

Article

Novel Technologies Assisted Extraction of Bioactive Compounds from Orange Peels Waste

Varvara Andreou ^{1,*}, Achilleas Ntafoulis ², Konstantinos Panagiotis Masouras ¹, Marianna Giannoglou ¹,
Maria Giannakourou ², Petros Taoukis ² and George Katsaros ^{1,*}

¹ Institute of Technology of Agricultural Products, Hellenic Agricultural Organization–DEMETER, 14123 Lykovrissi, Attica, Greece; takmasouras@chem.uoa.gr (K.P.M.); giannoglou@chemeng.ntua.gr (M.G.)

² Laboratory of Food Chemistry and Technology, School of Chemical Engineering, National Technical University of Athens, 15772 Zografos, Attica, Greece; achilleasdaoulis@gmail.com (A.N.); mgian@chemeng.ntua.gr (M.G.); taoukis@chemeng.ntua.gr (P.T.)

* Correspondence: vandreou@chemeng.ntua.gr (V.A.); gkatsaros@elgo.gr (G.K.);
Tel.: +302102845940 (V.A. & G.K.)

Abstract

The study evaluated the conventional extraction (CE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and pulsed electric fields-assisted extraction (PEFAE) of bioactive compounds from orange peel waste. The effect of extraction time (0–120 min), ethanol concentration (0, 50 & 80%), and temperature (25–70 °C) were investigated, while response surface methodology (RSM) was applied for each process optimization. MAE achieved the highest total phenolic content (TPC) (6.36 mg/g w.m.) under optimized conditions (50 kJ microwave energy, 50% ethanol, 60 min), representing approximately 12% higher recovery compared to CE (TPC: 5.29 mg/g w.m.; 50 °C, 55% EtOH, 80 min). UAE (90% amplitude, 66.4 W/kg) resulted in the highest flavonoid recovery (0.48 mg/g w.m.) using 50% ethanol at 50 °C for 90 min, while PEFAE (4 kV/cm, 1000 pulses) for 75 min extraction time exhibited the same TPC yield as CE and the highest antioxidant activity (1.12 mg/g w.m.) using only water at room temperature. RSM analysis confirmed that ethanol concentration and extraction time significantly affected extraction performance. These findings demonstrate the potential of green extraction technologies for sustainable valorization of citrus processing waste and recovery of high added-value compounds.

Keywords: orange peels waste; ultrasound-assisted extraction; microwave-assisted extraction; pulsed electric fields-assisted extraction; response surface methodology; phenolic compounds



Academic Editor: Dario Donno

Received: 29 May 2026

Revised: 30 June 2026

Accepted: 1 July 2026

Published: 4 July 2026

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1. Introduction

The global production of citrus fruits, particularly oranges (*Citrus sinensis*), generates substantial quantities of peel waste, which account for approximately 30–40% of the total fruit mass [1]. These residues are primarily produced by the juice and food processing industries and are traditionally considered low-value agro-industrial byproducts. However, orange peel represents a rich source of bioactive compounds, including polyphenols, flavonoids, carotenoids, and essential oils, which exhibit significant antioxidant, antimicrobial, and health-promoting properties [2,3]. The valorization of citrus processing waste is of increasing importance, as it aligns with circular economy principles and contributes

to the sustainable production of added-value ingredients for applications in the food, nutraceutical, and pharmaceutical industries [4,5]. Among the various bioactive constituents of orange peel, polyphenols and flavonoids are of particular interest due to their strong antioxidant activity and associated biological effects. Consequently, the recovery of these compounds from citrus waste offers a promising and sustainable approach to produce natural antioxidants and functional ingredients [6]. In current industrial practice, citrus peel residues are typically subjected to drying before further utilization [7]. This process is highly energy intensive and contributes significantly to industrial operating costs. Furthermore, the dried peel is mainly used for low-value applications such as animal feed, resulting in the underutilization of valuable bioactive compounds present in the raw material [8]. As a result, a large portion of these compounds remain unexploited or may degrade during processing (usually the drying step). Therefore, the extraction of bioactive compounds prior to the drying stage represents a promising alternative approach, enabling the recovery of high-value compounds while improving the overall valorization of citrus processing byproducts. Conventional extraction (CE) techniques, such as solvent maceration and Soxhlet extraction, have been extensively employed for the recovery of bioactive compounds from citrus peel [9–12]. The extraction of bioactive compounds from plant matrices is influenced by several factors, including solvent type and polarity, extraction temperature, extraction time, particle size, and the physicochemical properties of the plant tissue [1,6,13]. Aqueous ethanol solutions are widely used due to their effectiveness in extracting a broad range of phenolic compounds while maintaining low toxicity and environmental impact [14]. However, these methods are often characterized by long extraction times, high solvent consumption, and relatively low extraction efficiency [15]. Furthermore, prolonged exposure to elevated temperatures may lead to the degradation of thermolabile compounds, reducing extract quality and antioxidant capacity [16]. These limitations have driven the development of alternative extraction technologies that are more efficient, environmentally friendly, and compatible with industrial applications. In recent years, green extraction technologies such as microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and pulsed electric fields-assisted extraction (PEFAE) have emerged as promising alternatives to conventional methods [16–18]. These technologies enhance extraction efficiency primarily by improving mass transfer and facilitating the release of intracellular compounds. Microwave-assisted extraction utilizes electromagnetic radiation to generate rapid volumetric heating through dipole rotation and ionic conduction, resulting in increased internal pressure within plant cells, cell wall disruption, and enhanced solute diffusion. This process facilitates the release of intracellular compounds into the surrounding solvent and significantly reduces extraction time [19]. Ultrasound-assisted extraction employs acoustic cavitation, a phenomenon involving the formation and collapse of microbubbles, which generates localized mechanical forces that disrupt plant tissues and improve solvent penetration and generally improving mass transfer phenomena [20]. Pulsed electric fields-assisted extraction is a non-thermal technique that involves the application of short, high-voltage pulses to biological tissues, inducing electroporation of cell membranes. This process increases membrane permeability by forming temporary or permanent pores, thereby facilitating the release of intracellular compounds into the surrounding solvent [21]. Compared to thermal extraction methods, PEFAE offers significant advantages, including reduced thermal degradation, improved extraction efficiency, lower energy consumption, and shorter processing times. These characteristics make PEFAE particularly suitable for the recovery of heat-sensitive bioactive compounds and for applications requiring high extract quality [20,22]. These emerging extraction techniques have been widely applied for the recovery of bioactive compounds from agro-industrial byproducts [18]. These technologies have demonstrated significant potential in enhancing

extraction efficiency, reducing processing time, and improving the recovery of valuable compounds from plant-based waste materials, including fruit peels [23–27], seeds [28–31], and pomaces [32–37]. Limited studies have compared these emerging technologies for the recovery of bioactive compounds from orange peel. Moreover, most studies have focused on dried peel, while extraction from fresh peel remains largely unexplored. The application of these techniques to fresh peel, prior to the drying stage, could enable the recovery of high-value bioactive compounds while maintaining the existing industrial processing workflow. A comprehensive understanding of the influence of extraction techniques and process parameters is crucial for optimizing recovery processes and facilitating the industrial-scale valorization of citrus processing waste. Therefore, the objective of this study is to evaluate and compare the efficiency of conventional extraction (CE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and pulsed electric fields-assisted extraction (PEFAE) for the recovery of polyphenols from orange peel waste. Furthermore, the flavonoids content and antioxidant activity of the obtained extracts are assessed, and the effects of key processing parameters are investigated. The findings of this study contribute to the development of efficient, sustainable, and scalable extraction strategies for the valorization of citrus byproducts, supporting the transition toward circular economy-based approaches in the food and bioprocessing industries.

2. Materials and Methods

2.1. Raw Material

Orange peels waste was collected from a fruit processing facility located in the Argolida region of Greece. The raw material originated from *Navel cv.* oranges at commercial maturity suitable for juice processing. The fruits were fully orange, with only very limited green areas observed in a small number of fruits. Fresh orange peel waste was used in all experiments. The waste consisted of orange peel residues containing both flavedo and albedo. The flavedo-to-albedo ratio was not strictly standardized, as the aim of the study was to evaluate real industrial orange peel waste; however, the approximate ratio was visually estimated to be around 50:50. After collection from the fruit processing facility, the material was manually separated, filtered to isolate the solid peel fraction, and immediately ground to ensure sample homogeneity (the remaining solid fraction was cut/ground into small pieces of approximately 2×2 cm). The ground fresh peel was stored under refrigeration at 4 °C only for short-term preservation and was used within 3 d after collection. For experiments requiring a longer experimental period, fresh batches were collected and processed following the same procedure. Batch-to-batch variability was considered limited, since all extraction experiments were performed within a total period of two weeks using raw material from the same processing period. No dried or lyophilized orange peel was used in the present study. The initial moisture content of the fresh peels was $82.3 \pm 3.5\%$ on a wet basis. After extraction, the extracts were stored under freezing conditions until further analysis.

2.2. Experimental Design

The ground orange peel waste was subjected to three different treatments, pulsed electric fields (PEF), ultrasound (US), and microwave (MW). Following PEF and MW pretreatments, solid–liquid solvent extraction was performed at a 1:10 ratio using ethanol/water mixtures for approximately 120 min in a temperature-controlled water bath under constant agitation at 160 rpm. For US, solvent extraction was performed simultaneously with the US treatment. Conventional solvent extraction was also carried out. All extracts were analyzed for total phenolic content (TPC), total flavonoids (TF), and antioxidant activity (AA).

2.2.1. Conventional Extraction (CE)

Conventional extraction (CE) involved macerating 3 g of ground orange peel waste in 30 mL of either 80% aqueous ethanol, 50% aqueous ethanol, or water (0% ethanol). CEs were conducted at 30, 50, and 70 °C, which are commonly reported as effective conditions for recovering bioactive compounds from citrus peels [38]. Aliquots (<1 mL) were withdrawn at specific time intervals to monitor extraction kinetics. The extraction process lasted up to 120 min, after which the extracts were filtered and stored for subsequent analysis. Longer extraction times did not result in higher yields of bioactive compounds (preliminary experiments). All CE experiments were conducted in duplicate (biological/experimental replicates).

2.2.2. Microwave-Assisted Extraction (MAE)

Microwave-Assisted Extraction (MAE) experiments were carried out using a laboratory microwave system (Izzy IZ-8005, 20 L, 1050 W; Benrubi S.A., Athens, Greece) with temperature control. Three grams (3 g) of ground orange peel were combined with either 80% aqueous ethanol, 50% aqueous ethanol, or water (0% ethanol). Three microwave power levels (204, 480, and 780 W) and three exposure times (0.5, 1.0, and 2.0 min) that corresponded to microwave applied energy ranging from 6.1 to 93.6 kJ were evaluated. After irradiation, the CE followed for each MW condition studied lasted up to 120 min. Aliquots (<1 mL) were withdrawn at specific time intervals to monitor extraction kinetics. The extracts were filtered and stored for subsequent analysis. All MW experiments were conducted in duplicate (biological/experimental replicates).

2.2.3. Ultrasound-Assisted Extraction (UAE)

Ultrasound-Assisted Extraction (UAE) (90% amplitude, 23.24 W) was performed using a BIOBASE Ultrasonic Cell Disruptor UCD Series device (BIOBASE Group, Jinan, Shandong, China) with temperature control. Three grams (3 g) of ground orange peel were suspended in 30 mL with either 80% aqueous ethanol, 50% aqueous ethanol, or water (0% ethanol). The extraction temperature was maintained at either 35 or 50 °C, while the extraction time was fixed at 120 min. Longer extraction times did not result in higher yields of bioactive compounds based on preliminary experiments. Aliquots (<1 mL) were withdrawn at specific time intervals to monitor extraction kinetics. The extracts were filtered and stored for subsequent analysis. All US experiments were conducted in duplicate (biological/experimental replicates).

2.2.4. Pulsed Electric Fields-Assisted Extraction (PEFAE)

Pulsed Electric Fields-assisted extraction (PEFAE) was performed using a pilot-scale system (Elcrack-5 kW, DIL, Quakenbruck, Germany) with a batch chamber (10 × 5 × 8 cm—length × height × width) and oscilloscope monitoring (Tektronix TDS 1012, Beaverton, OR, USA). Three grams (3 g) of ground orange peel were initially mixed with water, as ethanol is not compatible with PEF treatment. Samples were placed in a stainless-steel treatment chamber, and PEF treatment was applied at 4 kV/cm with 1000 pulses, a pulse width of 15 µs, 20 Hz frequency and 21 kJ/kg energy input. The PEFAE conditions were selected based on our previous study [39] on orange peel waste, in which treatment at 4 kV/cm and 1000 pulses resulted in a high cell disintegration index of approximately $Z = 0.78$. Preliminary extraction trials also showed that less intense PEF treatments, which resulted in lower cell disintegration index values, either led to lower phenolic recovery or required longer extraction times to achieve comparable extraction yields based on preliminary experiments. Therefore, 4 kV/cm and 1000 pulses (21 kJ/kg) were selected as a fixed PEFAE pretreatment condition. The PEF intensity was kept constant, while

the extraction time and ethanol concentration were evaluated. After PEF pretreatment, the three different mixtures ethanol/water were restored (80% EtOH, 50% EtOH and 0% EtOH), and the CE followed for up to 120 min. Small aliquots (<1 mL) were periodically collected to monitor compounds release. All PEF experiments were conducted in duplicate (biological/experimental replicates).

2.3. Determination of Bioactive Compounds

Total phenolic content (TPC) (mg caffeic acid/100 g wet mass (w.m.)), total flavonoids (TF) (mg catechin/100 g w.m.) and antioxidant activity (AA) (mg Trolox equivalent/100 g w.m.) were measured for all samples studied. TPC were quantified using the Folin–Ciocalteu method [40]. Briefly, 100 μ L of appropriately diluted extract were mixed with 7.9 mL of deionized water and 500 μ L of Folin–Ciocalteu reagent. After vortexing and incubation for 1.5 min, 1.5 mL of saturated Na_2CO_3 solution was added, and the mixture was vortexed again. The reaction mixture was incubated in a water bath at 40 °C for 30 min, and the absorbance was measured at 765 nm. A reagent blank was prepared by replacing the extract with deionized water. Quantification was performed using caffeic acid as the calibration standard, and the results were expressed as mg caffeic acid equivalents per mL of extract (mg CAE/mL). TF were determined using the aluminum chloride colorimetric assay [41]. In a 10 mL volumetric flask, 1 mL of diluted extract was mixed with 4 mL of deionized water and 0.3 mL of 5% NaNO_2 . After 5 min, 0.3 mL of 10% AlCl_3 was added, followed after 1 min by 2 mL of 1 M NaOH. The volume was then adjusted to 10 mL with deionized water, and the absorbance was measured at 510 nm. A reagent blank was prepared by replacing the extract with deionized water. Quantification was performed using catechin as the calibration standard, and the results were expressed as mg catechin equivalents per mL of extract. AA was assessed via the DPPH assay [42]. Briefly, 3.9 mL of DPPH solution were mixed with 100 μ L of diluted extract and incubated in the dark at 25 °C for 15 min. The absorbance was then measured at 515 nm. A reagent blank and a control solution without extract were used for blank correction. Quantification was performed using Trolox as the calibration standard, and the results were expressed as Trolox equivalents. All analytical spectrophotometric measurements were performed in triplicate for TPC, TF, and AA.

2.4. Extraction Optimization–Response Surface Methodology (RSM)

Response Surface Methodology (RSM) was applied to evaluate the influence of the extraction parameters on the response variable (Y), defined as the concentration of phenolic compounds in the extracts. For the CE process, three independent variables were investigated: extraction temperature, extraction time, and ethanol concentration (% v/v). Similarly, for UAE, the independent variables examined were extraction temperature, extraction time, and ethanol concentration (% v/v). In the case of MAE, the variables studied were microwave energy applied (MW power $\times$ MW time), extraction time, and ethanol concentration (% v/v). For PEFAE, two independent variables were considered: extraction time and ethanol concentration (% v/v). For each extraction technique, a second-order polynomial model was employed to describe the relationship between the independent variables and the response variable (Y). The experimental data were analyzed using multiple regression analysis to estimate the coefficients of the quadratic models. The adequacy and statistical significance of the developed models were evaluated using analysis of variance (ANOVA). Contour plots were generated to visualize the effects of the independent variables and their interactions on the response. In addition, prediction profiles were constructed to assess the individual contribution of each factor to the predicted response. Process optimization was performed using a desirability function approach to determine the combination of experi-

mental conditions that maximized the response. The response surface analysis revealed the presence of a plateau region around the predicted optimum. This plateau was further evaluated using prediction intervals (PI), which allowed the identification of a range of experimental conditions capable of producing statistically similar response values. Based on this plateau region, the final optimal conditions were selected not solely based on the maximum predicted response but also considering practical aspects such as energy consumption and process efficiency. Therefore, the selected optimal conditions correspond to a point within the plateau that represents the most energetically and economically favorable operating conditions while maintaining high extraction performance.

