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What Can Be Done to Get More—Extraction of Phenolic Compounds from Plant Materials

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Abstract

Polyphenols are a large group of compounds of natural origin, known for their health-promoting effects on the human body. In plant materials, they can be present in the form of glycosides or aglycones, to a different extent bounded to the sample matrix. Their distribution in different parts of the plant may also vary. The extraction process is one of the most important and difficult stages of sample preparation for the analysis of polyphenol compounds. The main goal is to choose the right extraction method to isolate polyphenols from plant samples with the highest possible efficiency and in unchanged forms. This review summarizes some aspects of different extraction methods for phenolic compounds proposed in the last 5 years. Efforts were made to look critically at each of the described extraction methodologies.

Αŀ

Abbreviations		PLE Pressurized liquid extraction	
Bmim	1-Butyl-3-methylimidazolium cation	SFE	Supercritical fluid extraction
CGA	Chlorogenic acid	SSE	Subcritical solvent extraction
CMC	Critical micellar concentration	SWE	Subcritical water extraction
CP	Cold plasma	UAE	Ultrasound-assisted extraction

DES Deep eutectic solvent

DPPH 2,2-Difenylo-1-pikrylohydrazyl **EAE** Enzyme-assisted extraction **EGCG** Epigallocatechin gallate Folin-Ciocalteu assay FC FΕ Flash extraction

HAE Homogenizer-assisted extraction **HHPE** High hydrostatic pressure extraction **HPSE** High-pressure solvent extraction

HBA Hydrogen-bond acceptor **HBD** Hydrogen-bond donor

ILIonic liquid

MAE Microwave-assisted extraction **MCAE** Mechanochemical-assisted extraction **PEFE** Pulsed electric fields extraction **PHSE** Pressured hot solvent extraction

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Introduction

Phenolic compounds are a large class of plant secondary metabolites with different molecular structures containing one or more aromatic rings with at least one hydroxyl group. They are ubiquitously found in foods and herbal medicines and exhibit various biological activities such as antioxidant, anti-inflammatory, and anticancer properties (Ashwin et al. 2021; Cakır and Güzel 2023; Rathod et al. 2023). The most commonly occurring classes of these compounds are flavonoids and phenolic acids. Increased intake of foods containing phenolics has been claimed to have beneficial effects in preventing cardiovascular, neurological, and liver diseases (Rudrapal et al. 2022; Khan et al. 2021; Zhang et al. 2022). These potent bioactivities render them powerful alternatives for pharmaceutical and medical applications. Due to their versatile benefits concerning human health, research studies focusing on plant-derived phenolics had been intensively performed in recent years (Bitwell et al. 2023; Calderón-Oliver and Ponce-Alquicira 2021; Ponphaiboon et al. 2023; Sridhar et al. 2021; Wiedemair and Zlöbl 2023). Their isolation and recovery from food by-products and wastes have also gained growing interest as they decrease the financial



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and environmental impact (Milczarek et al. 2022; Pagano et al. 2021; Ebrahimi and Lante 2022; Patra et al. 2022). Plant extracts containing a high content of phenolic compounds have found several applications in the food industry as flavors, coloring, antioxidants, preservatives, nutrient enhancers, and packing additives (Awad et al. 2022; Bolouri et al. 2022; Nalhiati et al. 2023; Olszewska et al. 2020).

Phenolic compounds can exist in plant materials such as aglycones or glycosides, free or matrix-bound compounds, and polymerized or monomer structures. Additionally, they are not universally dispensed within the different parts of plants and exhibit varied stability. These difficulties have contributed to challenging extraction processes. Before the extraction processes, several unit operations are performed to increase the yield, and washing, cutting, milling, grinding, and drying are examples of these pretreatment steps (Krakowska-Sierpawska et al. 2023; Ebrahimi et al. 2022; Garcia et al. 2021). These operations can affect the final yield of extraction as sample components can undergo irreversible chemical changes such as hydrolysis, oxidation, or condensation. Drying the plant materials is often suggested before extraction to reduce moisture and degradation of bioactive components. The freeze-drying of sugar beet leaves and gridding under liquid nitrogen reduced polyphenol oxidase activity and retained higher amount of polyphenols (Ebrahimi et al. 2022).

The choice of the appropriate extraction process is of great importance to obtain the highest possible collection of analytes. On the other side, high selectivity and enrichment of a target analyte are necessary for determination of specific compounds. Despite some disadvantages, conventional solid-liquid extraction methods are still the most commonly used due to their simplicity and wide range of applications. Several methods, such as maceration, infusion, decoction, percolation, and digestion, were used to isolate the bioactive phenolics from different parts of plants. Modern extraction alternatives represent ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), subcritical water extraction (SWE), enzyme-assisted extraction (EAE), and pulsed electric fields extraction (PEFE) (Bitwell et al. 2023; Chaves et al. 2020; Mastellone et al. 2021).

The selection of solvent relies on the specific nature of the targeted polyphenols, type of solvent, and its physicochemical characteristics, such as its polarity, viscosity, reactivity and safety concerns, legislature compatibility for food usages, and potential reusability. The solvent boiling temperature should be as low as possible to prevent degradation by heat. Solvents commonly used for plant material extraction are polar (e.g., water, alcohols, acetonitrile), intermediate polar (e.g., acetone, dichloromethane), and nonpolar (e.g., n-hexane, ether, chloroform) solvents. The usage of methanol is often questionable because of its high

toxicity to humans, so ethanol is more attractive since it has similar chemical properties to methanol and is less toxic. As an alternative to toxic and volatile organic solvents, ionic liquids (ILs), deep eutectic solvents (DES), supercritical and subcritical fluids, and enzyme-assisted extraction have been proposed (Chemat et al. 2019; García-Roldán et al. 2023; Essien et al. 2020).

