Extraction Methods of Major Phytochemicals

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Contributor: Ashwani Kumar , Nirmal P , Mukul Kumar , Anina Jose , Vidisha Tomer , Emel Oz , Charalampos Proestos , Maomao Zeng , Tahra Elobeid , Sneha K , Fatih Oz

Scientific studies have established a relationship between the consumption of phytochemicals such as carotenoids, polyphenols, isoprenoids, phytosterols, saponins, dietary fibers, polysaccharides, etc., with health benefits such as prevention of diabetes, obesity, cancer, cardiovascular diseases, etc. This has led to the popularization of phytochemicals. Foods containing phytochemicals as a constituent (functional foods) and the concentrated form of phytochemicals (nutraceuticals) are used as a preventive measure or cure for many diseases. The health benefits of these phytochemicals depend on their purity and structural stability. The yield, purity, and structural stability of extracted phytochemicals depend on the matrix in which the phytochemical is present, the method of extraction, the solvent used, the temperature, and the time of extraction.

phytochemicals bioactive compounds extraction methods solvents Functional foods

1. Introduction

Phytochemicals are plant-based bioactive compounds produced by plants for their protection. They can be derived from various sources such as whole grains, fruits, vegetables, nuts, and herbs, and more than a thousand phytochemicals have been discovered to date. Some of the significant phytochemicals are carotenoids, polyphenols, isoprenoids, phytosterols, saponins, dietary fibers, and certain polysaccharides. These phytochemicals possess strong antioxidant activities and exhibit antimicrobial, antidiarrheal, anthelmintic, antiallergic, antispasmodic, and antiviral activities [1][2]. They also help to regulate gene transcription, enhance gap junction communication, improve immunity, and provide protection against lung and prostate cancers [3][4][5][6][7]. The recent focus on translational research has enhanced the dimensions of functional foods. Phytochemicals, after extraction from various sources, find profound application in the development of functional foods and nutraceuticals. Phytochemicals exhibit variations in their affinity for solvents and tolerance to heat. The selection of the solvent also affects the quality of the recovered phytochemical and its application in the development of food and nutraceutical products. The solvents can be divided into green solvents [water, ethanol, glycerol, fatty acids/oils, acetic acid, ionic liquids, carbon dioxide (CO_2), deep eutectic solvents and natural deep eutectic solvents (NADES), etc.] and other solvents such as acetone, chloroform, butanol, methanol, ethyl acetate, methyl acetate, benzene, hexane, cyclohexane, etc. [8]. Loss in functional properties can occur with the use of non-compatible solvents and varied exposure to different temperatures. Additionally, extraction efficiency depends upon the matrix in which the phytochemical is present. Several matrix-related characteristics, such as matrix type, structure, pre-treatment, particle size, and solid–liquid ratios influence the extraction efficiencies of phytochemicals and extraction techniques [9]. To ensure quality products, phytochemicals must be extracted from the source crop in

a manner that retains their natural structure and properties. Hence, it is imperative to select a suitable method of phytochemical extraction. Some of the widely used conventional methods are maceration, percolation, decoction, reflux extraction, and Soxhlet extraction, and the novel methods are pressurized liquid extraction (PLE), high hydrostatic pressure extraction (HHP), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), pulsed electric field extraction (PEF), vibro-cavitation extraction, extraction under vacuum-oscillating boiling conditions, extractions in mills, extraction in rotary-pulsation apparatus (RPE), liquid gas extraction (LGS), enzyme-assisted extraction (EAE), supercritical fluid extraction (SFE), and natural deep eutectic solvent extraction (NADES) [8][10].

2. Overview of Major Phytochemicals and Related Health Benefits

The type and concentration of phytochemicals in the source crop vary according to intrinsic and extrinsic factors such as crop type, variety, soil, and environment (region, altitude, and season) of cultivation. This section discusses major phytochemicals, their characteristics, and associated health benefits. A detailed list of major phytochemicals, their sources, and their health benefits is also provided in **Table 1**.

Table 1. Major phytochemicals, their sources, active sites, and the related health benefits.

Phytochemical	Sources	Active Site	Health Benefits	References
Carotenoids				
α-carotene	Mango, pear, peach, pumpkin, butternut squash, green bean, okra, avocado, chard, collard greens, tangerine, banana	Pulp of mango, tangerine, avocado, butternut squash, and pumpkin; the green part of okra, chard, collard greens	Regulates gene transcription, protects against lung and prostate cancer, good for eye health	[7][11][12]
β-carotene	Red pepper, carrot, spinach, peaches, brussel sprout, grapefruit, sour cherries, papaya, mango, romaine lettuce	Green parts of plants, flowers, roots, and stems of plants; pulp of mango, grapefruit, papaya, etc.	Enhancement of gap junction communication, enhances immunity	[6][13][14]

Phytochemical	Sources	Active Site	Health Benefits	References
Lutein	Asparagus, spinach, kale, green beans, orange pepper, lettuce, broccoli, parsley, pistachio nuts	Leaves of spinach, lettuce, parsley; flower part of broccoli; essential oil of pepper; middle lamella of nuts	Improves immunity, good for eye health	[15]
Lycopene	Tomato, sweet potato, pink grapefruit, pink guava, watermelon, apricot, papaya, rosehip	Skin and pulp of tomato, grapefruit, watermelon, apricot, guava	Improves eyesight, reduces pain, and strengthens bones	[16][17]
Xanthophylls	Pumpkin, papaya, pepper, mushroom	Young leaves of papaya, pumpkin; essential oil of pepper	Antioxidant properties, boosts eye health and blood flow	[18][19]
Cryptoxanthin	Apricot, papaya, peach, cashew apples, seabuckthorn, mandarin, tangerine, lemon	Skin and pulp of cashew apple and citrus fruits	Maintains pulmonary health, prevents arthritis and inflammation; improves immune response	[20][21]
Fucoxanthin	Brown seaweeds, Bacillariophyta, Chromophyta, Macroalgae, Microalgae	Chloroplasts of brown seaweeds	Antioxidant, anti-inflammatory, antihypertensive, anticancerous, antidiabetic, antiobesity and radioprotective properties	[10][22]
Polyphenols				