2.5. Calculation of Energy Input, Ethanol Consumption, and TPC-Normalized Efficiency for Each Extraction Technique

A quantitative comparison of the extraction methods was performed based on the energy input, ethanol consumption, and TPC-normalized efficiency. The energy input was estimated as calculated nominal process energy, since direct electricity consumption was not monitored during the experiments. For CE, the total energy was calculated as the sum of the nominal water bath energy, estimated from the rated power (1.6 kW) and extraction time, and the minimum theoretical heating energy required to raise the extraction mixture to the target temperature, calculated as $Q = m \cdot C_p \cdot \Delta T$. For MAE, the total supplied microwave energy was used directly from RSM optimization. For UAE, the energy input was calculated from the electrical power of the ultrasound device (90% amplitude, 23.24 W) and the extraction time. For PEFAE, the total energy was calculated as the sum of the specific PEF energy input (21 kJ/kg), expressed per kg, and the nominal pulse energy estimated from the rated power of the PEF unit (5 kW), the number of pulses, and the pulse width. The calculated energy values were then normalized per mg of recovered TPC. Solvent consumption and ethanol use were also expressed per mg of recovered TPC to support a quantitative discussion of the sustainability-related performance of each extraction method.

2.6. Statistical Analysis

All extraction experiments were carried out in duplicate independent extraction runs (biological/experimental replicates). Each extract was subsequently analyzed in triplicate spectrophotometric measurements for TPC, TF, and AA. Therefore, the values reported in the manuscript represent the mean $\pm$ standard deviation of six combined measurements (2 biological & 3 analytical replicates). Data were analyzed by ANOVA, and differences were considered significant at $p < 0.05$. Response surface methodology (RSM) was applied to optimize CE parameters. One-way or factorial ANOVA was used to assess significant differences and interactions between MAE, UAE and PEFAE treatments regarding extraction time, ethanol concentration and extraction temperature. Analyses were performed using Statistica 7 (StatSoft, Tulsa, OK, USA), and mean comparisons were conducted using Duncan's test at a 5% significance level.

3. Results

3.1. Conventional Extraction (CE) of Orange Peel Waste

Figure 1 visualizes the interaction effects between the independent variables on the extraction of TPC from orange peel waste.

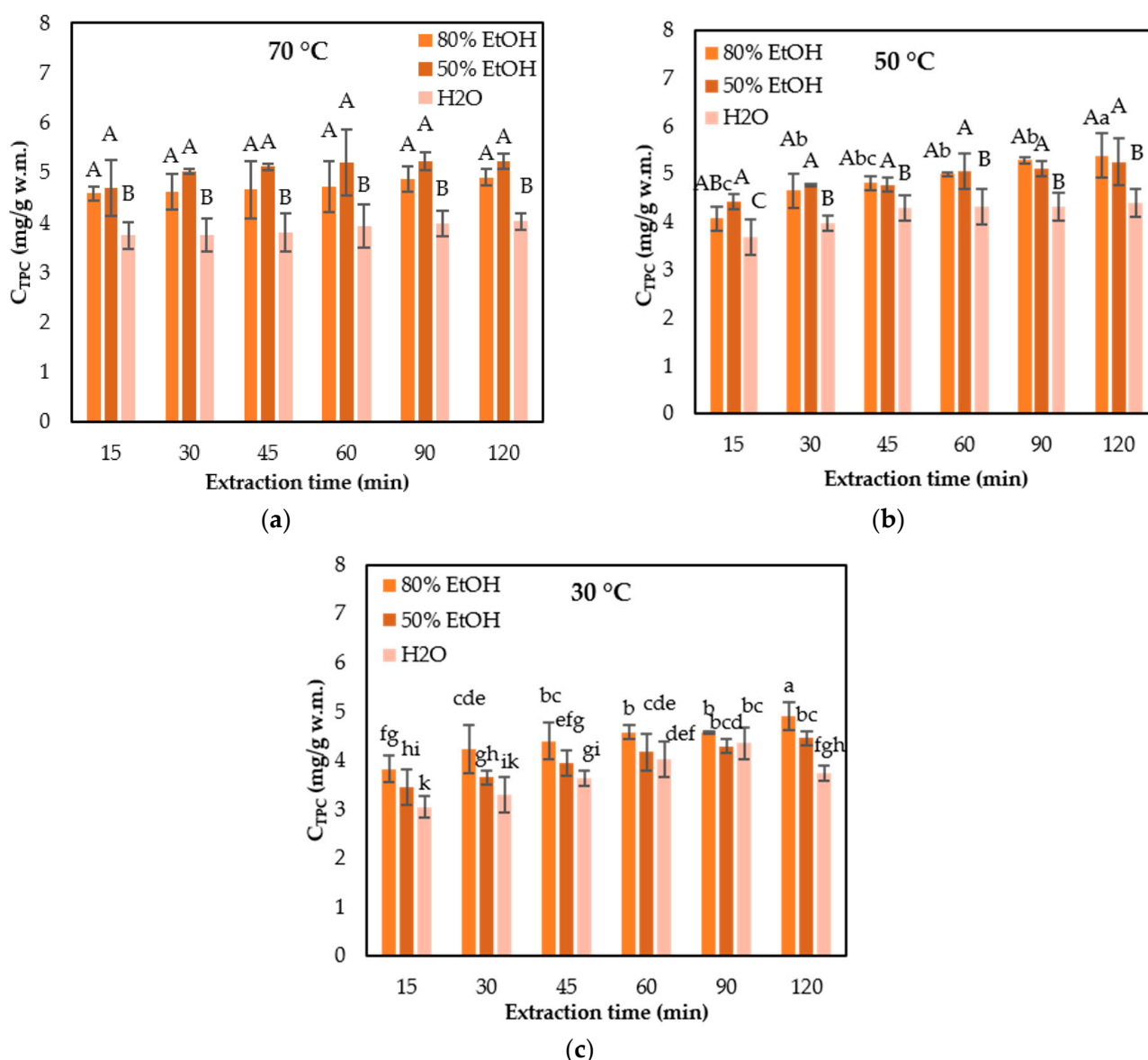


Figure 1. Concentration of TPC (mg/g w.m.) as a function of extraction time (0–120 min) for 80, 50, and 0% ethanol in conventional extraction at (a) 70, (b) 50, and (c) 30 °C. Error bars represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). For Figure 1a,b different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (extraction times) based on the results obtained from one-way ANOVA analysis for each variable. Different uppercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (ethanol concentration), based on the results obtained from one-way ANOVA analysis for each variable. For Figure 1c different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (extraction time and ethanol concentration), based on the results obtained from factorial ANOVA analysis for the variables time and ethanol concentration.

The graphical analysis revealed that the response increased progressively with increasing temperature and ethanol concentration up to a certain level, beyond which the response tended to stabilize or slightly decrease. This behavior suggests that excessively high extraction conditions do not significantly improve extraction efficiency. In addition, the interaction between extraction time and ethanol concentration showed a noticeable effect on the extraction of TPC, as moderate extraction times combined with intermediate ethanol concentrations favored higher response values, indicating the existence of an op-

timal extraction region within the experimental design space. The experimental results demonstrated that the extraction conditions significantly influenced the C_{TPC} values obtained. Overall, C_{TPC} values ranged from approximately 3.05 to 5.38 mg/g w.m., indicating a strong dependence on temperature, ethanol concentration, and extraction time. Among the examined parameters, ethanol concentration exhibited the most pronounced effect on C_{TPC} . Increasing ethanol concentration from 0% to 50% and 80% resulted in a substantial increase in C_{TPC} values across all temperatures and extraction times. Extractions performed with 50% and 80% ethanol consistently produced higher C_{TPC} values. For instance, at 70 °C and 45 min, C_{TPC} increased from 3.42 mg/g w.m. (0% ethanol) to 5.24 mg/g w.m. (80% ethanol), an increase of over 53%. Extraction with water (0% ethanol) resulted in the lowest phenolic recovery, with values generally below 4.1 mg/g w.m. Temperature also influenced extraction efficiency. Increasing the extraction temperature led to higher yields of total extractable TPC. However, no significant differences were observed between 50 and 70 °C, suggesting that the extraction process approaches a plateau region at higher temperatures, increasing from ~3.59–4.70 mg/g w.m. at 30 °C to ~4.42–5.86 mg/g w.m. at 50–70 °C. Similarly, extraction time showed a positive effect on C_{TPC} up to approximately 90–120 min, where the highest values were typically observed. For example, at 50% ethanol and 50 °C, C_{TPC} values increased from 4.42 mg/g w.m. at 15 min to 5.74 mg/g w.m. at 120 min, representing an increase of nearly 30%. Overall, the results indicate that moderate to high ethanol concentrations combined with extended extraction times favor higher phenolic recovery, while temperature mainly enhances the extraction process up to a certain level.

The complete experimental datasets for total flavonoids (TF) and antioxidant activity (AA) obtained from orange peel waste extracts under the conventional extraction (CE), as affected by extraction time, ethanol concentration and temperature are presented in Tables A1 and A2 in Appendix A. For conventional extraction, total flavonoid content was strongly affected by ethanol concentration, extraction temperature, and extraction time. In most cases, 80% ethanol resulted in substantially higher TF values than 50% ethanol or water, indicating that the less polar hydroethanolic solvent favored flavonoid recovery from orange peel waste. TF generally increased with extraction time, with the highest value observed at 30 °C, 80% EtOH, and 120 min, suggesting that prolonged extraction under mild temperature conditions was favorable for flavonoid release. In contrast, antioxidant activity did not follow the same trend as TF, especially at 70 °C and 50 °C, where higher values were often observed at shorter extraction times. At 30 °C, antioxidant activity tended to increase with time, suggesting better preservation or gradual release of antioxidant compounds under milder thermal conditions.

3.2. Microwave-Assisted Extraction (MAE) of Orange Peel Waste

MAE significantly enhanced extraction efficiency compared to CE. Table 1 presents the effect of three main parameters on extraction yield: (i) extraction time (0–120 min), (ii) solvent concentration (0, 50, and 80%), and (iii) microwave energy input (6.12–93.6 kJ). The measured values ranged from approximately 2.41 to 6.42 mg/g w.m. A statistically significant ($p < 0.05$) increase was observed with increasing extraction time under all microwave conditions and solvent concentrations. At higher microwave energy levels, the increase is more pronounced and tends to plateau after 90 min. At later extraction times (≥ 60 min), the difference between 50% and 80% becomes less pronounced (often <5–10%), suggesting partial convergence toward equilibrium solubility conditions.

Table 1. TPC concentration (mg/g w.m.) during MAE of orange peel waste for extraction time ranged from 0 to 120 min, solvent concentration equal to 0, 50, and 80% and microwave energy input ranged from 6.12 to 93.6 kJ.

Microwave Energy Applied		6.1 kJ			12.2 kJ		
Extraction Time (min)		80% EtOH	50% EtOH	0% EtOH	80% EtOH	50% EtOH	0% EtOH
0		2.69 ± 0.26 ^B	2.99 ± 0.10 ^D	2.41 ± 0.05 ^E	2.82 ± 0.16 ^j	3.27 ± 0.15 ^{ij}	2.81 ± 0.14 ^j
15		3.63 ± 0.15 ^{AB}	3.63 ± 0.02 ^C	2.87 ± 0.06 ^D	3.84 ± 0.16 ^{gh}	3.92 ± 0.02 ^{gh}	3.20 ± 0.28 ^{ij}
30		3.95 ± 0.06 ^{AB}	4.03 ± 0.04 ^{BC}	3.23 ± 0.07 ^C	4.18 ± 0.11 ^{defg}	4.36 ± 0.31 ^{cdefg}	3.50 ± 0.08 ^{hi}
45		4.21 ± 0.07 ^{AB}	4.28 ± 0.15 ^{AB}	3.53 ± 0.11 ^B	4.45 ± 0.09 ^{bcdef}	4.62 ± 0.15 ^{abcde}	3.87 ± 0.16 ^{gh}
60		4.38 ± 0.11 ^{AB}	4.40 ± 0.37 ^{AB}	3.70 ± 0.10 ^{AB}	4.66 ± 0.13 ^{abcd}	4.77 ± 0.08 ^{abc}	3.97 ± 0.27 ^{fgh}
90		4.60 ± 0.16 ^{AB}	4.66 ± 0.26 ^A	3.85 ± 0.12 ^A	4.85 ± 0.02 ^{abc}	4.87 ± 0.03 ^{abc}	4.04 ± 0.35 ^{fg}
120		4.70 ± 0.15 ^B	4.76 ± 0.11 ^A	3.95 ± 0.13 ^A	4.93 ± 0.01 ^{ab}	4.99 ± 0.05 ^a	4.10 ± 0.19 ^{efg}
		14.4 kJ			23.4 kJ		
0		3.18 ± 0.17 ^{jk}	3.03 ± 0.15 ^k	3.08 ± 0.15 ^k	3.55 ± 0.13 ^{lm}	3.55 ± 0.18 ^{lm}	3.22 ± 0.10 ^m
15		3.90 ± 0.03 ^{fghi}	3.93 ± 0.15 ^{fghi}	3.50 ± 0.27 ^{ijk}	4.50 ± 0.15 ^{defghi}	4.03 ± 0.27 ^{jk}	3.87 ± 0.15 ^{kL}
30		4.27 ± 0.04 ^{cdefg}	4.27 ± 0.10 ^{cdefg}	3.67 ± 0.35 ^{hij}	4.68 ± 0.03 ^{bcdefg}	4.48 ± 0.05 ^{efghij}	4.08 ± 0.16 ^{ijk}
45		4.66 ± 0.08 ^{abcd}	4.41 ± 0.05 ^{bcdef}	3.79 ± 0.04 ^{ghi}	4.89 ± 0.07 ^{bcde}	4.66 ± 0.06 ^{cdefgh}	4.16 ± 0.17 ^{ijk}
60		4.93 ± 0.11 ^{ab}	4.50 ± 0.14 ^{bcde}	3.92 ± 0.08 ^{fghi}	5.01 ± 0.02 ^{abc}	4.76 ± 0.10 ^{bcdefg}	4.22 ± 0.10 ^{hijk}
90		5.04 ± 0.11 ^a	4.75 ± 0.18 ^{abc}	4.04 ± 0.37 ^{efgh}	5.13 ± 0.15 ^{ab}	4.82 ± 0.21 ^{bcdef}	4.31 ± 0.11 ^{ghijk}
120		5.12 ± 0.02 ^a	4.88 ± 0.21 ^{ab}	4.19 ± 0.16 ^{defgh}	5.37 ± 0.23 ^a	4.94 ± 0.02 ^{abcd}	4.37 ± 0.24 ^{fghij}
		24.5 kJ			28.8 kJ		
0		3.06 ± 0.15 ^L	3.53 ± 0.10 ^k	3.11 ± 0.14 ^L	3.39 ± 0.15 ^D	3.53 ± 0.18 ^C	3.15 ± 0.15 ^D
15		4.13 ± 0.09 ^{hij}	4.01 ± 0.03 ^{ij}	3.51 ± 0.04 ^k	4.25 ± 0.26 ^C	4.20 ± 0.40 ^{BC}	3.64 ± 0.08 ^{CD}
30		4.52 ± 0.07 ^{efgh}	4.42 ± 0.04 ^{fghi}	3.71 ± 0.03 ^{jk}	4.64 ± 0.37 ^{BC}	4.34 ± 0.15 ^{AB}	3.76 ± 0.01 ^{BC}
45		4.84 ± 0.15 ^{bcde}	4.58 ± 0.01 ^{defg}	3.98 ± 0.07 ^{ij}	4.89 ± 0.40 ^{ABC}	4.64 ± 0.37 ^{AB}	3.83 ± 0.36 ^{ABC}
60		5.09 ± 0.06 ^{abc}	4.79 ± 0.13 ^{cdef}	4.10 ± 0.05 ^{hij}	5.11 ± 0.29 ^{AB}	4.72 ± 0.17 ^{AB}	4.04 ± 0.22 ^{ABC}
90		5.21 ± 0.15 ^{ab}	4.96 ± 0.37 ^{abcd}	4.21 ± 0.15 ^{ghij}	5.24 ± 0.17 ^{AB,}	4.90 ± 0.35 ^{AB}	4.26 ± 0.29 ^{AB}
120		5.35 ± 0.09 ^a	5.14 ± 0.15 ^{abc}	4.26 ± 0.15 ^{ghij}	5.45 ± 0.16 ^A	5.02 ± 0.12 ^A	4.38 ± 0.08 ^A

Table 1. Cont.

