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Plasma-Assisted Extraction of Bioactive Compounds from Tomato Peels and Sugar Beet Leaves Monitored by Electron Paramagnetic Resonance Spectroscopy

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Featured Application

High-voltage electrical discharge (HVED) represents a promising non-thermal, environmentally friendly technology with strong potential for future applications in the extraction of valuable bioactive compounds from agricultural by-products. Its ability to enhance mass transfer, reduce solvent use, and operate under mild conditions makes HVED an attractive tool for developing sustainable extraction methods in the food, nutraceutical, and biorefinery sectors. In parallel, electron paramagnetic resonance (EPR) spectroscopy offers considerable potential as an advanced analytical technique for real-time monitoring of radical formation, oxidative processes, and the overall impact of emerging extraction technologies on product quality. The combined use of HVED and EPR provides new insights into the design, optimization, and quality control of next-generation green extraction processes, supporting the transition toward a circular and resource-efficient bioeconomy.

Abstract

Agricultural by-products, such as tomato peels and sugar beet leaves, represent valuable sources of bioactive compounds that can be efficiently recovered using advanced extraction techniques. This study investigated the efficiency of high-voltage electrical discharge (HVED) extraction of bioactive compounds and antioxidant properties from tomato peel (TP) and sugar beet leaves (SBLs). The target compounds were total phenolic content (TPC), lycopene, β-carotene, and chlorophylls. HVED treatments of 1, 3, and 5 min were applied using 30% and 50% methanolic solutions. A 5 min treatment enhanced the extraction of lycopene (2.04 mg/100 mL) and β-carotene (1.14 mg/100 mL) in the 50% methanolic solution, while the shorter 3 min treatments increased TPC (0.117 mg GAE/mL in TP; 0.280 mg GAE/mL in SBLs) and chlorophyll content (25.47 mg/100 mL). For both TP and SBLs, the more concentrated methanolic solvent (50%) was more efficient in extracting bioactive components than the 30% solution. Electron paramagnetic resonance (EPR) spectroscopy confirmed increases in antioxidant activity in all treated samples, with the highest values of 45.27% for TP and 53.16% for SBLs. As a direct and sensitive technique for detecting free-radical scavenging, EPR proved highly suitable for evaluating the impact of HVED treatments. Overall, HVED demonstrated strong potential as a green and effective method for enhancing the recovery of valuable bioactives and antioxidant properties from tomato and sugar beet by-products.

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1. Introduction

The food industry generates a considerable number of by-products after processing plant or animal ingredients. The global Food Waste Index was 1.05 billion tonnes in 2022, as reported in the UNEP Food Waste Index Report 2024 [1]. Food waste includes shells, seeds, whey, bones, skin, and oil cake. Every year, millions of tonnes of tomatoes are processed to produce commercial products such as ketchup, juice, and sauce [2,3]. The main problem with tomato processing is waste. Globally, approximately 60 million tonnes of tomatoes are wasted annually. Recycling this waste reduces economic and energy costs and increases product yield [3,4]. An important source of waste is by-products from the tomato and sugar beet processing industries. These waste materials are valuable resources used for animal feed, fermentation, and the production of biofuels, chemicals, and food ingredients. Major waste products include peels, seeds, beet pulp, molasses, and other liquids, which can be transformed by various methods into new products [5,6].

By-products are usually rich in valuable bioactive compounds that can be valorized for various purposes, increasing sustainability and reducing waste [7]. In the food industry, by-products such as tomato peels and sugar beet leaves are often processed through extraction procedures to isolate compounds like carotenoids, chlorophylls, polyphenols, and vitamins, which can then be used as food supplements. These bioactive compounds exhibit strong antioxidant, anti-cancer, and anti-inflammatory properties [8,9]. Because of their valuable role in the human diet, it is important to preserve these compounds during processing. Tomato by-products, especially seeds and peels, are rich in lycopene. Lycopene is a natural pigment with a characteristic red color, produced by its distinctive chemical structure of 11 conjugated bonds, which allows lycopene to isomerize into 72 possible conformations. In addition to its strong antioxidant properties, lycopene plays an important role in protecting the human body from cancer and cardiovascular diseases [10]. The stability of lycopene depends on temperature, exposure to light, and processing technology. The development of new non-thermal technologies should enable better lycopene extraction with higher yield and efficiency [11,12].

There are numerous methods for processing by-products, most of which are thermal-based. Traditional extraction methods involve the use of large amounts of organic solvents, which are usually toxic, and the extraction process is often time-consuming. The development of novel, non-thermal technologies should reduce extraction time and the use of organic solvents during processing. Recently, pretreatments and treatments such as pulsed electric fields (PEF), high-voltage electrical discharge (HVED), as well as microwave-assisted extraction (MWE), ultrasound-assisted extraction (UAE), and supercritical fluid extraction (SFE) have been increasingly used for the extraction of bioactive components [13–15]. Non-thermal techniques can increase extraction without compromising nutrient quality [16]. Green technologies such as MWE, HVED, PEF, and UAE are the preferred methods for this process [17,18]. Sustainability will be at the forefront of research activities to achieve the 2030 Sustainable Development Goals (SDGs).

The aim of this work was to investigate the properties of cold-plasma technology as a non-thermal technology in the extraction of chlorophylls, carotenoids, and polyphenols from tomato peels and sugar beet leaves. The use of cold-plasma technology on sugar beet leaf by-products has largely been unexplored. On the other hand, to optimize the cold-plasma procedures as much as possible, monitoring the quality must be adjusted

and applied to this specific technology. Therefore, in our work, Electron Paramagnetic Resonance (EPR) spectroscopy, an advanced analytical technique that provides innovative solutions, was used for quality control. As one of the most informative and selective methods for food analysis, EPR offers a highly sensitive approach for detecting and characterizing free radicals and paramagnetic species in various food systems [19,20]. The EPR technique is based on the interaction of unpaired electrons with an external magnetic field, enabling the identification of short-lived species that are often undetectable by conventional analytical methods. EPR spectroscopy can be applied to monitor and quantify changes in foods processed by innovative technologies such as ionizing radiation, high pressure, pulsed electric fields, ultrasound, and microwaves. It also enables the evaluation of oxidation processes and the assessment of antioxidant activity in complex food systems [21,22]. Furthermore, EPR spectroscopy has emerging potential for tracing the origin and transformation of food waste, thereby supporting sustainable approaches in the food industry.

The aim of this study was to optimize the cold-plasma extraction process regarding solvent ratio, treatment time, and energy input for two types of by-products. In this study, tomato peel and sugar beet leaves were selected as raw materials for the isolation of bioactive compounds because they are readily available and often underutilized by-products of the food industry [8,23]. The tomato and sugar beet industries generate substantial amounts of by-products and waste due to seasonal overproduction and strict market standards, contributing to economic losses and environmental impact. However, these by-products have significant potential for valorization [24]. Although they have high antioxidant potential, these plant residues are rarely utilized in practice and therefore represent a valuable resource for obtaining bioactive substances [25,26]. Their use in this research supports sustainability efforts, reduces organic waste, and promotes more efficient biomass valorization. The determination of bioactive components and EPR analyses of inherently present radicals and antioxidant activity were conducted. Paramagnetic ions, such as Fe^{2} and Mn^{2+}, were monitored by EPR spectroscopy in the samples before plasma treatment. Furthermore, antioxidant activity was determined by EPR after treatment. The results show that the antioxidant activity depends on the type of treatment.