Phytochemical	Sources	Active Site	Health Benefits	References
Flavones	Parsley, oregano, rosemary, green olive, pumpkin, watermelon, bell pepper, honey, fava beans, chickpea, field pea	Essential oils of spices, pulp of watermelon and pumpkin	Action against free radicals, protective effects against cardiovascular diseases, cancers, and other age-related diseases	[23][24]
Flavanones	Grapefruit, pumelo, mandarin, lemon	Pulp of citrus fruits	Protective effects against cardiovascular diseases, prevention of inflammation and allergies	[25][26]
Flavanols	Chocolate, tea, grapes	Green and black tea leaves	Action against free radicals, prevention of inflammation and allergies	[27][28]
Anthocyanidins and anthocyanins	Blueberry, cranberry, pomegranate, red grapes, black soybean, purple corn, red cabbage, raspberry	Flesh of berries, skin of grapes, corn fiber	Protective effects against cardiovascular diseases, prevention of inflammation and allergies	[26][29]
Polyphenol amides	Oats, chili, pepper	Capsaicinoids in chili pepper, avenanthramides in oats	Prevention of inflammation and allergies	[23][30]
Isoprenoids				
Limonene	Lemon, lime, orange	Oil of orange	Anti-inflammatory, antioxidant, and anti-stress properties, as well as a	[31][32][33]

Phytochemical	Sources	Active Site	Health Benefits	References
			neuroprotective role in Alzheimer's disease	
Myrcene	Mango, guava, thyme, parsley, bay leaves, lemongrass, cardamom, sweet basil, juniper	Essential oil extract of lemongrass, juniper, cardamom	Anxiolytic, antioxidant, anti-aging, anti-inflammatory, and analgesic properties	[34]
Pinene	Cannabis, turpentine tree, ironwort, sage plant	Oil of cannabis, ironwort, and sage plants	Antibacterial, antitumor, anti-inflammatory, and sedative properties	[35][36]
Phytosterols				
Campesterol	Banana, pomegranate, pepper, coffee, grapefruit, cucumber, onion, oat, potato, lemongrass	Pulp of bananas, pomegranate, grapefruit; essential oil of pepper, lemongrass, etc.	Used in the treatment of allergy, asthma, psoriasis, rheumatoid arthritis, chronic fatigue syndrome, migraine, and menstrual disorders	[37][38][39]
Sitosterol	Avocado, hazelnut, walnut, soybean, olive, canola	Oil of hazelnut, walnut, olive, canola, soybean	Used in the treatment of an enlarged bladder; reduces the risk of cardiovascular disease, promotes anti-cancer properties	[28][37][38]
Stigmasterol	Soybean, calabar bean, and rapeseed	Oil of soybean, calabar bean, and rapeseed	Has a protective effect against gastric and duodenal ulcers, neurological disorders	[37][38][40]

Phytochemical	Sources	Active Site	Health Benefits	References
Campestanol	Soybean, olive, hazelnut, flax, cashew	Oil of soybean, olive, hazelnut, flax, and cashew	Prostate health, hair growth, reduce LDL cholesterol	[37][38][41]
Sitostanol	Pepper, banana, pomegranate, soybean, olive	Oil of pepper, soybean, and olive; pulps of banana and pomegranate	Reduces chance of heart attack and stroke, improves hair growth	[37][38][42]
Stigmastanol	Hazelnut, olive, corn	Oil of hazelnut and olive, as well as corn fiber	Reduces chance of heart attack and stroke, antioxidant activity	[37][38][43]
Saponins				
Dammarane	Black gram, garden pea, pigeon pea	Middle lamella of peas and legumes	Exhibits hypoglycemic, virucidal, and antifungal activity	[44][45]
Tirucallane	Sunflower, almond, walnut	Oil of almond, sunflower, and walnut	Has an effect on the transverse tubular system and sarcoplasmic reticulum at lower concentration (10µg/mL), has an effect on skin inflammation and diarrhea	[46][47]
Oleanane	Common bean, black gram, almond	Middle lamella of legumes and oil of almond	Antimicrobial and hypolipidemic activities; aids in the treatment of chronic diseases	[48]
Dietary fiber				

Phytochemical	Sources	Active Site	Health Benefits	References
Pectin	Apples, apricots, cherries, oranges, carrots, citrus fruits, rose hip	Peels of citrus fruits, middle lamella of cell walls of fruits	Lowers LDL cholesterol; cures diarrhea; promotes the generation of peripheral regulatory T cells	[49][50][51]
Cellulose	Rice, wheat, sisal, jute, hemp, corn, flasks	Rice husk, wheat straw, kernels of corn	Improves insulin sensitivity, gut microbial viability and diversity; reduces the level of bad cholesterol; reduces free radical damage to cells	[52][53][54]
Lignin	Flaxseeds, parsley, carrots, horseradish), Wheat, tomatoes, berries, broccoli, cabbage, green beans, peaches, peas, Brazil nuts, apples	Seeds of tomatoes and berries, stems of cabbage and broccoli, bran of wheat	Lowers the risk of cancer, reduces hot flashes in postmenopausal women, protects from cardiovascular diseases	[55][56][57]
Hemicelluloses	Rice, wheat, nuts, legumes, whole grains	Bran of rice and wheat, middle lamella of legumes, nuts	Improves metabolites from gut microflora; reduces cardiovascular risk	[58][59]
Polysaccharides				
Amylose	Corn, rice, quinoa, potato, oats, arrowroot	Starchy endosperm of corn, rice, potato, and oats;	Cures immunodeficiency, cancer, inflammation, hypertension, hyperlipidemia	[60]

