

Review

Environmentally Friendly Techniques for the Recovery of Polyphenols from Food By-Products and Their Impact on Polyphenol Oxidase: A Critical Review

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Abstract: Even though food by-products have many negative financial and environmental impacts, they contain a considerable quantity of precious bioactive compounds such as polyphenols. The recovery of these compounds from food wastes could diminish their adverse effects in different aspects. For doing this, various nonthermal and conventional methods are used. Since conventional extraction methods may cause plenty of problems, due to their heat production and extreme need for energy and solvent, many novel technologies such as microwave, ultrasound, cold plasma, pulsed electric field, pressurized liquid, and ohmic heating technology have been regarded as alternatives assisting the extraction process. This paper highlights the competence of mild technologies in the recovery of polyphenols from food by-products, the effect of these technologies on polyphenol oxidase, and the application of the recovered polyphenols in the food industry.

Keywords: bioactive compounds; food wastes; enzymatic browning; nonthermal technologies; ultrasound; microwave; pulsed electric field; ohmic heating; cold plasma; pressurized liquid extraction



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1. Introduction

As stated by previous studies, food by-products are approximately one-third of the total food production, and this amount is increasing annually. These wastes can give rise to the generation of a significant quantity of greenhouse gases, causing many problems such as climate change [1]. Nonetheless, food by-products are still regarded as principal sources for the extraction of food bioactive compounds [2–6]. Bioactive compounds are found numerously in nature and are extensively used in the food industry [7]. The recovery of these valuable compounds decreases the financial and environmental impact caused by food wastes. Thus, the challenge for the contemporary time is likely to find processing methods that are able to reuse by-products in industries and recover the nutrients still contained in the food wastes [8,9].

Polyphenols are the most pervasive bioactive compounds found in agroindustry by-products [10,11], various fruits [12,13], seeds [14], cereals [15], nuts [16], vegetables [17], tea [18,19], coffee [20], etc. These compounds are a group of secondary metabolites derived from phenylalanine, and they own one or more phenolic rings with one or several bound hydroxyl groups. Polyphenols act as natural antioxidants, enhancing the nutritional value of food by retarding the oxidation of lipids [8,21,22]. To date, many studies have classified polyphenols as nutritional sources possessing a highly beneficial impact on the prevention of many diseases, including diabetes, obesity, atherosclerosis, hyperlipidemia, hypertension, Alzheimer's, and thrombosis [21,23]. Therefore, it is crucial to recover these compounds from herbal food wastes and use them also in the production of functional foods and ingredients.

However, there is a large challenge regarding the efficient extraction of polyphenols from food by-products, since conventional extraction methods consume plenty of time

and require a relatively large amount of solvent and energy. Indeed, extract quality, extraction rate, final cost, extraction yield, consumer, and environmental protection are some determining factors in choosing the more useful technology for recovering polyphenolic compounds [24]. Another challenge is to prevent climate change caused by the emission of heat from different industries [25]. Hence, there is a growing demand for novel extraction methods with less solvent consumption, shorter time, and higher extraction yield [7]. Recently, many sustainable technologies such as microwave [26], ultrasound [27], cold plasma [28], pulsed electric field [29], pressurized liquid [30], and ohmic technology [31] have been utilized in the pre-treatment and extraction process of polyphenols. In these techniques, the temperature is not the decisive factor, and thus they require lower energy and have a lower effect on the loss of sensitive compounds, resulting in better recovery of bioactive compounds [32].

Therefore, it is critical to study the efficiency of mild technologies in the extraction of polyphenols from wastes produced in the food industry and agricultural processes. In addition, the control of polyphenol oxidase during the extraction of polyphenolic compounds is another crucial issue for maintaining their nutritional attributes. Accordingly, this paper seeks to review up-to-date information regarding the valorization of food by-products as rich sources of polyphenols in the extraction process using novel technologies that could be useful also for avoiding the impact of enzymatic browning on the polyphenolic compounds recovered.

2. Application of Polyphenols Recovered from By-Products

As the consumers' perspective on healthy eating habits has changed over the past few years, food products with added health-promoting compounds are becoming more attractive [15]. In addition, the negative view of consumers over synthetic additives has raised their interest in natural compounds [22]. This perspective could be a strategy for developing new functional foods [33]. In this regard, polyphenols have many applications in developing innovative products in various technological and industrial fields (Figure 1). Cisneros-Yupanqui et al. (2020) used the polyphenols obtained from the by-products of the winemaking industry (grape pomace) for delaying the oxidation of corn oil and enriching its antioxidant properties for health-promoting purposes [10]. Moreover, Cisneros-Yupanqui et al. (2021), in another study, reported that red chicory leaves, which are rich in phenolic compounds, could be utilized in the formulation of a functional jam, which has health-promoting effects on people suffering from dysphagia [34]. Jirasuteeruk et al. (2019) employed the polyphenols extracted from mango peel to inhibit the enzymatic browning of potato puree. They reported that the mango peel extract has a competitive inhibitory effect on potato PPO compared to ascorbic or citric acid [35].

In addition, some polyphenols (e.g., anthocyanins) could be employed as natural food dyes due to their coloring properties [36]. Since the synthetic dyes used in the food industry may pose potential health risks to consumers, it is recommended that natural dyes such as polyphenols be used as an alternative [37]. In respect of this, when polyphenols are used as a colorant agent in food products, they could provide antioxidant properties together with color, making the product a potential functional food [38]. Moreover, the instability of natural colors is an important issue to address, and the comparison between different plant sources is necessary. In this respect, Ghareaghajlou et al. (2021) reported that anthocyanins isolated from red cabbage represent the color in a broad range of pH values compared to anthocyanins recovered from other natural sources. Furthermore, polyphenols are used as components of smart packaging [25]. For instance, anthocyanins, catechins, theaflavins, etc., have pH-sensitive properties, and when they are used in food packaging, they show different colors in different pH solutions. This feature could be used to monitor the freshness of fish [39,40]. Among novel technologies used for the recovery of anthocyanins, the ultrasound-assisted extraction (UAE) method is the most commonly applied approach, having a satisfying efficiency [41].

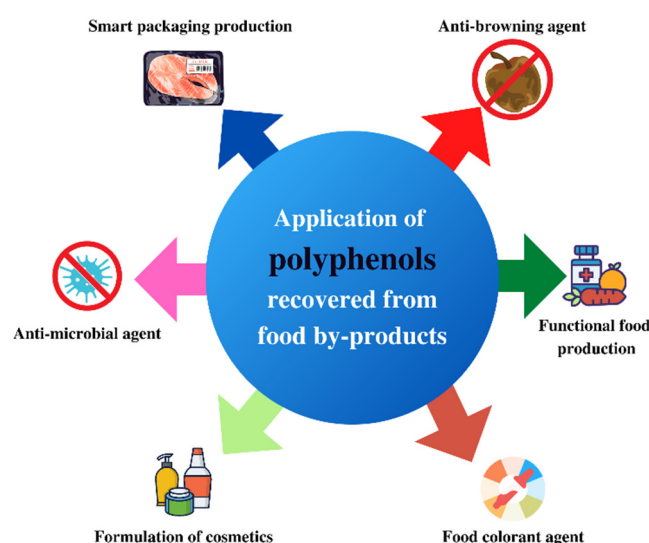


Figure 1. Industrial applications of polyphenols.

