitating downstream separation and purification phases,

Table 2
Total phenolic content (TPC) and antioxidant activity (FRAP) of extracts from untreated and PEF (E = 3 kV/cm, W_T = 5 kJ/kg)-treated artichoke bract discs of variable size (D = 0.5–3 cm). Percentage increases in TPC and FRAP values experienced by PEF-treated samples as compared to untreated ones are also presented. Extraction time and temperature were set at 120 min and 25 °C, respectively.

D (cm)	$TPC_{untreated}$	$TPC_{PEF\text{-}treated}$	TPC increase after PEF (%)	$FRAP_{untreated}$	$FRAP_{PEF\text{-}treated}$	FRAP increase after PEF (%)
	(mg_{GAE}/100 g FW)			($mmol_{AAE}$/100 g FW)		
0.5	$17.61 \pm 1.24^{e,A}$	37.26 ± 1.66^{iB}	112	$1.25 \pm 0.04^{cd,A}$	$2.28 \pm 0.41^{h,B}$	83
0.8	$8.81 \pm 0.11^{c,A}$	$32.31 \pm 2.77^{h,B}$	267	$0.96 \pm 0.02^{bc,A}$	$2.10 \pm 0.36^{gh,B}$	119
1	$6.33 \pm 0.10^{bc,A}$	$28.21 \pm 0.81^{g,B}$	355	$0.55 \pm 0.02^{ab,A}$	$1.95 \pm 0.15^{gh,B}$	254
1.5	$4.32 \pm 0.20^{ab,A}$	$26.78 \pm 0.35^{g,B}$	520	$0.57 \pm 0.02^{ab,A}$	$1.80 \pm 0.03^{fg,B}$	216
2	$2.72 \pm 0.14^{a,A}$	$21.48 \pm 0.52^{f,B}$	690	$0.33 \pm 0.02^{a,A}$	$1.54 \pm 0.12^{ef,B}$	367
3	$2.89 \pm 0.06^{a,A}$	$13.33 \pm 1.76^{d,B}$	361	$0.14 \pm 0.01^{a,A}$	$1.34 \pm 0.02^{de,B}$	836

Data are means ± standard deviations (n = 9). Different lowercase letters in the same column indicate significant differences among the mean values ($p \leq 0.05$). For each tested response, different uppercase letters in the same row indicate significant differences between the mean values ($p \leq 0.05$).

Table 3
Correlation coefficient among cell disintegration index (Z_p) of artichoke bract tissues, total phenolic content (TPC), and antioxidant activity (FRAP) of extracts from untreated and PEF (E = 3 kV/cm, W_T = 5 kJ/kg)-treated artichoke bract discs of different sizes (D = 0.5–3 cm). Extraction time and temperature were set at 120 min and 25 °C, respectively.

	Z_p	TPC (untreated)	TPC (PEF-treated)	FRAP (untreated)	FRAP (PEF-treated)
Z_p	1	0.833	0.975	0.945	0.934
TPC (untreated)	0.833	1	NA	0.923	NA
TPC (PEF-treated)	0.975	NA	1	NA	0.984
FRAP (untreated)	0.945	0.923	NA	1	NA
FRAP (PEF-treated)	0.934	NA	0.984	NA	1

NA means Not Applicable.

thereby enhancing overall extraction efficiency.

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CRediT authorship contribution statement

Daniele Carullo: Conceptualization, Data curation, Investigation, Writing - original draft. **Serena Carpentieri:** Data curation, Formal analysis, Investigation, Writing - original draft. **Giovanna Ferrari:** Conceptualization, Funding acquisition, Supervision, Writing - review & editing. **Gianpiero Pataro:** Conceptualization, Funding acquisition, Supervision, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

Adekanmbi, E.O., Srivastava, S.K., 2019. Dielectric characterization of bioparticles via electrokinetics: the past, present, and the future. Appl. Phys. Rev. 6 (4) https://doi.org/10.1063/1.5113709.

