Chapter 4

Microwave-Assisted Extraction of Bioactive and Nutraceuticals

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Abstract

The increasing consumer awareness about the link between nutrition and health has led the food industry to produce fortified food with bioactive compounds. Considering that not all bioactive compounds are freely available and in the light of increasing attention to preserve environmental resources, the new trend consisted of waste recovery of industrial food processing residues with active potential. Currently, clean label and eco-friendly extraction methods have realized reputation accounts for the removal of solvent usage and reduction in energy consumption. In this context, microwave-assisted extraction (MAE) evolved as a novel procedure for the extraction of bioactives and nutraceuticals. With higher extraction efficiency, this process was noted to consume less time and energy, and interestingly, the bioactive compound's functionality has not degraded. In this chapter, MAE's potential as an eco-friendly technique was explored. To improve its efficiency, microwave-assisted extraction has been coupled with conventional techniques. Accessible data stress the significance of various hybrid techniques: microwave/conventional ones for the extraction of bioactive compounds. Information about this topic could help students and scientific researchers who are engaged in chemical engineering, chemistry, and meat technology communities to approach the complex theme of microwave-assisted extraction.

Key words Microwave-assisted extraction, Bioactive compounds, Nutraceuticals, Eco-friendly, Hybrid technique extraction

1 Introduction

Nowadays, extraction and production of various bioactive compounds have gained momentum as there is increased demand for herbal products globally. This is because they are safe, possess various biological activities as compared to synthetic formulations, and are cost-effective. To provide higher recoveries and greater reproducibility, the chief tendencies in analyte extraction was to reduce solvent and energy consumption and to provide higher

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recoveries and greater reproducibility. The implementation of traditional extraction methods may be more time-consuming, requiring large volumes of solvents, and are principally related with the degradation of heat-sensitive compounds [1]. In this sense, to overcome the drawbacks of these extraction methods, it is decisive to explore contemporary techniques. Green solvent extraction methods have been developed, including microwave-assisted extraction (MAE), which has gained a wide attention due to its various advantages, namely, a reduced solvent consumption, a shorter operation time, and an enhanced recovery yield [2]. Numerous comparative studies have shown that MAE allowed better performance in terms of compound recoveries [3–7]. By applying MAE, plentiful kinds of compounds, comprising essential oils, antioxidants, pigments, and other organic compounds have been successfully isolated from various natural plant resources [5, 8–10]. These previous studies displayed that MAE could be a promising alternative to conventional extraction of plant pigments (carotenoids and anthocyanins) [9, 11], polyphenols, polysaccharides [12–14], essential oils [5, 15, 16], and proteins and lipids [17]. Compared to maceration and Soxhlet extraction, it was established that MAE approach was more effective [9]. It was found that the obtained extracts using MAE had a greater concentration of volatile terpenoids (α- and β-pinene) [18]. Microwaves have electric and magnetic fields since they are electromagnetic devices [19]. MAE employs microwave radiation to heat solvents and facilitates the transfer of target compounds from the sample matrix to the extractant by inducing polar molecules, ions, and dipoles movement and rotation [20]. Microwaves can penetrate the sample and incite cell molecules to absorb their energy, resulting in an increase in temperature and pressure. Then, it facilitates the cell's rupture and the reachability of the components into the solvent solution [21]. In fact, there are several categories of MAE such as solvent-free MAE (SFM), focused-MAE (FMAE), ionic liquid-based MAE (ILMAE), ultrasonic MAE (UMAE), microwave hydro-distillation (MHD), microwave hydro-diffusion and gravity (MHG), and microwave-assisted subcritical extraction (MASE) [12, 22–27]. The extraction techniques employed for this purpose are highly dependent on the following factors: microwave power, time, solvent, sample-to-solvent ratio, temperature, and matrix characteristics [28]. The aim of this chapter is to spotlight the versatility of MAE in the recovery of bioactive compounds and nutraceuticals from various types of vegetal materials using different techniques of MAE with a special focus on the factors that could influence its processing and efficiency.

2 Mechanism of MAE Process

Microwaves have electric and magnetic fields since they are electromagnetic devices. These fields lead to a heating effect via two mechanisms, dipolar rotation and ionic conduction [19].