3. Different Extraction Methods of Polyphenols

Methods employed in the recovery of polyphenols from food by-products could be classified into mild and conventional approaches (Figure 2). Conventional approaches comprise solvent extraction, Soxhlet extraction, squeezing or cold pressing, and steam distillation. The most prominent mild technique-assisted methods include ultrasound, microwave, pulsed electric field, ohmic heating, cold plasma, and pressurized liquid extraction. Depending on the type of these techniques, they could be used either as the main extraction method or as a pretreatment before other extraction techniques [42–44]. For instance, Tzima et al. (2021) employed the pulsed electric field as a pretreatment step before the UAE of polyphenols from fresh rosemary and thyme by-products. In fact, because of the ability of the pulsed electric field to electroporate cell envelopes, it could be used as a pretreatment to facilitate the recovery of polyphenols using a subsequent conventional or novel extraction method [45]. Furthermore, the usage of organic solvents or water mixtures in combination with some nonthermal extraction methods could provide promising results [45,46].

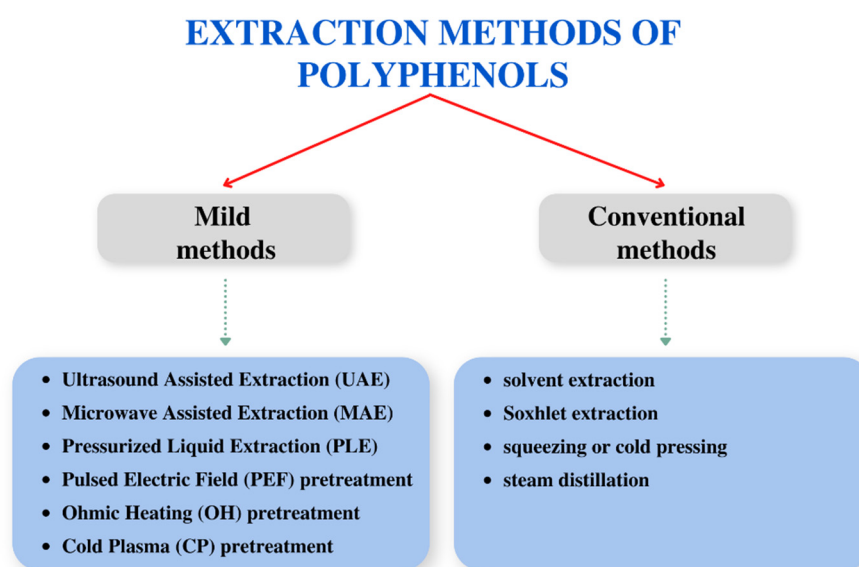


Figure 2. Classification of polyphenols' extraction methods.

3.1. Ultrasound-Assisted Extraction (UAE)

In this process, mechanical vibrations are produced by the passage of ultrasound waves through a liquid medium. The transmission of sound waves induces compression and rarefaction areas in the medium. This leads to the formation of many bubbles exploding at the next level. The collapse of these bubbles causes a phenomenon named acoustic cavitation. In other words, the growth and collapse of bubbles by ultrasonic effect is known as the acoustic cavitation phenomenon. The rarefaction phase is the growth of bubbles due to the reduction in local pressure, while the compression phase is the decrease in the bubble surface area. During acoustic cavitation, both the rarefaction and compression phases occur simultaneously. When the size of bubbles increases to above their critical extent, they implode and cause the cavitation phenomenon [47].

The cavitation phenomenon is divided into two categories, namely, stable cavitation and transient cavitation. When the acoustic pressure is inadequate and the bubbles do not reach their critical size, it is called stable cavitation. Conversely, when the pressure is adequate to generate the implosion, it is called transient cavitation. The growth and collapse of bubbles and release of energy at the molecular level can result in arising a high temperature (up to 5000 K) and pressure (up to 1000 atm) [47]. Indeed, UAE involves physical and chemical forces different from those involved in conventional solvent extraction. The cavitation resulting from the physical forces causes the breakdown of the plant cell membrane and enhances the extraction process of polyphenols [48]. In other words, the cavitation may cause hydration and swelling of herbal food matrices, which produce micro-bubbles and micro-jets rupturing the cell of the food matrix and favoring the penetration of the solvent into them. This could simplify the release of polyphenols, resulting in an increased extraction yield [49]. Ultrasound processes could be employed using various types of tools and apparatus depending on the aim of its usage. This variation could be ranged from basic ultrasonic water baths to more advanced high-power ultrasonic generators [47]. Figure 3 illustrates a simplified schematic of the extraction of polyphenols using UAE.

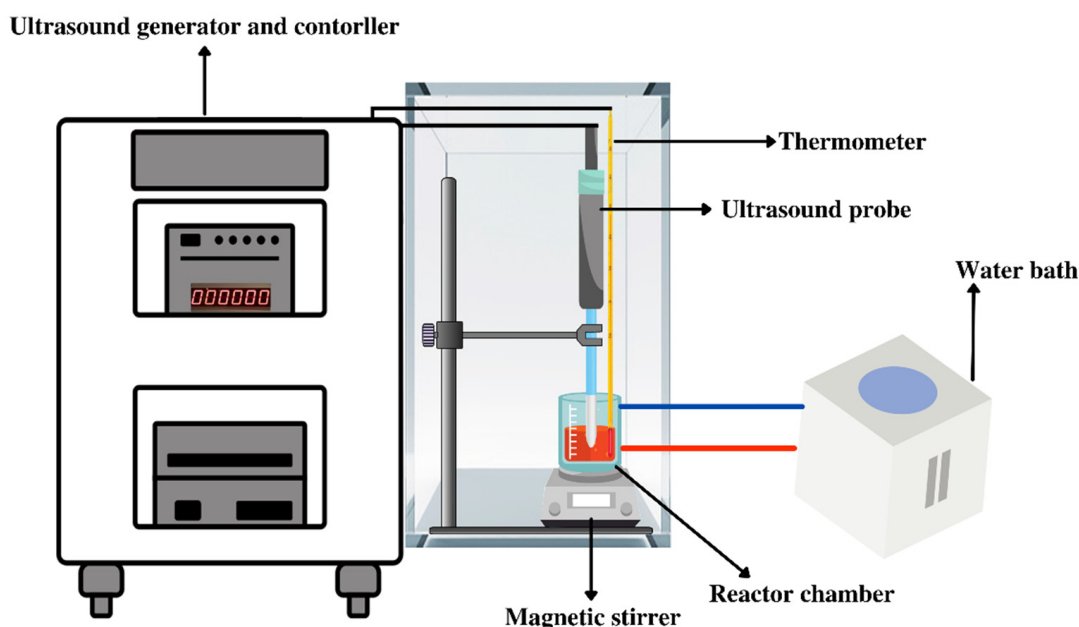


Figure 3. Simplified schematic of ultrasound assisted extraction of polyphenols.

The efficacy of the UAE is impacted by some factors, including extraction time, acoustic intensity, solid/solvent ratio, solvent type, temperature, and the height of the solvent around the sample within the extraction container [48]. Depending on the food matrix, volume, and ultrasound processing states, the temperature of the sonicated medium

increases with the formation of hot areas due to cavitation. As shown in Figure 4, ultrasound waves are divided into two groups according to their usage in the food industry [47].