Currently, many benchtop EPR devices are commercially available, enabling easy monitoring and analysis at both laboratory and industrial scales. Here we present the potential use of a benchtop EPR device in innovative quality control concepts throughout the entire process “from farm to fork”, i.e., during processing.

2. Materials and Methods

2.1. Chemicals

All chemicals used in the analyses were HPLC grade. Methanol and n-hexane were purchased from VWR (Vienna, Austria). Folin–Ciocalteu reagent was obtained from Merck (Kenilworth, NJ, USA), while gallic acid was obtained from Thermo Scientific™ Chemicals (Waltham, MA, USA). Acetone and sodium carbonate were purchased from Kemika (Zagreb, Croatia), and 96% ethanol, 30% hydrogen peroxide, and L (+)-ascorbic acid were obtained from Gram-mol (Zagreb, Croatia). Iron (II) chloride tetrahydrate and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were purchased from Sigma (St. Louis, MO, USA).

2.2. Sample Origin and Preparation

2.2.1. Origin and Preparation of the Tomato Peels

A plum-type tomato (*Solanum lycopersicum* L., cultivar “Roma”) was obtained from the Croatian Eco Farm “Vrtni centar Baković”, Sveti Filip i Jakov (43.9818° N, 15.4269° E), Croatia. The tomato peels were separated with a sharp knife after blanching in hot water

for 10 s and immersing in cold water for approximately 30 s. The peels were then dried in a dryer (Elektrokovina, Zagreb, Croatia) at 70 °C until a constant mass was reached. The peels were ground in a mill to a powder consistency and stored in plastic cups at room temperature in a desiccator until analysis.

2.2.2. Origin and Preparation of the Sugar Beet Leaves

The sugar beet leaves (SBLs) (*Beta vulgaris* var. *saccharifera* L.), a herbaceous species of the Chenopodiaceae family, were received from the Croatian family business "Hrgović" in Kapinci (45.8126° N, 17.7008° E), Croatia. The SBLs were dried in a dryer at 50 °C to a constant mass, ground in a mill to a powder consistency, and stored in plastic cups at room temperature in a desiccator until analysis.

2.3. Transitional Metal Ions Detection by EPR Spectroscopy in Dried Raw Materials

EPR spectra of dried tomato peel and dried sugar beet leaves were recorded using a benchtop Bruker Magnettech ESR5000 spectrometer (Bruker BioSpin, Rheinstetten, Germany) operating at X-band frequency and equipped with N_2 temperature control. Technical specifications are provided in Table 1.

Table 1. Bruker Magnettech ESR5000 technical specifications.

Operating Frequency	X-Band
Microwave power	1 μW–100 mW
Sensitivity	5×10^{10} spins/mT (5×10^{9} spins/G)
Concentration sensitivity	20 pM
Field homogeneity	−/+5 μT (50 mG) within sample region
Field stability	1.0 μT/h (10 mG/h)
Sweep resolution (field and time)	≥250,000 points
Magnetic field range	10–650 mT (100–6500 G)
Modulation frequency	10 kHz and 100 kHz
Compact size	397 × 262 × 192 mm (45 kg)

EPR spectroscopy was used to detect the presence of transition metals in the dried tomato peel and SBLs before cold-plasma processing. In EPR tubes, 53.76 mg of SBLs and 18.20 mg of tomato peel powder were weighed. EPR spectra were recorded using the following parameters: the magnetic field was swept from 100 to 450 mT, the modulation amplitude was 0.5 mT, the microwave power was 5 mW, and the microwave frequency was 100 kHz. Measurements were performed at 293.15 K and 153.15 K.

2.4. Plasma Extraction Treatment of Bioactive Compounds from Tomato Peels and Sugar Beet Leaves

The plasma extraction treatment of bioactive compounds from the prepared samples (tomato peels and sugar beet leaves) was performed using High-Voltage Electrical Discharge (HVED). The experimental setup for HVED (Figure 1) consisted of a closed Plexiglas box containing a glass reactor (up to 1000 mL, depending on the sample amount) with a point-to-point electrode configuration. A high-voltage electrode (a stainless steel medical needle) was placed in the liquid phase at the bottom of the reactor. A stainless steel electrode in the gas phase above the liquid surface served as the ground. The reactor was equipped with a rubber plug and adapted openings for electrodes and gas injection. The high-voltage pulse generator IMPEL HVG60/1 (IMPEL GROUP Ltd., Zagreb, Croatia) transmitted rectangular pulses through the HV electrode, with parameters set by the user

(treatment time, pulse frequency, high voltage). The technical specifications of the generator are provided in Table 2. Argon, used as the operating gas, was continuously injected through the HV electrode at the bottom of the reactor. Complete or partial ionization occurs when a high-voltage electrical current passes through a neutral fluid (in liquid and gas phases), resulting in electrical discharges, or plasma with increased conductivity.

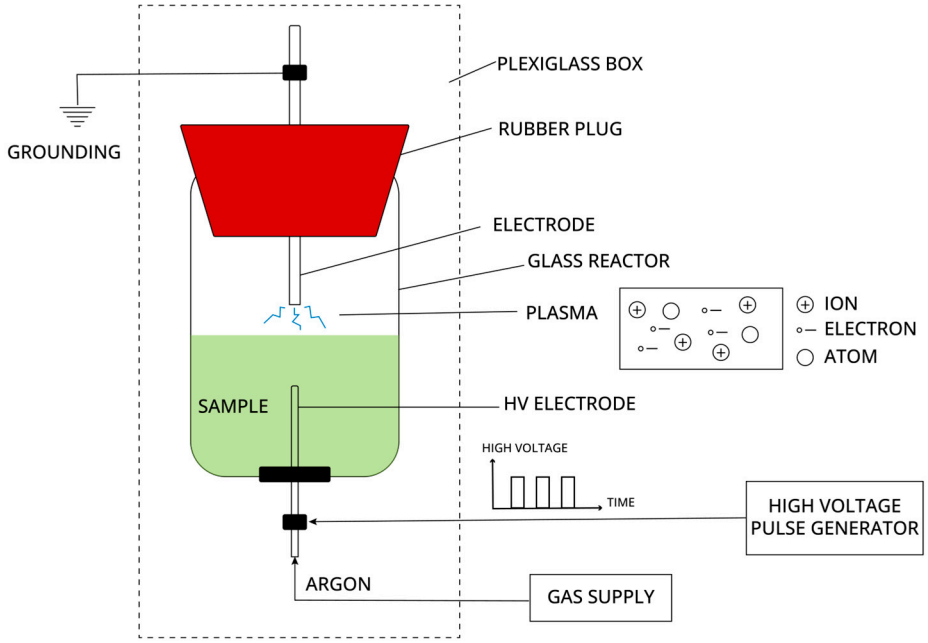

Figure 1. Experimental setup for High-Voltage Electrical Discharge (HVED).