	46.8 kJ			57.6 kJ		
0	3.92 ± 0.15^F	3.60 ± 0.01^F	3.15 ± 0.09^C	4.66 ± 0.24^B	4.28 ± 0.15^D	3.93 ± 0.15^D
15	4.42 ± 0.13^E	4.16 ± 0.04^E	4.16 ± 0.15^B	5.16 ± 0.15^{AB}	4.85 ± 0.16^{CD}	4.15 ± 0.11^{CD}
30	4.78 ± 0.15^{DE}	4.34 ± 0.15^{DE}	4.29 ± 0.17^{AB}	5.24 ± 0.26^{AB}	5.12 ± 0.36^{BC}	4.44 ± 0.22^{BCD}
45	5.01 ± 0.08^{CD}	4.54 ± 0.30^{CD}	4.42 ± 0.18^{AB}	5.49 ± 0.18^{AB}	5.30 ± 0.26^{ABC}	4.60 ± 0.33^{ABC}
60	5.14 ± 0.27^{ABC}	4.66 ± 0.22^{BC}	4.55 ± 0.25^{AB}	5.69 ± 0.40^A	5.46 ± 0.12^{AB}	4.76 ± 0.27^{ABC}
90	5.27 ± 0.16^{AB}	4.87 ± 0.16^{AB}	4.70 ± 0.25^A	5.91 ± 0.46^A	5.63 ± 0.11^{AB}	4.89 ± 0.25^{AB}
120	5.48 ± 0.15^A	5.04 ± 0.15^A	4.79 ± 0.20^A	6.11 ± 0.54^A	5.83 ± 0.22^A	5.10 ± 0.15^A
	93.6 kJ					
0	4.68 ± 0.01^D		4.56 ± 0.10^D		3.99 ± 0.08^E	
15	5.44 ± 0.04^C		5.09 ± 0.11^C		4.34 ± 0.31^D	
30	5.82 ± 0.02^{BC}		5.47 ± 0.20^B		4.51 ± 0.15^{CD}	
45	5.94 ± 0.37^B		5.63 ± 0.11^{AB}		4.78 ± 0.30^{BC}	
60	6.02 ± 0.15^{AB}		5.74 ± 0.21^{AB}		5.13 ± 0.10^{ABC}	
90	6.20 ± 0.15^{AB}		5.82 ± 0.05^{AB}		5.29 ± 0.21^{AB}	
120	6.42 ± 0.16^A		5.99 ± 0.08^A		5.54 ± 0.09^A	

± represents the standard deviation from six combined measurements (2 biological & 3 analytical replicates). Small letters indicate the significant differences ($p < 0.05$) between different extraction times and different solvent concentrations (factorial ANOVA analysis) for each microwave condition applied (energy input). Capital letters indicate the significant differences ($p < 0.05$) between different extraction times (one-way ANOVA analysis) for each microwave condition applied (energy input) and each solvent concentration.

Regarding solvent concentration, intermediate and higher levels (50% and 80%) generally result in higher values compared to 0%. In many cases, the 80% solvent concentration yields the highest extraction efficiency, especially at longer extraction times and higher energy inputs, suggesting improved solubilization of target compounds due to optimized solvent polarity. At later extraction times (≥ 60 min), the difference between 50% and 80% becomes less pronounced (often $< 5\text{--}10\%$), suggesting partial convergence toward equilibrium solubility conditions. Microwave energy input also plays a critical role. A progressive increase in the measured values is observed as energy increases from 6.12 to 93.6 kJ, with the highest responses consistently obtained at the highest energy levels (46.8–93.6 kJ). The increased microwave energy enhances extraction efficiency, likely due to intensified matrix disruption and improved mass transfer. The rapid heating effect of microwaves likely facilitated cell wall disruption, enhancing solvent penetration and solute diffusion. At 90 min and 80% ethanol, increasing microwave energy from 6.12 kJ to higher levels resulted in a progressive enhancement of the response variable. Overall, the results suggest that optimal extraction conditions are achieved at high microwave energy (≥ 46.8), high solvent concentration (80%), and longer extraction times (≥ 90 min), where maximum values are obtained. However, the relatively small differences between 90 and 120 min in several cases indicate that equilibrium may be approached, which is important for process optimization in terms of reducing extraction time and energy consumption.

The complete experimental datasets for total flavonoids (TF) and antioxidant activity (AA) obtained from orange peel waste extracts under the microwave assisted extraction (MAE), as affected by extraction time, ethanol concentration and microwave energy applied are presented in Tables A3 and A4 in Appendix A. For microwave-assisted extraction, both microwave energy input and extraction time strongly affected total flavonoid recovery and antioxidant activity. In general, TF increased with extraction time within each microwave energy level, indicating that prolonged MAE promoted the gradual release of flavonoid compounds from the orange peel matrix. However, the effect of microwave energy was not linear, since the highest energy input did not always result in the highest TF values. Higher TF recoveries were mainly observed at lower to intermediate microwave energies, particularly in the presence of 80% EtOH, suggesting that moderate microwave treatment combined with hydroethanolic solvent favored flavonoid solubilization while avoiding excessive thermal or structural degradation. Antioxidant activity also generally increased with extraction time, but its maximum values were observed at intermediate microwave energy, especially at 23.4 kJ, rather than at the highest energy levels. This indicates that excessive microwave energy may negatively affect the antioxidant effectiveness of the extracts, possibly due to degradation, oxidation, or transformation of thermosensitive antioxidant compounds.

3.3. Ultrasound-Assisted Extraction (UAE) of Orange Peel Waste

The combined effect of ultrasounds and temperature of 50 °C led to the highest yields of extracted TPC in significantly shorter times compared to yields at 35 °C (data not shown). Based on those preliminary experiments, the ultrasounds experiments were conducted at 50 °C, since at 35 °C the yield of TPC was half of that of 50 °C. The concentration of total phenolic compounds during ultrasounds (90% amplitude) assisted extraction for 80, 50% EtOH concentration and H₂O for up to 120 min at 50 °C extraction temperature is illustrated in Figure 2.

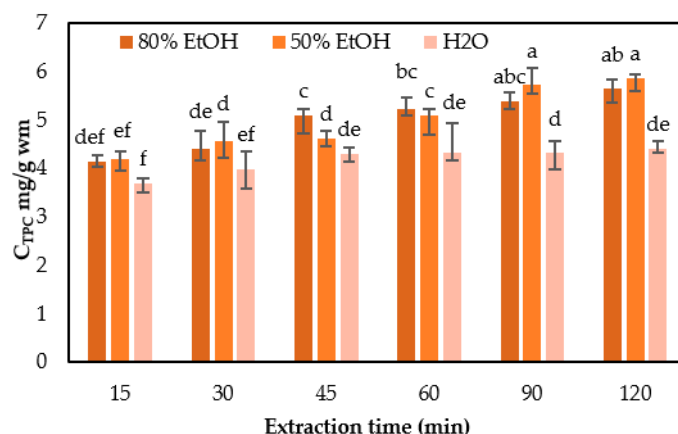


Figure 2. TPC concentration (mg/g w.m) during ultrasound (90% amplitude) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 50 °C extraction temperature. Error bars represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

The results demonstrate that both ethanol concentration and extraction time significantly influenced the recovery of total phenolic compounds (C_{TPC}). Across the conditions examined, C_{TPC} values ranged from approximately 3.55 to 5.93 mg/g w.m. The lowest phenolic recovery was observed with water as solvent extraction. In contrast, the highest values were obtained at 50% ethanol and 120 min, reaching up to 5.93 mg/g w.m., corresponding to an increase of approximately 65–70% compared to the lowest extraction conditions. The use of 50 and 80% EtOH concentrations resulted in similar TPC yields. Increasing ethanol concentration from 0% to 50% resulted in a substantial improvement in phenolic extraction at all extraction times. For example, at 90 min, C_{TPC} values increased from approximately 4.06–4.56 mg/g w.m. at 0% ethanol to 5.38–6.06 mg/g w.m. at 50% ethanol, representing an increase of about 30–40%. Generally, C_{TPC} values increased progressively with extraction time. For instance, using 80% ethanol, C_{TPC} increased from approximately 4.01–4.25 mg/g w.m. at 15 min to 5.47–5.83 mg/g w.m. at 120 min, corresponding to an increase of about 35–40%. However, the increase in C_{TPC} became less pronounced after 90 min, suggesting that the extraction process gradually approached equilibrium. Overall, the results indicate that moderate ethanol concentrations (around 50%) combined with longer extraction times (90–120 min) provide the most efficient conditions for phenolic recovery, whereas extractions performed without ethanol result in significantly lower phenolic yields.

The complete experimental datasets for total flavonoids (TF) and antioxidant activity (AA) obtained from orange peel waste extracts under the ultrasounds assisted extraction (UAE), as affected by extraction time and ethanol concentration are presented in Tables A5 and A6 in Appendix A. For ultrasound-assisted extraction, total flavonoid recovery was clearly influenced by ethanol concentration and extraction time. The highest TF values were obtained with 50% EtOH, reaching a maximum around 90 min, whereas 80% EtOH resulted in lower flavonoid recovery and water was generally the least effective solvent. This suggests that an intermediate ethanol concentration provided a more favorable polarity for the ultrasound-assisted release and solubilization of flavonoid compounds. TF values increased mainly up to 60–90 min and then tended to reach a plateau, indicating that prolonged sonication beyond this point did not substantially enhance flavonoid extraction. In contrast, antioxidant activity did not follow the same pattern as TF: the highest AA value was observed at short extraction time with 80% EtOH, while 50% EtOH showed higher AA after longer extraction times. This indicates that ultrasound extraction affected not only

the amount of flavonoids recovered, but also the qualitative composition and antioxidant efficiency of the extracted compounds.

3.4. Pulsed Electric Fields-Assisted Extraction (PEFAE) of Orange Peel Waste

Generally, the results obtained proved that PEF pretreatment significantly enhanced the TPC recovery from orange peel waste (Figure 3).

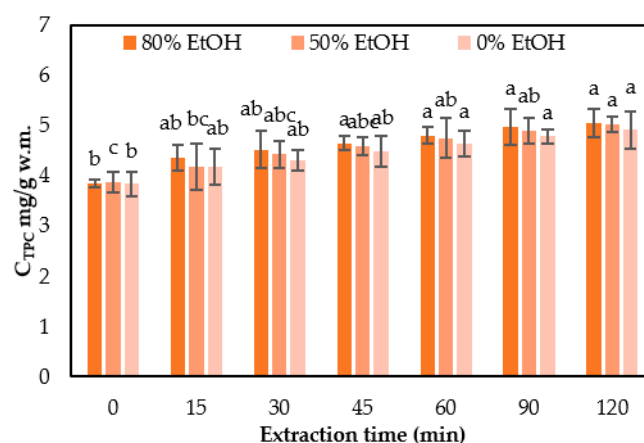


Figure 3. TPC concentration (mg/g w.m.) during pulsed electric fields (4.0 kV/cm, 1000 pulses) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 25 °C extraction temperature. Error bars represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Across all experimental conditions, C_{TPC} values ranged approximately from 3.59 to 5.34 mg/g w.m. As extraction time increased, C_{TPC} values gradually increased for all ethanol concentrations. At intermediate extraction times (45–90 min), C_{TPC} values generally ranged between 4.18 and 5.32 mg/g w.m., suggesting that the extraction process becomes progressively more efficient with time before approaching a plateau. C_{TPC} values obtained at 50% ethanol after 90 min (approximately 4.63–5.16 mg/g w.m.) were comparable to those achieved using water (0% ethanol) after 120 min (approximately 4.55–5.28 mg/g w.m.). This observation suggests that the use of moderate ethanol concentrations can reduce the required extraction time while achieving similar phenolic recovery. Statistical analysis further indicated that ethanol concentration did not have a statistically significant effect ($p > 0.05$) on C_{TPC} under the examined conditions, whereas extraction time appeared to exert a more pronounced influence on phenolic recovery. PEF pretreatment proved to be an effective and “green” method for the extraction of phenolic compounds from orange peel by-products for the relevant industry, as it uses water as the extraction medium and operates at room temperature.

The complete experimental datasets for total flavonoids (TF) and antioxidant activity (AA) obtained from orange peel waste extracts under the pulsed electric fields assisted extraction (PEFAE), as affected by extraction time and ethanol concentration are presented in Tables A7 and A8 in Appendix A. For pulsed electric field-assisted extraction, both total flavonoid recovery and antioxidant activity increased progressively with extraction time, indicating that the electroporation-induced increase in cell membrane permeability promoted the gradual release of bioactive compounds. The highest TF value was obtained after 120 min with 80% EtOH, while 50% EtOH and water also showed continuous increases with time, suggesting that PEFAE was able to enhance flavonoid extraction even under low- or non-ethanolic conditions. Antioxidant activity followed a similar increasing trend, with

the highest value again observed at 120 min using 80% EtOH. However, the differences among solvent systems were less pronounced for antioxidant activity than for TF, especially at longer extraction times. This suggests that PEFAE may favor the release or preservation of antioxidant compounds even in aqueous media, supporting its potential as a mild, non-thermal extraction technique with reduced organic solvent requirements.

3.5. Optimization of CE, MAE, UAE and PEFAE of Orange Peel Waste

The optimization of the extraction processes was performed using response surface methodology (RSM), and quadratic models were developed to describe the dependence between TPC and the processing parameters of each extraction technique. The optimization was intentionally performed using TPC as the primary response variable. TPC was selected because phenolic recovery was the main objective of the extraction process and because the extracted TPC values were approximately one order of magnitude higher than the corresponding TF content, making TPC the most representative and robust response for process optimization and comparison among the extraction methods.

In all cases, the models showed satisfactory goodness of fit, with coefficients of determination (R^2) ranging from 0.8298 to 0.8596 (CE: 0.8556, MAE: 0.8596, UAE: 0.8433, PEFAE: 0.8179), indicating adequate representation of the experimental data. The analysis of variance (ANOVA) confirmed that all models were statistically significant ($p < 0.0001$), with high F-values (CE: 47.47, MAE: 379.10, UAE: 94.96, PEFAE: 48.72), demonstrating their suitability for prediction and optimization. Appendix B provides the supplementary statistical information used to assess the adequacy of the quadratic response surface models developed for the different extraction methods. The model adequacy criteria, including (R^2), adjusted (R^2), RMSE, model significance, and lack-of-fit tests, are summarized to support the reliability of the fitted models beyond the coefficient of determination alone (Table A9). In addition, diagnostic plots are presented for each model, including normal probability plots of residuals, observed versus predicted values, and residuals versus predicted values (Figure A1). These plots were used to evaluate the normality of residuals, the agreement between experimental and predicted responses, and the absence of systematic trends in the residual distribution. Together, these statistical indicators and diagnostic plots provide a more complete assessment of model performance within the investigated experimental domain.

The linear and quadratic terms of the examined variables significantly influenced TPC yield in most cases, indicating the presence of curvature in the response surfaces and the existence of optimal operating regions.

The CE method yielded moderate phenolic content, with a maximum experimental value of 5.38 mg/g w.m. obtained at 50 °C, 80% ethanol, and 120 min. The correlation between TPC and the independent variables (temperature— T , ethanol concentration—%EtOH, extraction time— t : X_1 , X_2 , X_3) was expressed by the following polynomial equation:

$$C_{TPC} = 0.434 + 0.104 \cdot T - 0.0009 \cdot T^2 + 0.019 \cdot (\%EtOH) - 0.0002 \cdot (\%EtOH)^2 + 0.025 \cdot t - 0.00009 \cdot t^2 - 0.000132 \cdot T \cdot t \quad (1)$$

Regarding the individual effects of the variables, the linear terms of extraction temperature (X_1) and extraction time (X_3) had a highly significant effect on TPC yield ($p < 0.001$). The linear term of ethanol concentration (X_2) was also significant ($p < 0.001$). In addition, the quadratic terms of temperature (X_1^2), ethanol concentration (X_2^2), and extraction time (X_3^2) significantly affected the extracted TPC yield ($p < 0.001$), indicating curvature in the response surface. Among the interaction terms, only the interaction between temperature and extraction time (X_1X_3) significantly influenced the response ($p < 0.01$), whereas the interactions X_1X_2 and X_2X_3 were not statistically significant ($p > 0.05$). Contour plots were

constructed to visualize the interaction effects between the independent variables on the conventional extraction of TPC from orange peel waste (Figure 4a,b).