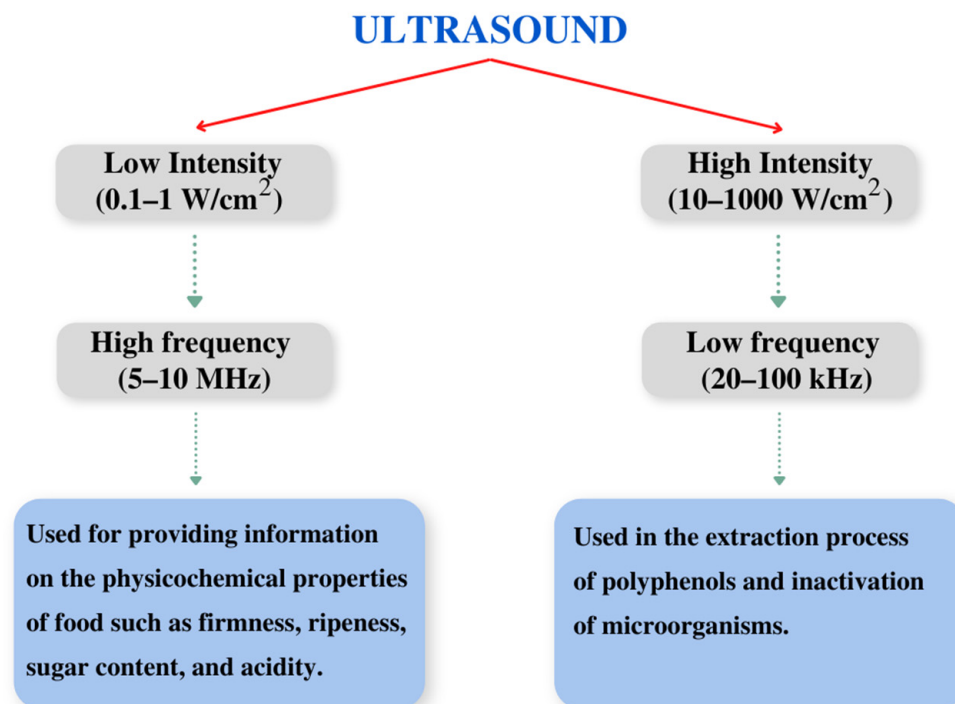


Figure 4. Classification of ultrasound waves according to their usage in food science.

The processing parameters in UAE are power, amplitude, frequency, intensity, treatment time, volume, and composition of food [50]. The intensity of ultrasound indicates the amount of power added per unit area [47]. This factor is calculated by the relation between the acoustic power and the surface area of the probe (Equation (1)) [51]. The higher the ultrasound intensity is applied to the solvent mixture, the more violently the cavitation bubbles implode [52]. Ultrasonic power can be determined calorimetrically using Equation (2). By fitting the temperature data versus time and extrapolating to the initial time, one can calculate dT/dt . Moreover, acoustic energy density (AED) is measured using the relation between acoustic power and the volume of the treatment medium (cm³ or mL) (Equation (3)).

$$I = \frac{P}{A} \quad (1)$$

$$P = mC_p \left(\frac{dT}{dt} \right)_{t=0} \quad (2)$$

$$AED = \frac{P}{V} \quad (3)$$

where I is the intensity, P is the power, A is the area, m is the mass, C_p is the specific heat capacity, and dT/dt is the initial rate of change in temperature during sonication [51].

There are plenty of merits stemming from the usage of UAE, including low temperature, optimized energy and mass transfer, selective extraction, effective mixing, small equipment size, high extraction rate, fast start-up, and improved production [53,54]. However, some studies have outlined that UAE might have some drawbacks, such as the reflection of the produced off-flavors in the food [55]. During the last few years, UAE has been one of the most utilized advanced techniques for the recovery of polyphenols from food by-products [56]. Table 1 highlights the recent research on the usage of UAE in the recovery of polyphenols from food by-products.

Table 1. Recovery of polyphenols from food by-products using UAE.

Food By-Product	Solvent	Solid/Solvent Ratio (<i>w/v</i>)	Power (W) (Based on Amplitude)	Frequency (kHz)	Extraction Time (min)	TPC ¹ in Final Extract (mg GAE ² /g)	Reference
Coffee silverskin	Methanol (80%)	1:50	NR ³	20	10	8.94 ± 0.01	[52]
Brazilian olive leaves	Water	0.5:25	247.5	20	29	80.51 ± 1.52	[57]
Potato peels	Methanol (80%)	1:10	100	33	900	4.24 ± 0.01	[58]
Tomato peel	Ethanol (70%)	1:50	380	30	15	36.43 ± 0.1	[59]
Lemon wastes	Water	1:100	250	43 ± 2	45	18.10 ± 0.24	[60]
Mango peels	Ethanol (50%)	1:30	NR	NR	10	35.5	[61]
Mango peels	Water	1:6	160	50	15	9.72	[35]
Beet leaves	Water	1:20	90	20	16	14.9	[62]
Pomegranate peels	Ethanol (70%)	1:70	140	40	30	69.89 ± 0.45	[63]
Grape seeds	Ethanol (61.76%)	1:30	250	28	20	25.96 ± 0.70	[64]
Olive leaves	Water	05:25	112.5	20	25	79.77	[65]
Lime peel	Ethanol (55%)	1.5:30	NR	NR	4	54 ± 0.2	[66]

¹ Total phenolic content; ² gallic acid equivalent; ³ not reported.

As declared by previous studies, increasing the extraction time could positively impact the extraction yield of polyphenols [65]. However, it is proven that the UAE with a higher amount of power and shorter times is more efficient because the long duration of sonication could cause the production of some free radicals from water, inducing the deterioration of polyphenols by activating radical chain reactions. In other words, the increase in the power in the UAE method could increase the extraction yield of polyphenols and shorten the time required for the extraction. In addition, the reduction of UAE time can lessen energy consumption significantly [62]. Increasing the extraction time higher than a threshold has no meaningful change in the phenolic content. This condition could be justified by Fick's second law of diffusion, stating that an equilibrium concentration between a solution and a solid matrix occurs after a certain period. Therefore, it is not necessary to increase the time for further extraction of polyphenols [61].

Since UAE is a nonthermal method, some works in the literature have proposed using thermal profiling to control the temperature of the extraction process [62,67]. However, when ultrasound is used as an extraction method, the heat generated in the medium solvent may be advantageous for enhancing extraction yield because it alters the membrane structure of herbal cells and simplifies the distribution of the extracted molecules into the solvent [62,68]. Nevertheless, due to the sensitivity of polyphenols to heat, their antioxidant activity may be lost when the temperature is greater than 55 °C [61].

In UAE, the polarity of the solvent could increase when there is a growth in the cavitation or bubble formation. This can result in an improved extraction yield [63]. As reported by Wen et al. (2019), the solvent has a substantial influence on the recovery yield of polyphenols. When the combination of organic solvents and water is employed as a medium in this process, the yield rises [52]. Grassino et al. (2020) reported that the extraction yield of polyphenols with 70% ethanol is higher than 96% ethanol. Indeed, the addition of water to pure ethanol provides better distribution of polyphenols and enhances the yield of this process [59]. Kaur et al. (2021) stated that solid/solvent ratio is a prominent factor in the extraction yield as the increase of the solid weight may decrease the recovery yield of polyphenols because of the extraction of other biomolecules (e.g., proteins and polysaccharides), which could dissolve in the solvent and influence the dissolution of polyphenols [61].

Kumari et al. (2017) reported that UAE with a low frequency and high power is much more efficient. Higher extraction yield of polyphenols at a lower frequency might be linked to the increased intensity of acoustic cavitation in the solvent because cavitation

intensity is inversely correlated with ultrasonic frequency. Enhanced extraction yield at a lower frequency may be associated with the production of larger but fewer cavitation bubbles collapsing with higher energy levels, which results in a more significant extent of cell disruption [58].