Table 2. Technical specifications of high-voltage pulse generator IMPEL HVG60/1 (IMPEL GROUP Ltd., Zagreb, Croatia).

Voltage	Up to 60 kV
Pulse frequency	Up to 300 Hz
Pulse width	Up to 2 μs
HV current	Up to 16 mA
Time of operation	Up to 30 min

Two grams of tomato peel powder were suspended in 100 mL of 30% methanol solution and treated under the following parameters: treatment time of 0, 1, 3, and 5 min, pulse frequency 120 Hz, and voltage 30 kV. Everything was repeated for tomato peel powder with the 50% methanol solution. Bioactive compounds from sugar beet leaves were extracted using the same parameters in 100 mL of 30% and in 100 mL of 50% methanol solution.

2.5. Isolation of Bioactive Components

2.5.1. Total Polyphenolic Content in Tomato Peel Extracts and SBL Extracts

Polyphenolic compounds were extracted using 30% and 50% methanol solutions in an HVED plasma reactor. To determine the total polyphenolic content (TPC) in tomato peel and SBL samples, the Folin–Ciocalteu (FC) assay was used. A volume of 2 mL of Milli-Q water was mixed with 100 μL of the sample and 200 μL of FC reagent. The mixture was incubated for 3 min, then 1 mL of 20% sodium carbonate solution was added and mixed. The samples were incubated at room temperature in the dark for 2 h, after which absorbance at 765 nm was measured using a Secomam UviLine 9400 spectrophotometer (Secomam Groupe Aqualabo, Alès, France). A gallic acid calibration curve (0.06–0.3 mg/mL) was

used to determine the total polyphenolic concentration in tomato peel and SBL samples, and results are expressed as mg/mL of gallic acid equivalents.

2.5.2. Total Carotenoid Content in Tomato Peel Extracts

After the treatment described in previous chapters, the methanolic layer was decanted from the samples, and the precipitate was suspended in 25 mL of a 4:6 (*v*/*v*) acetone:hexane solution to isolate carotenoid compounds. The prepared suspension was vortexed for 15 min at 3000 rpm using a Vortex V-1 Plus (Biosan, Riga, Latvia) and centrifuged with a Rotina 380 R centrifuge (Hettich, GmbH & Co. KG, Tuttlingen, Germany) at 2500 rpm for 10 min. Lycopene and beta-carotene concentrations were measured using a Secomam UviLine 9400 spectrophotometer (Secomam Groupe Aqualabo, Alès, France) at wavelengths of 663, 645, 505, and 453 nm, and concentrations were calculated using the following equations [27]:

$$c_{lyc} = -0.0458\ A_{663} + 0.204\ A_{645} + 0.372\ A_{505} - 0.0806\ A_{453} \quad (1)$$

$$c_{betac} = 0.216\ A_{663} - 1.22\ A_{645} - 0.304\ A_{505} + 0.452\ A_{453} \quad (2)$$

where A_{663}, A_{645}, A_{505}, and A_{453} correspond to absorbance at 663, 645, 505, and 453 nm, respectively. The results are expressed as mg/100 mL.

2.5.3. Total Chlorophyll Content in SBLs

After the HVED plasma treatment, the methanolic layer was separated, and the precipitate was suspended in 25 mL of 80% acetone solution. The prepared suspension was vortexed for 15 min at 3000 rpm and centrifuged at 2500 rpm for 10 min. The total chlorophyll content was then measured using a Secomam UviLine 9400 spectrophotometer (Secomam Groupe Aqualabo, Alès, France) at 663 and 645 nm, and the concentration was calculated using the following equation [28]:

$$\text{total chlorophyll content} = 7.15A_{665} + 18.71A_{645} \quad (3)$$

where A_{665} and A_{645} correspond to absorbances at 665 and 645 nm, respectively. The results are expressed as mg/100 mL.

2.6. EPR Detection of Antioxidant Activity in Tomato Peel and Sugar Beet Leaf Powder Extracts

Antioxidant activity was determined using the EPR-DPPH assay. Antioxidants from tomato peels and SBLs react with the DPPH free radical, resulting in a decrease in DPPH concentration and EPR-DPPH signal intensity, which corresponds to the antioxidant activity of the investigated samples. The results are expressed as the percentage (%) reduction in EPR-DPPH signal intensity. A volume of 970 µL of 15 mM DPPH solution (in 96% ethanol) was mixed with 30 µL of the sample (tomato peel or SBL). Measurements were performed over 30 min with the following parameters: the magnetic field was swept from 331 to 343 mT, the modulation amplitude was 0.2 mT, the microwave power was 10 mW, and the microwave frequency was 100 kHz.

2.7. Statistical Analysis

Statistical analysis was performed using Systat v.13.2.01 (Grafiti LLC, Palo Alto, CA, USA). Measurements were performed in triplicate, and results are expressed as the mean of three consecutive measurements ± standard deviation (SD). Differences between results were determined using a *t*-test for the effect of two solvents, and a one-way analysis of variance (ANOVA) with Tukey's honestly significant difference (HSD) test was used for other analyses. A *p*-value of $p \leq 0.05$ was considered statistically significant.

3. Results and Discussion

3.1. Transition Metals in Sugar Beet Leaf and Tomato Peel Powders

3.1.1. Sugar Beet Leaf Powder

Paramagnetic transition metal ions play a crucial role in agricultural and environmental systems. They are involved in key processes such as adsorption–desorption, transport, bioavailability, and potential phytotoxicity within the soil–water–plant system. The metals most commonly detected in soils and plants using EPR spectroscopy include iron, copper, manganese, vanadium, and molybdenum [29]. EPR analysis provides valuable insights into the oxidation states of these metal ions, as well as their complexation behavior, symmetry, and coordination environment. In this study, the EPR detection of transition metals in SBL powder revealed the presence of iron and manganese ions, with signals attributed to Mn^{2+} ranging from 200 mT to 400 mT, Fe^{3+} from 100 mT to 220 mT, and a free radical signal around 340 mT (Figure 2), which is consistent with spectra obtained in the research of Costa et al. [29] and Segnini et al. [30]. The measurements performed at 293.15 K (black line) exhibited a low EPR signal intensity. In order to enhance the EPR signal intensity, the measurement temperature was decreased by 293.15 K to 153.15 K (red line).