(i) Dipolar rotation refers to the phenomenon that occurs when molecules with uneven distribution of charge, known as a dipole moment, attempt to align themselves with the alternating electric field produced by microwaves. The oscillation of these dipolar molecules results in collisions with other molecules in the surrounding medium, which then generates heat. This process happens quickly and repeatedly, making it an efficient way to convert electromagnetic energy to thermal energy [29].

On the other hand, (ii) *ionic conduction* is defined as a process that occurs when charged particles, such as ions and electrons, move through a medium in response to an electric field produced by microwaves. This movement or migration generates friction between the ions and the medium, which results in the generation of heat. The degree of heat generated by this process depends on factors such as the strength of the electric field and the conductivity of the medium [20].

The relative contribution of these two mechanisms to the overall heating of the sample is largely dictated by temperature. Specifically, as the contribution of dipole rotation decreases, the temperature of the sample increases, while the contribution of ionic conduction increases. It means that if a sample contains both polar molecules and ions, then as it is heated by microwave energy, the heating will initially be dominated by dipole rotation. The relative contribution of these two mechanisms also depends on the mobility and concentration of the ions within the sample [20]. Consequently, these mechanisms induced the destruction of hydrogen bonds in organic molecules, which increased solvent penetration into the plant matrix [30] and thereby dissolution of extractable molecules. In fact, microwave-assisted extraction (MAE) may be summarized in two main steps as Chemat et al. [21] and Vinatoru et al. [20] mentioned:

1. Penetration of the solvent into the plant cell by diffusion: Initially, in the equilibrium phase, solubilization and partitioning phenomena come into play, which leads to the detachment of the substrate from the particle's outer surface at a relatively consistent rate. This step is then followed by an intermediate phase of transition to diffusion, where resistance to mass transfer begins to appear at the interface between the solid and liquid phases. During this period, a mass transfer occurs through convection and diffusion.

2. Cell rupture and leaching out of cell components into the solvent solution: When the dielectric loss tangent of the plant cell is higher than that of the solvent, the vegetal material could absorb more electrical energy, which can lead to an increase in the temperature of the plant material and subsequently an increase in cell pressure. As the extract is removed mainly through diffusion, it is typically regarded as the limiting step of the process.

Throughout the extraction process, a variety of forces and relationships can be observed, including dispersion forces, interstitial diffusion, driving forces, and chemical interactions, with the persistence and strength of these phenomena often closely linked to the solvent's properties, such as solubilization power, solubility in water, purity, and polarity [21].

3 Factors Affecting MAE

Numerous types of compounds, including essential oils, antioxidants, pigments, and other organic compounds, have been effectively isolated from various natural plant resources using MAE [5, 8–10]. The extraction techniques employed for this purpose are highly dependent on the following factors (Fig. 1).

3.1 Microwave Power

In a reaction medium, the amount of electromagnetic energy that is transformed into heat depends, in a practical sense, on the permittivity and permeability of the chemical compounds or mixture, as well as the intensity of the electromagnetic field [21].

Microwaves belong to the electromagnetic spectrum, and their frequency range spans from 300 MHz (classified as radio radiation) up to 300 GHz. In scientific research, two specific frequencies are usually utilized: 2.45 GHz, which is commonly used in laboratory equipment, and 915 MHz, which is mostly used in industrial equipment [20, 31]. It has been shown that the range of power delivered was between 60 and 960 W (Table 1). Increasing