3.2. Microwave-Assisted Extraction (MAE)

MAE is an efficient advanced extraction technique integrating the conventional solvent extraction and microwave approach to recover polyphenols from food wastes. Table 2 represents the recent research in this area. This technique has plenty of advantages due to its rapid extraction rate, short extraction time, low solvent volume, and excellent product quality at a reasonable cost [66,69]. However, this method has some drawbacks, including high technical complexity, less control on the energy input, reduction of heat-sensitive bioactive compounds, high preliminary cost, and inadequate extraction yield [70].

Microwave technology is nonionizing electromagnetic radiation containing a frequency of 300 MHz to 300 GHz and a wavelength of 1–1000 mm [71,72]. Microwaves transfer energy from electromagnetic waves into thermal energy without contacting the food. The electromagnetic waves are generated from an emitter exposed to the food matrix [73]. The fundamental principles of microwave technique during the extraction process lie in the movement of energy by an electric field via two corresponding mechanisms [74]. Indeed, MAE is based on the interaction of microwave energy with the polar molecules (e.g., water) in the solvent surrounding the sample to generate heat. The increase in the temperature is due to the ionic conduction and dipole rotation, which results in increased cell wall destruction and efficient extraction of polyphenols [26,75]. Microwaves selectively warm polar molecules with low molecular weight and high dielectric constant, while nonpolar molecules remain static in the microwave electric field [74]. This selective heating may induce the formation of high-temperature microzones named hotspots compared to the temperature of the reaction medium. Eventually, the temperature of the matrix increases quickly, resulting in increasing the chemical reaction rates [76].

Table 2. Recovery of polyphenols from various food by-products using MAE.

Food By-Product	Solvent	Solid/Solvent Ratio (w/v)	Power (W)	Temperature (°C)	Time (s)	TPC ¹ in Final Extract (mg GAE ² /g)	Reference
Lime peel	Ethanol (55%)	1.5:30	140	Lower than 60	45	35 ± 0.5	[66]
Apple pomace	Ethanol (62.1%)	1:22.9	650.4	70	53.7	≈0.62	[77]
Avocado seeds	Ethanol (60%)	1:20	400	NR ³	180	82.36 ± 1.05	[26]
Pomegranate peels	Water	5:100	600	NR	60	87.81 ± 0.83	[63]
Blueberry leaves	Ethanol (30%) + citric acid (1.5 M)	0.5:80	142.1	NR	1440	128.760 ± 1.2961	[75]
Brazilian olive leaves	Water	0.5:25	1000	86	180	104.22 ± 0.61	[57]
Apple skins	Ethanol (68%)	2:20	NR	150	5400	50.4 ± 3.4	[78]
Olive leaves	Ethanol (70%)	0.5:25	1000	65	300	157.62	[65]

¹ Total phenolic content; ² gallic acid equivalent; ³ not reported.

The most prominent parameters in MAE are extraction temperature, solvent composition, extraction time, microwave power, and solid/solvent ratio [69]. The extraction yield of MAE could be improved by raising microwave power and time to a certain extent. However, when these two factors are higher than a threshold, the possibility of a significant loss in heat-sensitive bioactive compounds increases [79]. The stability of the extractive compounds is a deciding factor in selecting the optimum temperature [80]. Overall, the quantity of total recovered polyphenols increases with rising temperature because at higher temperatures, a decrease in solvent viscosity and an increase in intermolecular interaction and intracellular pressure occur, which give rise to a higher molecular motion improving the solubility of polyphenols in the solvent [65]. At low temperatures, the long exposures to microwave radiations diminish the extraction yield due to the loss of the chemical structure

of bioactive compounds [81]. The extraction time starts from only a few seconds to several minutes to prevent oxidative stress and thermal degradation. When a longer exposure time is needed, the thermal degradation of the matrix could be prohibited through the extraction cycle. This can be controlled by providing renewed solvent to the repetitive extraction cycle to guarantee the culmination of extraction [82].

The proper solvents may increase the extraction yield. Many solvents could be utilized in MAE, including water, methanol, acetone, ethanol, and their mixture [69]. They should be chosen according to the food matrix under extraction. This choice depends on the solubility of the compound in the solvent, the penetration power of solvent into the matrix, as well as its dielectric constant. Organic solvents, such as ethanol, acetone, and methanol, could be effectively employed in the extraction process. Since the presence of water could enhance the penetration power of solvent into the food matrix and increase heating efficiencies, water-based solvents are the most preferred ones for the extraction of bioactive compounds [83,84]. In this respect, Rodsamran et al. (2019) reported that pure ethanol gave the lowest yield of polyphenols from lime peel waste, while 50–60% ethanol concentration (diluted with water) resulted in higher yields of polyphenols. Since polyphenols are polar/hydrophilic molecules, the addition of water to the organic solvents increases their polarity index, which results in higher recovery of polyphenols in the extraction process. In addition, water could increase the dielectric constant of the solvent and absorption of microwave energy, leading to a higher temperature inside the sample, which leads to the disruption of cells and easier release of polyphenols [66]. In MAE, ethanol can increase the degree of sample cell membrane breakage and improve phenolic compounds solubility. However, as ethanol concentration increases, the polarity of the solvent changes, which may lead to increased impurities being extracted, therefore reducing the number of polyphenols extracted. Moreover, increased diffusion resistance due to the coagulation of proteins at high ethanol concentrations may inhibit the dissolution of polyphenols and affect the extraction rate [26].

It should be kept in mind that the selection of solvent for MAE is not consistent with the traditional extraction process. For instance, diethyl ether is not proper for MAE as a solvent, while it is employed in conventional methods [85]. Moreover, a modifier could be used to improve the performance of the solvent. For instance, acetone can be added as a modifier to methanol in the extraction of curcumin from *Curcuma longa* using MAE [24]. Furthermore, room-temperature ionic liquids are becoming one of the most attractive solvent modifiers in the extraction process due to their outstanding solvent properties. They have many advantages, including good low vapor pressure, thermal stability, wide liquid range, miscibility with organic solvents and water, and perfect solubility of different bioactive compounds [22,37]. Thus, they are preferable for unstable compounds since high solvent power can improve the extraction efficiency, and it lessens the overexposure to microwave heating [86].

In MAE, the sample should be in dried and powder form for an optimum extraction yield. If the particle size is too small, it causes a problem in the extraction and may require an extra washing stage [87]. Recently, microwave-assisted drying and extraction technique has emerged as a novel notion where microwave drying is combined with a condenser. The vapors (containing polyphenols) vaporized from the sample (to be dried) pass through the condenser and condense to the liquid extract. Using this approach, the bioactive compounds are extracted without the use of external solvents. The technique is suitable for the sustainable extraction of bioactive compounds from different fruits, vegetables, and herbal foods during drying [88]. Figure 5 shows a simplified schematic of extraction of polyphenols using MAE technique with the help of a condenser.