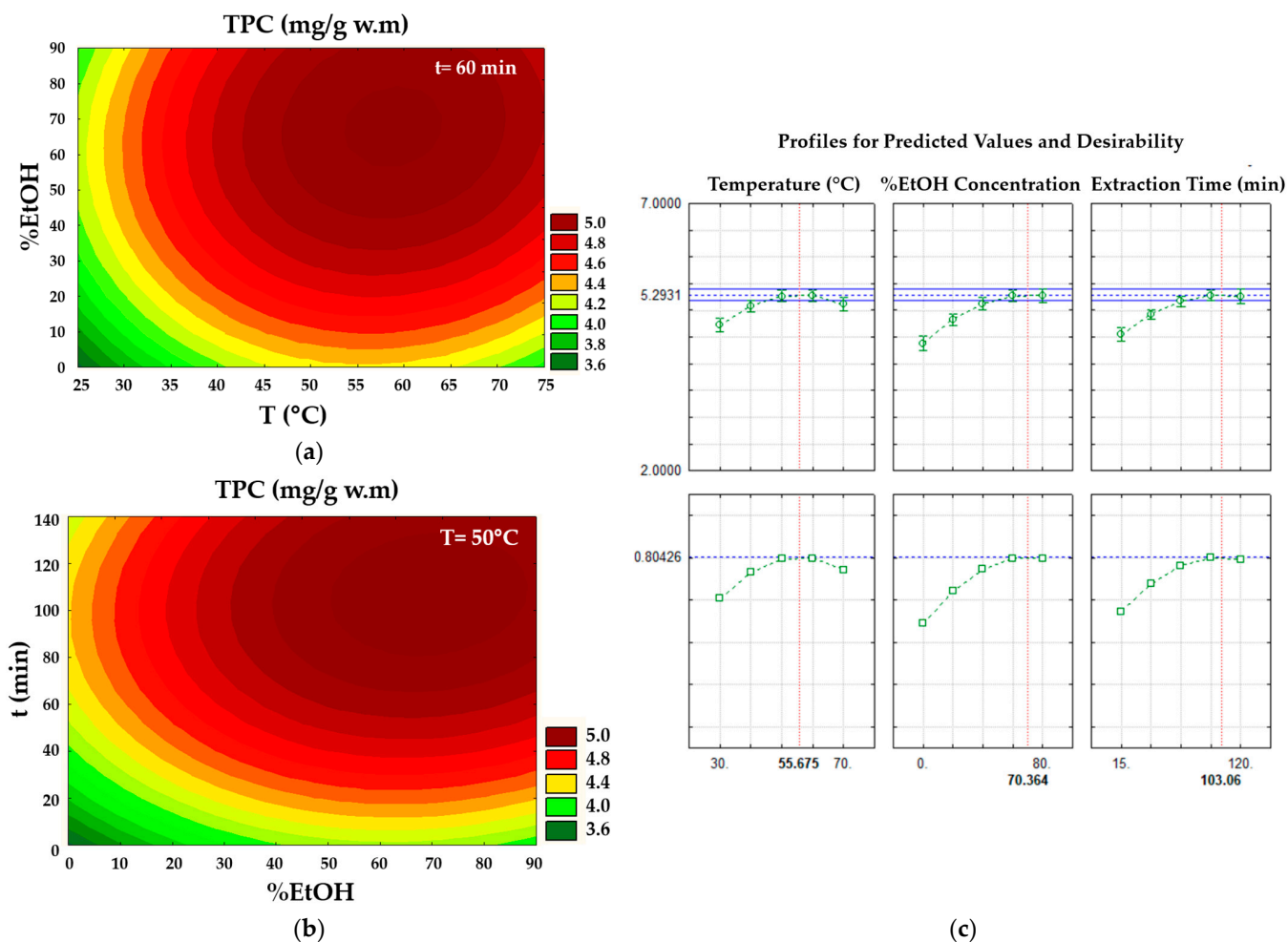


Figure 4. (a,b) Contour plots showing the effects of extraction temperature, extraction time and ethanol concentration on the extracted total phenolic compounds from orange peels waste during conventional extraction. (a) Extraction temperature vs. ethanol concentration (extraction time equal to 60 min). (b) Ethanol concentration vs. extraction time (extraction temperature equal to 50 °C). (c) Profiles for predicted values of extracted total phenolic compounds and the desirability level for different factors for optimum extracted TPC concentration yield for conventional extraction of orange peels waste.

The MAE method yielded moderate phenolic content, with a maximum experimental value of 6.42 mg/g w.m. obtained at 93.6 kJ microwave energy input, 80% ethanol, and 120 min. The correlation between TPC and the independent variables (microwave energy applied—E, ethanol concentration—%EtOH, extraction time—t: X1, X2, X3) was expressed by the following polynomial equation:

$$C_{TPC} = 2.5810 + 0.0221 \cdot E - 0.000064 \cdot E^2 + 0.0113 \cdot \%EtOH - 0.000063 \cdot \%EtOH^2 + 0.0280 \cdot t - 0.000147 \cdot t^2 + 0.000029 \cdot E \cdot \%EtOH + 0.000046 \cdot \%EtOH \cdot t \quad (2)$$

Regarding the individual effects of the variables, all three linear terms—microwave energy (X1), ethanol concentration (X2), and extraction time (X3)—had a highly significant effect on TPC yield ($p < 0.0001$). Among them, microwave energy (X1) exhibited the strongest influence ($F = 1361.85$), followed by extraction time (X3) ($F = 1191.49$) and ethanol concentration (X2) ($F = 689.75$), highlighting the dominant role of energy input in the MAE

process. The quadratic terms of all variables were also statistically significant. Specifically, the quadratic effects of extraction time (X_3^2) showed a strong contribution ($F = 346.47$, $p < 0.0001$), while both microwave energy (X_1^2) and ethanol concentration (X_2^2) had lower but still significant effects ($F \approx 16.05$, $p < 0.001$). This indicates the presence of curvature in the response surface and suggests that optimal conditions exist within the studied range. In terms of interaction effects, the interaction between ethanol concentration and extraction time ($X_2 \times X_3$) was highly significant ($F = 29.36$, $p < 0.0001$), demonstrating a strong synergistic effect between these two parameters. The interaction between microwave energy and ethanol concentration ($X_1 \times X_2$) was also significant ($F = 5.24$, $p < 0.05$), although to a lesser extent. In contrast, the interaction between microwave energy and extraction time ($X_1 \times X_3$) was not statistically significant ($p > 0.05$), indicating that these two factors act more independently within the studied range. Contour plots were constructed to visualize the interaction effects between the independent variables on the MAE of TPC from orange peel waste (Figure 5a,b).

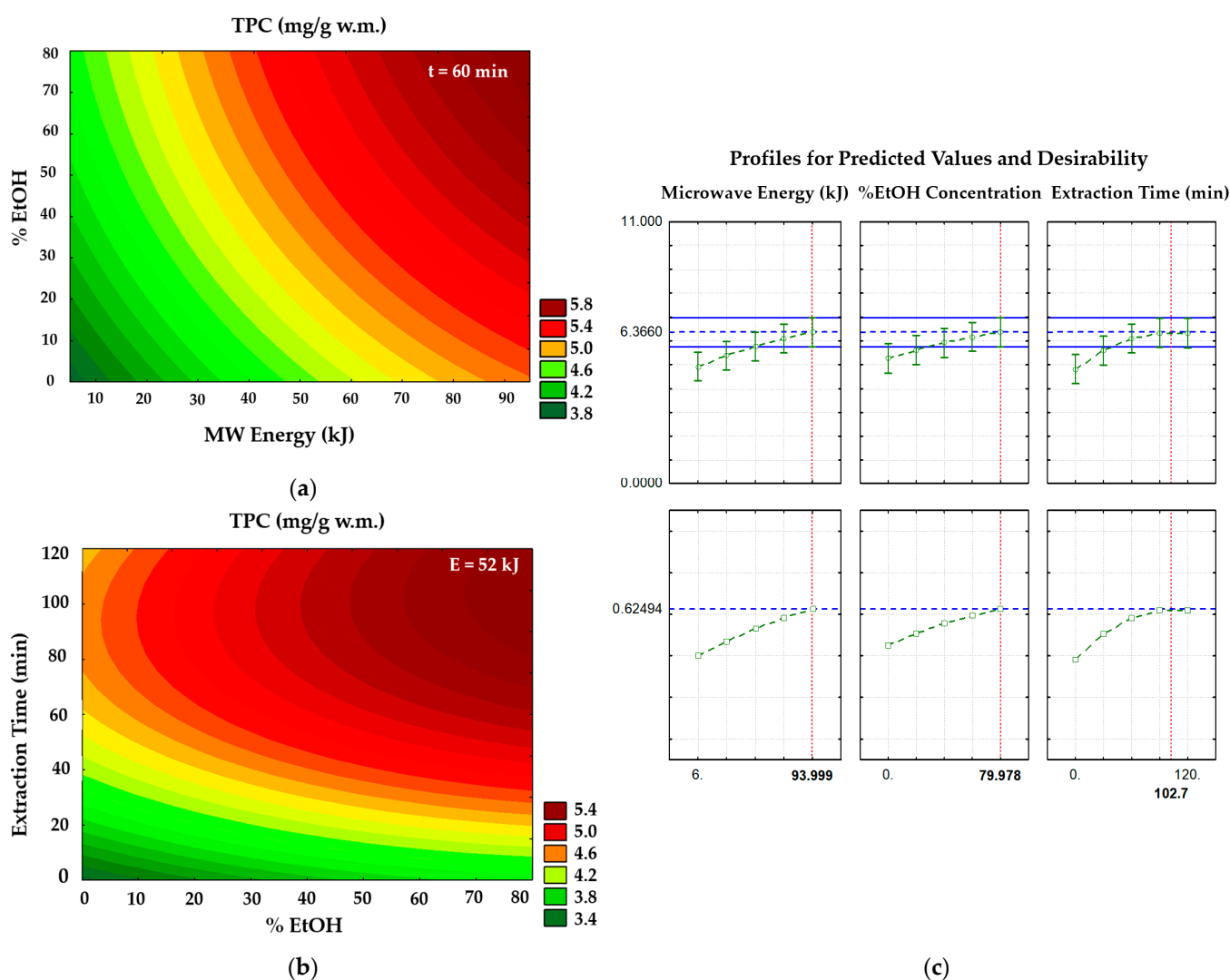


Figure 5. (a,b) Contour plots showing the effects of microwave energy, ethanol concentration and extraction time on the extracted total phenolic compounds from orange peels waste during MAE. (a) Microwave energy vs. ethanol concentration (extraction time equal to 60 min). (b) Ethanol concentration vs. extraction time (microwave energy equal to 52 kJ). (c) Profiles for predicted values of extracted total phenolic compounds and the desirability level for different factors for optimum extracted TPC concentration yield for MAE of orange peels waste.

The UAE method yielded moderate phenolic content, with a maximum experimental value of 5.85 mg/g w.m. obtained at 50 °C, 50% ethanol, and 120 min. The correlation between TPC and the independent variables (ethanol concentration—%EtOH, extraction time— t : X_1 , X_2) was expressed by the following polynomial equation:

$$C_{TPC} = 3.3500 + 0.0210 \cdot \%EtOH + 0.0228 \cdot t - 0.000220 \cdot \%EtOH^2 - 0.000114 \cdot t^2 + 0.000114 \cdot \%EtOH \cdot t \quad (3)$$

Regarding the individual effects of the variables, the linear terms of the ethanol concentration (X_1) and the extraction time (X_2) had the highest effect on TPC yield ($p < 0.001$). The quadratic terms of ethanol concentration (X_1^2) and of the extraction time (X_2^2) were also significant ($p < 0.001$). In addition, the interaction between extraction time and ethanol concentration (X_1X_2) affected the extracted TPC yield ($p < 0.05$). Contour plots were constructed to visualize the interaction effects between the independent variables on the UAE of TPC from orange peel waste (Figure 6a).

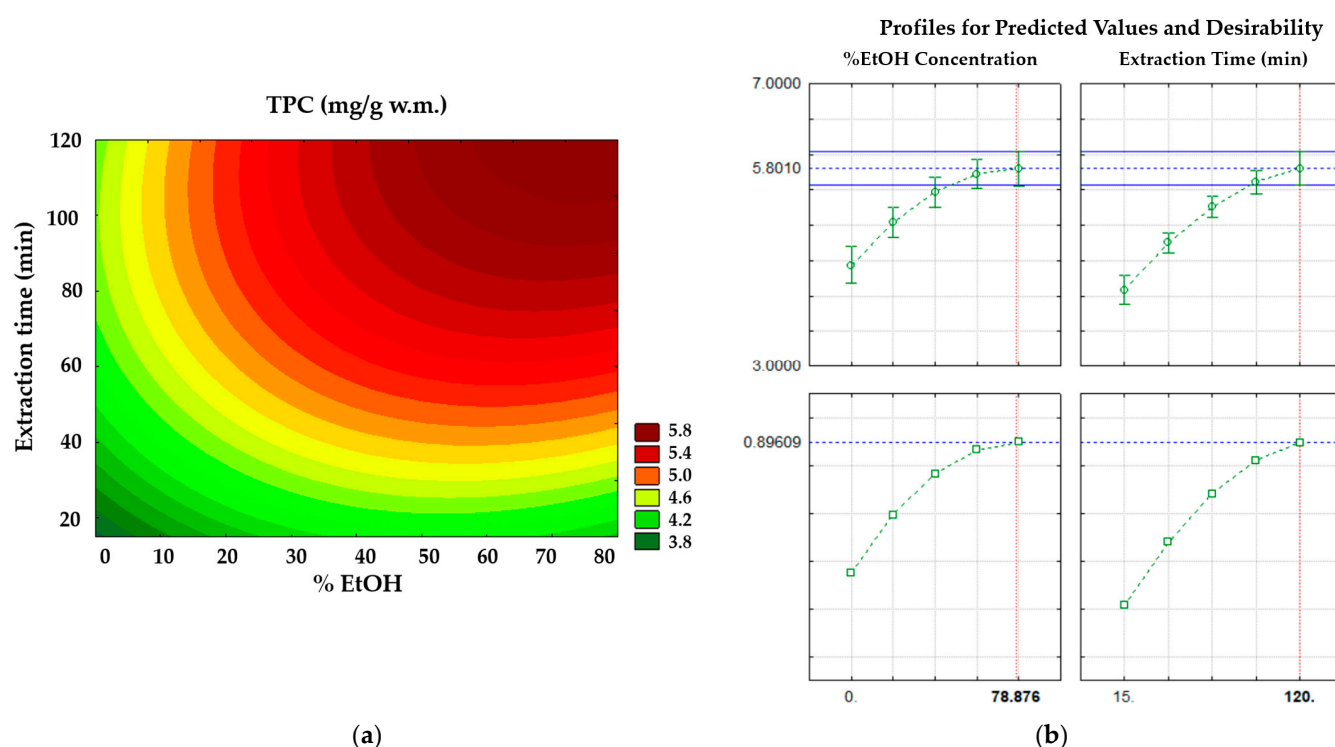


Figure 6. (a) Contour plot showing the effects of ethanol concentration and extraction time on the extracted total phenolic compounds from orange peels waste during UAE. (b) Profiles for predicted values of extracted total phenolic compounds and the desirability level for different factors for optimum extracted TPC concentration yield for UAE of orange peels waste.

The PEFAE method yielded moderate phenolic content, with a maximum experimental value of 5.33 mg/g w.m. obtained at 80% ethanol and 120 min. The correlation between TPC and the independent variables (ethanol concentration—%EtOH, extraction time— t : X_1 , X_2) was expressed by the following polynomial equation:

$$C_{TPC} = 3.8446 + 0.0184 \cdot t - 0.000082 \cdot t^2 \quad (4)$$

Regarding the individual effects of the variables, the linear and quadratic terms of the extraction time (X_2 , X_2^2) had the highest effect on TPC yield ($p < 0.001$). The linear term of ethanol concentration (X_1) did not affect the TPC yield ($p > 0.05$). The quadratic terms of ethanol concentration (X_1^2) and the interaction between two variables (X_1X_2) had no effect

on the extracted TPC yield ($p > 0.05$). Consequently, only extraction time had significant effect on TPC yield, while the ethanol concentration was not significant. Contour plots were constructed to visualize the interaction effects between the independent variables on the extraction of TPC from orange peel waste (Figure 7a).