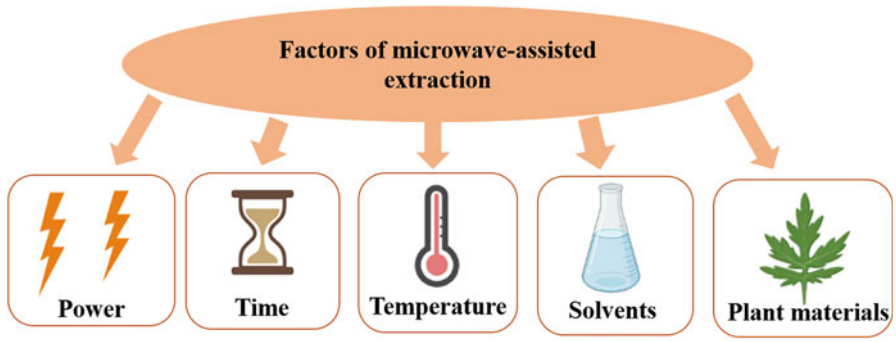

Fig. 1 Factors affecting MAE

Table 1
MAE extraction of different analytes from various vegetal materials

Materials	Solvent	Ratio	Temperature	Time	Power	Analyte	References
Mango peel	Diluted acidic solution (distilled H_2O, with 2 M HCl)	2 g/600 mL	–	3 min	700 W	Pectin: 1485.78 mg/mol	[58]
Sapindus mukorossi	40% Ethanol	1 g/19 mL	–	13 min	425 W	Saponins yield: 280.55 mg/g	[59]
Red cabbage	50% Ethanol	1 g/20 mL	–	10 min	600 W	Total monomeric anthocyanin content: 220 mg cyanidin-3-glycoside/L	[10]
Melissa officinalis L	25.9% Ethanol	300 mg/10 mL	–	29 min	400 W	Rosmarinic acid: 49.5 mg RA/g of DW	[60]
Melastoma sanguineum fruit	31.33% Ethanol	0.5 g/30 mL	52.24 °C	45 min	500 W	TPC: 39.02 mg GAE/g of DW	[55]
Chaya (*Cnidoscolus aconitifolius* Mill.) leaves	99.8% Ethanol	1 g/20 mL	140 °C	10 min	850 W	TPC: 57 mg GAE/g	[61]
Tomato pericarps	100% Ethanol	45 g/L	180 °C	20 min	200 W	TPC: 66.8 mg GAE1/g TFC: 3.89 mg CE/g	[62]
Hibiscus sabdariffa	60% Ethanol	3 g/30 mL	164 °C	22 min	850 W	Flavonoids yield: 55%	[63]
Banana peel	Water	2 g/100 mL	–	6 min	960 W	TPC: 50.55 mg GAE/g DM	[64]
Apple dust by-product	40% Ethanol		–	15 min	600 W	TPC: 36.99 mg GAE/g DW	[65]
Grape juice waste	Water	1 g/18.43 mL	–	2.23 min	428 W	Total monomeric anthocyanin yield: 1.32 mg/g	[66]
Onion peels	Choline chloride: urea: water (1:2:4)	1 g/54.97 mL	–	15.03 min	100 W	TPC: 80.45 mg GAE/g DW	[67]

(continued)

Table 1
(continued)

Materials	Solvent	Ratio	Temperature	Time	Power	Analyte	References
Pomelo peels	10 mmol/L [HO3S (CH2)4 mim] HSO4 aqueous solution	1 g/26 mL	–	15 min	331 W	Yield of pectin: 291.60 mg/g Yield of naringin: 8.38 mg/g	[68]
Spruce (*Picea abies*) bark	50% Ethanol	–	–	4 min	300 W	Catechin: 1.91 mg/g of VM Epicatechin: 6.72 mg/g of VM α-pinene: 2198.33 μg/g of VM β-pinene: 2997.66 μg/g of VM Camphene: 71.6 μg/g of VM Myrcene: 105.46 μg/g of VM Limonene: 101.07 μg/g of VM	[18]
Quercus cerris bark extracts	Water	10 g/ 200 mL	–	30 min	850 W	TPC: 382.26 mg GAE/g of DW Total tannin content: 49.14%	[4]
Quercus cerris bark extracts	70% Ethanol	10 g/ 200 mL	–	18 min	650 W	TPC: 403.73 mg GAE/g of DW Total tannin content: 45.68%	[4]
Avocado *(Persea americana Mill.)* seeds	58% Ethanol	1 g/20 mL	–	5 min	400 W	TPC: 82.36 mg GAE/g TFC: 19.93 mg QE/g	[69]
Olive tree leaves	Solvent-free	5 g	–	2 min	250 W	TPC: 2.480 ppm Oleuropein yield: 0.060 ppm	[70]
Coleus aromaticus leaves	Solvent-free	500 g	–	30 min	500 W	Essential oil yield: 0.54%	[16]
Lagenaria siceraria fruit	Solvent-free	20 g	–	60 s	480 W	TPC: 288.9 mg GAE/g DW TFC: 214.1 mg rutin equivalent/g DW	[22]
Pegagan (*Centella Asiatica* L.) leaves	Solvent-free	20 g	–	60 min	450 W	Yield: 4.5474%	[1]