Non-extractable phenolic compounds are still overlooked in several studies and remain in the plant material after the conventional extraction. The polyphenols identified in such extracts, named extractable polyphenols, are often used as the basis or calculations of dietary intake and bioavailability studies. Non-extractable phenolics are high molecular polymeric compounds, or single phenolic compounds associated with macromolecules, mainly polysaccharide constituents of dietary fiber and protein (Carboni Martins et al. 2022; González-Sarrías et al. 2017; Ding et al. 2020). They are not absorbed and reach the colon, where they are extensively fermented by the action of the microbiota, releasing low molecular weight phenolics. To enhance the liberation of bound phenolic compounds, alkaline hydrolysis, enzymatic treatment, or fermentation has been used (Krakowska-Sierpawska et al. 2023; Kornpointner et al. 2022).

Polyphenols extraction efficiency is often calculated as the total phenolic content using the Folin-Ciocalteu (FC) assay. However, FC reagent is not specific only for phenolic compounds, and it could simultaneously oxidize several non-phenolic organic compounds (e.g., sugars) or some inorganic substances, giving elevated apparent phenolic content (Lawag et al. 2023; Pico et al. 2020).

In recent years, several works have been published on polyphenol isolation methods, but they usually focus only on selected aspects of this process. Some of them relate to polyphenol isolation from specific matrices, e.g., tea leaves (Shaukat et al. 2023) or plant material in general Hu et al. (2022), others on chromatographic separation and detection methods (Ponphaiboon et al. 2023). A lot of attention is also paid to the recovery of phenolics from food industry by-products (Pagano et al. 2021; Ebrahimi et al. 2022; Gil-Martín et al. 2022).

This overview discusses the main advantages and limitations of the reported analytical procedures regarding extraction, determination, and separation of polyphenols from different plant materials. The influence of the sample matrix and choosing an appropriate solvent was discussed, as this aspect has not been brought to light thoroughly.

Water and Conventional Organic Solvents

The conventional extraction techniques of polyphenolic compounds apply infusion, decoction, percolation, digestion, and Soxhlet extraction process using the condensed



vapors of the solvent (Abubakar and Haque 2020; Gonfa et al. 2020). Infusions are generally made from the softer parts of plants, including flowers, tops, and leaves, while decoctions are applied to prepare an extract from the hard or woodier parts, such as bark, lichens, hard fruits, larger seeds, and roots. The decoction technique is rather suitable for heat-stable compounds as it involves boiling plant samples for some period of time. These techniques, however, need a large volume of solvents, are time-consuming, and often involve an organic solvent recovery step via an evaporation.

Polyphenols are generally more hydrophilic than lipophilic, and their relative lipophilicity depends on the number of contained hydroxyl groups. Thus, they can be readily extracted by solvents such as methanol, ethanol, acetonitrile, acetone, and ethyl acetate or by their mixtures with water. The presence of water is helpful in enhancing the swelling of plant material, which is favorable to increasing the contact surface area between the plant matrix and a solvent, which increases the extraction yield (Plaskova and Mlcek 2023; Dróżdż and Pyrzynska 2018; Dobros et al. 2022; Herrera-Rocha et al. 2022). According to Nakilcioğlu-Taş and Ötleş, aqueous acetone has been generally good for the extraction of higher molecular weight flavanols, whereas methanol is more effective for lower molecular weight polyphenols (Nakilcioğlu-Taş and Ötleş 2020). The most efficient solvent for the extraction of phenolics from turmeric, torch ginger, and lemongrass turned out to be the one containing 80% acetone, while for the recovery of these compounds from curry leaf, 80% ethanol was the most appropriate (Sepahpour et al. 2018). Solvent containing 80% acetone showed higher values of total phenolics and flavonoids as well as antioxidant activity of Opuntia stricta fruit in comparison to that containing ethanol (El Mannoub 2023).

High processing temperatures should be avoided as many phenolic compounds are subject to degradation or undergo undesirable oxidation. It was found that even for the preparation of a simple tea infusion as a domestic preparation, the type of water used for brewing significantly affects its sensory properties (Franks et al. 2019). Higher efficiency of more bitter catechins was determined using purified bottled water or deionized water, in comparison to using tap water for extraction.

Glycerol, a cheap, non-toxic, and abundant green solvent, can be an alternative in extraction of polyphenols, replacing petroleum-derived solvents (El Mannoub 2023; Kowalska et al. 2021; Eyiz et al. 2020). As opposed to ethanol, glycerol is not flammable and displays low volatility, and like ethanol, it easily mixes with water. In peppermint and nettle leaves, glycerol-water systems (30.5% at 50 °C and 12.5% at 20 °C, respectively) were better extractants than classical solvents such as water or ethanol (Navarro-Baez et al. 2022).

Accelerated Solvent Extraction

High Pressure and Temperature

The extraction technique with common solvents can be accelerated using elevated pressure and temperature, and thereby increasing the extraction efficiency in a shorter time. Pressurized liquid extraction (PLE) involves sample treatment with high pressure (usually up to 600 MPa) at temperatures from 40 to 200 °C for short periods (5-10 min) (Bitwell et al. 2023; Calderón-Oliver and Ponce-Alquicira 2021; Alara et al. 2021; Navarro-Baez et al. 2022). This technique is also known as high hydrostatic pressure extraction (HHPE), high-pressure solvent extraction (HPSE), pressured hot solvent extraction (PHSE), and subcritical solvent extraction (SSE), among others. Aqueous methanol or ethanol and acidic solutions are mainly used in this technique. The effects on plant materials depend on the pressure level (Navarro-Baez et al. 2022; López-Gámez et al. 2021; Grassino et al. 2020). Intensive PLE treatments (> 150 MPa) could cause loss of cell integrity and irreversible damage to plant materials that enhance the extractability of intracellular components. In addition, it inhibits microorganisms and enzymes related to food oxidation. Under mild pressure treatment, the biosynthesis of some metabolites could be activated, as a plant stress response caused by pressure that increases extraction yields (Jacobo-Velázquez et al. 2021).