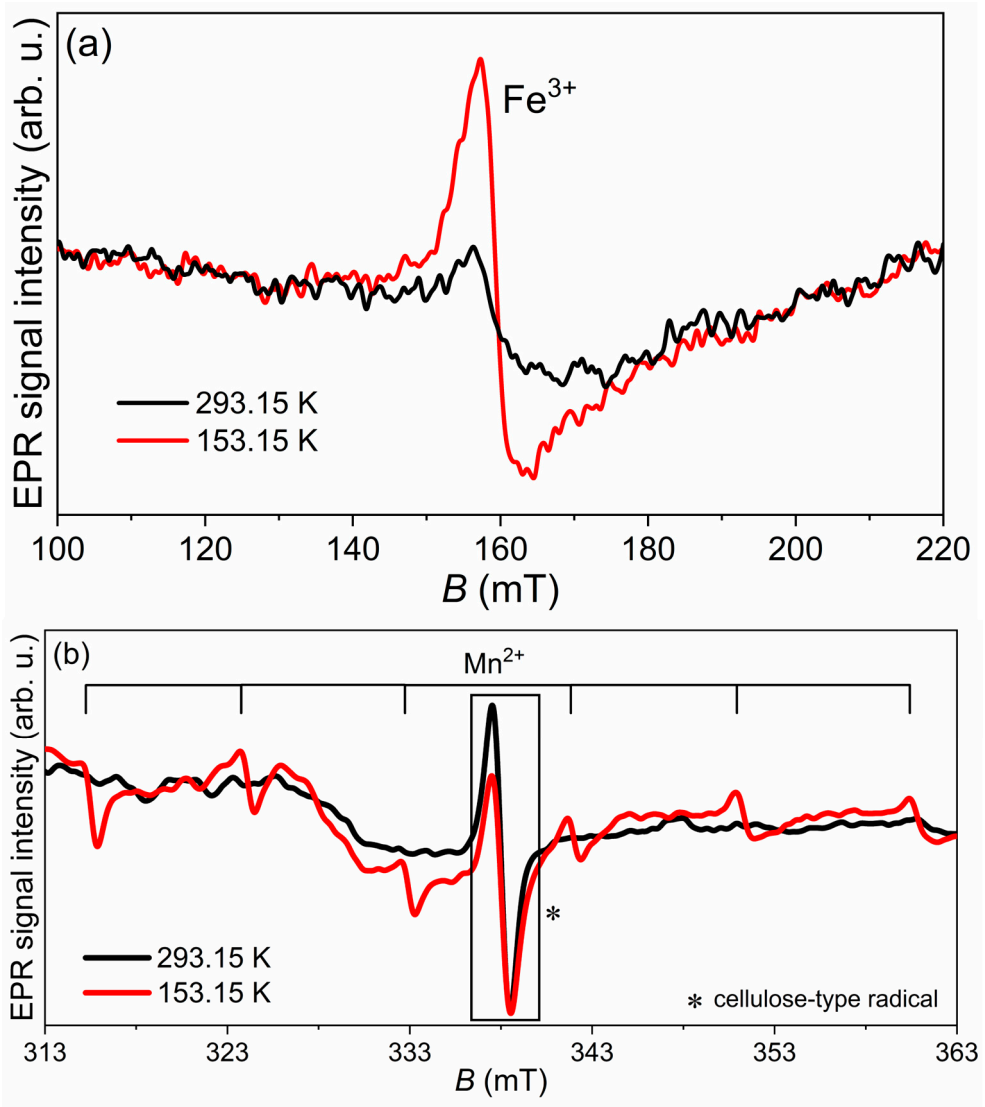

Figure 2. EPR spectra of SBL powder samples monitored at 293.15 K (black line) and 153.15 K (red line): (**a**) EPR spectra of Fe^{3+} ion, (**b**) EPR spectra of Mn^{2+} ion and cellulose radical (shown in insert).

Figure 2a shows the EPR signal of the Fe^{3+} ion in the SBLs sample (g = 4.25), while Figure 2b shows lines, which, according to Costa et al. [29] and Takeshitaet et al. [31], based on their coupling constant and g value, correspond to the manganese ions (S = 5/2) and the cellulose radicals that are often present in vegetable products. The presence of the cellulose-type radical (g = 2.005) indicates changes or damage to the proteins, lipids, and polysaccharide system, as cellulose occurs in two areas of the plant in addition to proteins, lipids, and polysaccharides [32]. The decrease in the EPR line of the cellulose radical type with decreasing temperature indicates that this radical is free at 293.15 K and can move freely in the lattice, whereas at 153.15 K the radical is frozen and its rotational movement is slower, resulting in lower EPR intensity [33].

The six spectral lines associated with Mn^{2+} ions can appear distorted [34]. This effect may result from the relatively low concentration of manganese (II) ions in the analyzed sample, leading to weak signal intensity and increased noise in the spectrum. Another possible explanation involves redox processes within the sample that alter the oxidation state of manganese. During redox reactions, Mn^{2+} ions may undergo oxidation or reduction, forming diamagnetic species that remain undetectable by EPR spectroscopy. Consequently, both the low concentration and the redox dynamics in the sample could contribute to the observed irregularities in the Mn^{2+} spectral lines.

The concentration and speciation of transition metal ions detected in the SBL powder are strongly influenced by environmental factors, including soil composition, metal availability, plant characteristics, and geographic origin [35]. Variations in these factors affect not only the abundance of metals such as iron and manganese, but also their redox behavior and interactions with organic radicals, as evidenced by the distorted Mn^{2+} lines and the presence of cellulose-type radicals observed in the EPR spectra. These results highlight EPR spectroscopy as a highly effective and sensitive method for analyzing both paramagnetic metal ions and associated organic radicals in plant-derived powders, providing insights into how soil characteristics, environmental conditions, and geographic origin influence metal bioavailability and distribution.

3.1.2. Tomato Peel Powder

The EPR spectrum of the tomato peel powder did not show any observable EPR signal corresponding to Fe^{2+} or Mn^{2+} ions; it only showed a typical cellulose-type signal (g = 2.005), as shown in Figure 3. This signal is expected, given the soil and type of plant growth, as well as whether the environment is clean or polluted, as discussed in the previous section.

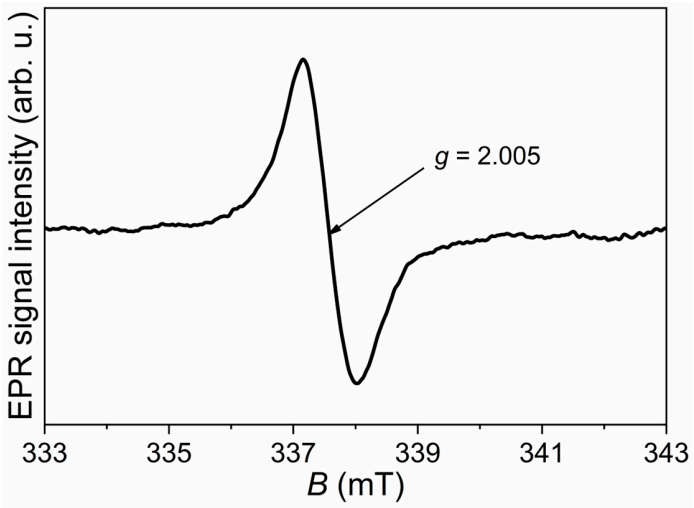

Figure 3. EPR signal of cellulose-type radical (g = 2.005) in dried tomato peel at 153.15 K.