Cinnamomum camphora leaves	Solvent-free	200 g	–	23 min	580 W	Essential oil yield: 3.51%	[5]
Black carrot pomace	19.8% Ethanol	1 g/ 9.3 mL	–	9.8 min	348.07 W	Total anthocyanin content: 753.4 mg/L TPC: 264.9 mg GAE/100 mL	[47]
Black currant	60% Ethanol	1 g/28.3 mL	–	3 min	551 W	Flavonols: 2323.3 μg/g Anthocyanins: 473.7 μg/g	[71]
Patchouli (*Pogostemon cablin*)	Water	0.15 g/mL	–	51.61 min	634.024 W	Patchouli oil yield: 2.8%	[72]
Ananas comosus peel	H_2SO_4 (0.5 N, pH 1.83)	1 g/10 mL	80 °C	2.5 min	600 W	Yield of pectin: 2.43% Anhydrouronic acid content: 54.61%	[73]
Brown seaweeds	0.1 M HCl containing 2 M $CaCl_2$	1 g/25 mL	–	1 min	560 W	Sugar content: 0.47 mg glucose equivalent/mg extract	[74]
Eucalyptus globulus bark	Water	1 g/48.5 mL	141 °C	15 s	500 W	TPC: 350 mg of GAE/g of extract	[75]
Curcuma longa	Choline chloride-citric acid (1:1)	1 g/20ML	–	6 min	60 W	Curcuminoids yield: 89.87 mg/g	[53]
Pineapple peel waste	50% Ethanol	1 g/20 mL	–	40 min	600 W	TPC: 14.188 mg GAE/g DW TFC: 12.925 mg QE/g DW Total Tannin Content: 371.25 mg TAE/g DW Protein content: 59.49 mg BSAE/g DW	[76]

Db dry basis, *DM* dry matter, *DW* dry weight, *VM* vegetal material, *TPC* total phenolic content, *TFC* total flavonoids content

microwave power from 180 to 300 W gave rise to high trans-lycopene and β-carotene contents [32]. Additionally, Vu et al. [33] reported that high phenolic compounds were acquired when the power was raised from 240 to 960 W. These radiations led to the disruption of cell wall and then cell membrane followed by the release of bioactive compounds [2]. Hence, it permitted the gradual efflux of plant exudates. Consequently, it affected the yield of bioactive compounds [33]. However, increasing microwave power beyond 300 W decreased the contents of trans-lycopene and β-carotene [32]. In fact, there is a risk of losing/degrading plant bioactive components caused by the usage of higher power with extended exposure [34]. Thus, microwave power and irradiation time are completely opposed [2].

Nisca et al. [4] found that the TPC of *Quercus cerris* bark extracts was improved when the microwave power was increased. The variation of microwave power from 200 to 850 W had a significant influence on the content of the total phenolics and tannins.

Microwaves could be influenced by different types of materials, which can be categorized as follows [35]:

Opaque materials: Conductive materials that possess free electrons, like metals, have a tendency to reflect electromagnetic waves, preventing them from passing through. These materials are utilized in constructing microwave applicants [36].

Transparent materials: Materials that have a low dielectric loss or insulating properties, such as ceramics and glass, only absorb and reflect electromagnetic waves to a minimal extent, thus enabling microwaves to pass through with minimal attenuation [37]. These materials are typically used in reactors that are placed within microwave applicants.

3.2 Extraction Time

Heating time is an essential factor that affects the extraction mechanism [19]. Furthermore, increasing time augmented extraction efficiency and quantity of analytes [2]. However, it also increases the possibility of the degradation of thermolabile compounds. For the extraction of different kinds of plant matrices, various time scales are required [38]. Sometimes, 60 s−60 min are required for the maximum production of analytes in MAE (Table 1). Several findings agreed that extended exposure to microwave irradiation resulted in a greater release of phenolic compounds from *Hibiscus sabdariffa*, *Aegle marmelos*, and *Myrtus communis* leaves [39–41]. The amount of polyphenols extracted increased by over 30% when the extraction time was changed from 5 to 29.5 min [42]. Whereas, Belwal et al. [8] reported that 2 min was suitable for MAE of alkaloids, berberine and palmatine with concentrations of 46.38 mg/g DW and 20.54 mg/g DW, respectively. In the investigation of Kadi et al. [9], it was noticed that the total

carotenoid content from *Citrus clementine* peel reached its maximum (186.55 μg/g DM) at 7.64 min; then the compounds of interest easily decomposed as a result of the long exposure. According to Samanta et al. [3], MAE technique has exhibited an increase in yield of 70% compared to other traditional methods, resulting in higher TFC and TPC levels, which was accomplished in a shorter time frame.