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Phytochemical	Sources	Active Site	Health Benefits	References
		powder of arrowroot		
Amylopectin	White potato, rice, oats, corn	Starchy endosperm of rice, white potato, oats, and corn	Improves intestinal health and increases gut microbiota	[60][61]
Resistant starch	Buckwheat, oats, lentils, peas, beans	Starchy endosperm of oats, buckwheat, and lentils	Cures hypercholesterolemia and obesity; improves gut microbiota	[62]
Arabinoxylan	Rice, barley, guar gum, wheat, finger millet	Starchy endosperm of rice, barley, wheat, and finger millet	Improves gastrointestinal health; reduces diabetics, cancer, and obesity	[63][64][65]

3. Phytochemical Extraction Methods

Extraction is defined as a process of removing or obtaining the desired compounds from the source material [66]. The solvents used for the extraction of phytochemicals can be divided into green solvents such as water, ethanol, glycerol, fatty oils, ionic liquids, acetic acid, isopropanol, supercritical CO_2, deep eutectic solvents, natural deep eutectic solvents, etc., and other organic solvents such as acetone, chloroform, butanol, methanol, ethyl acetate, methyl acetate, benzene, hexane, cyclohexane, etc. The green solvents are termed so due to their nontoxic, biodegradable, recyclable, and renewable nature. These solvents also have a high flash point. Among the green solvents, water is the most used and universal solvent. It is a non-selective solvent and can separate all the hydrophilic substances such as saponins, phenolics, polysaccharides, etc. The extraction efficiency of water can be enhanced by superheating, as superheating decreases the dielectric constant of water and provides better penetration. Superheated water is also a better solvent for the extraction of lipophilic substances such as essential oils as the polarity of water decreases significantly between 100–374 °C. Ethanol is selective in action and is used for the extraction of polyphenols and triterpenes. The extraction efficiencies of ethanol can be modified by using water and acid. Glycerol has high thermal stability (boiling point 290 °C). It is too viscous at low temperatures and hence has a low solubility. It can be used as an extraction solvent above 60 °C or with other co-solvents. Glycerol is not a good solvent for hydrophobic compounds such as fatty acids and oils but is a selective solvent for polyphenolic extraction. Fatty oils are a good solvent for the extraction of hydrophobic substances and can be used for the extraction of carotenoids, coumarins, tocopherols, flavonoids, etc. The major oils used for extraction are soybean, almond, olive, sunflower, etc. Acetic acid buffer can be used for the extraction of phenolics and

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anthocyanins. Isopropanol is a green alternative to n-hexane. It can be used for the extraction of oils, alkaloids, gums, and natural resins. Supercritical CO_2 is used for the extraction of lignans, anthocyanins, and essential oils. Ionic liquids are non-volatile, non-inflammable salts with low melting points (below 100 °C). The melted salts form a liquid that is composed of ions that have high thermal stability, high conductivity, high heat capacity, low flammability, and low or negligible vapor pressure. These can be used for the extraction of a wide range of organic and inorganic compounds such as flavonoids, alkaloids, saponins, lignans, etc. Deep eutectic solvents are a mixture of two or more pure compounds having a eutectic point temperature below an ideal liquid mixture. Deep eutectic solvents are used for the extraction of alkaloids, flavonoids, saponins, and phenolic compounds, and the most commonly used deep eutectic solvents for the extraction of phytochemicals are type III (choline chloride and urea in ratio 1:2) and type IV (choline chloride and zinc chloride in ratio 1:2). NADES solvents are made up of natural metabolites such as organic acids, amino acids, sugars, polyols, and choline derivatives. These solvents have a low volatility and melting point and a broad polarity range. These solvents can be used for the extraction of compounds that are poorly soluble in water. Among the organic solvents, acetone has low toxic potential and is a suitable solvent for the extraction of alkaloids, oils, etc. Ethyl acetate is non-toxic and is used for the extraction of flavonoids, total phenolics, etc. Methanol and chloroform have inherent toxicity. Methanol is used for the extraction of flavonoids, saponins, tannins, etc. Chloroform is used with other solvents such as ethanol and fatty acids for the extraction of alkaloids and anthocyanins. Butanol can be used for the extraction of saponins, total phenolics, and flavonoids; however, it is mainly used for the purification of fractions of individual compounds. n-hexane is a solvent with low acute toxicity. It can be used for the extraction of flavonoids, carbohydrates, anthra-glycosides, and saponins. Methyl acetate is a volatile solvent that is produced by acetic acid esterification with methanol or as a byproduct during methanol carboxylation. It is used mainly for the extraction of phytosterols and tocopherols. Benzene is a potentially dangerous chemical. It can be used for the extraction of flavonoids, phytosterols, alkaloids, and volatile oils. Cyclohexanes are used for the extraction of fats, waxes, and oils [10][67][68][69]. The detailed considerations for the suitable solvent for the extraction of phytochemicals have been discussed by Kim and Wijesekra [70].

The extraction efficiency can be further enhanced by optimizing extraction conditions such as the choice of solvent, temperature, and time. The extraction process starts with cell lysis, followed by the collection of extract, isolation, purification using chromatographic techniques to separate bioactive compounds from the mixture, and identification of phytochemicals using spectrophotometry [71][72][73][74]. Various extraction methods and their mechanism of action are discussed in detail in this section and are also provided in **Table 2**.

Table 2. The optimized conditions for the extraction of phytochemicals using various extraction methods.