Ameer, K., Shahbaz, H.M., Kwon, J.H., 2017. Green extraction methods for polyphenols from plant matrices and their byproducts: a review. Compr. Rev. Food Sci. Food Saf. https://doi.org/10.1111/1541-4337.12253.

Angersbach, A., Heinz, V., Knorr, D., 1999. Electrophysiological model of intact and processed plant tissues: cell disintegration criteria. Biotechnol. Prog. 15 (4), 753–762. https://doi.org/10.1021/bp990079f.

Arshad, R.N., Abdul-Malek, Z., Roobab, U., Munir, M.A., Naderipour, A., Qureshi, M.I., El-Din Bekhit, A., Liu, Z.W., Aadil, R.M., 2021. Pulsed electric field: a potential alternative towards a sustainable food processing. Trends Food Sci. Technol. 111, 43–54. https://doi.org/10.1016/j.tifs.2021.02.041.

Ayala-Zavala, J.F., Vega-Vega, V., Rosas-Domínguez, C., Palafox-Carlos, H., Villa-Rodriguez, J.A., Siddiqui, W., Dávila-Aviña, J.E., González-Aguilar, G.A., 2011. Agro-industrial potential of exotic fruit byproducts as a source of food additives. Food Res. Int. 44, 1866–1874.

Barba, F.J., Parniakov, O., Pereira, S.A., Wiktor, A., Grimi, N., Boussetta, N., Saraiva, J. A., Raso, J., Martin-Belloso, O., Witrowa-Rajchert, D., Lebovka, N., Vorobiev, E., 2015. Current applications and new opportunities for the use of pulsed electric fields in food science and industry. Food Res. Int. 77, 773–798. https://doi.org/10.1016/j.foodres.2015.09.015.

Battipaglia, G., De Vito, F., Donsì, F., Pataro, G., Ferrari, G., 2009. Enhancement of polyphenols extraction from involucral bracts of artichokes. In: Proceeding of the International Conference Bio & Food Electrotechnologies – BFE 2009. Université de Technologie de Compiegne, Compiegne, France, pp. 40–44, 978-2-913923-31-7.

Benzie, I.F., Strain, J.J., 1996. The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. Anal. Biochem. 239, 70–76.

Bobinaitė, R., Pataro, G., Lamanauskas, N., Šatkauskas, S., Viškelis, P., Ferrari, G., 2015. Application of pulsed electric field in the production of juice and extraction of bioactive compounds from blueberry fruits and their by-products. J. Food Sci. Technol. 52 (9), 5898–5905. https://doi.org/10.1007/s13197-014-1668-0.

Bouras, M., Grimi, N., Bals, O., Vorobiev, E., 2016. Impact of pulsed electric fields on polyphenols extraction from Norway spruce bark. Ind. Crop. Prod. 80, 50–58. https://doi.org/10.1016/j.indcrop.2015.10.051.

Bucic-Kojic, A., Planinic, M., Tomas, S., Bilic, M., Velic, D., 2007. Study of solid–liquid extraction kinetics of total polyphenols from grape seeds. J. Food Eng. 81, 236–242. https://doi.org/10.1016/j.jfoodeng.2006.10.027.

Cao, X., Rosentrater, K.A., 2015. Energy Consumption of Corn Stover Size Reduction. https://doi.org/10.13031/aim.20152188476.

Carpentieri, S., Soltanipour, F., Ferrari, G., Pataro, G., Donsì, F., 2021a. Emerging green techniques for the extraction of antioxidants from agri-food by-products as promising ingredients for the food industry. Antioxidants 10, 1417. https://doi.org/10.3390/antiox10091417.

Carpentieri, S., Mazza, L., Nutrizio, M., Jambrak, A.R., Ferrari, G., Pataro, G., 2021b. Pulsed electric fields- and ultrasound-assisted green extraction of valuable compounds from Origanum vulgare L. and Thymus serpyllum L. Int. J. Food Sci. Technol. 1 https://doi.org/10.1111/ijfs.15159. –9.