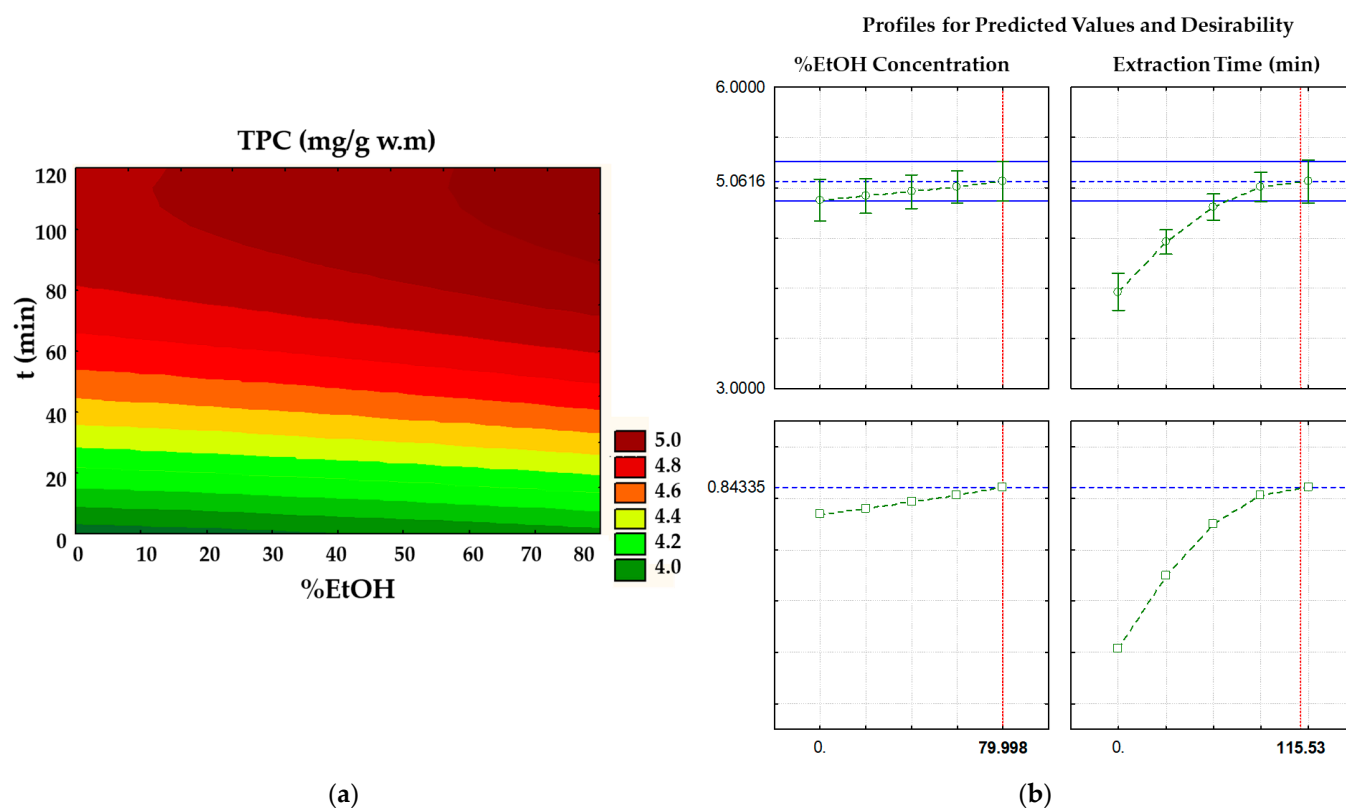


Figure 7. (a) Contour plot showing the effects of ethanol concentration and extraction time on the extracted total phenolic compounds from orange peels waste during PEFAE. (b) Profiles for predicted values of extracted total phenolic compounds and the desirability level for different factors for optimum extracted TPC concentration yield for PEFAE of orange peels waste.

Optimization using a desirability function approach revealed that, although maximum predicted responses were obtained under relatively high processing conditions, a plateau region was identified in all cases. Within this region, variations in process parameters did not result in statistically significant differences in TPC, indicating that the extraction processes are robust and allow flexibility in the selection of operating conditions. Based on the prediction intervals (PIs) obtained from the prediction profiles, a plateau region around the predicted optimum was identified for each extraction technique. Within this region, several combinations of the examined variables can produce statistically similar response values. Therefore, the final optimal conditions were selected within the plateau regions, considering not only the maximization of TPC but also energy efficiency and process sustainability.

For CE, the optimization analysis predicted that the maximum response could be achieved at 56 °C, 70% ethanol concentration, and an extraction time of approximately 103 min, with a predicted TPC value of 5.29 mg/g w.m (Figure 4c). Specifically, the extraction temperature can vary between 50 and 56 °C, the ethanol concentration between 55 and 70% (v/v), and the extraction time between 80 and 103 min without causing a statistically significant change in the predicted response (based on PIs obtained from the prediction profiles). Therefore, the final optimal conditions were selected within this plateau region, considering not only the maximization of the predicted response but also practical

considerations such as energy consumption and process efficiency. The selected operating conditions were 50 °C, 55% ethanol, and 80 min, representing a more energy-efficient and economically favorable extraction process while maintaining high phenolic recovery. Under these optimized CE conditions, the concentration of recovered flavonoids was 0.29 mg/g w.m., while the antioxidant capacity of the extracts was 0.98 mg/g w.m.

For MAE, the optimization analysis predicted that the maximum response could be achieved at 94 kJ microwave energy applied, 80% ethanol concentration, and an extraction time of approximately 103 min, with a predicted TPC value of 6.36 mg/g w.m. (Figure 5c). Specifically, the microwave energy applied between 50 and 94 kJ, the ethanol concentration between 50 and 80% (v/v), and the extraction time between 60 and 103 min did not cause a statistically significant change in the predicted response. The selected operating conditions were 50 kJ microwave energy input, 50% ethanol, and 60 min, representing a more energy-efficient and economically favorable extraction process while maintaining high phenolic recovery. Under these optimized MAE conditions, the concentration of recovered flavonoids was 0.23 mg/g w.m., while the antioxidant capacity of the extracts was 0.85 mg/g w.m.

For UAE, the optimization analysis predicted that the maximum response could be achieved at 50 °C, 79% ethanol concentration, and an extraction time of approximately 120 min, with a predicted TPC value of 5.80 mg/g w.m. (Figure 6b). Specifically, the ethanol concentration between 50 and 80% (v/v), and the extraction time between 90 and 120 min did not result in a statistically significant change of the predicted response. The selected operating conditions were 50 °C, 50% ethanol, and 90 min, representing a more energy-efficient and economically favorable extraction process while maintaining high phenolic recovery. Under these optimized UAE conditions, the concentration of recovered flavonoids was 0.48 mg/g w.m., while the antioxidant capacity of the extracts was 0.77 mg/g w.m.

For PEFAE, the optimization analysis predicted that the maximum response could be achieved at 80% ethanol concentration, and an extraction time of approximately 115 min, with a predicted TPC value of 5.06 mg/g w.m. (Figure 7b). Specifically, the ethanol concentration between 0 and 80% (v/v), and the extraction time between 75 and 115 min retained almost unaffected the predicted response. The selected operating conditions were 0% ethanol (only water), and 75 min, representing a more energy-efficient and economically favorable extraction process while maintaining high phenolic recovery. Under these optimized PEFAE conditions, the concentration of recovered flavonoids was 0.35 mg/g w.m., while the antioxidant capacity of the extracts was 1.12 mg/g w.m.

For all extraction techniques that are optimized, validation experiments confirmed the adequacy of the developed models, as the experimental values were in good agreement with the predicted responses. Under these optimized conditions for each extraction method, the recovered flavonoid content and antioxidant activity further supported the effectiveness of the selected parameters for each extraction technique.

4. Discussion

4.1. Comparison of Extraction Methods of Orange Peel Waste

The selection of the extraction method should consider not only the extraction yield but also other operational parameters such as energy consumption, solvent use, scalability, and selectivity toward target compounds. The comparative results (Table 2) highlight that the extraction technique significantly affects total phenolic content (TPC), flavonoid recovery, and antioxidant activity (Table 2).

Table 2. TPC (mg/g w.m), TF (mg/g w.m) and AA (mg/g w.m) of orange peels waste extracts under optimal conditions using different extraction techniques, such as CE, MAE, UAE and PEFAE.

Extraction Method	Optimal Conditions	TPC (mg/g w.m.)	TF (mg/g w.m.)	AA (mg/g w.m.)
CE (Conventional Extraction)	50 °C, 55% EtOH, 80 min	5.29 ± 0.13 ^c	0.29 ± 0.09 ^a	0.98 ± 0.10 ^a
MAE (Microwave-Assisted Extraction)	50 kJ, 50% EtOH, 60 min	6.36 ± 0.05 ^a	0.23 ± 0.07 ^{ab}	0.85 ± 0.11 ^a
UAE (Ultrasound-Assisted Extraction)	50 °C, 50% EtOH, 90 min	5.80 ± 0.17 ^b	0.48 ± 0.12 ^a	0.77 ± 0.08 ^b
PEFAE (Pulsed Electric Fields-Assisted Extraction)	25 °C, 0% EtOH, 75 min	5.06 ± 0.11 ^c	0.35 ± 0.10 ^a	1.12 ± 0.07 ^a

± represent standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significant differences ($p < 0.05$) between different extraction methods (one-way ANOVA analysis).

Among the evaluated methods, microwave-assisted extraction (MAE) achieved the highest TPC (~6.36 mg/g w.m.), confirming its superior extraction efficiency, 12% higher TPC yield compared to with conventional extraction (~5.29 mg/g w.m.). This performance is attributed to rapid internal heating and enhanced cell disruption, which improves mass transfer and facilitates phenolic release. However, despite its high TPC yield, MAE exhibited relatively lower flavonoid content (~0.23 mg/g w.m.) and antioxidant activity (~0.85 mg/g w.m.), indicating differences in extract composition. Ultrasound-assisted extraction (UAE) resulted in the highest flavonoid recovery (0.48 mg/g w.m., 66% higher than with CE), suggesting that acoustic cavitation enhances the selective extraction of specific phenolic subclasses. Nevertheless, UAE showed the lowest antioxidant activity (~0.77 mg/g w.m.), indicating that flavonoid concentration alone does not fully determine antioxidant capacity. The TPC yield achieved with UAE was 10% increased under milder conditions (lower temperature and ethanol concentration) compared to with conventional extraction. Pulsed electric fields-assisted extraction (PEFAE) produced the highest antioxidant activity (1.12 mg/g w.m.), despite yielding a lower TPC values (~5.06 mg/g w.m.) compared to MAE and UAE. Nevertheless, PEFAE achieved comparable TPC values to CE while using only water as extraction medium and without thermal input, highlighting its strong environmental advantages. Conventional extraction (CE) showed moderate performance across all response variables. Although widely applied, its efficiency was lower compared to intensified techniques, mainly due to longer extraction times and higher energy requirements.

Additionally, these green technologies indicate a clear trade-off between extraction efficiency and sustainability. To support a quantitative comparison of the sustainability-related performance of the extraction methods, the calculated energy demand and ethanol consumption were normalized per mg of extracted TPC (Table 3).

Table 3. Quantitative comparison of TPC recovery, ethanol use (mL/mg of extracted TPC), extraction conditions, and calculated energy efficiency of the different extraction methods (kJ/mg of extracted TPC).

Extraction Method	Recovered TPC (mg/g w.m.)	EtOH (%)	Time (min)	Temperature (°C)	Energy Calculation	Energy (kJ/mg Extracted TPC)	V _{EtOH} (mL/mg Extracted TPC)
CE	5.29	55	80	50	$1.6 \text{ kW} \times 80 \text{ min} + mC_p\Delta T$	484.14	1.04
MAE	6.36	50	60	25	50 kJ	2.62	0.79
UAE	5.80	50	90	50	$23.24 \text{ W} \times 90 \text{ min}$	7.21	0.86
PEFAE	5.06	0	75	25	$21 \text{ kJ/kg} + 5 \text{ kW} \times 1000 \text{ p} \times 15 \mu\text{s}$	0.05	0.00

Among the tested techniques, MAE resulted in the highest TPC yield (6.36 mg/g w.m.), indicating the most effective recovery of phenolic compounds under the selected conditions. However, when energy input was considered, PEFAE showed the most favorable performance, requiring only 0.05 kJ per mg of extracted TPC, while also operating at 25 °C and without ethanol addition. In comparison, MAE required 2.62 kJ/mg TPC and 0.79 mL EtOH/mg TPC, representing a good compromise between high phenolic recovery and reduced solvent use. UAE showed intermediate TPC recovery but higher energy demand (7.21 kJ/mg TPC), mainly due to the longer extraction time. UAE requires higher energy input, which may increase operational costs and limit its sustainability on an industrial scale. CE exhibited the highest calculated energy requirement (484.14 kJ/mg TPC), reflecting the prolonged operation of the water bath and the need for thermal processing. Additionally, both UAE and MAE use intermediate ethanol concentrations and milder thermal conditions enhance their environmental profile compared to CE. PEFAE stands out as the most energy-efficient and environmentally friendly technique. The process operates at room temperature and can utilize water as the extraction solvent (0% ethanol), significantly reducing both energy demand and solvent-related environmental impact. Although the TPC yield (~5.06 mg/g w.m.) is lower compared to MAE, PEFAE achieved the highest antioxidant activity (1.12 mg/g w.m.), indicating selective extraction of highly bioactive compounds. Furthermore, the shorter effective extraction time (75 min within the plateau region) and the absence of thermal degradation risks further enhance its sustainability profile.

Overall, the investigated emerging technologies enhance the recovery of high added-value compounds from citrus waste, enabling its valorization while improving process efficiency and sustainability. Therefore, the selection of the most appropriate extraction technique should consider not only extraction yield but also energy consumption, solvent use, and environmental impact, especially in the context of large-scale industrial applications.

4.2. Comparison with Literature

The enhanced performance of the assisted extraction techniques is attributed to distinct physical and physicochemical mechanisms that facilitate cell disruption, solvent penetration, and mass transfer. These mechanisms directly influence not only extraction yield but also the selectivity and composition of the recovered phenolic compounds. In microwave-assisted extraction (MAE), dielectric heating plays a dominant role. The interaction of microwave radiation with polar molecules and ions induces dipole rotation and ionic conduction, leading to rapid volumetric heating of the plant matrix. This internal heating generates localized pressure gradients within plant cells, causing cell wall rupture and enhanced permeability. As a result, the diffusion of phenolic compounds into the solvent is significantly accelerated. Moreover, the rapid heating reduces solvent viscosity and surface tension, further improving mass transfer. However, excessive energy input may lead to thermal degradation or transformation of heat-sensitive phenolic compounds, which could explain the lower antioxidant activity observed despite the higher total phenolic content [43–47]. Ultrasound-assisted extraction (UAE) is primarily governed by acoustic cavitation. The formation, growth, and implosive collapse of microbubbles generate intense local shear forces, micro-jets, and shockwaves. These phenomena mechanically disrupt plant tissues, increase the accessible surface area, and enhance solvent penetration into cellular structures [20,47–51]. Additionally, cavitation improves the mixing and reduces the boundary layer thickness around solid particles, facilitating faster mass transfer. The selective increase in flavonoid recovery observed in this study suggests that ultrasound may preferentially enhance the release of compounds located in specific cellular compartments or bound forms. In pulsed electric fields-assisted extraction (PEFAE), the key mechanism is electroporation. The application of short high-voltage pulses induces the

formation of pores in cell membranes, increasing their permeability without significant thermal effects [21,22,52–54]. This non-thermal mechanism allows the release of intracellular compounds while preserving their structural integrity. The high antioxidant activity observed in PEFAE extracts indicates that this technique may favor the extraction of highly bioactive and less degraded phenolic compounds [53]. Furthermore, the absence of heating minimizes oxidation reactions and prevents the degradation of sensitive molecules, contributing to the preservation of functional properties [52,54]. Beyond the individual mechanisms, all intensified techniques share a common effect: the reduction in internal and external mass transfer limitations. By disrupting cell structures and enhancing solvent–matrix interactions, these technologies shift the extraction process from diffusion-controlled to more kinetically favorable regimes. This explains the reduced extraction times and improved efficiencies observed compared to conventional extraction.