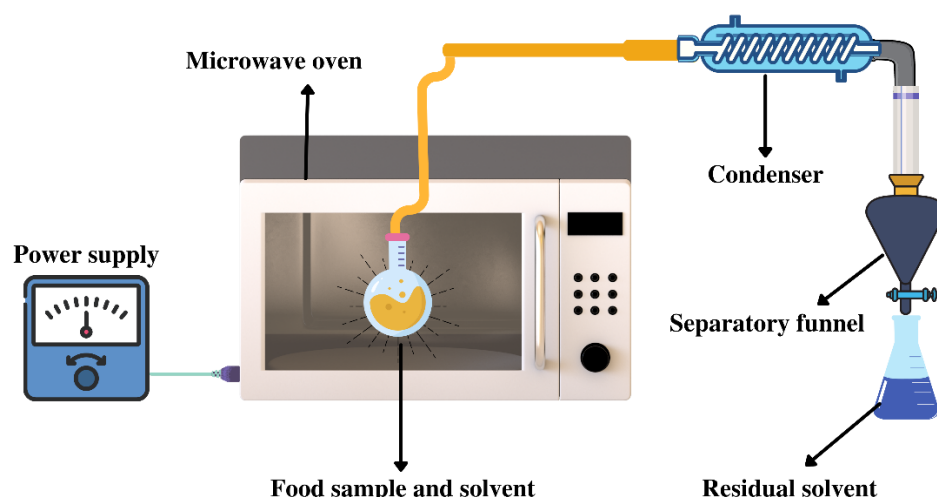


Figure 5. Simplified schematic of MAE with the help of a condenser.

As reported by Rosa et al. (2021), MAE has a better performance compared to the UAE method in the extraction of polyphenols from Brazilian olive leaves. This could be attributed to the greater cell wall disruption under microwave processing, resulting in faster release of the cell compounds into the solvent. Moreover, the temperature applied in MAE could enhance the permeation and the solubilization processes to wash the intracellular compounds out of the matrix. However, many plant compounds are sensitive to high temperatures, and the use of microwave energy during the extraction may result in poor extraction yield because of the deterioration of these compounds. Nevertheless, the rising temperature in a shorter extraction time may increase the extraction yield, as it reaches a maximum before decreasing [57]. Furthermore, Rosa et al. (2019) reported that when MAE is used as pretreatment in UAE, the recovery of polyphenols increases significantly compared to when each method is used separately [65].

3.3. Pressurized Liquid Extraction (PLE)

PLE is another green method frequently used for the recovery of polyphenols from food by-products. Many advantages could be attributed to this technique such as short extraction time, elevated pressure and temperature, automatization of process, and low consumption of solvents [56,89,90]. The equipment required for PLE method are relatively simple, including a solvent container, an oven holding the extraction cell, a pump, blocking valves, and a collecting vial. However, there are some commercially available PLE apparatus [30]. Figure 6 illustrates a simplified schematic of the equipment used in the PLE process.

Firstly, the food sample is placed into the extractor and reaches the desired temperature using an oven, and the pressure is adjusted to the required level. This technique could be carried out also at room temperature. Afterwards, the solvent is transferred to the extraction cell using a pump. When the desired temperature and pressure are reached, the extraction process begins. This process could have more than one extraction cycle, and for each new cycle, the extracting solvent is renewed. The blocking valves are critical for controlling the extraction pressure. Inert gases (e.g., nitrogen) may be used for purging the apparatus by removing the residual solvent [30,44].

The temperature in PLE ranges from room temperature to 200 °C, but the temperatures higher than 140–150 °C may lead to the degradation of thermosensitive compounds or formation of undesirable products due to Maillard reaction. Since high pressure is applied in this process, the temperature could exceed the boiling point of the solvent. Indeed, the high pressure, ranging from 8 to 15 MPa, maintains the solvent in the liquid state [24]. This allows improved solubility and mass transfer between the food matrix and the solvent, leading to better extraction of polyphenols. In addition, it could decrease the viscosity of

the extractant, which results in improved soaking of the food matrix. This causes the high solubility of the polar compounds. Moreover, the temperature can lead to the improved diffusion rate of polyphenols by the breakage of bonding forces between molecules, causing a better recovery rate [91]. The recent research on the extraction of polyphenols using PLE method is provided in Table 3.

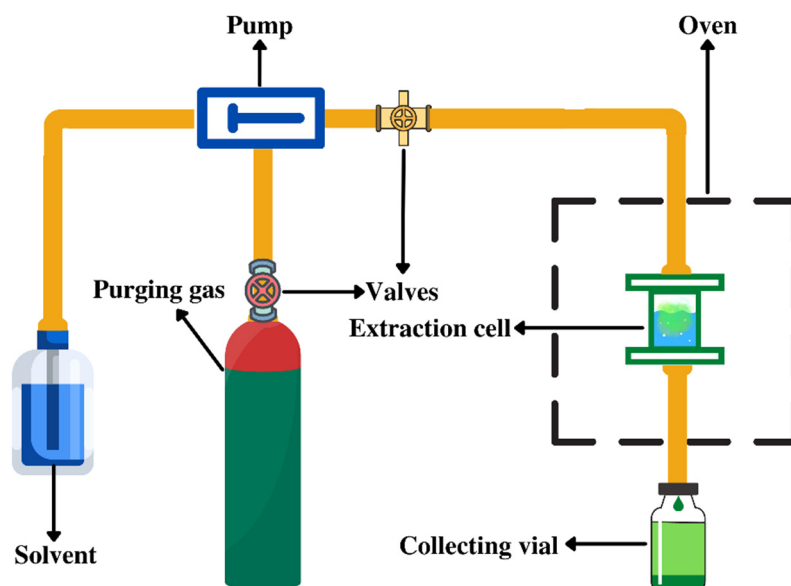


Figure 6. Simplified schematic of extraction of polyphenols using PLE.

Table 3. Recovery of polyphenols from various food by-products using PLE.

Food By-Product	Solvent	Pressure (MPa)	Temperature (°C)	Time (min)	TPC ¹ in Final Extract (mg GAE ² /g)	Reference
Burdock roots	Ethanol (70%)	NR ³	NR	105	12.13 ± 0.34	[56]
Cocoa bean hulls	Ethanol (70%)	10	70	20	9.6 ± 0.3	[92]
Pomegranate peel	Ethanol (77%)	10.34	200	20	17 ± 3.6	[93]
Mulberry pulp	Methanol (74.6%)	10.13	99.4	10	2.18	[94]
Jaboticaba skin	Ethanol (99.5%)	5	120	15	18.7 ± 0.4	[95]
Parsley flakes	Ethanol (50%)	6.9	40	5	22.9	[96]
Grape marc	Ethanol (50%)	10	100	40	65.68 ± 2.24	[97]

¹ Total phenolic content; ² gallic acid equivalent; ³ not reported.

3.4. Pulsed Electric Field (PEF) Pretreatment

PEF is an emerging technology based on the usage of an external electric field that yields reversible or irreversible electroporation in cell membranes. The electroporation phenomenon induces cell transformation or rupture under the utilization of a few to several hundred short pulses with a period ranging from microseconds to milliseconds [29,98]. In other words, electroporation refers to the exposure of cells to transmembrane electrical pulses [99]. Involving an external electric field in cells gives rise to the formation of pores in the membrane. Since pore formation is a dynamic process according to the intensity of the PEF, electroporation can be reversible or irreversible. When the generated pores are smaller than the membrane area and are produced with a low-intensity PEF, the electric breakdown is reversible [100]. The rise in the electric field strength and treatment duration induces an increase in the intensity of the treatment, which results in the transformation of reversible permeabilization to the irreversible disruption of the cell membrane. The irreversible disruption of the cell membrane facilitates the usage of treated compounds in various

areas, including the extraction of bioactive compounds from different sources [98,101]. When PEF is used as a pretreatment in the extraction, diffusion of polyphenols from the cell matrix into aqueous media occurs under mild conditions, and it does not need additional solvents [29]. The efficiency of PEF pretreatment depends on some factors, including extraction time, treatment temperature, pulse frequency, pulse shape, specific energy input, electric field strength, pH, pulse width, food matrix density and size, and chemical properties of extracting by-product [102]. PEF has some key advantages, such as the maintenance of the quality of the extracted products and the possibility of its application on a continuous industrial scale [103,104]. Figure 7 displays the simplified schematic of extraction of polyphenols using PEF pretreatment.