Despite the disadvantages of PLE, such as low analytes selectivity and the need for costly advanced instrumentation, this technique had been utilized for the extraction of polyphenolic compounds from different plant matrices (López-Gámez et al. 2021; Grassino et al. 2020; Jacobo-Velázquez et al. 2021; Gonçalves et al. 2018; Dobroslavić et al. 2022; Liu et al. 2016; Jin et al. 2023). The PLE conditions applied yielded different breaking matrix-analyte interactions leading to an increase in the number of compounds. Gonçalves et al. evaluated the extraction yield and composition of phenolic compounds from fruit and vegetable residues and have found that the highest phenolic acids content was achieved at high temperatures (200 °C, 50% ethanol), while lower temperatures (40–63 °C) with the same solvent composition were more efficient for extraction of flavonoids (Gonçalves et al. 2018). Low-pressure treatment of Lonicera caerulea berry for a long period of time (400 MPa/20 min) demonstrated higher contents of anthocyanins and phenolics than that with higher pressure for a shorter time (600 MPa/10 min) (Liu et al. 2016).

The PLE technique makes better use of water, a cheap, nontoxic, easily available solvent with tunable selectivity, due to the involvement of high temperature and pressure (Bitwell et al. 2023; Jin et al. 2023; Deans et al. 2020; Essien et al. 2020). As the temperature rises above the water boiling value, its dielectric constant decreases, which allows the



extraction of less polar compounds. Additionally, due to the decrease in the viscosity, better penetration into matrices could be obtained. The pressure of 5 MPa ascertains a liquid water state in the temperature range of 100–250 °C (Jin et al. 2023). This technique, also called subcritical water extraction (SWE), was used for the extraction of phenolics, hydrolysable tannins, and flavonoids from *Phyllanthus tenellus* plant at 121°C (Mohd Jusoh et al. 2019), polyphenolic compounds from *Stevia rebaudiana* leaves at 160 °C (Bursać Kovačević et al. 2018), garlic phenolics (Krstić et al. 2023), and rutin from the aerial parts of common buckwheat at 120 °C (Kim and Lim 2019).

Ultrasounds and Microwaves

To improve solvent penetration within the sample, water or organic solvents are also supported by the action of ultrasounds or microwaves. In ultrasound-assisted extraction, mechanical effect of acoustic cavitation disrupts the plant cell wall and facilitates the release of compounds (Korobijchuk et al. 2023; Venkateswara Rao et al. 2021; Vinatoru et al. 2017; Abi-Khattar et al. 2022). Ultrasonic treatment promotes mass transfer not only by releasing intracellular substances into the extraction medium but also by strengthening the diffusion of the solvent within the cells due to thermal and chemical changes (Bitwell et al. 2023). The

benefits of ultrasound assisted extraction (UAE) are mainly the reduction of extraction time and solvent consumption. However, the formation of free radicals at irradiation higher than 20 kHz may affect the extraction of heat-labile compounds (Calderón-Oliver and Ponce-Alquicira 2021).

Dobros et al. investigated the effect of different extraction techniques—maceration: 1 g of the plant was soaked in 20 mL of 50% EtOH for 30 min at room temperature without mixing; decoction: 1 g of the plant was boiled with 10 mL of water for 30 min; and UAE: 1 g of the plant was sonicated with 20 mL of 50% EtOH for 30 min-on the content of the prominent phenolic compounds (e.g., caffeic and rosmarinic acids, ferulic acid glycoside, morin, coumarin, and its derivative herniarin) from different cultivars of lavender (Dobros et al. 2022). The results are presented in Fig. 1 for one type of Lavendula angustifolia (cultivar Betty's Blue) as an example. Similar contents of caffeic acid were determined in all prepared extracts. However, for other individual phenolic compounds, statistically significant differences were found between the extraction methods. The maceration procedure gave a much lower amount of rosmarinic acid than decoction and application of ultrasound. Using these two techniques, similar results were obtained. The content of ferulic acid glucoside after ultrasound extraction was twice higher than in the case of a decoction or maceration process, while the efficiency of morin extraction

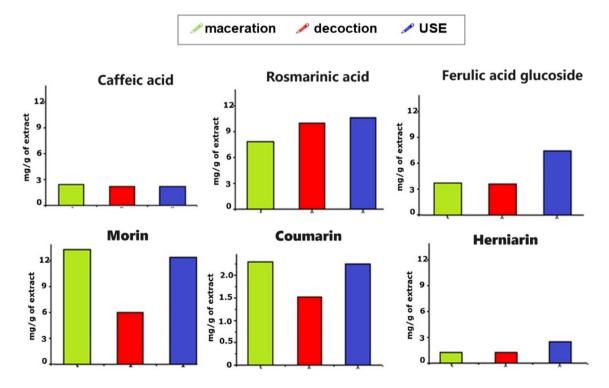


Fig. 1 The content of phenolic compounds and coumarins found in the extracts of lavender plant using water–ethanol (80:20, v/v) for maceration and UAE techniques, and water for decoction. The con-

tent of ferulic acid glucoside was expressed as ferulic acid equivalents (Dobros et al. 2022)



decreased in the order: maceration < UAE < decoction. The efficiencies for coumarin extraction using maceration and UAS were similar and higher than for decoction. On the contrary, herniarin (a methoxy derivative of coumarin) was best extracted with sonication, while decoction and maceration gave similar results. For other cultivars of *Lavendula angustifolia* and *Lavandula* × *intermedia*, similar associations were reported. The presented results indicate that the best extraction method should be chosen considering the specific compounds and not the whole groups.

The use of microwave-assisted extraction (MAE) can also facilitate the phenolics extraction process from various plant materials (Bitwell et al. 2023; Chaves et al. 2020; Kaur Kla et al. 2016; Raghunath et al. 2023; Alchera et al. 2022; Araújo et al. 2020; Rodsamran and Sothornvit 2019). This technique utilizes the direct effect of microwave energy to facilitate the partition of analytes from the sample into the solvent. The synergistic combination of heat and mass transfer, working in the same direction, is the primary cause of the process acceleration and an increase in the extraction yield. The efficiency of MAE depends on several factors, such as solvent properties, sample material, the components being extracted, power, and time of microwave application. Solvents with high dielectric constant, such as water, methanol, and ethanol, rapidly absorb microwave energy resulting in a faster process. Considering solvent affinity to phenolic compounds and its microwave energy absorption capacity, aqueous ethanol up to 70% usually increases extraction efficiency (Calderón-Oliver and Ponce-Alquicira 2021). The MAE technique has been useful for phenolic acids and flavonoids, but tannins and anthocyanins may be degraded due to the high temperature (Calderón-Oliver and Ponce-Alquicira 2021; Pico et al. 2020).