Extraction Method	Solvent	Temperature	Pressure	Time Consumed	References
Maceration	Water, aqueous and non-aqueous solvent	Room temperature or	Atmospheric pressure	3–7 days or up to months	[74][75][76]

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Extraction Method	Solvent	Temperature	Pressure	Time Consumed	References
		cold method (4–15 °C)			
Percolation	Water, aqueous and non-aqueous solvent	Room temperature or under heat (35–70 °C)	Atmospheric pressure	2–24 h	[77][78]
Decoction	Water	Atmospheric pressure	1–2 h	65–70 °C	[79][80]
Reflux extraction	Water, aqueous and non-aqueous solvent	60–100 °C	Atmospheric pressure	15 min–2 h	[81][82][83]
Soxhlet extraction	Organic solvents	65–100 °C	Atmospheric pressure	6–24 h	[84][85]
Pressurized liquid extraction	Water, aqueous and non-aqueous solvent	50–200 °C	50–300 psi	5–20 min	[86][87][88]
Microwave-assisted extraction	Water, aqueous and non-aqueous solvent	40–120 °C	Atmospheric pressure	30 s–20 min	[89]
Ultrasound-assisted extraction	Water, aqueous and non-aqueous solvent	20–80 °C	Atmospheric pressure	10–60 min	[90][91]
Pulsed electric field extraction	Water, aqueous and non-aqueous solvent	20–50 °C	1.32–1.64 bar or atmospheric	5 min–48 h	[92][93][94]

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Extraction Method	Solvent	Temperature	Pressure	Time Consumed	References
			pressure		
Enzyme-assisted extraction	Water, aqueous andnon-aqueous solvent	33–67 °C	Atmospheric pressure	20 min–4 h	[95][96]
Supercritical fluid extraction	Supercritical Fluids such as S-CO_2, S-H_2O	40–80 °C	35–70 MPa	10–60 min	[97][98]
High hydrostatic pressure extraction	Water, ethanol, glycerol, silicon oil, or a mixture of solvents	Below 45 °C	100–1000 MPa	3–15 min	[99][100] [101]
Liquid gas extraction	Liquified petroleum gas (propane, n-butane), dimethyl ether	35 °C	Room temperature or low pressure 200–1000 kPa	20 min	[10][102]
Natural deep eutectic solvent extraction	Deep eutectic solvents such as reline, ethaline, glycerine, etc.	25–105 °C	Atmospheric pressure	30–60 min	[103][104] [105]

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4. Suitability of the Methods for the Extraction of Various Bioactive Compounds

4.1. Carotenoids

Various methods have been adopted by scientists for the extraction of carotenoids. This section discusses the literature on the conditions and suitability of these methods. Yaqoob et al. [106] studied the extraction of carotenoids

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biomass was resuspended in a citrate phosphate McIlvaine buffer of pH 7.0 to a final concentration of about 10^8 cells/mL. This was treated for 150 µs at an electric field of 15 kV/cm and total specific energy of 37.12 kJ/kg. This treatment irreversibly electroporated 90% of the cells. Then, the PEF-treated samples were incubated in ethanol at two conditions, i.e., 24 h at 20 °C and pH of 7.0 and 24 h at 25 °C and pH of 8.0. The yield of carotenoids was 240 µg/g d.w. and 375 µg/g d.w., respectively, at either incubation condition.

4.2. Polyphenols

Pavlić et al. [112] studied the NADES extraction of polyphenols from dried wild thyme (*Thymus serpyllum* L.) dust. To the extraction, 0.05 g of sample was mixed with 20 different NADES each at a sample-to-solvent ratio of 1:20 mL/mL and the extraction was carried out for 60 min at 50 °C in a water bath placed on a magnetic stirred hot plate. To perform the separation of extract from solvent, 4 mL of water was added and centrifuged at 4000 rpm for 15 min. The use of L-proline (Pro)–glycerin (Gly)–water (H_2O) NADE solvent at a mixture ratio of 1:2:1 with a water content of 5.68% extracted out the highest polyphenols compared to other NADE solvents. The yield of polyphenols was 71.43 mg GAE/g when 1 g of sample was extracted using 28 g Pro-Gly-H_2O solvent. Popovic et al. [113] studied the green extraction of polyphenols from sour cherry (*Prunus cerasus* L.) pomace using NADES. To perform the extraction, 300 mg of freeze-dried sample was mixed with 4 mL deep eutectic solvent [1:1 M choline chloride (ChCl) as HBA and malic acid, urea, or fructose as HBD] and the extraction was carried out at 50 °C for 45 min with a stirring speed of 650 rpm. The obtained extract had 3238.32 µg/g of total phenols, 2442.93 µg/g of total anthocyanins, 418.00 µg/g of total flavonoids, and 377.39 µg/g of total phenolic acids. Froehlich et al. [114] optimized UAE for the extraction of phytochemicals from dried leaves of clove (*Syzygium aromaticum*) using 99.5% ethanol. It was found in the study that extraction using a solvent-to-sample ratio of 35 mL/g at 70 °C and amplitude of 85% for 25 min gave the highest yield. This resulted in a total extract yield of 14.63%, and the yield of eugenol was 2.94 g/kg of leaves. Domínguez-Rodríguez et al. [115] studied EAE of non-extractable bioactive polyphenol from sweet cherry (*Prunus avium* L.) pomace. In this study, 0.38 g of sweet cherry pomace was extracted using 1 mL methanol at different pH (2–10), temperature (30–70 °C), and enzyme concentrations (1–120 µL/g) for 10–300 min. The optimized conditions were a pH of 10, a temperature of 70 °C, an enzyme concentration of 2 µL/g, and an extraction time of 18.4 min. The recovery of polyphenols at the optimized conditions was 1.1 mg GAE/g sample. Hwang et al. [116] studied the PEF extraction of naringin and hesperidin from dried *Citrus unshiu* peels. For this, 30 g of the sample was immersed in distilled water and was treated at a 5 kW pulse generator, 50 Hz pulse frequency, and 3 kV/cm electric field for 60 and 120 s at room temperature. The total yield of extract was higher in the sample treated for 120 s, and the yield of naringin was 3.95 mg/100 g and 6.16 mg/100 g for the sample, respectively. Negative analysis of phenolic metabolism and antioxidant activity of extractions from *Lycium barbarum* and its [117] using different methods. Food Sci. Technol. 2022, 42, e71022. highest recovery of total anthocyanins (3.30 mg/g DW) was obtained for NADESs prepared using lactic acid and glucose in the ratios 8:1, followed by NADESs prepared using choline chloride: glycine (4:6) (3.30 mg/g DW), glycine: glucose (8:1) (3.06 mg/g DW) and tartaric acid: glycine (4:1) (3.03 mg/g). The anthocyanin content of extracts based on NADES was significantly higher than ethanol (1.16 mg/g DW), except for NADESs prepared using tartaric acid: glycine (1:2) (0.81 mg/g DW). Grdiša et al. [118] studied the extraction efficiency of pyrethrins from dried flower heads of Dalmatian pyrethrum (*Tanacetum cinerariifolium/Trevir. Sch. Bip.*) using maceration,