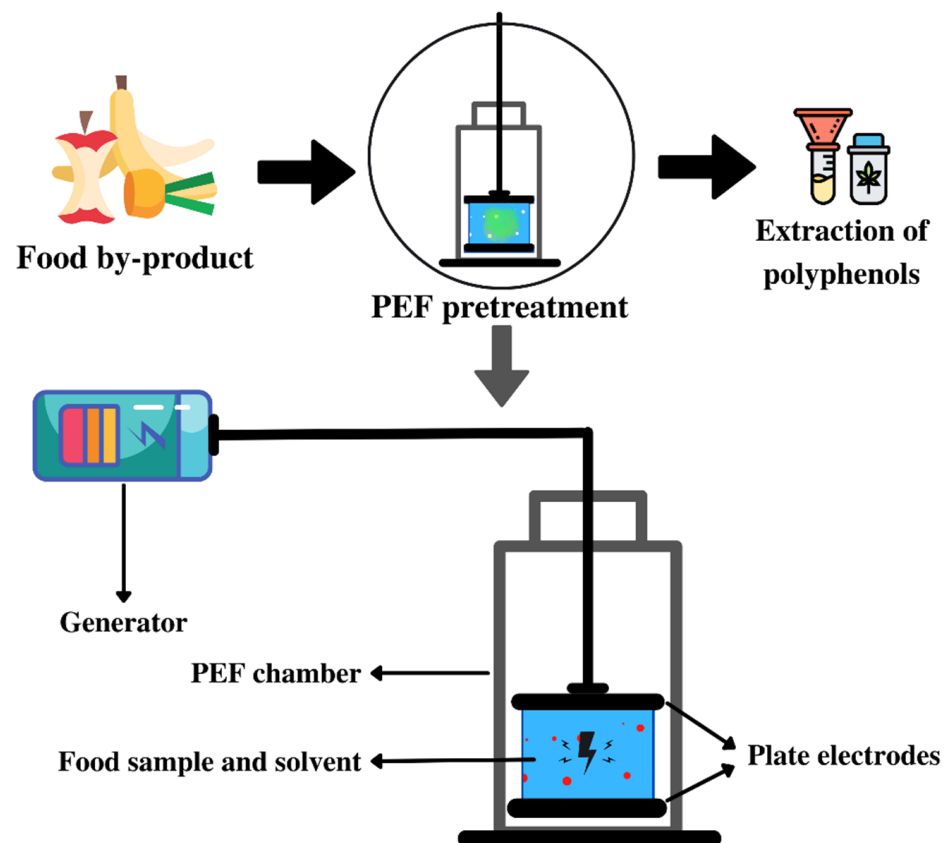


Figure 7. Simplified schematic of extraction of polyphenols using PEF pretreatment.

Peiró et al. (2019) evaluated the influence of PEF pretreatment in the recovery of polyphenols from lemon residues using pressing extraction technique, and they reported that the electric field intensity of 7 kV/cm and applying 30 pulses in the duration of 3 μ s increased the efficiency of polyphenol extraction by 300% compared to the untreated extracts [105]. Moreover, Rajha et al. (2019) used different nonthermal treatments, including PEF, in the extraction process from pomegranate peels, and they concluded that PEF with the electric field of 10 kV/cm selectively improved the recovery of ellagic acid compared to other methods [106]. According to the research conducted by Parniakov et al. (2016), the application of a two-step extraction procedure can allow a noticeable enhancement of the extraction yields of phenolic compounds (+400%) from mango peels. These two steps comprise a two-stage pretreatment of PEF as the first step and the aqueous extraction as the second step [107]. Table 4 provides broader details on the usage of recovery of polyphenols by the pretreatment of PFE.

Table 4. Recovery of polyphenols from various food by-products using PFE pretreatment.

Food By-Product	Solvent	Electric Field Strengths (kV/cm)	Pulse Duration (μ s)	Number of Pulses	TPC ¹ in Final Extract (mg GAE ² /g)	Reference
Lemon residues	Water	7	3	30	1.61	[105]
Pomegranate peels	Water	10	NR ³	n	39 \pm 2	[106]
Mango peels	NR	13.3	8.3	300	2.169	[107]
Borage leaves	Acidic water	5	3	20	Lower than 1.2	[108]
Orange peels	Water	10	70	n	22	[109]
Sesame cake	Water	13.3	10	up to 700	Lower than 4	[110]
Spearmint leaves	Mannitol	4.5	10	99	Lower than 10	[111]

¹ total phenolic content; ² gallic acid equivalent; ³ not reported.

3.5. Ohmic Heating (OH) Pretreatment

OH is a thermal–electrical method developed in the past few decades [112]. In the extraction process, OH reduces the treatment time, which this feature causes the least thermal damage to the polyphenols. However, this technology has some disadvantages, including its inefficiency in non-conductive and non-homogeneous food matrices. Moreover, the application of OH in foods with a high quantity of proteins may lead to deposit formation on the surface of electric-supplying electrodes, resulting in an electrical arcing [113].

This technique works on the basis of the contact of an electrode with a food matrix flowing an alternating electrical current (AC) with a frequency of 50 Hz to 100 kHz through it. This AC generates heat inside the food due to its natural electrical resistance. The food is heated rapidly in several seconds to a few minutes, and the amount of this heat depends on the voltage difference and electrical conductivity of the food [114]. The food matrix acts as the element of the electric circuit allowing the AC to flow. The yielded energy in this method is directly proportionate to the square of the electric field strength and the electrical conductivity of the food matrix. The most prominent factors in OH technology are the electrical field strength, the electrical conductivity of the food matrix, frequency, the type of waveform (Sine, Square, Triangle, Pulsed), concentration, electrodes, and particle size. This rapid heating approach has been used for many food products (e.g., dairy products, fruits, vegetables, and meat products) and is practical in the food industry for blanching, sterilization, pasteurization, evaporation, cooking, thawing, starch gelatinization, fermentation, and by-product utilization (i.e., extraction of bioactive compounds) [112,113,115]. OH can induce an electro and thermal-permeabilization of cell membranes, causing disturbances on their permeability and structural alterations, which contributes to the release of higher amounts of phenolic compounds [116]. Although OH has been applied for some time, there are few studies on its influence as an extraction method of polyphenols [87]. A simplified schematic of extraction of polyphenols using OH pretreatment is shown in Figure 8.

Coelho et al. (2019) reported that when OH is used as a pretreatment (70 °C for 15 min using 70% ethanol as a solvent) in the extraction of polyphenols from tomato by-products, rutin is recovered 77% higher than with conventional methods. The application of OH pretreatments was reported to induce the permeabilization of cell membranes and to facilitate the extraction of polyphenols with ethanol addition [31]. Kutlu et al. (2021) used OH as a pretreatment for extracting polyphenols from cornelian cherry before using UAE, and they reported that the highest extracted TPC was 7.52 mg GAE/g compared to the maceration and UAE methods [117]. It is also reported that the usage of OH as a pretreatment in the extraction of anthocyanins from grape skins increases the efficiency of extraction significantly [118]. Table 5 provides more information about the research conducted in this area.