3.2. Plasma Technology Extractions of Bioactive Component from Tomato Peel and SBL Powders

3.2.1. Total Polyphenolic Content

The total polyphenolic content in tomato peel and SBL samples was determined using the FC assay. Statistical analysis shows a significant impact of HVED treatment ($p = 0.000$) and solvent concentration ($p = 0.000$), as well as the combination of treatment time and solvent concentration ($p = 0.001$), on the extraction of polyphenolic compounds from tomato peel samples (Table 3).

Table 3. Multifactorial ANOVA testing effects of time, solvent, and combination of time and solvent impact on bioactive composition and antioxidant activity of tomato peel extract. * indicates $p < 0.05$.

	Time			Solvent			Time × Solvent		
	df	**F**	***p***	**Df**	**F**	***p***	**df**	**F**	***p***
TPC	3	99.57	0.000 *	1	72.86	0.000 *	3	15.81	0.001 *
Lycopene	3	41.57	0.000 *	1	36.57	0.000 *	3	1.81	0.223
Beta-carotene	3	11.67	0.003 *	1	2.88	0.128	3	2.10	0.179
Antioxidant activity	3	1.24	0.356	1	0.01	0.940	3	0.22	0.881
AAE	3	1.24	0.358	1	0.01	0.940	3	0.22	0.881

The lowest concentration (0.092 mg/mL; 0.052 mg/mL) in both extracts (30% and 50% MeOH) was found in the control (HVED untreated) sample. The highest concentration in both 30% and 50% MeOH extracts was observed after 3 min of treatment (Table 4). A 5 min treatment caused a slight decrease in the concentration of polyphenols in tomato peel samples compared to other HVED treatments. High electrical voltage combined with a longer treatment duration causes the degradation of the chemical structure of these valuable compounds. In contrast, a 1 min treatment had the opposite effect. Among all samples, a statistically significant impact of solvent concentration on the isolation of polyphenolic content was observed in the 5 min treatments, where a decrease in TPC was noted. However, it is important to note that higher concentrations of solvent increase the extraction of polyphenols, and all HVED treatments significantly increase TPC in tomato peel samples compared to the control (HVED untreated) sample.

Table 4. Total polyphenolic content in tomato peel samples. The results are expressed as mean value ± SD. Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters indicate significant differences within rows, and lowercase letters within columns.

HVED Duration (min)	GA Equivalents (mg/mL) 30% Methanolic Extracts	GA Equivalents (mg/mL) 50% Methanolic Extracts
0	0.092 ± 0.004^{aB}	0.052 ± 0.003^{aA}
1	0.106 ± 0.002^{abA}	0.114 ± 0.004^{bA}
3	0.110 ± 0.006^{bA}	0.117 ± 0.005^{bA}
5	0.099 ± 0.001^{abA}	0.111 ± 0.000^{bB}

Statistical analysis indicated that neither HVED treatment ($p = 0.310$), solvent concentration ($p = 0.059$), nor their interaction ($p = 0.166$) had a statistically significant effect on the extraction of polyphenols from SBL samples (Table 5). However, the highest total polyphenolic content (TPC) in the SBL samples was observed after a 3 min treatment when 30% methanol was used as the solvent (Table 6). All treated samples with 30% methanol contained higher concentrations of polyphenols compared to the control sample. In contrast, when 50% methanol was used, a negative effect of the HVED treatment and solvent concentration was observed, with all treated samples containing lower concentrations of

polyphenols than the control. Although statistical analysis shows no significant impact of the HVED treatment and solvent concentration, it can be observed that a higher solvent concentration and a longer treatment duration caused a decrease in TPC. Compared to 1 min and 5 min treatments, the 3 min treatment resulted in a greater increase in TPC with 30% methanol and a smaller decrease with 50% methanol. These results indicate the need to optimize HVED parameters when solvent concentration is increased. Among the treated samples, the greatest decrease in TPC was recorded after 1 min of treatment, while the highest increase was observed after 3 min.

Table 5. Multifactorial ANOVA testing effect of time, solvent, and time × solvent impact on bioactive composition and antioxidant activity of SBLs extracts. * indicates $p < 0.05$.

	Time			Solvent			Time × Solvent		
	df	**F**	***p***	**Df**	**F**	***p***	**df**	**F**	***p***
TPC	3	1.41	0.310	1	0.003	0.059	3	0.001	0.166
Chlorophylls	3	20.90	0.000 *	1	1603.92	0.000 *	3	13.14	0.000 *
Antioxidant activity	3	0.493	0.697	1	0.108	0.751	3	0.887	0.488
AAE	3	0.493	0.697	1	0.108	0.751	3	0.887	0.488

Table 6. Total polyphenolic content in SBL samples. The results are expressed as mean value ± SD. Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters indicate significant differences within rows, and lowercase letters within columns.

HVED Duration (min)	GA Equivalents (mg/mL) 30% Methanolic Extracts	GA Equivalents (mg/mL) 50% Methanolic Extracts
0	0.256 ± 0.024^{aA}	0.270 ± 0.007^{aA}
1	0.274 ± 0.002^{aA}	0.201 ± 0.042^{aA}
3	0.280 ± 0.004^{aA}	0.260 ± 0.022^{aA}
5	0.277 ± 0.017^{aA}	0.249 ± 0.038^{aA}

The results are consistent with the findings of Abbaspour, Mogaddam, and Ghasempour [36], who reported that extraction efficiency strongly depends on the applied energy input and treatment duration. Moderate HVED exposure enhances cell wall disruption and facilitates polyphenol release, whereas excessive energy or prolonged treatment can lead to degradation of these compounds due to the formation of reactive oxygen and nitrogen species (ROS and RNS). This trend confirms that optimal HVED parameters are essential for maximizing polyphenol yield in both samples (tomato peel and sugar beet leaves) while maintaining their structural stability. Similarly, Li et al. [37] highlighted in their review that careful optimization of HVED conditions is crucial to achieve high extraction efficiency without compromising compound stability, further supporting the trends observed in this study. The impact of solvent type (water, methanol, and ethanol) and concentration (70% and 100%) on the extraction of TPC from sugar beet was investigated in the research of Mohammed et al. [38]. The highest concentration of polyphenols was achieved when 100% methanol was used as the solvent. Similar results were obtained in the research of Mostapha, Hayette, and Zina [39], in which concentrated methanol extracted a higher concentration of polyphenols in tomato peel extracts compared to a 50% methanol solution. The effect of solvent polarity was investigated in the research of Babbar et al. [40], where the highest polyphenolic concentration was obtained when methanol was used rather than other non-polar solvents such as n-hexane, chloroform, and ethyl-acetate. Compared to the literature, these findings confirm that the correct solvent was used for the extraction of polyphenolic compounds. However, these findings suggest that further research is required

to identify the optimal methanol concentration for maximum extraction of polyphenolic compounds and to optimize HVED parameters (voltage, time, and pulse duration).