Applications of mathematical and statistical methods to the analysis of chemical data, like experimental design, response surface analysis, and principal component analysis, have been often used for determining the optimum extraction conditions (Sridhar et al. 2021; Tomasi et al. 2023; Nguyen-Kim et al. 2021).

A comparison of the extraction efficiency from *Passiflora* fruit for some flavones (orientin, isoorientin, and isovitoxin) was reported using homogenizer-assisted extraction (HAE), USE, and MAE techniques with 70% ethanol as a solvent (da Silva et al. 2020). As can be seen from Fig. 2, HAE using 5 mL of solvent and the mixer at the rotation of 15.360 rpm for 2 min, gave the highest extraction efficiency for these compounds. Alcántara et al. also showed that conventional extraction with 50% ethanol impacted higher flavonoids from olive leaves and total phenolics from fig leaves, while UAE extracted more carotenoids compared to conventional extraction (Alcántara et al. 2020). Although the benefits of ultrasound-assisted extraction are underlined in several reviews, the results described above show that ultrasound

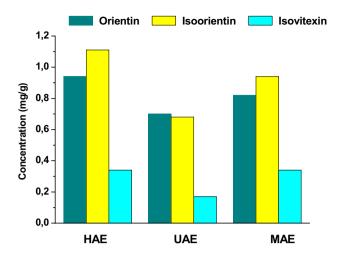


Fig. 2 Comparison of extraction efficiency for flavone compounds from Passiflora fruit samples using different techniques and 50% ethanol as a solvent. HAE, homogenizer-assisted extraction; UEA, ultrasound-assisted extraction; MAE, microwave-assisted extraction (da Silva et al. 2020)

application is not efficient for every plant equally, as well as for the extraction of certain compounds.

Supercritical Fluids

The properties of supercritical fluid are between gases and liquids. This combination of gas-like mass transfer properties and liquid-like solvating power makes supercritical fluid a good candidate for extraction. Water at a temperature above 374 °C and a pressure above 22.1 MPa is considered to be in the supercritical state. However, supercritical water extraction (SWE) was mostly applied to cellulosic and lignin materials (Marcus 2019).

Carbon dioxide is the most widely used in supercritical fluid extraction (SFE) due to its mild critical temperature (31.1 °C) and non-oxidative medium that allows extraction of thermally unstable and easily oxidized compounds. It is also cheap and easy to obtain and lacks solvent residue after extraction. Unlike SWE, where temperature is the critical factor, variations in pressure have a significant effect using CO₂. An increase in pressure increases the fluid diffusion coefficient enabling higher solvent penetration into the samples (Essien et al. 2020; Alara et al. 2021; Uwineza and Waskiewicz 2020). Thus, there is the possibility to choose different pressure-temperature combinations for better extraction conditions. However, low polarity of CO₂ is more suitable for the extraction of non-polar compounds. In the case of polar compounds such as polyphenols, a small percentage of polar co-solvents (methanol, ethanol, acetone, ethyl acetate, and lactate) is added (Alara et al. 2021).

The maximum extraction of phenolic compounds from rosehip flowers was achieved using supercritical CO₂ with



25% ethanol as a co-solvent at a temperature of 60 °C and pressure of 28 MPa (Kasapoĝlu et al. 2023). Da Porto extracted polyphenols from white grape seeds at 40 °C, 8 MPa, and 20% ethanol (Da Porto and Natolino 2017). The results presented by Atwi-Ghaddar et al. showed that while the temperature was an insignificant parameter, a higher percentage of water (up to 20% in ethanol) favored the extraction of catechins from Camellia sinensis leaves (Atwi-Ghaddar et al. 2023). The SFE results were compared with ultrasound-assisted extraction in equivalent conditions such as 30% of modifier and 15 min of extraction time. Three ratios of ethanol to H₂O in the mixtures were explored: 80:20, 50:50, and 0:100. The results presented in Fig. 3 show that different yields were obtained from both techniques; SFE provided higher extraction yields for caffeine, epigallocatechin gallate (EGCG), and total catechins compared with UAE. In addition, SFE affords a lower water content that favors compound stability.

On the other side, in a recent study comparing the efficiency of SFE and HPLE in screening phenolic compounds in sweet cherry stems, supercritical CO_2 fluid (at optimal conditions of 30 MPa, 15% ethanol, 40 °C for 1 h) was found to be less efficient than HPLE (10.3 MPa in 15% ethanol at 176 °C for 20 min) (Nastić et al. 2020). HPLE recovered 37.3% of phenolic acids and flavonoids with different polarities compared to only 4.4% in SFE.

Ionic Liquids

Over the last few years, ionic liquid solvents (ILs) have received great interest as alternatives to traditional organic solvents (Ventura et al. 2017; Gong et al. 2023; Usuki and Yoshizawa-Fujita 2019). They are a class of chemicals with melting points lower than 100 °C and composed entirely of an asymmetric large-size organic cation with an anion of weak coordination properties. The unique physicochemical properties of ILs include very low to negligible vapor pressure, high thermal stability, and conductivity as well as those which can be changed by proper cat-ion-anion combination or by incorporating different functional groups in their structures, such as density, viscosity, hydrophobicity, polarity, and acid-base properties. ILs can interact with bioactive compounds via hydrogen bonding, π - π interactions, ion-dipole, ion-induced dipole, and permanent dipole interactions as well as dispersion forces (Zhang et al. 2021). Many studies have demonstrated the ability of ILs for the extraction of polyphenolic compounds from plant materials, in either simply solid-liquid extraction or MAE and USE (Sentkowska and Pyrzynska 2021; Guo et al. 2019a, 2019b; Dróżdż and Pyrzynska 2019; Ettoumi et al. 2022; Hao et al. 2020; Wang et al. 2019; Mastellone et al. 2020). The recent applications of ILs for the extraction of poly-phenolic compounds from plant materials are shown in Table 1.