Carpentieri, S., Augimeri, G., Ceramella, J., Vivacqua, A., Sinicropi, M.S., Pataro, G., Bonofiglio, D., Ferrari, G., 2022a. Antioxidant and anti-inflammatory effects of extracts from pulsed electric field-treated artichoke by-products in lipopolysaccharide-stimulated human THP-1 macrophages. Foods 11, 2250. https://doi.org/10.3390/foods11152250.

Carpentieri, S., Ferrari, G., Pataro, G., 2022b. Optimization of pulsed electric fields-assisted extraction of phenolic compounds from white grape pomace using response surface methodology. Front. Sustain. Food Syst. 6 https://doi.org/10.3389/fsufs.2022.854968.

Carpentieri, S., Ferrari, G., Pataro, G., 2023. Pulsed electric fields-assisted extraction of valuable compounds from red grape pomace: process optimization using response surface methodology. Front. Nutr. 10, 1158019 https://doi.org/10.3389/fnut.2023.1158019.

Christaki, E., Bonos, E., Florou-Paneri, P., 2012. Nutritional and functional properties of cynara crops (globe artichoke and cardoon) and their potential applications: a review. Int. J. Appl. Sci. Technol. 2 (2), 64–70. http://www.ttjbiotech.com/pdf/2012nutritional.pdf.

Donsì, F., Ferrari, G., Pataro, G., 2010a. Applications of pulsed electric field treatments for the enhancement of mass transfer from vegetable tissue. Food Eng. Rev. 2 (2), 109–130. https://doi.org/10.1007/s12393-010-9015-3.

Donsì, F., Ferrari, G., Fruilo, M., Pataro, G., 2010b. Pulsed electric field-assisted vinification of aglianico and piedirosso grapes. J. Agric. Food Chem. 58 (22), 11606–11615. https://doi.org/10.1021/jf102065v.

Fincan, M., 2015. Extractability of phenolics from spearmint treated with pulsed electric field. J. Food Eng. 162, 31–37. https://doi.org/10.1016/j.jfoodeng.2015.04.004.

Francavilla, M., Marone, M., Marasco, P., Contillo, F., Monteleone, M., 2021. Artichoke biorefinery: from food to advanced technological applications. Foods 10 (1). https://doi.org/10.3390/foods10010112.

Frontuto, D., Carullo, D., Harrison, S.M., Brunton, N.P., Ferrari, G., Lyng, J.G., Pataro, G., 2019. Optimization of pulsed electric fields-assisted extraction of polyphenols from potato peels using response surface methodology. Food Bioprocess Technol. 12 (10), 1708–1720. https://doi.org/10.1007/s11947-019-02320-z.

Galanakis, C.M., 2012. Recovery of high added-value components from food wastes: conventional, emerging technologies and commercialized applications. Trends Food Sci. Technol. 26, 68–87. https://doi.org/10.1016/j.tifs.2012.03.003.

Galanakis, C.M., 2015. In: Galanakis, C.M. (Ed.), Food Waste Recovery: Processing Technologies and Industrial Techniques, 978-0-12-800351-0.

Genovese, J., Kranjc, M., Serša, I., Petracci, M., Rocculi, P., Miklavčič, D., Mahnič-Kalamiza, S., 2021. PEF-treated plant and animal tissues: insights by approaching with different electroporation assessment methods. Innovat. Food Sci. Emerg. Technol. 74, 102872 https://doi.org/10.1016/j.ifset.2021.102872.

Gerschenson, L.M., Deng, Q., Cassano, A., 2021. Chapter 4 - conventional macroscopic pretreatment. In: Food Waste Recovery, second ed., pp. 71–87. https://doi.org/10.1016/B978-0-12-820563-1.00003-2.