3.3 Extraction Solvent and Sample-to-Solvent Ratio

The proper selection of extraction solvent is one of the key elements that have a significant impact on MAE's total output. The properties of the solvent (nature, polarity, solubilization, purity, etc.) are other variables that affect the process of extraction [2]. Several forces, such as the physicochemical interactions, may be strongly associated with the properties of the solvent [21]. Typically, when a solvent has a high dielectric constant and dielectric loss, it tends to have a greater ability to absorb microwave energy. Essentially, the capacity of the solvent to absorb this energy increases as the dielectric constant and dielectric loss increase, resulting in a faster heating rate for the solvent relative to the plant material [43]. In order to measure relative solubility, the Hildebrand solubility parameter scale is commonly used. This parameter is the measure of the cohesive energy between the solvent and the matrix in a solution [20]. In fact, δ is linked to the hydrogen-bonding capacity, the polarity, and the dispersion coefficient. As a result, there is a significant correlation between the polarity and the Hildebrand solubility parameter (δ) [20]. The solvent volume is also a crucial component to take into account since it needs to be sufficient to ensure that the entire sample is submerged in the solvent during the whole irradiation process [21]. In addition, the selected solvent must be more selective toward the target analyte than the other matrix constituents [44]. MAE can use water as a solvent for both polar and nonpolar compounds, making it an attractive option for more environmentally friendly extraction processes [43]. By blending different solvents, it is possible to alter the properties of the solvent, resulting in differing selectivity for various compounds [43]. In fact, the use of an ethanol-water mixture as an extraction solvent facilitated the recovery of TPC due to its high dielectric constant and dissipation factor, which enables the effective absorption of microwave energy. Furthermore, this solvent mixture increased the penetration of the solvent into the sample matrix, thereby enhancing heating efficiency. These results are in line with the findings of Nisca et al. [4], who carried out an optimization of extraction parameters for aqueous and hydroalcoholic extractions, and the total polyphenolic and tannin contents were determined. The results indicated that the optimal extraction conditions for aqueous (30 min at 850 W) and hydroalcoholic (18 min at 650 W) extracts were different. The hydroalcoholic bark extract exhibited a higher yield of total polyphenols (403.73 mg GAE/g

dried weight) compared to the aqueous extract that had a lower level of tannins. Hence, MAE may yield higher levels of polyphenols when mixtures of solvents are used due to the increased solubility of target compounds and better penetration into the plant material [4].

In addition, optimizing the solvent-to-solid ratio (S/S) is a crucial parameter. It is necessary to ensure that the solvent volume is adequate to fully immerse the sample during the entire irradiation process, particularly when dealing with a matrix that may expand during the extraction process [45]. As the ratio of sample to solvent increased from 2:100 g/mL to 8:100 g/mL, the TPC decreased by nearly 50% [33]. The reason coming behind these results is that when a smaller sample ratio is utilized, the plant material swells, which leads to an increase in the contact area between the plant matrix and the solvent [46]. While Kumar et al. [47] stated that the retrieval of phenolic content showed a notable upsurge when the ratio of solvent to solid (S/S) increased, achieving the maximum at 20:1 and declining afterward at higher levels. Hence, 20:1 was considered the best ratio for subsequent process parameters. The range of 10–30 (v/w) was then used to refine the process parameters through response surface methodology (RSM) optimization. Choosing the appropriate ratios can have significant difficulty in MAE. This decision is typically influenced by various factors, including the solvent's selectivity toward the target analyte, its ability to absorb microwaves, its interaction with the sample matrix, and its compatibility with the analytical methods used downstream [48].