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UAE, and matrix solid-phase dispersion (MSPD). A sample size of 0.25 g was used in all three extraction methods. Maceration extraction was performed using different solvents, i.e., acetone, ethanol, and ethyl acetate at different volumes, i.e., 5, 7, 9, and 11 mL, at different extraction times, i.e., 0.5, 1, 2, and 3 h, at the stirrer rotational speed of 200, 300, 400, and 500 rpm. UAE was carried out using 5 mL acetone at 50 °C for 60 min at 1200 W and 35 kHz. In MSPD, the sample was mixed with 0.50 g of florisil and 0.40 g of Na_2SO_4, after which florisil was activated at 160 °C and washed with n-hexane and methanol. It was then treated with solvents such as acetone and ethyl acetate at 1:1 (*v/v*) and extracted using a solid phase extractor. It was found in the study that the highest extraction efficiency of pyrethrin was obtained in maceration (0.62%), followed by MSPD (0.59%) and UAE (0.49%). Sharma et al. [119] optimized MAE for the extraction of phytochemicals such as phenols, flavonoids, ascorbic acid, and tannins from dried fruits of *Ficus racemosa*. The optimized conditions for the extraction were sample to water ratio of 1:15, pH of 3.5, microwave power of 360.55 W, and extraction time of 30 s. These extraction conditions resulted in the extraction of 31.19 mg/100 mL of ascorbic acid, 35.14 mg/100 mL of gallic acid, 14.06 mg/100 mL of tannic acid, 50.86 mg/100 mL of chlorogenic acid, 36.96 mg/100 mL of quercetin. Oroian et al. [120] evaluated the extraction efficiency of flavonoids and polyphenols from crude pollen (collected from a local beekeeper in Suceava County, Romania) using UAE. To perform the extraction, 30 g of pollen sample was mixed with 1 liter of 80% methanol and extraction was carried out at 40.85 °C and 100% amplitude for 14.30 min. The extraction of total phenols and total flavonoids was 366.1 mg GAE/100 g and 592.2 mg QE/g of the sample, respectively. De Queiroz et al. [121] optimized the MAE for the extraction of phenols and tannins from the dried stem bark of *Stryphnodendron adstringens*. The extraction was carried out by adding 0.075 g of sample in 1 mL of water and heating it at 106–134 °C for 0.48–2.12 min. These conditions extracted out 15.91–18.69% tannins and 16.36–22.12% phenols from the studied sample. In a study conducted by Azman et al. [67] on the extraction of free and bound phenolics from dried black currant (*Ribes nigrum* L.) skins, it was found that acetic buffer solvent resulted in the highest free anthocyanin (1712.3 mg/100 g), free hydroxycinnamic acid (268 mg/100 g), total phenolic content (3702 mg GAE/100 g), and DPPH inhibition activity (60.7%) compared to other solvents, i.e., water, methanol and a mixture of methanol and water. The use of acetic acid as a co-solvent with other solvents such as water and ethanol has also been reported to extract the phytochemicals from colored vegetables [122]. Jamaludin et al. [99] optimized the extraction of bioactive compounds from noni fruits using high hydrostatic pressure. This study was carried out in two parts. In the first part, the effect of each extraction parameter (ethanol concentration, pressure, and extraction time) was studied individually on the yield of bioactive compounds (scopoletin, alizarin, and rutin), and in the second part, the combined effect of the extraction parameters was studied on the yield of bioactive compounds using the Box-Behnken Design of RSM. The highest yield of bioactive compounds, i.e., scopoletin (82.4%), alizarin (77.2%), and rutin (82.2%), were found at 544 MPa, with an extraction time of 15 min and ethanol concentration of 65%. The extraction of phenyletanes and phenylpropanoids of *Rhodiola rosea* L. using NADES was studied by Shikov et al. [123]. The highest concentration of total phenyletanes and phenylpropanoids (26.10 mg/g) was obtained using NADES prepared using L-lactic acid, fructose, and water in the ratios 5:1:11 mL/mol when the particle size of *Rhodiola rosea* L. rhizome was in between 0.5–1 mm and the extraction was carried out for 154 min at 22 °C and extraction modulus of 40. Razboršek et al. [103] performed choline chloride-based UAE (NADES) extraction of phenolic compounds from chokeberry (*Aronia melanocarpa*) and compared the results with those obtained from 80% methanol extract. The highest total phenols (36.15 (mg GAE/g DW)) and total flavonoids