Gominho, J., Curt, M.D., Lourenço, A., Fernández, J., Pereira, H., 2018. Cynara cardunculus L. as a biomass and multi-purpose crop: a review of 30 years of research. Biomass Bioenergy 109, 257–275. https://doi.org/10.1016/j.biombioe.2018.01.001.

Guida, V., Ferrari, G., Pataro, G., Chambery, A., Di Maro, A., Parente, A., 2013. The effects of ohmic and conventional blanching on the nutritional, bioactive compounds and quality parameters of artichoke heads. LWT -. Food Sci. Technol. 53 (2), 569–579. https://doi.org/10.1016/j.lwt.2013.04.006.

Ilahy, R., Tlili, I., R'him, T., Rached, Z., Arfaoui, K., Pék, Z., Lenucci, M.S., Daood, H., Helyes, L., 2022. Assessment of the phenolic and flavonoid content in certain globe artichoke (Cynara scolymus L.) cultivars grown in Northern Tunisia. Turk. J. Agric.-Food Sci. Technol. 10 (6), 1125–1129. https://doi.org/10.24925/turjaf.v10i6.1125-1129.4921.

Jaeger, H., Schulz, M., Lu, P., Knorr, D., 2012. Adjustment of milling, mash electroporation and pressing for the development of a PEF assisted juice production in industrial scale. Innovative Food Sci. Emerging Technol. 14, 46–60. https://doi.org/10.1016/j.ifset.2011.11.008.

Lattanzio, V., Kroon, P.A., Linsalata, V., Cardinali, A., 2009. Globe artichoke: a functional food and source of nutraceutical ingredients. J. Funct.Foods 1, 131–144. https://doi.org/10.1016/j.jff.2009.01.002.

Lavecchia, R., Maffei, G., Paccassoni, F., Piga, L., Zuorro, A., 2019. Artichoke waste as a source of phenolic antioxidants and bioenergy. Waste Biomass Valorization 10 (10), 2975–2984. https://doi.org/10.1007/s12649-018-0305-y.

Llorach, R., Espín, J.C., Tomás-Barberán, F.A., Ferreres, F., 2002. Artichoke (Cynara scolymus L.) byproducts as a potential source of health-promoting antioxidant phenolics. J. Agric. Food Chem. 50, 3458–3464.

López-Salas, L., Borrás-Linares, I., Quintin, D., García-Gomez, P., Giménez-Martínez, R., Segura-Carretero, A., Lozano-Sánchez, J., 2021. Artichoke by-products as natural source of phenolic food ingredient. Appl. Sci. 11 (9) https://doi.org/10.3390/app11093788.

Luengo, E., Álvarez, I., Raso, J., 2013. Improving the pressing extraction of polyphenols of orange peel by pulsed electric fields. Innovative Food Sci. Emerging Technol. 17, 79–84. https://doi.org/10.1016/j.ifset.2012.10.005.

Martín-García, B., Tylewicz, U., Verardo, V., Pasini, F., Gómez-Caravaca, A.M., Caboni, M.F., Dalla Rosa, M., 2020. Pulsed electric field (PEF) as pre-treatment to improve the phenolic compounds recovery from brewers' spent grains. Innovative Food Sci. Emerging Technol. 64, 102402 https://doi.org/10.1016/j.ifset.2020.102402.

Moreira, S.A., Alexandre, E.M.C., Pintado, M., Saraiva, J.A., 2019. Effect of emergent non-thermal extraction technologies on bioactive individual compounds profile from different plant materials. Food Res. Int. 115, 177–190. https://doi.org/10.1016/j.foodres.2018.08.046.

Odriozola-Serrano, I., Soliva-Fortuny, R., Gimeno-Añó, V., Martín-Belloso, O., 2008. Modeling changes in health-related compounds of tomato juice treated by high-intensity pulsed electric fields. J. Food Eng. 89, 210–216. https://doi.org/10.1016/j.jfoodeng.2008.04.019.