The different trends observed for TPC, TF, and AA indicate that the extraction techniques did not only affect the total amount of extracted phenolic compounds, but also the qualitative composition and antioxidant effectiveness of the extracts. This distinction is important because TPC, TF, and AA are based on different analytical principles and do not necessarily describe the same chemical fraction. Under the tested conditions, MAE produced the highest TPC, most likely due to rapid volumetric heating, increased solvent penetration, and enhanced disruption of the plant matrix. However, the lower AA observed for MAE compared with PEFAE suggests that the larger phenolic pool recovered by MAE may have contained compounds with lower radical-scavenging efficiency, or that part of the thermosensitive antioxidant fraction may have been degraded, oxidized, or transformed during microwave treatment. UAE resulted in the highest TF value, indicating that acoustic cavitation may have favored the release of flavonoid-rich fractions or flavonoids associated with specific cellular structures. Nevertheless, this increase in TF was not accompanied by the highest AA, suggesting that the extracted flavonoids may not necessarily have been the most active radical-scavenging species, or that their antioxidant contribution depended on their specific molecular form. In contrast, PEFAE produced lower TPC than MAE and UAE but the highest AA. This may indicate that the non-thermal electroporation mechanism promoted the selective release and/or better preservation of compounds with higher antioxidant efficiency, even if the total amount of Folin-reactive compounds was lower. Therefore, antioxidant activity cannot be explained solely by total phenolic or flavonoid concentration but should also be interpreted in relation to the phenolic profile and the structure–activity relationships of the extracted compounds. These results suggest that MAE was the most effective technique for maximizing total phenolic recovery, UAE was more favorable for flavonoid extraction, whereas PEFAE provided the most active antioxidant extract under mild, non-thermal conditions. It should be noted that the Folin–Ciocalteu assay is not specific exclusively to phenolic compounds, as other reducing substances naturally present in citrus peel may also contribute to the measured response. Therefore, TPC values should be interpreted as an estimate of total reducing phenolic-like capacity rather than as a direct quantification of individual phenolic compounds. Future studies should include HPLC or UPLC profiling of major orange peel phenolics, such as hesperidin, narirutin, naringin, ferulic acid, and caffeic acid derivatives, to better explain differences in antioxidant activity and extraction selectivity.

The present findings are consistent with previous studies reporting the effectiveness of intensified extraction techniques for phenolic recovery from orange peel waste. Reported values for total polyphenols in orange processing waste vary widely (36.3–66,360 mg GAE/kg), mainly due to differences in raw material composition, extraction conditions, and solvent systems. García-Martín et al. [44] proved that MAE (500 W, 1/20 m/v sample-to-solvent ratio) assisted extraction is highly efficient (20.2 g/100 OW) for

phenolic recovery from orange waste with optimal yields achieved using aqueous ethanol solutions (50% ethanol, 75 °C, 10.8–15 min). Petrotos et al. [55] reported that MAE was optimized at industrial scale using response surface methodology, considering microwave power, solvent ratio, and extraction time to maximize the phenolic recovery from orange pomace. The optimum total polyphenols yield was 37,667 mg GAE Kg⁻¹ of dry orange pomace with microwave power 4484.7 W for 15 min and with water to solid orange pomace ratio equal to 19.6). Boukroufa et al. [56] highlight the effectiveness of UAE as an intensification technique for phenolic extraction from orange peel waste. UAE (0.956 W/cm², 59.83 °C) significantly enhanced polyphenol recovery compared to conventional extraction, achieving an increase of approximately 30% in TPC yield (50.02 mg GA/100 g dm). El Kantar et al. [57] showed that UAE (30 min at 50 °C) significantly improved polyphenol recovery (up to 112% compared to conventional extraction) from orange waste, particularly when combined with infrared pretreatment (20 min). These results demonstrate the strong synergistic effect of pretreatment and UAE on extraction efficiency. De Miera et al. [58] showed that MAE (373 K, 6 min, 80% ethanol) led to efficient phenolic recovery (7.2 ± 0.1 mg gallic acid equivalent GAE/g) from orange peel waste, while UAE (10 W for 6 min, 80% ethanol) also demonstrated high performance in phenolic compounds yield (8.8 ± 0.0 mg GAE/g OPW) with lower energy consumption (~18 kJ). Both techniques significantly reduced extraction time compared to conventional extraction. Luengo et al. [59] proved that PEF treatment (7 kV/cm, 20 pulses of 3 µs) significantly enhanced polyphenol extraction from orange peels through electroporation effects, with total polyphenol extraction yield increased by 159% compared to when untreated, without using organic solvents. Irina et al. [60] investigated that MAE (80% ethanol, m/v: 5 g: 50 mL, 10 s, 35 °C, 170 W) resulted in high concentration of total phenolic content (2.363 ± 0.014 g GAE/100 g orange peel powder) extracted from orange peel powder and MAE also enabled efficient recovery of key flavonoids such as hesperidin and neohesperidin. UAE (80% ethanol, m/v: 5 g: 50 mL, 30 min, 35 °C, 125 W) was also examined and led to phenolic recovery equal to approximately 2.1 g GAE/100 g orange peel powder. Angoy et al. [61] combined MAE with centrifugal force and they presented enhanced recovery of phenolics and essential oils. The process significantly reduced extraction time compared to hydrodistillation. Belsi et al. [62] investigated that UAE could enable efficient recovery of polyphenols (approximately 12.3 mg GAE/g dm) from waste orange peels using water as a green solvent (80% amplitude, 15 min, duty cycle 2/2) with reduced processing time. Khan et al. [63] used UAE that achieved improved extraction yield of phenolic compounds (275.8 mg of gallic acid equivalent/100 g FW) from orange peels compared to conventional techniques under optimized conditions (150 W, 40 °C, ethanol:water 4:1) using central composite design. Haya et al. [64] showed that the highest phenolic compounds extraction yield from orange peels (115.52 mg GAE/g dm) achieved at 25 °C with a stirring speed of 400 rpm in neutral medium (pH~6) and a particle size of 605 µm. Hernández-Carranza et al. [65] optimized the phenolic recovery from orange peel waste using RSM with optimal values around 60 °C for 12 h. The study confirmed that higher temperatures enhance mass transfer and phenolic recovery (729 mg GAE/100 g dm). M'hiri et al. [66] confirmed that MAE (200 W, 180 s) had the highest total phenolic and flavonoid contents from orange peels, followed by UAE (125 W, 30 min, 35 °C) and conventional extraction. The results confirmed the rapid extraction capability of MAE, achieving high yields within very short processing times.

In the present study, the differences in extraction performance among MAE, UAE, and PEFAE can be attributed to the balance between thermal and non-thermal effects, the intensity of cell disruption, and the degree of selectivity toward specific phenolic compounds. These mechanistic insights are essential for optimizing extraction processes and tailoring them to specific industrial applications, particularly in the context of sustain-

able valorization of agro-industrial waste. Additionally, these novel assisted extraction technologies offer advantages such as improved extraction efficiency, reduced extraction time and decreased solvent usage compared to the conventional method. These pretreatments led to enhanced extractability of high added value compounds from citrus waste increasing recovery yields and making byproduct valorization commercially attractive for the industry.

5. Conclusions

This study demonstrated that emerging extraction technologies significantly enhance the recovery of bioactive compounds from orange peel waste compared to conventional extraction. Among the evaluated methods, microwave-assisted extraction (MAE) achieved the highest total phenolic content, confirming its superior extraction efficiency due to intensified mass transfer and rapid heating effects. Ultrasound-assisted extraction (UAE) provided a competitive alternative, offering high extraction yields under comparatively milder conditions and lower energy input. Pulsed electric fields-assisted extraction (PEFAE), although yielding slightly lower total phenolic content, exhibited the highest antioxidant activity and operated under non-thermal conditions using water as the extraction medium, highlighting its strong potential as a sustainable and environmentally friendly technique. Conventional extraction, while widely used, showed lower efficiency and higher energy demand, confirming the advantages of process intensification. The results highlight the potential for valorization of citrus peel byproducts as a source of natural antioxidants and functional ingredients. Integrating green extraction technologies into agro-industrial processes can contribute to waste minimization, circular economy practices, and the development of value-added nutraceutical and functional food products. Future research should focus on the scale-up of these extraction processes, techno-economic evaluation, and life cycle assessment to determine their industrial feasibility. In addition, further studies on the stability, bioavailability, and functionality of the extracted compounds are necessary to support their incorporation into nutraceutical and functional food applications.

Author Contributions: Conceptualization, V.A.; methodology, V.A. and M.G. (Marianna Giannoglou); software, V.A. and A.N.; validation, A.N., K.P.M. and V.A.; formal analysis, V.A. and M.G. (Marianna Giannoglou); investigation, A.N., K.P.M. and V.A.; resources, G.K.; data curation, A.N., K.P.M. and V.A.; writing—original draft preparation, V.A.; writing—review and editing, P.T., M.G. (Maria Giannakourou), and G.K.; visualization, P.T., M.G. (Maria Giannakourou) and G.K.; supervision, P.T., G.K. and M.G. (Maria Giannakourou); project administration, G.K.; funding acquisition, G.K. All authors have read and agreed to the published version of the manuscript.

Funding: A part of this research was co-financed by the European Regional Development Fund of the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call Greece 2.0 (project code: TAEDK-06176).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data supporting the findings of this study are available from the corresponding author upon reasonable request.

Acknowledgments: The authors would like to acknowledge the technical support provided by the laboratory staff and express their gratitude to Institute of Technology of Agricultural Products ELGO DEMETER for providing research facilities and resources essential for this study.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Appendix A

This appendix presents the complete experimental datasets for total flavonoids (TF) and antioxidant activity (AA) obtained from orange peel waste extracts under the different extraction techniques investigated in this study. Tables A1–A8 include the results for conventional extraction (CE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and pulsed electric field-assisted extraction (PEFAE), as affected by extraction time, ethanol concentration, temperature, microwave energy input, or PEF treatment conditions, depending on the extraction method. These supplementary tables provide the full experimental basis supporting the response surface modelling, optimization, and comparative interpretation of TF recovery and AA discussed in the main manuscript.

Table A1. Concentration of TF (mg/g w.m.) of orange peel waste extracts as a function of extraction time (0–120 min) for 80, 50, and 0% ethanol in conventional extraction at 70, 50, and 30 °C.

Conventional Extraction (CE)						
Total Flavonoids (mg/g w.m.)						
Extraction Temperature (°C)	70 °C			50 °C		
Ethanol Concentration (%)						
Extraction time (Min)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)
15	0.361 ± 0.036 klm	0.022 ± 0.014 u	0.027 ± 0.002 u	0.475 ± 0.048 ghij	0.040 ± 0.001 tu	0.057 ± 0.001 stu
30	0.403 ± 0.040 ijk	0.112 ± 0.036 qrst	0.137 ± 0.013 opqrst	0.522 ± 0.052 efgh	0.201 ± 0.024 mnopqr	0.057 ± 0.011 stu
45	0.454 ± 0.045 hijk	0.163 ± 0.010 opqr	0.154 ± 0.022 opqrs	0.559 ± 0.056 defg	0.298 ± 0.036 klm	0.193 ± 0.008 nopqr
60	0.467 ± 0.047 ghij	0.167 ± 0.001 opqr	0.163 ± 0.008 opqr	0.529 ± 0.059 efgh	0.210 ± 0.033 mnopq	0.186 ± 0.010 nopqr
90	0.496 ± 0.050 fghi	0.175 ± 0.025 opqr	0.205 ± 0.010 mnopq	0.615 ± 0.025 cde	0.297 ± 0.030 klm	0.203 ± 0.009 mnopq
120	0.556 ± 0.055 defg	0.209 ± 0.002 mnopq	0.218 ± 0.003 mnop	0.678 ± 0.036 bc	0.277 ± 0.012 lmn	0.195 ± 0.022 nopqr
Extraction temperature (°C)	30 °C					
Ethanol Concentration (%)						
Extraction time (min)	80% EtOH		50% EtOH		0% EtOH (H ₂ O)	
15	0.525 ± 0.036 ^{efgh}		0.102 ± 0.014 ^{rstu}		0.027 ± 0.008 ^u	
30	0.581 ± 0.014 ^{cdef}		0.187 ± 0.036 ^{nopqr}		0.129 ± 0.036 ^{pqrst}	
45	0.632 ± 0.008 ^{bcd}		0.281 ± 0.011 ^{lmn}		0.193 ± 0.022 ^{nopqr}	
60	0.653 ± 0.036 ^{bcd}		0.383 ± 0.025 ^{jkl}		0.207 ± 0.011 ^{mnopq}	
90	0.725 ± 0.013 ^{ab}		0.401 ± 0.028 ^{ijk}		0.229 ± 0.047 ^{mno}	
120	0.792 ± 0.010 ^a		0.405 ± 0.033 ^{ijk}		0.231 ± 0.024 ^{mno}	

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (different extraction times, ethanol concentrations and extraction temperatures) based on the results obtained from factorial ANOVA analysis.

Table A2. Antioxidant activity (mg/g w.m.) of orange peel waste extracts as a function of extraction time (0–120 min) for 80, 50, and 0% ethanol in conventional extraction at 70, 50, and 30 °C.

Conventional Extraction (CE)						
Antioxidant activity (mg/g w.m.)						
Extraction Temperature (°C)	70 °C			50 °C		
	Ethanol Concentration (%)					
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)
15	1.283 ± 0.125 ^a	1.256 ± 0.065 ^a	0.930 ± 0.147 _{bcde}	1.141 ± 0.075 ^a	0.994 ± 0.033 _{abc}	0.705 ± 0.028 _{def}
30	1.023 ± 0.145 _{abc}	1.082 ± 0.012 ^{ab}	0.737 ± 0.098 _{defg}	1.026 ± 0.089 ^{ab}	0.916 ± 0.124 _{abcd}	0.664 ± 0.091 ^{ef}
45	0.900 ± 0.015 _{bcdef}	0.940 ± 0.055 _{bcd}	0.668 ± 0.111 _{efg}	0.931 ± 0.050 _{abcd}	0.861 ± 0.136 _{bcde}	0.627 ± 0.088 ^{ef}
60	0.759 ± 0.025 _{cdefg}	0.900 ± 0.078 _{bcdef}	0.650 ± 0.152 ^{fg}	0.859 ± 0.088 _{bcde}	0.801 ± 0.042 _{bcdef}	0.613 ± 0.090 ^f
90	0.627 ± 0.036 ^g	0.771 ± 0.098 _{cdefg}	0.622 ± 0.025 ^g	0.744 ± 0.029 _{def}	0.769 ± 0.048 _{cdef}	0.668 ± 0.079 ^{ef}
120	0.672 ± 0.012 _{efg}	0.630 ± 0.015 ^g	0.599 ± 0.069 ^g	0.856 ± 0.076 _{bcde}	0.737 ± 0.022 _{def}	0.728 ± 0.082 _{def}
Extraction temperature (°C)	30 °C					
	Ethanol Concentration (%)					
Extraction time (min)	80% EtOH		50% EtOH		0% EtOH (H ₂ O)	
15	0.810 ± 0.066 _{cdef}		0.751 ± 0.044 _{defg}		0.618 ± 0.028 ^g	
30	0.888 ± 0.024 _{abcde}		0.810 ± 0.026 _{cdef}		0.691 ± 0.026 ^{fg}	
45	0.957 ± 0.078 _{abc}		0.870 ± 0.061 _{bcdef}		0.728 ± 0.032 ^{efg}	
60	0.995 ± 0.056 _{abc}		0.925 ± 0.125 _{abcd}		0.879 ± 0.071 _{abcde}	
90	1.050 ± 0.063 ^{ab}		0.985 ± 0.058 _{abc}		0.916 ± 0.097 _{abcd}	
120	1.063 ± 0.030 ^a		0.998 ± 0.069 ^{ab}		0.939 ± 0.034 _{abc}	

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (different extraction times and ethanol concentrations) for each extraction temperature based on the results obtained from factorial ANOVA analysis.

Table A3. Concentration of TF (mg/g w.m.) during MAE of orange peel waste for extraction time ranged from 15 to 120 min, solvent concentration equal to 0, 50, and 80% and microwave energy input ranged from 6.12 to 93.6 kJ.

Microwave Assisted Extraction (MAE)						
Total Flavonoids (mg/g w.m.)						
Microwave Energy Applied	6.1 kJ			12.2 kJ		
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH	80% EtOH	50% EtOH	0% EtOH
15	0.543 ± 0.015 _c	0.011 ± 0.025 _k	0.005 ± 0.005 _k	0.192 ± 0.017 _{fg}	0.002 ± 0.002 _k	0.015 ± 0.001 _{ik}
30	0.589 ± 0.036 _{bc}	0.074 ± 0.015 _i	0.010 ± 0.002 _k	0.298 ± 0.014 _{de}	0.035 ± 0.008 _{ik}	0.044 ± 0.022 _{ik}

Table A3. Cont.