3.2.2. Total Carotenoid Content in Tomato Peel Extracts

It is well known that lycopene and beta-carotene are non-polar compounds; therefore, their extraction should be carried out using low-polarity solvents, such as n-hexane and chloroform, or mixtures of polar and non-polar solvents, including acetone–n-hexane, methanol–chloroform, n-hexane–ethanol–acetone, and ethyl lactate–ethanol–acetone [41]. Consequently, methanol alone is not suitable for lycopene extraction. For this reason, after the HVED treatment, the methanolic layer was decanted, and the extraction of lycopene and beta-carotene was subsequently performed using an acetone:n-hexane (4:6) mixture. From the results presented in Figure 4, it can be observed that a higher concentration of lycopene was extracted from the precipitate obtained after the HVED treatments with 50% methanol. Considering that lycopene is a non-polar compound, while methanol is a polar solvent, a lower amount of lycopene was extracted during the HVED treatment in the methanol layer. Consequently, a higher concentration of lycopene remained in the residue, from which it was subsequently released by adding an acetone:n-hexane solvent mixture. The opposite effect was noticed when a 30% methanol solution was used in HVED treatments. The same observation is noticed for the extraction of beta-carotene.

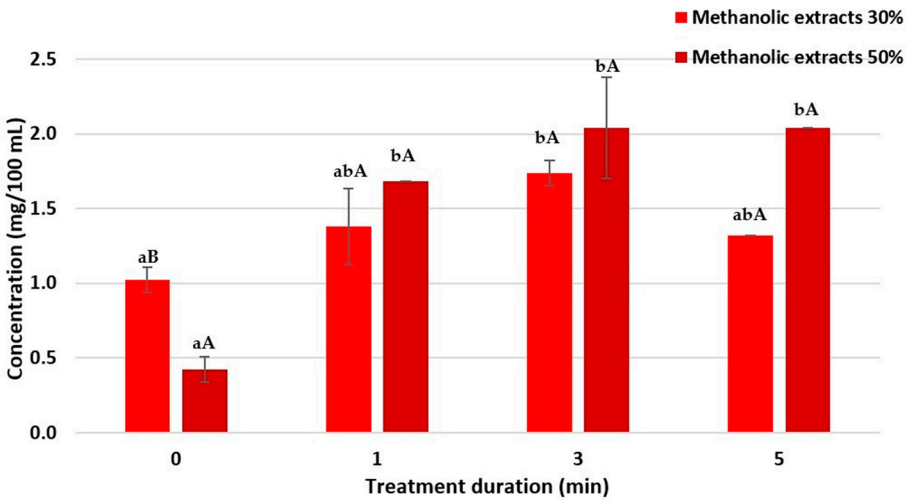

Figure 4. Lycopene content (mg/100 mL) in tomato peel extracts after HVED treatments (0, 1, 3, and 5 min). Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters denote significant differences for solvents, whereas lowercase letters denote significant differences for treatments.

Statistical analysis showed that both HVED treatment time ($p = 0.000$) and methanol concentration ($p = 0.00$) significantly affected lycopene yield, while their interaction was not significant ($p = 0.223$), as can be seen in Table 3. This indicates that each factor independently influenced lycopene extraction efficiency. Among all treated samples, the 3 min treatment resulted in the highest lycopene content, reaching 1.74 mg/100 mL for the treatments with 30% methanol and 2.04 mg/100 mL for the 50% methanol. The lowest concentrations were obtained in the control (HVED untreated) samples, measuring 1.02 mg/100 mL for 30% methanol and 0.42 mg/100 mL for 50% methanol.

Similar results were obtained for beta-carotene extraction. Statistical analysis revealed a significant impact ($p = 0.003$) of HVED treatments on beta-carotene extraction, while solvent concentration ($p = 0.128$) and the interaction between solvent and treatment time ($p = 0.179$) did not have a significant effect (Table 3). With 30% methanol, the highest beta-carotene concentration (1.02 mg/100 mL) was obtained after 3 min of treatment,

while with 50% methanol, the highest concentration (1.14 mg/100 mL) was achieved after 5 min. Longer treatment durations combined with higher solvent concentrations enhanced beta-carotene extraction.

The results of this study demonstrate that HVED treatment significantly enhanced the extraction of lycopene and beta-carotene from tomato peels (Figures 4 and 5), with the highest concentrations obtained using 50% methanol as the solvent. Specifically, 3 min HVED treatments yielded the greatest recovery of lycopene, while beta-carotene extraction was further improved with longer exposure (up to 5 min). These findings are consistent with previous studies on the application of HVED plasma for carotenoid extraction. For example, Buniowska et al. (2015) [42] reported that HVED applied to orange peel enhanced the release of carotenoids and other pigments, although excessive energy could lead to their degradation. Similarly, studies on carrot juice treated with high-voltage cold plasma showed increased levels of beta-carotene and other carotenoids, indicating that controlled electrical discharge can improve pigment extraction [43].

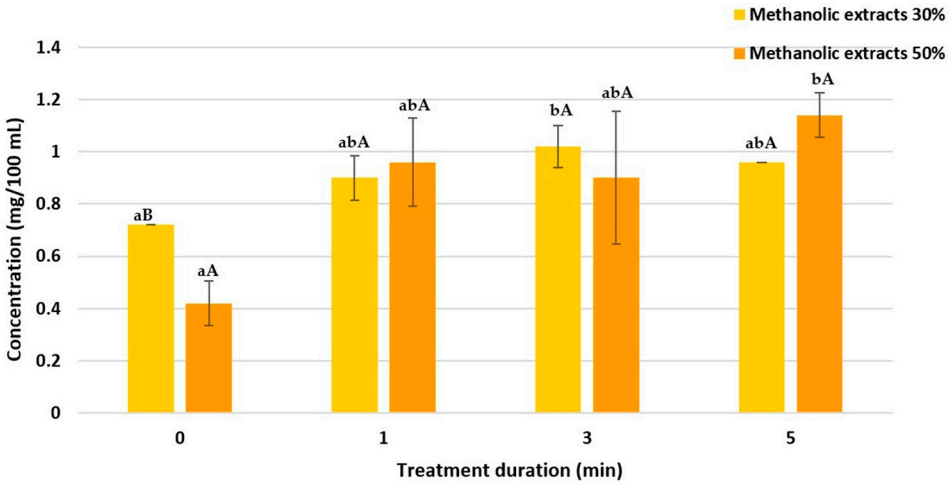

Figure 5. Total beta-carotene (mg/100 mL) in tomato peel extracts after HVED treatments (0, 1, 3, and 5 min). Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters denote significant differences for solvents, whereas lowercase letters denote significant differences for treatments.

The improved extraction efficiency observed after HVED treatment can be attributed to the disruption of tomato peel cell structures, leading to increased permeability and solvent accessibility to lycopene and beta-carotene from chromoplasts [44]. However, it should be noted that carotenoids are sensitive to oxidation and isomerization under plasma conditions. Therefore, prolonged HVED exposure or excessive energy input could lead to degradation and decreased pigment stability. Compared with conventional solvent extraction or ultrasound-assisted extraction, HVED offers the advantage of shorter extraction time and lower solvent consumption, while achieving comparable or even higher carotenoid yields [37].