According to Zhang et al., promising ILs can be found within five cation-based IL classes such as ammonium, imidazolium, pyridinium, pyrrolidinium, and phosphonium (Zhang et al. 2019). Among them, imidazolium ionic liquids are the most frequently used due to their greater ability to

Fig. 3 Comparison between supercritical fluid extraction (SFE) and ultrasound-assisted extraction (UAE) for green tea leaf compounds (total catechins represent all the catechins including EGCG) (Atwi-Ghaddar et al. 2023)

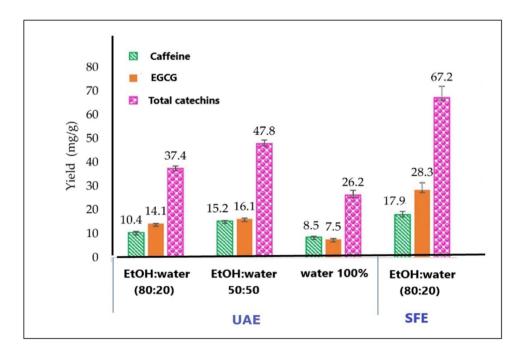




Table 1 Recent applications of ILs for the extraction of phenolic compounds from plant materials

Materials	Analytes	Type of ILs	Extraction conditions	Ref.
Blueberry fruits	Chlorogenic acid	[Bmim]Cl	60 min at room temperature, sonication for 5–20 min	85
Tea	Total phenolic content	[C ₃ mim]FeCl ₄	7 min with microwaves (200 W)	86
Heather flowers	Total phenolic content	[Bmim]Cl	30 min, room temperature	87
Carya cathayensis husk	Total phenolics and flavonoids	$[C_4C_1im]BF_4$	30 °C, ultrasonic power (350 W), for 9 min	88
Glycyrrhiza uralensis	Isoliquiritigenin	[Bmim]Br	60 °C, sonication for 120 min	89
Chrysanthemum spp.	Luteolin, acacetin, chlorogenic acid	[Hmim]BF ₄	65 °C for 30 min	90
Vitis vinifera leaves	Quercetin, rutin, quercetin-3-O-glucoside	$[C_{16}C_4im]Br$	30 min at 70 °C and microwaves (50 W)	91

dissolve cellulose and break the cell wall. Thus, the target components within the cell could be effectively dissolved. Besides the effect on the disruption of plant matrix structure, the aromatic structure of imidazolium cation allows strong interactions with polarizable and aromatic analytes (Ventura et al. 2017).

The type of anion of ionic liquid may also significantly influence the extraction yields (Zhang et al. 2021; Dróżdż and Pyrzynska 2019; Berthod et al. 2018). Usually, small inorganic or organic anions (e.g., tetrafluoroborate, hexafluorophosphate, chloride, and bromide) are combined with organic cations. The 1-butyl-3-methylimidazolium (Bmim) cation with three different types of anions (Cl $^-$, BF $_4^-$, and PF $_6^-$) was studied for extraction from the heather flower (Dróżdż and Pyrzynska 2019). The differences in the total flavonoid content were apparent and increased in the order: [Bmim]PF $_6$ < [Bmim]BF $_4$ < [Bmim]Cl. The results with Cl $^-$ and BF $_4^-$ anions significantly increased with the increase

of ionic liquid concentrations, while those obtained for all studied concentrations of [Bmim]PF₆ were not statistically different. The result using [Bmim]Cl was higher (156.9 \pm 4.1 μ mol/g as catechin equivalent) than reported for 60% ethanol (145.0 \pm 4.5 μ mol/g) and ethyl acetate (144.0 \pm 1.5 μ mol/g) under similar conditions (Dróżdż et al. 2017).

Faster extraction of bioactive compounds using ionic liquids can be achieved through ultrasound-assisted extraction. The mixture of ethanol and water (60:40, v/v) was compared with [Bmim]Cl and [Bmim]BF₄ ionic liquids for the extraction of chlorogenic acid (CGA) from different plant matrices such as green tea leaves, heather flowers, and blueberry fruits (Sentkowska and Pyrzynska 2021). The results are depicted in Fig. 4. Using a 60% EtOH aqueous solution the highest CGA content was obtained in heather and green tea, while [Bmim]Cl ionic liquid proved to be a good solvent for blueberry fruits with statistically insignificant differences (p < 0.05) with prolonged

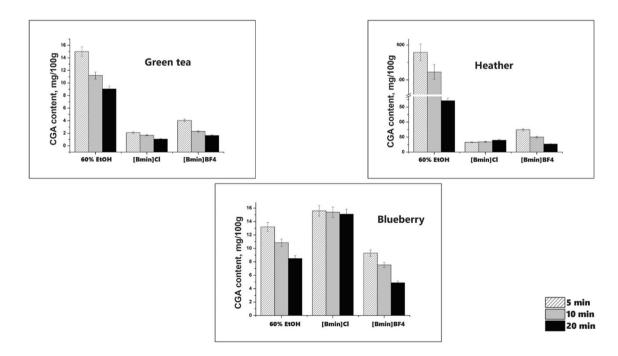


Fig. 4 Effect of sonication time on the content of chlorogenic acid using different extractants (Sentkowska and Pyrzynska 2021)

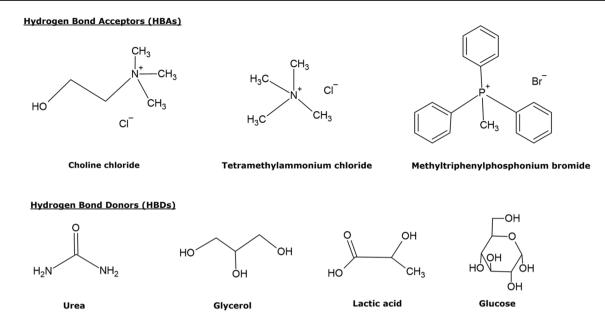
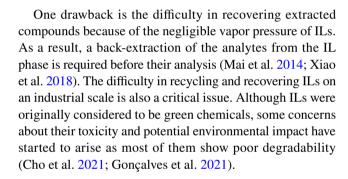


Fig. 5 The chemical structures of common HBAs and HBDs for the composition of DESs

ultrasonic time. However, a significant decrease using 60% EtOH and [Bmim]BF₄] for these samples was recorded. HPLC analysis showed that CGA under these conditions was degraded to caffeic acid (Nastić et al. 2020).