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(4.71 mg rutin/g DW) were obtained for NADES prepared using choline chloride, fructose, and water in the ratios 2:1:1. This was significantly higher than 80% methanol, i.e., 27.11 mg GAE/g DW for total phenols and 3.37 mg rutin/g DW for flavonoids. The application of methyl acetate under pressurized conditions for the extraction of Crambe seed oil has been reported to have higher phytosterol and tocopherol values compared to the Soxhlet method [124]. Castro-López et al. [125] studied polyphenol extraction from pomegranate (*Punica granatum*) peels, walnut (*Juglans regia*) shells, hojasen (*Cassia fistula*) leaves, and moringa (*Moringa oleifera*) leaves using different extraction methods, i.e., maceration, decoction, UAE, and MAE. For maceration, 0.2 g of sample was treated with 10 mL deionized water at a sample-to-solvent ratio of 1:50 at room temperature in a magnetic stirrer for 2 h. For decoction, 0.2 g of sample was treated with 10 mL deionized water at a sample-to-solvent ratio of 1:50 in an oven at 60 °C for 2 h. The UAE was carried out at 25 °C in a sonicated water bath for 60 min using a sample-to-solvent (deionized water) ratio of 1:50. For MAE, deionized water at a sample-to-solvent ratio of 1:50 was used at 550 W and 70 °C for 90 s. Higher polyphenol content was obtained using MAE followed by decoction, UAE, and maceration methods. Total polyphenol yields of 6.4–18.92 mg GAE/g, 1.17–12.8 mg GAE/g, 2.73–15.19 mg GAE/g, and 1.68–12.69 mg GAE/g were obtained for pomegranate peel, walnut shell, moringa leaves, and hojasen leaves, respectively. Jovanović et al. [126] extracted polyphenols from the air-dried aerial part of *Thymus serpyllum* L. using maceration, heat-assisted extraction (HAE), and UAE. Maceration was carried out using ethanol and water solutions containing 30%, 50%, 70%, and 96% ethanol. The particle sizes of the powder used for extraction were 0.3, 0.7, and 1.5 mm, and to perform the extraction, solid-to-solvent ratios of 1:10, 1:20, and 1:30 were used for the extraction times of 5, 15, 30, 60 and 90 min. In HAE, solvent concentrations and sample-to-solvent ratios were the same as that of maceration; however, the extraction was carried out at 80 °C for 5, 15, and 30 min in an incubator shaker. In UAE, solvent type, solid-to-solvent ratio, particle size, and extraction time were similar to those of HAE. The extraction was carried out at 25 °C and 80% amplitude and a 750 W output ultrasonic processor with a 20 kHz converter having a solid titanium probe of 19 mm diameter. The total phenolics extracted using maceration, HAE, and UAE were 19.56 mg GAE/L, 22.60 mg GAE/L, and 24.94 mg GAE/L, respectively. Porto and Natalino [127] studied the SFE of polyphenols from dried white grape Marc (*Vitis vinifera*) seeds. They used 100 g of sample in an SFE pilot plant (SCF100 series 3 PLC-GR-DLMP, Separeco S.R.L, Pinerolo, Italy) equipped with a 1 L extraction vessel, and the extraction was carried out for 13 min at a pressure of 80 bar, a temperature of 40 °C, and a CO_2 flow rate of 6 kg/h along with 57% *v/v* of ethanol–water (20% *w/w*) mixture as co-solvent. The total polyphenol yield in this extraction was 7132 mg GAE/100 g DM. The extraction of phlorotannins from brown algae using NADES is reported by Obluchinskaya et al. [128]. The study reported that the use of aqueous NADES solutions (50–70%) based on choline chloride with added lactic or malic acid and betaine and malic acid gave a 6–72% yield of phlorotannins. Sharif and Bennet [129] compared maceration and reflux methods for the extraction of polyphenols from freeze-dried ginger rhizomes using various solvents viz. ethanol, methanol, and acetone. For maceration, 10 g of sample was used with 300 mL of solvent and placed in an orbital shaker for 8 h. In the reflux extraction, a 2.5 g sample was extracted with 50 mL solvent at 90 °C for 30 min. The total phenolic content obtained using ethanol for the maceration and reflux extraction were 263 and 205 mg/100 g GAE, respectively. In the case of acetone, the total phenolic yield was 216 mg/100 g GAE for maceration and 184 mg/100 g GAE for reflux extraction, whereas using methanol, it was 148 mg/100 g GAE for maceration and 95 mg/100 g GAE for reflux. This shows the lower extraction efficiency of reflux extraction compared to maceration.