Overall, previous studies, together with the results of the present experiments, suggest that HVED has the potential to be an effective method for extracting carotenoids from plant matrices. However, its efficiency depends on treatment parameters, such as treatment duration, energy input, and solvent concentration, which should be carefully optimized to prevent degradation of sensitive compounds.

3.2.3. Total Chlorophyll Content in SBL Extracts

Depending on treatment duration and solvent concentration, HVED treatments had varying impacts on the extraction of chlorophylls from SBLs, as shown in Figure 6. When a

30% methanol solution was used, HVED treatments had a statistically significant (p = 0.000) decreasing effect on chlorophyll isolation. The highest chlorophyll concentration was obtained in the control sample, while the lowest was found in the 3 min treated sample. It can also be observed that longer treatment durations further decreased chlorophyll concentrations. An opposite effect was observed with HVED treatments when a 50% methanol solution was used as the solvent. The highest concentration was found in the 3 min treated sample, and the lowest in the 5 min treated sample. It can be concluded that prolonged treatments caused chlorophyll degradation, while shorter durations resulted in better extraction of these compounds. In this case, solvent concentration played a crucial role, as confirmed by statistical analysis (p = 0.000). As shown, a higher concentration (50%) of methanol increased the extraction of total chlorophyll content compared to 30% methanol extracts.

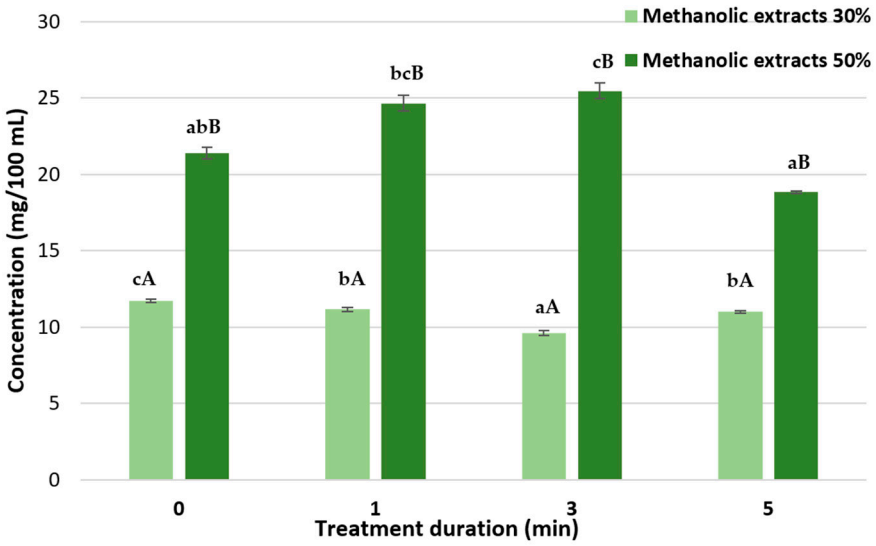

Figure 6. Total chlorophyll content (mg/100 mL) in SBL extracts after HVED treatments (0, 1, 3, and 5 min). Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters denote significant differences for solvents, whereas lowercase letters denote significant differences for treatments.

Unlike carotenoids, chlorophylls are amphiphilic compounds, and their extraction is typically performed using an acetone solution. As with carotenoid extraction, after HVED treatment, the methanol layer was decanted, and the precipitate was filled with an 80% acetone solution to extract total chlorophylls. It was observed that when a 50% methanol solution was used, a higher concentration of chlorophylls remained in the precipitate. This can be explained by the amphiphilic nature of chlorophyll molecules, which possess a polar porphyrin head and a non-polar phytol tail [45]. Due to this dual character, chlorophylls are more efficiently extracted with solvents of intermediate polarity, such as acetone, rather than highly polar solvents like methanol [46,47]. Consequently, the methanolic phase extracted lower amounts of chlorophylls, while a higher amount remained in the residue. According to Hu, Tanaka A., and Tanaka R. (2013) [48], selecting an appropriate solvent for chlorophyll extraction can be challenging, as organic solvents may induce the conversion of chlorophyll to chlorophyllide. This conversion can also be triggered by certain extraction methods that promote enzymatic hydrolysis of chlorophyll through the enzyme chlorophyllase [48]. Since chlorophyllide has the same absorption spectrum as chlorophyll, conventional spectrophotometric methods cannot differentiate between them, which is why the decrease in total chlorophyll content is related to HVED treatments.

However, it is necessary to perform HPLC analysis to distinguish chlorophylls and their corresponding chlorophyllides formed during HVED treatment.

The observed decrease in chlorophyll content with increasing HVED treatment duration in the SBL samples is consistent with the findings reported by Mohan et al. (2025) [49], who demonstrated that high-voltage electrical discharge treatments can enhance the release of bioactive compounds under moderate conditions, whereas prolonged exposure may lead to pigment degradation due to the generation of reactive species. Similarly, Chalise et al. (2024) [50] reported that plasma-activated water treatments can reduce chlorophyll stability when plant tissues are exposed for extended periods. These studies support our observations that longer HVED treatments combined with lower solvent concentrations caused a significant decrease in chlorophyll content, while shorter treatments combined with higher solvent concentrations resulted in the highest pigment retention. Overall, these results emphasize the need for careful optimization of HVED parameters to maximize extraction efficiency while minimizing degradation of sensitive compounds, such as chlorophylls.

3.2.4. Antioxidant Activity of Tomato Peel and SBL Extracts

EPR measurements of the antioxidant activity of the tomato peel and SBL samples revealed a mainly positive effect of HVED on their antioxidant properties, as shown in Tables 7 and 8. According to the literature, these properties depend on the concentration of bioactive components present in samples. In tomato peel, antioxidant activity is primarily associated with carotenoids, polyphenols, and vitamin C [51], whereas in SBLs, it is mainly linked to their high content of polyphenols, betalain pigments, and vitamin C [52]. Statistical analysis showed no impact of the HVED treatment ($p = 0.356$), the solvent concentration ($p = 0.940$), or the combined effect of HVED and solvent concentration ($p = 0.881$) on the antioxidant activity of tomato peel samples. The same was observed for the SBL samples, where HVED treatment ($p = 0.358$), solvent concentration ($p = 0.940$), and their combined effect ($p = 0.881$) had no significant impact on antioxidant activity. Although statistical analysis showed no significant effect, results of EPR and DPPH measurements demonstrated that HVED treatment generally improved the antioxidant activity of both tomato peel and SBL samples, although the effect varied depending on solvent concentration and treatment duration. In tomato peel samples, the increased antioxidant activity is consistent with higher levels of bioactive compounds, particularly lycopene, beta-carotene, polyphenols, and vitamin C. For instance, lycopene content was highest after 3 min of HVED treatment for 30% methanol (1.74 mg/100 mL) and 50% methanol (2.04 mg/100 mL), while beta-carotene also increased under similar conditions. These enhancements in carotenoid content are associated with higher DPPH reduction values, with the highest percentage of reduction observed after 1 min (45.27%) for 30% methanol and 5 min (41.90%) for 50% methanol extracts. Although EPR-DPPH measurements indicated an increase in antioxidant activity after HVED treatment, the ascorbic acid equivalents (AAE) remained constant (0.01 mg/mL) across all treated and untreated samples. This can be explained by the fact that AAE reflects the overall antioxidant activity in terms of ascorbic acid equivalents, rather than the absolute concentrations of individual bioactive compounds. Variations in carotenoids, polyphenols, betalains, and vitamin C can offset each other, resulting in a stable AAE value despite differences in individual compound levels.