45	0.602 ± 0.015 ^b	0.112 ± 0.011 ^{ghi}	0.019 ± 0.002	0.387 ± 0.065 ^{bc}	0.087 ± 0.006 ^{hi}	0.120 ± 0.015 ^{gh}
60	0.623 ± 0.022 ^b	0.129 ± 0.019 ^{fgh}	0.078 ± 0.015 ^{hi}	0.403 ± 0.025 ^{abc}	0.142 ± 0.011 ^{gh}	0.184 ± 0.016 ^g
90	0.729 ± 0.010 ^a	0.167 ± 0.001 ^{ef}	0.133 ± 0.026 ^{fg}	0.429 ± 0.014 ^{ab}	0.171 ± 0.025 ^g	0.264 ± 0.017 ^{ef}
120	0.741 ± 0.026 ^a	0.230 ± 0.014 ^d	0.205 ± 0.011 ^{de}	0.467 ± 0.036 ^a	0.180 ± 0.029 ^g	0.344 ± 0.020 ^{cd}
14.4 kJ			23.4 kJ			
15	0.247 ± 0.015 ^D	0.011 ± 0.008 ^D	0.158 ± 0.010 ^A	0.374 ± 0.017 ^{defg}	0.171 ± 0.017 ^k	0.230 ± 0.014 ^{ik}
30	0.302 ± 0.028 ^{CD}	0.108 ± 0.025 ^C	0.192 ± 0.017 ^A	0.403 ± 0.016 ^{cde}	0.218 ± 0.022 ^{ik}	0.285 ± 0.025 ^{ghi}
45	0.353 ± 0.036 ^{BCD}	0.112 ± 0.017 ^C	0.200 ± 0.026 ^A	0.441 ± 0.006 ^{cd}	0.251 ± 0.036 ^{hik}	0.361 ± 0.022 ^{defg}
60	0.391 ± 0.096 ^{ABC}	0.158 ± 0.036 ^{BC}	0.238 ± 0.015 ^A	0.640 ± 0.030 ^b	0.306 ± 0.025 ^{fghi}	0.412 ± 0.019 ^{cde}
90	0.458 ± 0.015 ^{AB}	0.180 ± 0.015 ^B	0.276 ± 0.36 ^A	0.725 ± 0.036 ^{ab}	0.332 ± 0.012 ^{efgh}	0.437 ± 0.065 ^{cd}
120	0.488 ± 0.022 ^A	0.251 ± 0.022 ^A	0.302 ± 0.015 ^A	0.741 ± 0.022 ^a	0.378 ± 0.036 ^{def}	0.484 ± 0.047 ^c
24.5 kJ			28.8 kJ			
15	0.061 ± 0.036 ^{hi}	0.066 ± 0.025 ^{hi}	0.019 ± 0.005 ⁱ	0.361 ± 0.015 ^C	0.154 ± 0.014 ^C	0.251 ± 0.011 ^C
30	0.091 ± 0.014 ^{ghi}	0.108 ± 0.036 ^{fgh}	0.120 ± 0.006 ^{efgh}	0.399 ± 0.022 ^C	0.171 ± 0.036 ^{BC}	0.260 ± 0.025 ^{BC}
45	0.108 ± 0.025 ^{fgh}	0.129 ± 0.033 ^{efgh}	0.163 ± 0.011 ^{defg}	0.433 ± 0.019 ^{BC}	0.188 ± 0.022 ^{BC}	0.294 ± 0.069 ^{ABC}
60	0.188 ± 0.012 ^{def}	0.142 ± 0.030 ^{defgh}	0.273 ± 0.014 ^{bc}	0.463 ± 0.010 ^{BC}	0.205 ± 0.015 ^{BC}	0.328 ± 0.017 ^{ABC}
90	0.201 ± 0.047 ^{cde}	0.180 ± 0.047 ^{def}	0.298 ± 0.036 ^b	0.496 ± 0.011 ^{AB}	0.235 ± 0.025 ^B	0.344 ± 0.020 ^{AB}
120	0.226 ± 0.025 ^{bcd}	0.192 ± 0.025 ^{cdef}	0.412 ± 0.014 ^a	0.564 ± 0.069 ^A	0.319 ± 0.022 ^A	0.361 ± 0.016 ^A
46.8 kJ			57.6 kJ			
15	0.222 ± 0.005	0.158 ± 0.012	0.167 ± 0.012	0.205 ± 0.010 ^{defg}	0.078 ± 0.011 ⁱ	0.061 ± 0.008 ⁱ
30	0.302 ± 0.022	0.205 ± 0.036	0.218 ± 0.025	0.251 ± 0.025 ^{cde}	0.133 ± 0.025 ^{ghi}	0.087 ± 0.019 ^{hi}
45	0.349 ± 0.014	0.239 ± 0.001	0.277 ± 0.036	0.311 ± 0.036 ^{bc}	0.158 ± 0.036 ^{fgh}	0.158 ± 0.022 ^{fgh}
60	0.3470 ± 0.016	0.289 ± 0.024	0.332 ± 0.015	0.378 ± 0.008 ^{ab}	0.184 ± 0.014 ^{efg}	0.188 ± 0.036 ^{defg}
90	0.391 ± 0.028	0.306 ± 0.011	0.357 ± 0.028	0.391 ± 0.019 ^a	0.201 ± 0.049 ^{defg}	0.264 ± 0.044 ^{cd}
120	0.416 ± 0.036	0.315 ± 0.018	0.391 ± 0.033	0.425 ± 0.008 ^a	0.235 ± 0.016 ^{cdef}	0.370 ± 0.022 ^{ab}
93.6 kJ						
15	0.171 ± 0.025 ^{ghi}		0.137 ± 0.010 ^{hi}		0.112 ± 0.014 ⁱ	
30	0.188 ± 0.012 ^{fgh}		0.175 ± 0.036 ^{gh}		0.167 ± 0.026 ^{ghi}	
45	0.205 ± 0.001 ^{efg}		0.209 ± 0.011 ^{efg}		0.201 ± 0.033 ^{fg}	
60	0.226 ± 0.036 ^{defg}		0.243 ± 0.015 ^{cdef}		0.247 ± 0.015 ^{cdef}	
90	0.239 ± 0.012 ^{cdef}		0.273 ± 0.019 ^{bcd}		0.289 ± 0.014 ^{abc}	
120	0.264 ± 0.020 ^{cde}		0.327 ± 0.007 ^{ab}		0.336 ± 0.009 ^a	

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (different extraction times and ethanol concentrations) for each microwave energy applied based on the results obtained from factorial ANOVA analysis. Capital letters indicate the significant differences ($p < 0.05$) between different extraction times (one-way ANOVA analysis) for each microwave condition applied (energy input) and each solvent concentration.

Table A4. Antioxidant activity (mg/g w.m.) during MAE of orange peel waste for extraction time ranged from 0 to 120 min, solvent concentration equal to 0, 50, and 80% and microwave energy input ranged from 6.12 to 93.6 kJ.

Microwave Assisted Extraction (MAE)						
Antioxidant Activity (mg/g w.m.)						
Microwave Energy Applied	6.1 kJ			12.2 kJ		
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH	80% EtOH	50% EtOH	0% EtOH
15	0.774 ± 0.078 D	0.627 ± 0.125 B	0.755 ± 0.025 C	0.815 ± 0.048 C	0.687 ± 0.096 B	0.820 ± 0.014 C
30	0.852 ± 0.036 CD	0.687 ± 0.048 AB	0.865 ± 0.014 B	0.875 ± 0.014 C	0.747 ± 0.074 AB	0.888 ± 0.058 BC
45	0.916 ± 0.022 BC	0.709 ± 0.074 AB	0.911 ± 0.025 AB	0.925 ± 0.022 BC	0.834 ± 0.056 AB	0.953 ± 0.069 ABC
60	0.962 ± 0.015 AB	0.769 ± 0.096 AB	0.939 ± 0.039 AB	0.994 ± 0.014 AB	0.870 ± 0.074 AB	1.003 ± 0.099 AB
90	0.998 ± 0.010 AB	0.820 ± 0.036 AB	0.971 ± 0.011 A	1.076 ± 0.036 A	0.926 ± 0.096 A	1.049 ± 0.048 AB
120	1.031 ± 0.009 A	0.870 ± 0.011 A	0.994 ± 0.074 A	1.104 ± 0.078 A	0.944 ± 0.036 A	1.091 ± 0.074 A
14.4 kJ			23.4 kJ			
15	0.934 ± 0.014 E	0.893 ± 0.065 D	0.783 ± 0.045 D	1.214 ± 0.066 D	1.182 ± 0.047 D	1.141 ± 0.065 D
30	1.017 ± 0.025 CE	0.957 ± 0.036 CD	0.801 ± 0.036 CD	1.287 ± 0.025 CD	1.237 ± 0.069 CD	1.205 ± 0.096 CD
45	1.081 ± 0.036 CD	1.031 ± 0.014 BC	0.875 ± 0.033 BCD	1.370 ± 0.036 C	1.306 ± 0.066 BCD	1.241 ± 0.055 BCD
60	1.150 ± 0.069 BC	1.104 ± 0.078 AB	0.943 ± 0.015 ABC	1.434 ± 0.015 BC	1.347 ± 0.030 BC	1.310 ± 0.044 ABC
90	1.218 ± 0.015 AB	1.163 ± 0.036 A	1.008 ± 0.085 AB	1.532 ± 0.088 AB	1.421 ± 0.096 AB	1.370 ± 0.036 AB
120	1.287 ± 0.036 A	1.232 ± 0.025 A	1.085 ± 0.069 A	1.608 ± 0.069 A	1.535 ± 0.011 A	1.457 ± 0.015 A
24.5 kJ			28.8 kJ			
15	0.814 ± 0.014 ij	0.709 ± 0.014 k	0.829 ± 0.069 hij	0.732 ± 0.017 ^f	0.789 ± 0.014 ef	0.985 ± 0.014 abcd
30	0.887 ± 0.036 ghij	0.797 ± 0.036 jk	0.916 ± 0.036 fgh	0.896 ± 0.022 bcde	0.833 ± 0.069 def	0.872 ± 0.069 cdef
45	1.085 ± 0.014 bc	0.898 ± 0.028 fghi	0.966 ± 0.022 defg	0.999 ± 0.014 abc	0.985 ± 0.036 abcd	0.845 ± 0.011 cdef
60	1.182 ± 0.058 ab	0.953 ± 0.011 efg	0.998 ± 0.015 cdef	1.037 ± 0.085 ab	1.078 ± 0.010 a	0.977 ± 0.069 abcd
90	1.214 ± 0.030 a	1.031 ± 0.036 cde	1.031 ± 0.022 cde	1.044 ± 0.079 ab	1.047 ± 0.046 ab	1.060 ± 0.075 a
120	1.232 ± 0.011 a	1.054 ± 0.047 cd	1.049 ± 0.008 cde	1.040 ± 0.069 ab	1.043 ± 0.022 ab	1.047 ± 0.088 ab

Table A4. *Cont.*

46.8 kJ							57.6 kJ		
15	0.572 ± 0.098 C	0.476 ± 0.008 B	0.434 ± 0.069 C	0.434 ± 0.048 C	0.453 ± 0.069 C	0.301 ± 0.033 D			
30	0.594 ± 0.069 BC	0.572 ± 0.056 AB	0.535 ± 0.033 BC	0.512 ± 0.055 BC	0.517 ± 0.044 BC	0.397 ± 0.096 CD			
45	0.677 ± 0.047 ABC	0.613 ± 0.036 AB	0.622 ± 0.047 AB	0.590 ± 0.096 ABC	0.581 ± 0.056 ABC	0.434 ± 0.044 C			
60	0.723 ± 0.044 ABC	0.663 ± 0.105 A	0.641 ± 0.056 AB	0.613 ± 0.055 ABC	0.636 ± 0.023 AB	0.494 ± 0.036 BC			
90	0.755 ± 0.056 AB	0.691 ± 0.063 A	0.660 ± 0.066 AB	0.654 ± 0.074 AB	0.686 ± 0.039 A	0.567 ± 0.009 AB			
120	0.796 ± 0.036 A	0.704 ± 0.056 A	0.696 ± 0.022 A	0.773 ± 0.069 A	0.718 ± 0.096 A	0.631 ± 0.014 A			
93.6 kJ									
15	0.792 ± 0.015 ^C		0.626 ± 0.056 ^C		0.563 ± 0.096 ^C				
30	0.805 ± 0.036 ^C		0.668 ± 0.069 ^{BC}		0.641 ± 0.047 ^{BC}				
45	0.847 ± 0.025 ^{BC}		0.723 ± 0.047 ^{ABC}		0.755 ± 0.015 ^{AB}				
60	0.906 ± 0.036 ^{AB}		0.782 ± 0.056 ^{ABC}		0.778 ± 0.066 ^{AB}				
90	0.920 ± 0.055 ^{AB}		0.814 ± 0.068 ^{AB}		0.796 ± 0.057 ^{AB}				
120	0.938 ± 0.002 ^A		0.856 ± 0.067 ^A		0.833 ± 0.088 ^A				

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different lowercase letters indicate significant differences ($p < 0.05$) among the different extraction conditions (different extraction times and ethanol concentrations) for each microwave energy applied based on the results obtained from factorial ANOVA analysis. Capital letters indicate the significant differences ($p < 0.05$) between different extraction times (one-way ANOVA analysis) for each microwave condition applied (energy input) and each solvent concentration.

Table A5. TF concentration (mg/g w.m) during ultrasound (90% amplitude) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 50 °C extraction temperature.

Ultrasounds Assisted Extraction (UAE)				
Total Flavonoids (mg/g w.m.)				
Extraction Temperature (°C)	50 °C			
Extraction Time (min)	Ethanol Concentration (%)			
	80% EtOH	50% EtOH	0% EtOH (H ₂ O)	
15	0.171 ± 0.035 de	0.209 ± 0.018 cd	0.057 ± 0.019 f	
30	0.185 ± 0.014 cd	0.331 ± 0.019 ab	0.095 ± 0.013 ef	
45	0.192 ± 0.027 cd	0.335 ± 0.027 ab	0.192 ± 0.028 cd	
60	0.221 ± 0.039 cd	0.378 ± 0.038 a	0.197 ± 0.025 cd	
90	0.268 ± 0.022 bc	0.382 ± 0.039 a	0.188 ± 0.026 cd	
120	0.265 ± 0.012 bc	0.379 ± 0.030 a	0.192 ± 0.050 cd	

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Table A6. Antioxidant activity (mg/g w.m) during ultrasound (90% amplitude) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 50 °C extraction temperature. Error bars represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Ultrasounds Assisted Extraction (UAE)			
Antioxidant Activity (mg/g w.m.)			
Extraction Temperature (°C)	50 °C		
Ethanol Concentration (%)			
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)
15	1.150 ± 0.014 ^a	0.971 ± 0.009 ^{cde}	1.012 ± 0.025 ^{bcd}
30	1.086 ± 0.011 ^{ab}	0.985 ± 0.008 ^{bcd}	0.856 ± 0.036 ^{fgh}
45	0.950 ± 0.036 ^{cdef}	0.930 ± 0.022 ^{defg}	0.783 ± 0.014 ^h
60	0.956 ± 0.074 ^{cdef}	0.833 ± 0.014 ^{gh}	0.930 ± 0.069 ^{defg}
90	0.844 ± 0.096 ^{fgh}	1.051 ± 0.011 ^{abc}	0.948 ± 0.014 ^{cdef}
120	0.860 ± 0.010 ^{efgh}	1.040 ± 0.012 ^{abcd}	0.950 ± 0.014 ^{cdef}

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Table A7. TF concentration (mg/g w.m) during pulsed electric fields (4.0 kV/cm, 1000 pulses) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 25 °C extraction temperature.