Guo et al. compared the extraction efficiency for green tea polyphenols using magnetic $[C_3mim]FeCl_4$ ionic liquid assisted with MAE and USE [Guo et al. 2019a, 2019b]. The highest value of total phenolic in microwave-assisted mode was obtained (159.8 mg/g) when the solid to liquid ratio was 1:20, microwave power was 200 W, and extraction time was 7 min. Compared with ultrasonic-assisted extraction (185.4 mg/g), its extraction efficiency was lower, but extraction time was shortened significantly from 300 to 7 min.

It should be mentioned that ionic liquid derivates, like IL-based surfactants, are capable of forming micellar aggregates when dissolved in water above their critical micellar concentration (CMC); thus, they present the inherent properties of ILs but with improved solvation properties due to the formation of micelles. CMC values are lower than conventional surfactants with similar structures and small amounts can be used to take advantage of their surface-active properties (El Seoud et al. 2021). For the extraction of flavonoids from several samples of *Passiflora* sp. and *Mangifera* sp. leaves, only 525 µL of the low cytotoxic decylguanidinium chloride solution at 930 mM concentration and microwave irradiation at 30 °C and 50 W for 10.5 min was required (Hao et al. 2020). Considering this interesting set of characteristics, the applications of IL-based surfactants in analytical extraction strategies have been proposed (Mastellone et al. 2020; Moučková et al. 2020).



Deep Eutectic Solvents

An alternative to ILs is using deep eutectic solvents (DESs). DESs are usually obtained by mixing a hydrogen-bond acceptor (HBA) and a hydrogen-bond donor (HBD) molecules in specific molar ratios to form a homogenous liquid (Martin et al. 2019; Huang et al. 2019; Serna-Vázquez et al. 2021; Mannu et al. 2021; Tang et al. 2021; Florindo et al. 2019). For their preparation elevated temperature (40–60 °C), freeze-drying, and gridding methods are mostly used (Huang et al. 2019; Meneses et al. 2019). DESs are characterized by a much lower melting point than the individual components. A small amount of water in these solvents is involved in the formation of a network of hydrogen bonds. A large number of compounds have been used to prepare deep eutectic solvents. At this time, the most common DESs are based on cholinium, phosphonium, or tetraalkylammonium halide salts mixed with carboxylic acid, urea, ethylene glycol, or carbohydrates of different types. The examples of the



common HBA and HBD molecules for the composition of DESs are presented in Fig. 5.

The majority of DESs that have been prepared and studied so far are classified in different ways. They are typically classified based on the nature of the HBD used as a combination of an ammonium or phosphonium salt with a metal chloride (type I), with a metal chloride hydrate (type II), and with alcohols, amides, or carboxylic or amino acids (type III). Type IV combines all the deep eutectics composed of only non-ionic, molecular HBAs and HBDs (Huang et al. 2019; Serna-Vázquez et al. 2021; Ijardar et al. 2022). In addition, there have been some debatable information appearing in the literature concerning the new classification for distinguishing different types of DESs as more non-ionic (hydrophobic) compounds were used as HBA in the synthesis of new DESs (Florindo et al. 2019; Cao and Su 2021; van Osch et al. 2020). Thus, the classical DESs containing choline chloride and urea (1:2) can be considered ionic (hydrophilic) DESs due to the presence of ionic HBA. Some special branches have also attracted significant attention and a new subclass of DESs has been proposed called natural deep eutectic solvents (NADESs). They are composed of two or more compounds that are generally plant-based primary metabolites such as sugars, alcohols, carboxylic acids, and amino acids (Cao and Su 2021; van Osch et al. 2020). Regardless of the type, the prevailing opinions hold that DESs are formed through self-associated intermolecular interactions, most likely caused by van der Waals interactions, hydrogen bonding, and/or ionic bonding.

Alhadid et al. proposed a simple approach for selecting possible substances sharing the same functionality and melting temperature based on their melting entropy and enthalpy (Alhadid et al. 2020). It was demonstrated for six hydrophobic eutectic systems composed of L-menthol and monocarboxylic acids with linear and cyclic structures. For similar melting temperatures, the melting enthalpy of cyclic acids with more rigid structures could form deeper eutectics.

Deep eutectic solvents have similar physical properties to ionic liquids, and they are practically non-volatile and non-flammable. In addition, they are cheaper and usually much easier to synthesize, and they are less toxic and often biodegradable (Lomba et al. 2021). Additionally, hydrogen bonding between DESs and phenolic compounds prevents their oxidative degradation DESs could stabilize them and prevent oxidative degradation (Lomba et al. 2021; Barbieri et al. 2020; Zannou et al. 2020; Dai et al. 2016).

DESs have been extensively studied for the extraction of polyphenolic compounds from different plants and food-processing waste (Mansur et al. 2019; Oomen et al. 2020; Wojciechowski et al. 2021; Vo et al. 2023; Zhang et al. 2022; Guo et al. 2019a, 2019b; Alrugaibah et al. 2021; Panic et al. 2019). Mansur et al. investigated the efficiency of 18 different choline chloride-based DESs on the extraction of

flavonoids from buckwheat sprouts coupled with the UAE technique (Mansur et al. 2019). ChCl/triethylene glycol with 20% (v/v) of water extracted significantly higher amounts of flavonoids than other DESs. The extracted flavonoids were recovered from DES extracts (yields >97%) by using solid-phase extraction with C18 microcolumn.

Several DESs were evaluated to extract phenolic compounds from rosemary (Rosmarinus officinalis L.) leaves by heating at 30 °C for 15 min (Vo et al. 2023). The extractants were prepared using choline chloride as the HBA and various HBD such as acetic acid, lactic acid, oxalic acid, 1,2-propanediol, ethylene glycol, glycerol, xylitol, sorbitol, xylose, and zinc chloride. For pure DESs, phenolic content was found only in four DESs, namely those composed of acetic and lactic acids, ethylene glycol, and 1,2-propanediol allowed the successful extraction. The addition of water (30%) allowed the use of all DESs with higher extraction yields than 70% ethanol but significantly lower extraction yields than pure ethanol. The best extraction conditions were obtained for ChCl/1,2-propanediol composition at 65 °C, with a 50% aqueous DES, resulting in the highest total phenolic yield of 78 mg GAE/g (Zannou et al. 2020).