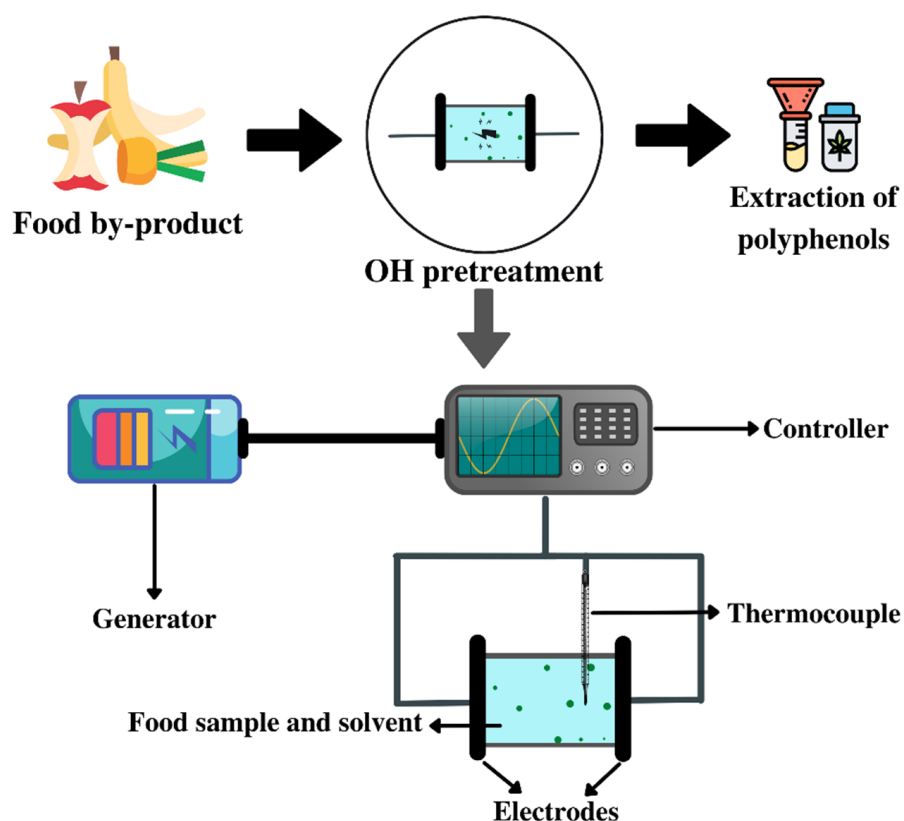


Figure 8. Simplified schematic of extraction of polyphenols using OH pretreatment.

Table 5. Recovery of polyphenols from various food by-products using OH pretreatment.

Food By-Product	Solvent	Voltage (V)	Electrical Field Strength (V/cm)	Temperature (°C)	Time (min)	TPC ¹ in Final Extract (mg GAE ² /g)	Reference
Yacon leaves (red, fresh)	NaCl solution (0.3%)	150	NR ³	NR	10	76.67 ± 21.67	[116]
Grape skins	Water	NR	16	100	1 s	3.2	[118]
Tomato peels	Ethanol (70%)	NR	NR	70	15	2.550 ± 0.072	[119]
Vine pruning residue	Ethanol (45%)	NR	840	80	60	3.1 ± 0.2	[120]
Stevia rebaudiana leaves	Water	NR	150	NR	1	84.36 ± 4.61	[121]

¹ Total phenolic content; ² gallic acid equivalent; ³ not reported.

3.6. Cold Plasma (CP) Pretreatment

After solid, liquid, and gas, plasma is the fourth state of matter. In general, plasma is classified as thermal and nonthermal. The thermal plasma is produced when a gas is heated and ionized at a high temperature (up to 20,000 K), while nonthermal plasma is generated as a result of an elastic collision of the gas particles, atoms, and electrons by the applied energy. This causes the transfer of kinetic energy to other particles, resulting in the cooling of the uncharged particles and neutral ions, which is faster than the energy transfer to the electrons. As a result, the electrons stay at a higher temperature, while the neutrons, ions, and radicals reach ambient temperature. This enables the gas bulk to stay at a low temperature. Thus, this type of plasma is called nonthermal. This condition makes it possible to treat thermolabile food components. Nonthermal CP is generated by ionization of some process gases (e.g., N₂, O₂, CO₂, or noble gases (He, Ar, or Ne) or their combinations) with a strong electric field under room temperature. The CP comprises reactive chemical species, such as ions, electrons, UV photons, atoms, molecules, and free

radicals. In general, these active agents can decompose covalent bonds and produce many chemical reactions [122]. Figure 9 shows the usage of a dielectric barrier discharge cold plasma apparatus as a pretreatment in the extraction of polyphenols from food by-products.

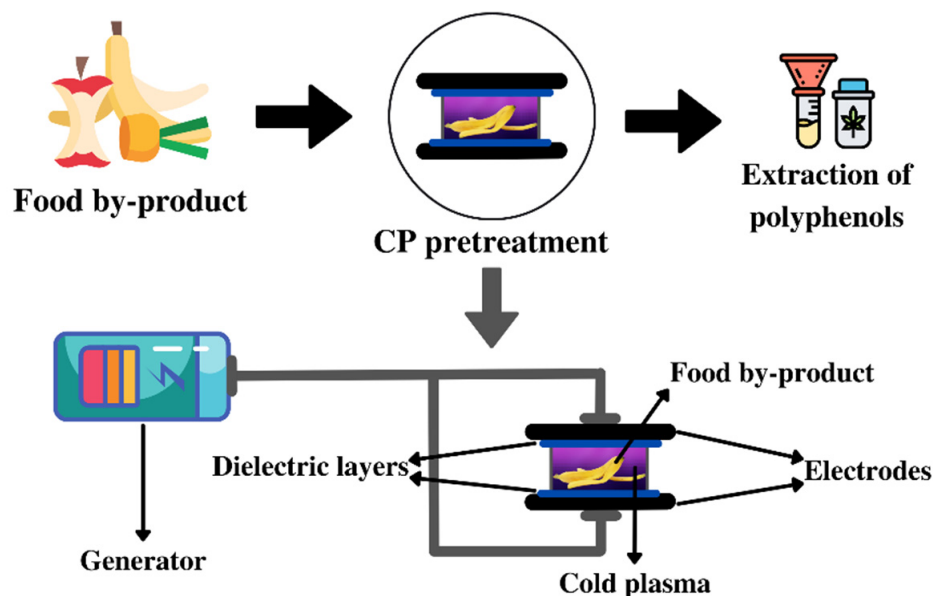


Figure 9. Simplified schematic of extraction of polyphenols using CP pretreatment.

Indeed, CP is another novel method that is used as a pretreatment before the extraction of polyphenols using conventional or novel methods [123]. It is stated in the literature that CP can significantly affect the amount of phenolic compounds. Armini et al. (2016) reported that after the treatment of CP, the phenolic content of green tea leaves was slightly increased [124]. Moreover, Hou et al. (2019) reported that CP increased TPC in blueberry juice as it breaks the covalent bonds and cell membrane [125]. In CP treatment, the source of plasma, time, various active species, power of treatment, and type of food matrix are prominent factors affecting the TPC. As an example, air CP generates many reactive oxygen species, causing oxidation of phenolic compounds [123,125]. Therefore, using a CP source that reduces the amount of reactive oxygen species and increases the extraction rate of phenolic compounds from plants is crucial [123].