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4.3. Phytosterols

De Aguiar et al. [130] studied the effect of thermal pre-treatment on the enzyme (protease)-assisted aqueous extraction and yield of phytosterols from sunflower (*Helianthus annuus* L.) seeds. The thermal pre-treatment was performed by immersing 150 g of whole seeds in distilled water at room temperature in the ratio of 1:3 (*w*/*v*). Excess water was removed after 3 h and the sample was placed in an oven with air circulation (Marconi, Model MA035) at 120 °C for 60 min. A commercial enzymatic preparation, i.e., Alcalase® 2.4 L FG was used as a source of protease. Enzymatic extraction for both thermally pre-treated and untreated samples was carried out for 3 h at 40 °C, pH of 8, and enzyme concentration of 8% *v*/*v*. The total yield of the oil using this method was 15.59%. The total yield of phytosterols in the extracted oil without thermal treatment was 149.41 mg/100 g oil, and the major phytosterols were campesterol (15.80 mg/100 g), stigmasterol (21.46 mg/100 g), γ-sitosterol (12.07 mg/100 g), and β-sitosterol (100.07 mg/100 g). The yield of total phytosterols from the thermally pre-treated samples was 133.66 mg/100 g. The major phytosterols obtained were campesterol (15.99 mg/100 g), stigmasterol (18.88 mg/100 g), γ-sitosterol (8.72 mg/100 g), and β-sitosterol (90.08 mg/100 g). Hien and Minh [131] compared UAE and enzyme-assisted UAE for the extraction of oil and phytosterols from dried pumpkin (*Cucurbita pepo* L.) seeds. The extraction was carried out using hexane for 4.5 h at a sample to solvent ratio of 1:6, frequency of 40 kHz, and temperature of 60 °C. In enzyme-assisted UAE, commercial enzyme Alcalase® 2.4 L FG with enzyme activity of 3.9 U/mL was used along with the above parameters. The oil extraction yield was 95.46% in UAE, while 91.87% was obtained from enzyme-assisted UAE. The phytosterol content was 2017.5 mg/100 mL in UAE-extracted oil and 2327.7 mg/100 mL in oil extracted from enzyme-assisted UAE. UAE was found to be effective for oil extraction, while the phytosterol extraction was more efficient with the enzyme-assisted UAE. Jalani et al. [132] extracted phytosterols from sludge palm oil (also known as palm acid oil) and empty fruit bunch (*Elaeis guineensis*) residual oil. In this study, a 5 g sample was extracted at 90 °C for 1 h with 50 mL of ethanol at the sample-to-solvent ratio of 1:10. The quantity of the phytosterols in the sludge palm oil was 500 ppm and the content of phytosterols in unsaponifiable form was 6.19%. In empty fruit bunch residual oil, phytosterol content was 450 ppm and the phytosterols in unsaponifiable form were 4.58%. Jafarian Asl et al. [133] compared Soxhlet and SFE extraction of phytosterols from rapeseed (*Brassica napus* L.) oil. Soxhlet extraction was carried out using a 15 mg sample in 150 mL ethanol at the sample to solvent ratio of 1:10 at different temperatures (40, 60, and 80 °C) for 1 h. SFE was carried out for 1 h with CO_2 and co-solvent ethanol having flow rates of 5 mL/min and 0.5 mL/min, respectively. This extraction was carried out at different pressures ranging from 100–400 bar. The highest yield (87%) of phytosterols was obtained in Soxhlet extraction at 40 °C and SFE at 350 bar. The lowest phytosterol yield (21%) was obtained with SFE at 100 bar. Ibrahim et al. [134] studied the MAE extraction of β-sitosterol from cocoa (*Theobroma cacao*) shell waste. The extraction was carried out using a 100 g sample with 300 mL, 99% ethanol in the ratio of 1:3 *w*/*v*, temperature of 70 °C, and 500 W power. The total extract was obtained based on the one factor at a time (OFAT) approach and was 35.6 mg/100 g extract.

4.4. Saponins

Li et al. [135] studied the UAE of saponins from powdered *Aralia taibaiensis* root bark. The extraction was carried out in a water-bath sonicator with different ethanolic concentrations (50, 60, 70, 80, and 90%), time durations (10,

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20, 30, 40, and 50 min), temperatures (40, 50, 60, 70, and 80 °C), sample to solvent ratios (5, 10, 15, 20, and 25 g/mL), ultrasonic power (100, 200, 300, 400, and 500 W) and number of extractions (1, 2, 3, 4, and 5). The highest total saponin (11.45%) content was obtained when 5 g of sample was extracted with 75 mL of 73% ethanol at 400 W and 61 °C for 34 min. Liu et al. [136] studied the EAE of saponins from powdered *Acanthopanax senticosus*. The extraction was carried out at different enzyme concentrations (1000, 5000, and 9000 U/g), time durations (45, 55, and 65 min), temperatures (40, 50, and 60 °C), and solvent pH values (5.4, 6, and 6.6). The highest extraction yield of saponins (17.8 mg/g sample), was obtained when 2 g of sample was extracted with 6963 U/g of enzyme mixture (cellulase and pectinase at a ratio of 2:3) at pH 6 and a temperature of 53.7 °C for 60 min. Yang et al. [137] studied the extraction of four bioactive steroidal saponins (protodioscin, protogracillin, pseudoprotodioscin, and pseudoprotogracillin) from the dried rhizome of *Dioscorea nipponica*, also known as Dioscoreae Nipponicae Rhizoma (DNR), using NADES. A mixture containing ChCl and malonic acid in a molar ratio of 1:1 with 30% water was used as the NADE solvent. For a sample weighing 50 mg, the optimal extraction conditions were 1 mL NADE solvent, an extraction time of 23.5 min, a liquid–solid ratio of 57.5 mL/g, a water content of 54%, and ultrasonic conditions of 300 W and 40 kHz. The recovery yield of four steroidal saponins was between 98.8 and 107.5% compared to standard steroidal saponins. The extract consisted of 64.99 mg/g of total saponins where 29.39 mg/g was protodioscin, 15.86 mg/g was protogracillin, 0.71 mg/g was pseudoprotodioscin, and 3.66 mg/g was pseudoprotogracillin. Ramli et al. [138] studied the MAE of saponins from dried furcraea (*Furcraea selloa var. marginata*) leaves using water, ethyl acetate, and ethanol. The extraction was carried out using a 3 g sample in 200 mL solvent at a ratio of 1:24, frequency of 2.45 GHz, and power of 1000 W at 90 °C for 9 min. The extraction yields obtained from aqueous, ethyl acetate, and ethanolic extract were 5.77%, 8.07%, and 6.67%, respectively. The saponin contents in the samples extracted using water, ethyl acetate, and ethanol were 0.0514 g/mL, 0.0453 g/mL, and 0.0344 g/mL extract, respectively.