Guo et al. found that efficiency for extraction of anthocyanins from mulberry utilising NADES with high-speed homogenization and cavitation-burst extraction decreased in the following order: Ch/citric acid, Ch/tartaric acid > Ch/lactic acid > Ch/glycerol > Ch/xylitol > Ch/mannitol > Ch/glucose > Ch/fructose, thus DESs with organic acids as the HBDs than those with sugars or sugar alcohols (polyols) (Guo et al. 2019a, 2019b). Among mixtures containing three components, the NADES comprising ChCl/citric acid/ glucose (with 30% of water) showed the best extraction efficiency of anthocyanins as compared to traditional ethanol/ water mixture. Similarly, Panic et al. extracted anthocyanins from grape pomace on a larger scale using ChCl/citric acid composition with 30% of water under simultaneous ultrasound/microwave-assisted extraction (MW power at 300 W, US power 50 W), for 10 min (Panic et al. 2019).

The recent applications of DESs for the extraction of polyphenolic compounds from plant materials are presented in Table 2.

Enzymes

An interesting approach that allows the release of polyphenolics, particularly those localized intracellularly or bound with components of cell walls, is the use of enzymes for the destruction of cell wall integrity (Bitwell et al. 2023; Nadar et al. 2018; Krakowska-Sieprawska et al. 2020) and facilitates the diffusion of phenolics from plant cells to the solvent. Enzymes, such as cellulase, glucanase, β -glucosidase, and pectinase, are mainly used for these purposes. The



Table 2 Recent applications of DESs for the extraction of polyphenolic compounds from plant materials

Materials	Analytes	Type of DESs	Extraction conditions	Ref.
Buckwheat (Fagopyrum esculentum Möench) sprouts	Rutin, vitexin, isovitexin, orientin, isoorientin	Choline chloride/triethylene glycol	40 min at 57 °C, ultrasounds (700 W)	Mansur et al. 2019
Baikal skullcap (Scutellaria baicalensis) bark	Baicalein, scutellarin, wogonin, oroxylin, and their glycosides	Proline/citric acid (1:1)	Stirring at 50 °C	Oomen et al. 2020
Rosemary (Rosmarinus officinalis L.) leaves	Total phenolics	Choline chloride/triethylene glycol (1:2)	150 min at 120 °C	Pontillo et al. 2021
Mangosteen (Garcinia mangostana L.) rinds	Total phenolic and flavo- noid contents	Lactic acid/1,2-propanediol	9 min at 57.5 °C, ultrasounds (300 W)	Vo et al. 2023
Siberian ginseng (Acantho- panax senticosus)	Total flavonoids	Lactic acid/glycerol (1:1)	73 min at 55 °C, ultrasounds (500 W)	Zhang et al. 2022
Mulberry (Fructus mori) leaves	Total anthocyanins	Choline chloride/citric acid/ glucose (1:1:1)	30 min, at 45 °C and negative pressure (-0.08 MPa)	Guo et al. 2019a, 2019b
Grape pomace (Vitis vinif- era cv. Plavac mali)	Total anthocyanins	Choline chloride: citric acid (1:1)	Simultaneous ultrasounds (50 W) and microwaves (300 W) for 10 min	Panic et al. 2019

effectiveness of enzyme-assisted extraction (EAE) depends largely on the type of enzyme or enzyme mixture used as well as the selection of optimal conditions for their operation (pH, ionic strength, temperature). In particular, extraction temperature plays an important factor since the enzyme activity may decrease at low or high temperatures (Calderón-Oliver and Ponce-Alquicira 2021).

Pretreatment of plant material of interest after the enzymes detach the phytochemicals from plant material is followed by other extraction methods. For example, flavonoids were obtained from pomelo peels by first treating the peels with 4.5% pectinase and then extraction of these phytochemicals was done using the USE method at a reduced optimal temperature of 30 °C (Anh et al. 2021). Similarly, pretreating of rosemary leaves with pectinolytic enzymes for 1 h before a 24-h conventional extraction with 50% ethanol was provided to obtain an extract with higher radical scavenging activity evaluated by DPPH assay than that without the enzyme pretreatment (Pontillo et al. 2021). It also led to increased extractability of phenolic compounds from olive pomace followed by MAE (Macedo et al. 2021). Using EAE, non-extractable polyphenols have been also recovered at relatively low temperatures from sweet cherry fruits (Domínguez-Rodríguez et al. 2021), citrus peels (Zarate Vilet et al. 2020), corn cob (Fernandez-Aulis et al. 2019), and green yerba mate (Heemann et al. 2019).

The comparison of three extraction techniques—maceration, supercritical fluid extraction (both for 120 min at 50 °C with 96% ethanol), and combination of enzymatic pretreatment with SFC, under the same conditions with the addition of 2.9% of Kenzyme® (commercially available enzyme mixture) and incubated at 45 °C for 90 min—was recently reported for evaluation of their efficiency to release

polyphenolic compounds from yerba mate leaves (Krakowska-Sieprawska et al. 2020). The extracts from yerba mate macerated with 96% ethanol had a much higher content of total phenolic compounds (135.5 mg GA/g DM) in comparison to the SFE technique (51.95 mg GA/g DM). The chromatographic analysis performed using HPLC-MS/MS of all studied extracts showed that SFE method combined with enzymatic degradation used allowed for a significant increase in the content of quercetin, caffeic acid, chlorogenic acid, and 4-hydroxybenzoic acid (4-HBA), in comparison with maceration and SFE extracts, which is shown in Figs. 5 and 6. A significant increase in rutin and chlorogenic acid was also observed for yellow lupine plant after enzymatic treatment (Krakowska-Sieprawska et al. 2020).