Table 7. Antioxidant activity of tomato peel samples expressed as % of reduction in DPPH-EPR signal intensity of mean ± SD. Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters indicate significant differences within rows, and lowercase letters within columns.

Antioxidant Activity of Tomato Peel Extracts				
HVED Duration (min)	30% Methanolic Extracts		50% Methanolic Extracts	
	% of Reduction in DPPH	AA Equivalents (μg/mL)	% of Reduction in DPPH	AA Equivalents (μg/mL)
0	34.60 ± 2.69^{aA}	4.74 ± 0.00^{aA}	28.00 ± 14.02^{aA}	3.10 ± 0.00^{aA}
1	45.27 ± 3.94^{aA}	7.38 ± 0.00^{aA}	41.20 ± 19.27^{aA}	6.37 ± 0.00^{aA}
3	42.28 ± 4.67^{aA}	6.64 ± 0.00^{aA}	40.58 ± 14.36^{aA}	6.22 ± 0.00^{aA}
5	44.34 ± 1.76^{aA}	7.15 ± 0.00^{aA}	41.90 ± 14.02^{aA}	6.54 ± 0.00^{aA}

Table 8. Antioxidant activity of SBL extracts expressed as % of reduction in DPPH-EPR signal intensity of mean ± SD. Statistically significant differences ($p < 0.05$) are indicated by different superscript letters: uppercase letters indicate significant differences within rows, and lowercase letters within columns.

Antioxidant Activity of SBL Extracts				
HVED Duration (min)	30% Methanolic Extracts		50% Methanolic Extracts	
	% of Reduction in DPPH	AA Equivalents (μg/mL)	% of Reduction in DPPH	AA Equivalents (50%) (μg /mL)
0	40.27 ± 9.54^{aA}	129.66 ± 0.06^{aA}	39.61 ± 11.20^{aA}	125.72 ± 0.07^{aA}
1	49.16 ± 2.68^{aA}	182.59 ± 0.02^{aA}	49.16 ± 2.68^{aA}	182.59 ± 0.02^{aA}
3	53.16 ± 1.82^{aB}	206.44 ± 0.01^{aB}	37.32 ± 0.33^{aA}	112.06 ± 0.00^{aA}
5	38.41 ± 4.15^{aA}	118.58 ± 0.02^{aA}	47.68 ± 26.90^{aA}	173.76 ± 0.16^{aA}

SBLs showed varying results in antioxidant activity. Specifically, when 30% methanol was used as the solvent, the highest DPPH reduction (53.16%) was observed after 3 min of treatment, whereas in samples with 50% methanol, the highest DPPH reduction (49.16%) was achieved after 1 min. Unlike tomato peel extracts, SBLs exhibited a more complex response. Total polyphenols in SBL samples remained relatively stable across treatments, but antioxidant activity, as measured by DPPH reduction, varied depending on the solvent and HVED duration. In SBLs, changes in antioxidant activity closely followed the concentrations of ascorbic acid equivalents (AAE), which varied with treatment conditions, while in tomato peel samples, AAE remained constant. The highest percentage of DPPH reduction for 30% methanol extracts (53.16%) was observed after 3 min, whereas for 50% methanol extracts, the maximum (49.16%) occurred after 1 min. Chlorophyll content, however, decreased with longer HVED treatments, suggesting that chlorophylls contributed minimally to antioxidant activity. These results indicate that, in SBLs, antioxidant properties are likely related to polyphenols, betalains, and vitamin C rather than chlorophylls. Similar trends were reported by Ebrahimi et al. (2022) [53], who observed that the antioxidant activity of sugar beet leaves depended strongly on extraction conditions and solvent composition, despite relatively stable polyphenol contents. Their findings support the results obtained in this study, indicating that solvent polarity and extraction or treatment parameters can alter the contribution of different bioactive compounds to overall antioxidant activity. Accordingly, the observed variation in ascorbic acid equivalents (AAE) in SBLs suggests that antioxidant activity is determined not only by polyphenols, but also

by the dynamic balance between phenolic compounds, betalains, and vitamin C under different HVED conditions.

4. Conclusions

Plasma technology, or HVED, is now recognized as an advanced non-thermal technology in many industries and is considered a modern technological standard. It has a wide range of applications, particularly in the food sector. Plasma can be generated under various conditions and in different environments. Monitoring, controlling, and adjusting plasma production parameters are important to ensure reproducible and reliable applications of advanced plasma technology in a stable environment.

In this experiment, HVED extraction proved to be an effective method for isolating bioactive compounds from tomato peel and sugar beet leaves (SBLs). The results show that HVED treatment duration and solvent concentration significantly influence the extraction of polyphenols, carotenoids, and chlorophylls. For tomato peel extracts, a 3 min HVED treatment was optimal for extracting total polyphenols and lycopene, while beta-carotene extraction improved with longer treatment times and higher solvent concentrations. In SBL extracts, HVED treatment did not have a statistically significant effect on total polyphenols, but it significantly influenced total chlorophyll content, with the highest concentrations observed in extracts obtained with higher solvent concentration after a 3 min treatment. These observations indicate that further optimization of HVED parameters may be necessary.

EPR measurements revealed the presence of Fe^{2+} and Mn^{2+} ions, as well as cellulose radicals in SBLs, while only cellulose radicals were detected in tomato peel, highlighting differences in radical composition between the plant matrices. HVED treatment generally improved the antioxidant properties of both extracts, with the highest activity observed after a 3 min treatment in most conditions, as confirmed by EPR-DPPH measurements.

These findings emphasize the need to optimize HVED parameters to maximize extraction efficiency and preserve bioactive compounds, highlighting the importance of dedicated optimization protocols for each extract type. EPR spectroscopy is highly suitable for monitoring and evaluating the impact of novel non-thermal technologies in food processing. EPR spectroscopy, through both direct and indirect methods, is indispensable for controlling food quality and guiding the development of new products, such as those based on the Mediterranean diet. Its advantages include the ability to determine key process parameters, monitor products during production and storage, and differentiate plant origin based on metal ion composition, which may reflect cultivation practices and soil characteristics.

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Abbreviations

The following abbreviations are used in this manuscript:

DPPH	2,2-diphenyl-1-picrylhydrazyl
EPR	Electron paramagnetic resonance
FC	Folin–Ciocalteu reagent
GA	Gallic acid
HVED	High-voltage electrical discharge
MWE	Microwave-assisted extraction
PEF	Pulsed electric field
SBLs	Sugar beet leaves
SDGs	Sustainable Development Goals
SFE	Supercritical fluid extraction
TP	Tomato peel
TPC	Total polyphenolic content
UAE	Ultrasound-assisted extraction

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