4.5. Isoprenoids

Lanjekar and Rathod [139] optimized the extraction of glycyrrhizic acid from *Glycyrrhiza glabra* (Liquorice root) powder using choline chloride (ChCl): lactic acid (1:1), ChCl: dextrose (2:1), ChCl: glycerol (1:1), ChCl: malic acid (1:1), ChCl: citric acid (1:1), ChCl: oxalic acid (1:1), and ChCl: succinic acid (1:1) as different natural deep eutectic solvents. In this extraction, 2 g licorice powder having a moisture content of 7.78% was treated with 20 mL NADES in an overhead stirrer for 60 min at 400 rpm and 30 °C. The mixture was then centrifuged at 8000 rpm for 10 min and analyzed using HPLC. The highest glycyrrhizic acid yield was 43.65 mg/g of the sample using ChCl: succinic acid as solvent. The yield of glycyrrhizic acid was 42.82 mg/g using ChCl: lactic acid as solvent, 23.25 mg/g using ChCl: dextrose, 14.37 mg/g using ChCl: glycerol, 30.67 mg/g using ChCl: citric acid, 36.70 mg/g using ChCl: malic acid, and 39.60 mg/g using ChCl: oxalic acid. The extraction of glycyrrhizic acid from liquorice roots using NADES has also been reported by Shikov et al. [140]. The yields of glycyrrhizic acid in NADES based on sucrose and lactic acid (3:1), sorbitol and lactic acid (3:1), and choline chloride and lactic acid (1:3) were higher (38–60 mg/g) than its yield in water (<30 mg/g). Rodrigues et al. [141] extracted triterpenoids from dried leaves of *Acacia dealbata* using SFE and Soxhlet extraction. The sample was dried using a forced convection oven at 35 °C for 72 h to a moisture content of 4.5% wt. For Soxhlet extraction, different solvents such as 99.5% ethanol, 99% hexane, 99% ethyl acetate, and 99% dichloromethane were used. In this study, 3 g of sample was used with 180

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mL solvent for 6 h at 30–70 °C. For SFE, CO_2, CO_2:ethanol (95:5 wt.%) and CO_2:ethyl acetate (95:5 wt.%) were used as solvents. For extraction, a 25 g sample was loaded into the extraction chamber of a lab-scale Speed Helix SFE System at a flow rate of 12 g/min for 6 h at 40–80 °C. From Soxhlet extraction, the highest total extraction yield obtained was 11.58% using ethanol as solvent. The highest triterpenoid yield obtained was 8201 mg/kg of extract using ethyl acetate as a solvent and 6259 mg/kg of extract using ethanol as a solvent. From SFE, the highest yield was 1.76% using CO_2 as solvent. The highest triterpenoid yield obtained was 4719 mg/kg of extract using CO_2: ethanol as solvent and a triterpenoid yield of 4366 mg/kg of extract using CO_2:ethyl acetate as solvent. Grdiša et al. [118] studied the extraction efficiency of pyrethrins from dried flower heads of Dalmatian pyrethrum (*Tanacetum cinerariifolium/Trevir./Sch. Bip.*) using maceration, UAE, and matrix solid-phase dispersion (MSPD). A sample size of 0.25 g was used in all three extraction methods. Maceration extraction was performed using different solvents, i.e., acetone, ethanol, and ethyl acetate at different volumes, i.e., 5, 7, 9, and 11 mL, at different extraction times, i.e., 0.5, 1, 2, and 3 h at stirrer rotational speeds of 200, 300, 400, and 500 rpm. UAE was carried out using 5 mL acetone at 50 °C for 60 min at 1200 W and 35 kHz. In MSPD, the sample was mixed with 0.50 g of florisil and 0.40 g of Na_2SO_4, (florisil was activated at 160 °C and washed with n-hexane and methanol). It was then treated with solvents such as acetone and ethyl acetate at 1:1 (*v/v*) and extracted using a solid phase extractor. It was found in the study that the highest extraction efficiency of pyrethrin was obtained in maceration (0.62%), followed by MSPD (0.59%) and UAE (0.40%).

4.6. Polysaccharides and Dietary Fiber

Gong et al. [142] studied the SFE of polysaccharides from dried fallen *Ginkgo biloba* leaf powder. For the extraction, a 20 g sample was placed in a supercritical extraction device and the extraction was carried out using SC-CO_2 and 15% co-solvent having strengths of 60, 70, and 80% and at the temperatures of 50, 60, and 70 °C, pressures of 35, 40, and 45 MPa, and time intervals of 80, 100, and 120 min. The extract was centrifuged at 6000 rpm for 15 min and analyzed. The highest yield (10.13 g/100 g) of polysaccharides was obtained with 68% co-solvent at the extraction conditions of 42 MPa, 63°C, and 99 min. García et al. [143] studied pressurized liquid extraction of total dietary fiber from dried pomegranate (*Punica granatum* L.) peel and fruit. For the extraction, 3.75 g of powdered sample was mixed with 11.25 g of sand and the extraction was carried out in a pressurized liquid extractor at 1500 psi for 20 min. The total dietary fiber extracted was analyzed using an enzymatic gravimetric method and a yield of 30% was obtained from the peel and 18% from the fruit. Hussain et al. [144] optimized the UAE of soluble dietary fiber from dried sea buckthorn (*Hippophae rhamnoides* L.) pomace powder. Samples were pretreated by mixing with 0.1% citric acid at the sample-to-acid ratio of 1:25 and incubated in a water bath at 80 °C for 1 h. Extraction was carried out using a Digital Sonifier® S450 CE (Richmond Newtown, VA, USA) attached with a 13 mm diameter disruptor horn. The extraction was carried out at different sonication temperatures (60, 70, and 80 °C), powers (100, 130 and 160 W), and time intervals (30, 45, and 60 min). The extract so obtained was further centrifuged at 6000 rpm for 10 min to obtain the dietary fiber. The highest extraction yield obtained was 17.82% under the conditions of 130 W and 70 °C for 45 min. Rivas et al. [145] optimized the SFE of dietary fiber from dried pomegranate (*Punica granatum* L.) peel. The extraction was carried out using 4.0 g powdered sample in a 100 mL stainless steel column with a CO_2 flow rate of 2 L/min and different extraction conditions such as pressure (250, 275, and 300 bar), temperature (45, 50, and 55 °C), and time (2, 3, and 4 h). Dietary fiber was estimated using the

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