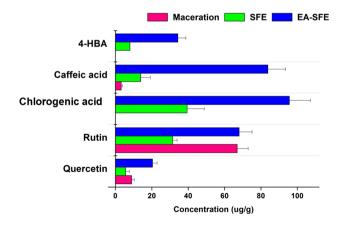


Fig. 6 Concentration of the main polyphenolic compounds extracted from yerba mate using different techniques (Krakowska-Sieprawska et al. 2020). Chlorogenic and 4-hydroxybenzoic (4-HBA) acids were not detected in macerated extracts



It is worth mentioning that the changes in the structure of the evaluated plant materials and the functional aspects of the quality of the extracts after enzymatic hydrolysis were rarely studied. Rafińska et al. found that enzymes not only acted on a plant surface but efficiently penetrated inside the fragments of tissue (Rafińska et al. 2022). Pectinase turned out to be particularly efficient in obtaining high-quality *Medicago sativa* extract with a low content of interfering compounds such as reducing sugars or short chains of polysaccharides.

Other Innovative Extraction Techniques

Various novel thermal and non-thermal extraction techniques have been recently investigated. These advanced techniques are more efficient in comparison to methods used so far in terms of solvent consumption, extraction time, and energy cost (Zia et al. 2022). Thus, these novel methodologies present the principles of green chemistry, as their application provides a positive environmental and social impact. They are particularly attractive to the food and pharmaceutical industries (Belwal et al. 2020).

Pulsed electric field (PEF) is used as a pretreatment technique to improve the extraction and preservation of bioactive compounds followed by a subsequent traditional or novel extraction step (Ranjha et al. 2021; Bocker and Silva 2022). PEF uses short and intense pulses of electricity to create pores in the cell membranes of plant tissues, allowing the release of intracellular substances and enhancing the mass transfer of solvents. In addition to increasing the yield of the extraction process, the PEF technique promotes minimal changes to the sensory and nutritional properties of the products as it is associated with the low temperatures used during the process (30-40 °C). It also inactivates microorganisms and enzymes that cause spoilage and degradation of food quality. There is increasing interest in the preparation of extracts using PEF from plant material, such as the extraction of polyphenols from onion bulbs (Liu et al. 2018a, 2018b), aromatic plant leaves (Carpentieri et al. 2022), cinnamon (Pashazadeh et al. 2020), and herbs (Athanasiadis et al. 2022).

Cold plasma (CP) is a new technology with promising potential to be used (López-Gámez et al. 2021; Pogorzelska-Nowicka et al. 2021; Kumar et al. 2023). The cellular damages and surface modifications of plant materials caused by plasma increase the extractability of hydrophilic compounds, which can facilitate phenolic recovery. Bao et al. investigated the effect of different gases (air, argon, helium, and nitrogen) on phenolic extraction from CP-treated tomato pomace (60 kV for 15 min) (Bao et al. 2022). It was found that plasma composition was more relevant than treatment time for extracting phenolic

content, and helium and nitrogen caused the most intensive disruption of cells. Application of CP increased the amount of caffeic acid for argon, helium, and nitrogen as well as for chlorogenic acid by argon, helium, and nitrogen plasmas. However, the concentrations of gallic acid, rutin, isoquercetin, and ferulic acid remained similar to untreated samples. Hou et al. mentioned the importance of CP treatment exposure time since the antioxidant potential of blueberry juice declined when the operation time was extended from 2 to 6 min (Hou et al. 2019). Cold plasma treatment has also been applied to improve the extraction of phenolics from green tea leaves (Keshavarzi et al. 2020) and apple cubes (Farias et al. 2020), as well as to improve the shelf life of food (Heydari et al. 2023; Subrahmanyam et al. 2023).

Methodologies, such as flash extraction (FE) and mechanochemical-assisted extraction (MCAE), could also be mentioned among the innovative techniques for the extraction of phenolic compounds. The high efficiency achieved by FC is mainly due to the ultrahigh-speed rotation of the specially designed cutter head and other effects caused by this, such as very rapid dynamic molecular permeation, drastic stir, and strong vibration (Qin and Xi 2022; Fan et al. 2022). It is not only able to achieve high yields in a very short time (about 1 min) with a simple operation but also possesses better industrial acceptability. Ethanol and methanol, and their mixtures with water are mostly used as solvents (Mo et al. 2020). MCAE applies high-energy mechanical force acting on the raw materials to enhance the release of intracellular molecules and dissociate the target components from natural products in aqueous media avoiding or considerably reducing the organic solvent consumption (Fan et al. 2022). Moreover, this green pretreatment method is performed under relatively low temperatures; it can reduce or avoid the loss of thermolabile compounds. Chemat et al. summarized the application of MCAE for the extraction of bioactive compounds, including phenolics (Chemat et al. 2020).

Conclusions

Due to their properties, phenolic compounds are widely used in the food, pharmaceutical, and cosmetics industries. The classic extraction methods such as maceration, Soxhlet extraction, and percolation still dominate, particularly on an industrial scale. Novel methods that apply ultrasounds, microwaves, supercritical fluids, or enzymes are becoming more popular because they allow the reduction of solvents and extraction time, as well as guarantee less thermal degradation of phenolic compounds. However, they also have some challenges as operational costs limit their wide applications.



The choice of extraction technique hinges on the plant matrix, targeted compounds, economic viability, and environmental impacts. Due to the importance of phenolic compounds, it should be expected that searching for innovative procedures for their extraction will be intensively developed in the coming years to achieve appreciable recovery yields from the plant materials.

In some cases, the combination of different extraction methods can be applied as a single extraction technique to obtain high phenolic yield from the source material. The simultaneous use of high-pressure extractions and hydrolysis with enzymatic assistance, as well as the combination of ultrasound and microwave, may promote the development of efficient technical and economic systems. Cold extraction techniques are also a viable alternative for the phenolics extraction from natural sources, preferable regarding the extraction of thermolabile compounds. That may overcome the disadvantages of individual techniques and provide benefits specifically related to specific compounds, matrices, or applications. The combination of novel extraction technologies to achieve synergistic effects, minimal degradation of active components, and enhanced extraction yields will probably be a topic in future research.

Declarations

Conflict of Interest The authors declare no competing interests.

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