Overall, CP has many applications in the food industry, including microbiological decontamination [126] and enzyme inactivation [127]. However, there is only limited research applying CP as a pretreatment before the extraction of polyphenols. Keshavarzi et al. (2020) evaluated the effect of CP pretreatment in the extraction of polyphenols from green tea leaves, and they concluded that the nitrogen dielectric barrier discharge CP (generation power: 15 W, time: 15 min) can increase the TPC of green tea by 41.14% [123]. It has also been reported that the treatment of CP could be used for the extraction of some phenolic compounds such as diosmetin in *Valerianella locusta* leaves [128]. Moreover, Bao et al. (2020) used CP pretreatment for improving the extraction of polyphenols from tomato pomace. They reported that He and N₂ plasmas increased the extraction yield of polyphenols by 10%, and usage of CP pretreatment was able to successfully increase the antioxidant activity of tomato pomace extracts [129]. The same authors used CP to enhance the extractability of polyphenols from grape pomace, and they concluded that it could raise the yield of extraction and improve the antioxidant capacity by a significant extent. These could result from the impact of CP on the disruption of the cell structures, reduction of the water contact angle, and acceleration of drying [130].

4. Control of Polyphenol Oxidase (PPO) Using Mild Technologies

PPO is a copper-containing oxidoreductase enzyme that catalyzes the oxidation of o-diphenols in the presence of oxygen and changes them into o-quinones, which generate dark pigments causing browning in foodstuffs [48,89,131]. Generally, in the plant cell, PPO is positioned in cytoplasmic organelles (e.g., thylakoid membrane of chloroplasts, mitochondria, and peroxisomes), while polyphenols as their substrates are localized in the vacuole and apoplast or cell wall compartment. The enzymatic browning happens after the breakdown of cell structure, and the subsequent interaction between PPO and polyphenols causes color change and a reduction in the antioxidant activity, leading to the deterioration of food nutritional properties [132]. In addition, the reaction between PPO and polyphenols give rise to their oxidation resulting in a decrease in TPC, which has a negative impact in the extraction process of polyphenols from food by-products [21]. Therefore, it is crucial to find an appropriate solution to reduce the drawbacks derived from PPO and prevent the oxidation of polyphenols during the extraction process.

To date, many strategies have been used to control the PPO activity in food matrices, including physical and chemical methods. These methods are utilized to eliminate the critical components for the enzymatic browning reaction, such as copper ion, oxygen, products, substrate (polyphenols), and even the enzyme itself (PPO) [132]. Nowadays, the research on sustainable food processing is concentrated on replacing these conventional anti-PPO treatments with nonthermal techniques [133]. Application of nonthermal technologies in the extraction of polyphenols may have different results for the enzymatic activity because they influence PPO by several factors, including the intensity, duration, mode of exposure, gas composition, electrical input, and degradation of enzymes or substrates [134]. Nonthermal techniques decrease the amount of PPO, resulting in the better maintenance of phenolic compounds [21]. Indeed, the reduction of PPO activity has a direct impact on the maintenance of polyphenols and could increase the recovery yield of polyphenols [28,132,135,136]. Lante et al. (2016) reported that raisins with a low PPO activity have a more quantity of polyphenols and accordingly have a richer nutritional value [137].

As an example, CP treatment may alter the composition of different enzymes [138]. In the CP process, the higher the frequency, the further the anti-PPO activity. The loss of this enzyme in the CP treatment may be due to the activity of free radicals produced in this process on protein bands. Another possible cause for the loss of enzymes is secondary structure modification and amino acid side chain modification of proteins, which is mediated by free radicals from CP [139]. Batista et al. (2020) evaluated the effect of CP on phenolic content and PPO that existed in avocado pulp combined with lime extract, and they reported that it can significantly reduce the activity of PPO and increase the phenolic content. This could be explained by the small reduction in the pH of the pulp due to the addition of lime extract, making it slightly more acidic and lessening the activity of PPO by the complexation of the Cu^{+2} group present in its active site. The combined impact of the pH decline and the extended treatment time and gas flow results in the generation of free radicals interacting with the released enzymes and causing structural changes in them. This leads to the loss of PPO activity. As a result of this reduction, the phenolic content of the food matrix is not used as the substrate of enzymatic browning and could be extracted with better quality [28]. To the best of our knowledge, there is no research evaluating the impact of nonthermal technologies on PPO during the extraction process of polyphenols from food by-products. Nevertheless, the literature on the impact of nonthermal technologies on PPO has proved that they can decrease PPO significantly (Table 6).

Table 6. Effect of different novel technologies on PPO.

Technique	Treated Food/Compound	Treatment Condition	Result	Reference
Flat sweep frequency and pulsed ultrasound	Mushroom PPO	The ultrasound was applied with a frequency that moved up and down within a predetermined range.	Treatment with dual-frequency of 22/40 kHz mode decreased PPO activity significantly.	[140]
Ultrasound	Potato	Power: 540 W, time: 15 min, temperature: 20 °C.	The optimal condition had the highest PPO inhibitory effect.	[141]
Ohmic heating	Water chestnut juice	Voltage: 220 V; electric field strength: 22, 27.5, and 36.7 V/cm; with a titanium electrode.	PPO activity decreased rapidly with ohmic heating treatment at the critical deactivation temperature (35 °C).	[142]
Ohmic heating	Coconut water	Electric field strength: 10 and 20 V/cm, time: 3–15 min.	At 90 °C, PPO activity decreased to about 10% of its initial activity at only 3 min.	[143]
Ultrasound	Spinach juice	Power: 180 W, frequency: 40 kHz, time: 21 min, temperature: 30 °C.	PPO activity decreased by 36%.	[144]
Cold plasma	Cut apple and potato	Time: 10 min, frequency: 2.45 GHz, power: 1.2 kW, gas flow: 20 L/min.	PPO activity was reduced by about 62% and 77% in fresh cut apple and potato tissue, respectively.	[145]
Pulsed electric field	Spinach juice	Electric field strength: 9 kV/cm, frequency: 1 kHz, treatment time: 335 µs.	PPO activity decreased by 44%.	[144]
Ultrasound and pulsed electric field	Spinach juice	Ultrasound was performed before pulsed electric field.	PPO activity decreased by 56%.	[144]
Microwave	Peach puree	Different power densities (4.4, 7.7, and 11.0 W/g) were applied, and cooking value was observed.	The PPO significantly decreased from around 50% to around 5% with increasing the cook value level, regardless of power density applied.	[146]

5. Conclusions

Since the conventional methods for the extraction of polyphenols have many drawbacks, the demand for the application of environmentally friendly techniques is rising significantly. Furthermore, the circular economy approach gives rise to finding an efficient and harmless method for their recovery from food by-products. Not only could this lessen the adverse effect caused by food waste on nature, but also it could save plenty of financial resources for industries. Among all the novel techniques used either as the pretreatment or as the main method of extraction, cold plasma is more ignored by the researchers, and there is not much research on its application in the recovery of polyphenols. In addition, there is no research evaluating the impact of nonthermal technologies on PPO during the extraction process of polyphenols. Therefore, it is imperative to focus on novel methods that can increase the efficacy of the extraction process by decreasing PPO activity. Moreover, it is important to assess and compare the best extraction conditions for maintaining the TPC and antioxidant capacity in the extract.

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