Pataro, G., Donsì, G., Ferrari, G., 2009. The effect of conventional and ohmic heating on the permeability of cell membrane in vegetables tissue, BFE. In: Vorobiev, E., Lebovka, N., Van Hecke, E., Lanoiselle', J.L. (Eds.), International Conference on Bio and Food Electrotechnologies, Compiegne, France. Universitè de Technologie de Compiegne: Compiegne, France, pp. 246–250, 2009.

Pataro, G., Bobinaitė, R., Bobinas, Č., Šatkauskas, S., Raudonis, R., Visockis, M., Ferrari, G., Viškelis, P., 2017. Improving the extraction of juice and anthocyanins from blueberry fruits and their by-products by application of pulsed electric fields. Food Bioprocess Technol. 10 (9), 1595–1605. https://doi.org/10.1007/s11947-017-1928-x.

Pataro, G., Carullo, D., Ferrari, G., 2019. Effect of PEF pre-treatment and extraction temperature on the recovery of carotenoids from tomato wastes. Chem. Eng. Trans. 75, 139–144. https://doi.org/10.3303/CET1975024.

Pataro, G., Carullo, D., Falcone, M., Ferrari, G., 2020. Recovery of lycopene from industrially derived tomato processing by-products by pulsed electric fields-assisted extraction. Innovat. Food Sci. Emerg. Technol. 63, 102369 https://doi.org/10.1016/j.ifset.2020.102369.

Peiró, S., Luengo, E., Segovia, F., Raso, J., Almajano, M.P., 2019. Improving polyphenol extraction from lemon residues by pulsed electric fields. Waste Biomass Valorization 10 (4), 889–897. https://doi.org/10.1007/s12649-017-0116-6.

Peleg, M., 1988. An empirical model for the description of moisture sorption curves. J. Food Sci. 53, 1216–1219. https://doi.org/10.1111/j.1365-2621.1988.tb13565.x.

Poojary, M.M., Passamonti, P., 2015. Extraction of lycopene from tomato processing waste: kinetics and modelling. Food Chem. 173, 943–950. https://doi.org/10.1016/j.foodchem.2014.10.127.

Raso, J., Frey, W., Ferrari, G., Pataro, G., Knorr, D., Teissie, J., Miklavčič, D., 2016. Recommendations guidelines on the key information to be reported in studies of application of PEF technology in food and biotechnological processes. Innovat. Food Sci. Emerg. Technol. 37, 312–321. https://doi.org/10.1016/j.ifset.2016.08.003.

Singh, A., Orsat, V., Raghavan, V., 2012. A Comprehensive Review on Electrohydrodynamic Drying and High-Voltage Electric Field in the Context of Food and Bioprocessing. Drying Technology: An International Journal 30, 1812–1820. https://doi.org/10.1080/07373937.2012.708912.

Sui, J.1, Fofonker, F., Bhattacharya, D., Javanmard, M., 2020. Electrical impedance as an indicator of microalgal cell health. Sci. Rep. 10, 1251. https://doi.org/10.1038/s41598-020-57541-6.

Turk, M.F., Baron, A., Vorobiev, E., 2010. Effect of pulsed electric fields treatment and mash size on extraction and composition of apple juices. J. Agric. Food Chem. 58 (17), 9611–9616. https://doi.org/10.1021/jf1016972.

Zuorro, A., 2014. Response surface methodology analysis of polyphenol recovery from artichoke waste. Am. J. Appl. Sci. 11 (9), 1463–1471. https://doi.org/10.3844/ajassp.2014.1463.1471.

Zuorro, A., Maffei, G., Lavecchia, R., 2016. Reuse potential of artichoke (Cynara scolimus L.) waste for the recovery of phenolic compounds and bioenergy. J. Clean. Prod. 111, 279–284. https://doi.org/10.1016/j.jclepro.2015.06.011.