Pulsed Electric Fields Assisted Extraction (PEFAE)			
Total Flavonoids (mg/g w.m.)			
Ethanol Concentration (%)			
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)
15	0.158 ± 0.025 ^{ijkl}	0.133 ± 0.004 ^{kL}	0.116 ± 0.018 ^L
30	0.188 ± 0.034 ^{ghij}	0.167 ± 0.006 ^{ijkl}	0.137 ± 0.026 ^{jkl}
45	0.230 ± 0.007 ^{efgh}	0.197 ± 0.021 ^{ghi}	0.184 ± 0.002 ^{hijk}
60	0.268 ± 0.006 ^{def}	0.226 ± 0.015 ^{fgh}	0.239 ± 0.021 ^{efg}
90	0.374 ± 0.023 ^b	0.281 ± 0.018 ^{cde}	0.294 ± 0.017 ^{cd}
120	0.429 ± 0.015 ^a	0.332 ± 0.013 ^{bc}	0.349 ± 0.014 ^b

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Table A8. Antioxidant activity (mg/g w.m) during pulsed electric fields (4.0 kV/cm, 1000 pulses) assisted extraction of orange peels waste for 80, 50% EtOH concentration and H₂O for up to 120 min at 25 °C extraction temperature.

Pulsed Electric Fields Assisted Extraction (PEFAE)			
Antioxidant Activity (mg/g w.m.)			
Ethanol Concentration (%)			
Extraction Time (min)	80% EtOH	50% EtOH	0% EtOH (H ₂ O)
15	0.976 ± 0.025 ^h	0.870 ± 0.004 ⁱ	0.806 ± 0.018 ^j
30	1.035 ± 0.017 ^{fg}	0.985 ± 0.016 ^{gh}	0.884 ± 0.016 ⁱ
45	1.090 ± 0.028 ^{de}	1.049 ± 0.018 ^{ef}	0.958 ± 0.012 ^h
60	1.150 ± 0.016 ^{bc}	1.108 ± 0.014 ^{cd}	1.053 ± 0.031 ^{ef}
90	1.122 ± 0.003 ^{cd}	1.146 ± 0.008 ^{bc}	1.081 ± 0.017 ^{def}
120	1.283 ± 0.020 ^a	1.182 ± 0.015 ^b	1.122 ± 0.004 ^{cd}

± represent the standard deviation of six combined measurements (2 biological & 3 analytical replicates). Different small letters indicate significantly different means ($p < 0.05$) between different extraction times and different ethanol concentrations (factorial ANOVA analysis).

Appendix B

This appendix provides the supplementary statistical information used to assess the adequacy of the quadratic response surface models developed for the different extraction methods. The model adequacy criteria, including (R^2), adjusted (R^2), RMSE, model significance, and lack-of-fit tests, are summarized to support the reliability of the fitted models beyond the coefficient of determination alone (Table A9). In addition, diagnostic plots are presented for each model, including normal probability plots of residuals, observed versus predicted values, and residuals versus predicted values (Figure A1). These plots were used to evaluate the normality of residuals, the agreement between experimental and predicted responses, and the absence of systematic trends in the residual distribution. Together, these statistical indicators and diagnostic plots provide a more complete assessment of model performance within the investigated experimental domain.

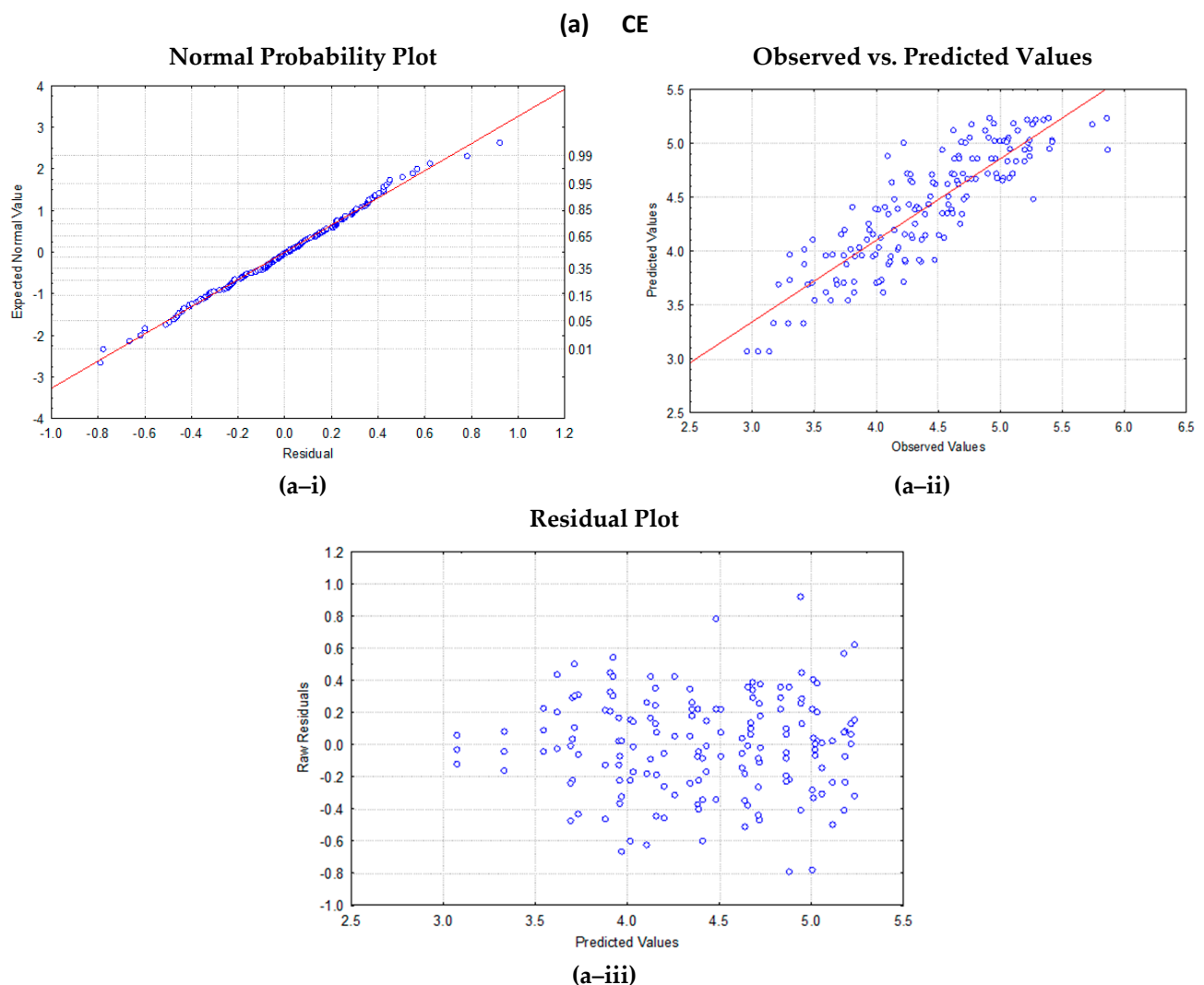
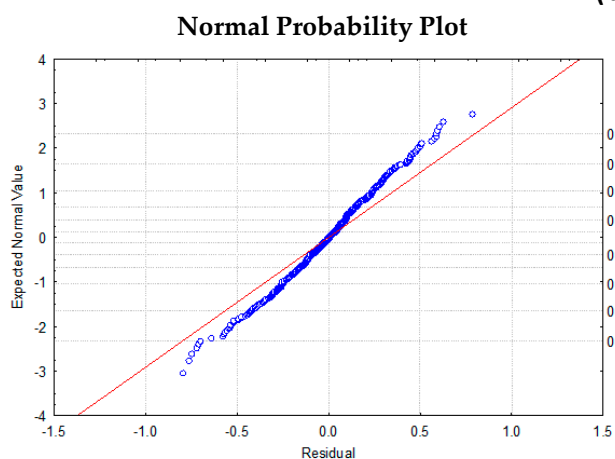
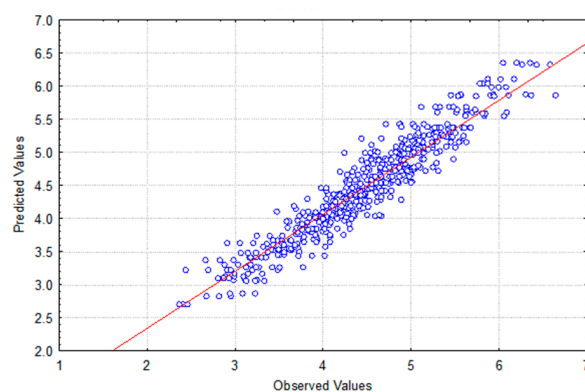
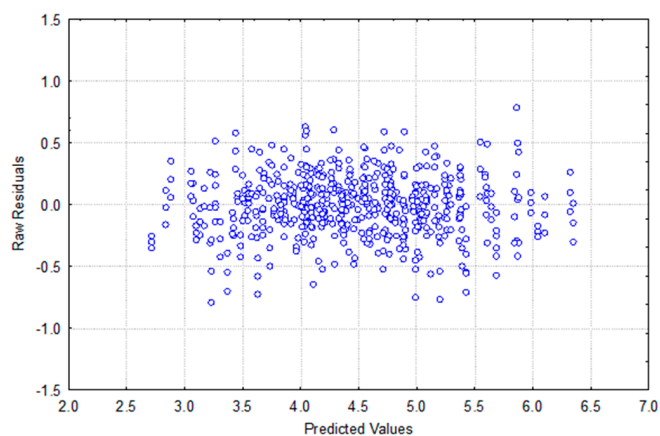
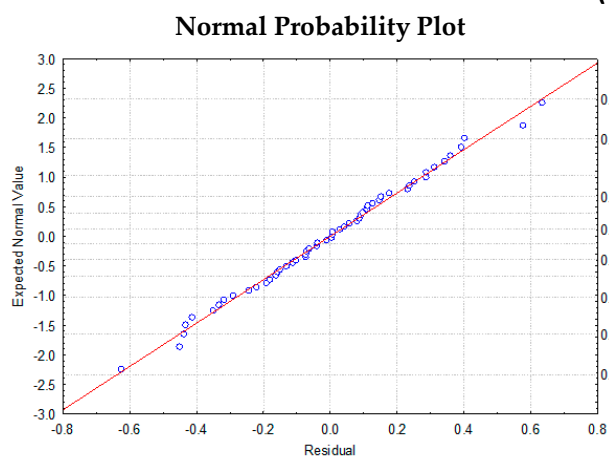
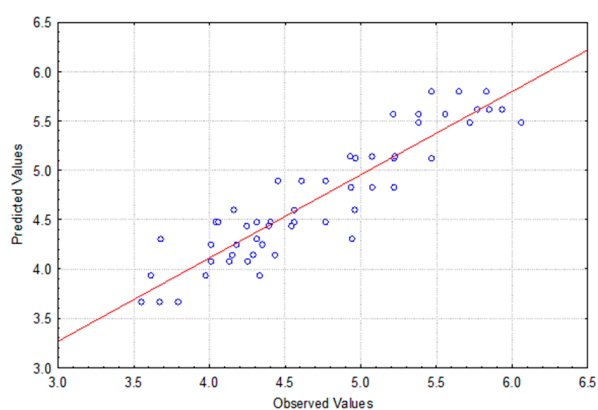


Figure A1. *Cont.*

(b) MAE**(b-i)****Observed vs. Predicted Values****(b-ii)****Residual Plot****(b-iii)****(c) UAE****(c-i)****Observed vs. Predicted Values****(c-ii)****Figure A1. Cont.**

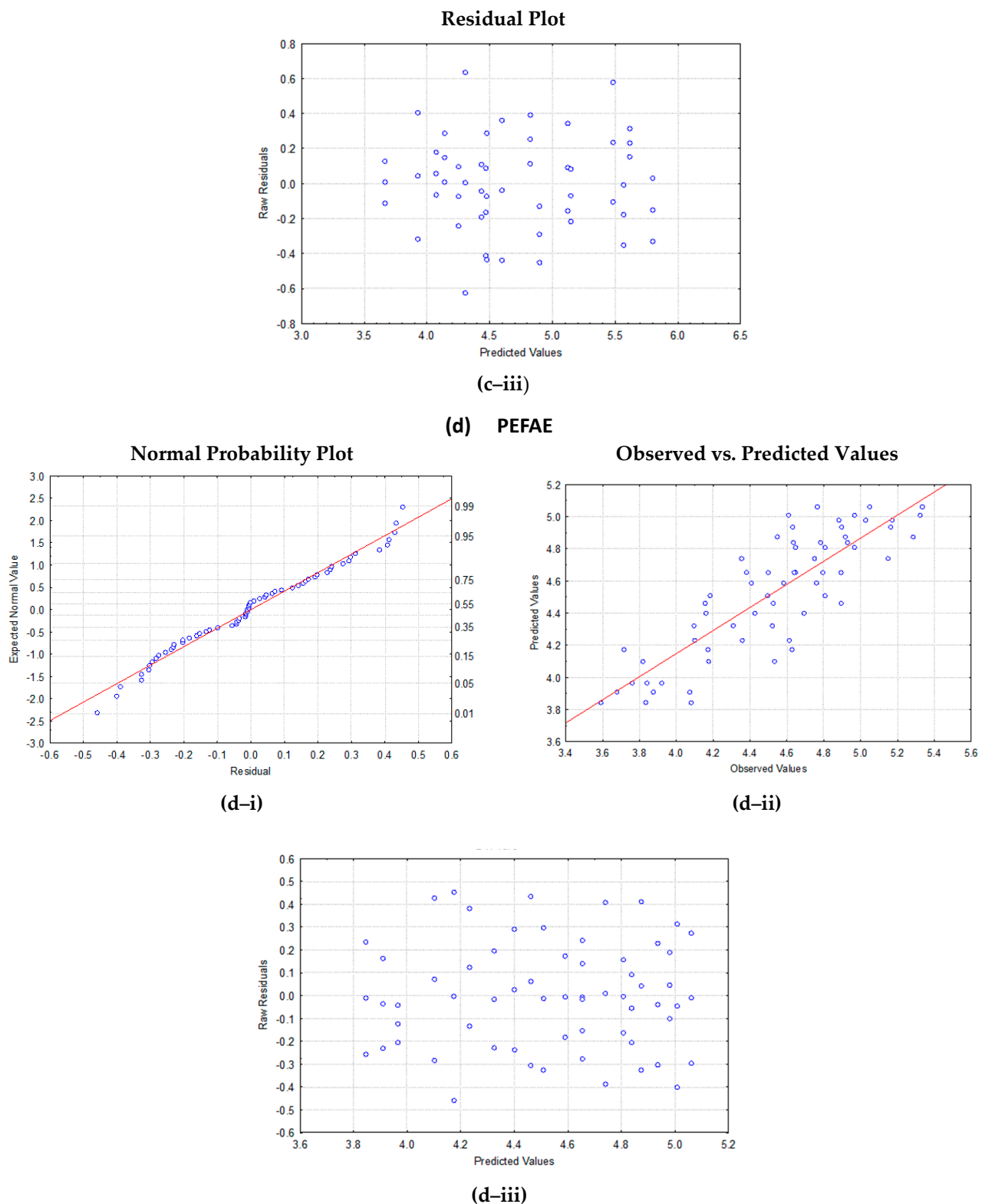


Figure A1. Diagnostic plots used to evaluate the adequacy of the quadratic response surface model fitted for (a) CE, (b) MAE, (c) UAE and (d) PEFAE. (i) Normal probability plot of raw residuals, used to assess the normality assumption; (ii) observed versus predicted values plot, used to evaluate the agreement between experimental and model-predicted responses; and (iii) residuals versus predicted values plot, used to examine the randomness of residual distribution and the absence of systematic trends. The approximately linear distribution of residuals in the normal probability plot, the satisfactory agreement between observed and predicted values, and the random scattering of residuals around zero support the adequacy of the fitted model within the studied experimental domain.

Table A9. Summary of model adequacy criteria for the quadratic response surface models fitted for each extraction method. R^2 represents the coefficient of determination, adjusted R^2 accounts for the number of fitted terms in the model, and RMSE denotes the root mean square error. Model significance was evaluated by ANOVA, while lack-of-fit was assessed by comparing the residual variation with the pure experimental error. A non-significant lack-of-fit test ($p > 0.05$) indicates that the quadratic model adequately describes the experimental data within the studied domain. CE: conventional extraction; UAE: ultrasound-assisted extraction; MAE: microwave-assisted extraction; PEFAE: pulsed electric field-assisted extraction.

Model Adequacy Criterion	CE	UAE	MAE	PEFAE
R^2	0.8556	0.8434	0.8596	0.8179
Adjusted R^2	0.8788	0.8271	0.8573	0.8019
RMSE	0.213	0.280	0.303	0.196
Model significance	$p < 0.0001$	$p < 0.0001$	$p < 0.0001$	$p < 0.0001$
Lack-of-fit test	Not significant	Not significant	Not significant	Not significant

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