3.4 Matrix Characteristics

MAE depends on the type of plant utilized as a raw material, which can produce a variety of valuable compounds as well as the composition of the chosen plant tissue/cell or part of the plant that incorporates different kinds of components. Moreover, bioactive and nutraceutical compounds are typically bound to other compounds within plant structures, such as polyphenols, which are uncommonly found in their unbound form. Instead, they are often covalently linked to the plant cell wall, may exist in waxes or on the exterior surfaces of plant organs, and are linked via glycosides [49]. For example, plants' leaves contain high content of phenols [50]. Moreover, Rahmawatii et al. [1] reported that the yield of extraction from Pegagan (*Centella Asiatica* L.) leaves is significantly impacted by the quantity of material present. Also, the particle size of the plant matrix is an important factor [51]. Several studies reported that the extraction yield improved when matrix particle size decreased [51–53]. According to Poureini et al. [52], the apigenin extraction yield was enhanced by decreasing the particle size from 0.75 to 0.10 mm. A similar trend was depicted by Patil et al. [53]. These authors detected an optimal range of particle size

between 0.150 and 0.212 μm for curcuminoids extraction. Thus, fine matrix particles promote the deeper penetration of the microwave [38]. This improvement can be explained by the raise of the contact area between the solvent and the plant matrix. In fact, reducing the size of the particles decreased the distance that the solvent needs to diffuse, which in turn accelerated the rate of mass transfer between the solute and the solvent [54].

The level of moisture in the sample matrix affects the extraction efficiency. The presence of water can increase the microwave-absorbing ability of the sample and facilitate heating by making the extractant more polar [40]. By utilizing, the RSM combined with a Box–Behnken design to extract essential oil from *Cinnamomum camphora* leaves by MAE, Liu et al. [5] showed that the optimal moisture content was found to be 60%.

3.5 Temperature

Increasing temperature until a certain level increases the extraction yield of some bioactive compounds. In fact, Zhao et al. [55] mentioned that the impact of extraction temperatures was examined while holding other variables constant (30% ethanol, 30 mL/g, 30 min, 500 W). As the temperature increased (20–50 °C), there was a significant increase in TPC value from 23.88 to 34.46 mg GAE/g DW. Elevated temperatures have the potential to accelerate intermolecular interactions and molecular movement, which may lead to increased solubility of solutes in the solvent [43]. Therefore, the TPC value depicted a marked improvement as the extraction temperature increased from 40 to 50 °C, but subsequently decreased as the temperature continued to rise. Kapoore et al. [48] noticed that an increase in temperature resulted in decreased yields of phycoerythrin, which confirmed that thermal damage can occur over 40 °C, while carotenoids degrade at temperatures over 60 °C. The extraction of phenolic acids from green tea was found to be more effective at a temperature of 100 °C, whereas the flavanols and flavonols, which are sensitive to high temperatures, displayed better extraction yield at a lower temperature of 80 °C [56]. In addition, according to these authors, the extraction of quercetin glycosides is more efficient at 80 °C compared to 100 °C. This finding could be explained by the fact that quercetin glycosides have an oxidizable catechol ring (B-ring), making them more susceptible to thermal degradation than kaempferol glycosides, which have a mono-phenolic B-ring. Moreover, when extracted using MAE at a temperature of 90 °C, the sulfated polysaccharides obtained from *Ulva prolifera* using an acidic solvent (0.05 M HCl) exhibited superior water- and oil-holding capacities. Conversely, the polysaccharides extracted at a higher temperature of 150 °C demonstrated the best foaming properties as well as the highest antioxidant and pancreatic lipase inhibition activities [57].

4 Some Techniques of MAE

Coupling MAE with other extraction methods was proved to have potential applications due to the popular effectiveness of MAE (Fig. 2).

4.1 Solvent-Free MAE (SFM)

This method involves using microwaves to perform a dry distillation on a fresh matrix, without adding any water or organic solvent. The process involves heating of the raw material with water to release the essential oil from glands, which is then carried away by steam produced from the matrix water. The distillate, made up of water and essential oil, is continuously condensed using a cooling system placed outside the microwave oven. Any excess water is returned inside the balloon to maintain the appropriate humidity level of the matrix [21]. This straightforward approach allows the efficient extraction of essential oils without the use of additional solvents. The findings of Iftikhar et al. [22] revealed that the SFM technique, which does not require solvents and utilizes a power setting of 480 W and a duration of 60 s, is an efficient approach for extracting antioxidant compounds from gourd fruit. Likewise, Wei et al. [15] mentioned that the combination of SFM and moisture regulation was a potent approach to extract essential oil from deciduous leaves of *C. longepaniculatum*. In addition, compared to conventional hydro-distillation, Liu et al. [5] depicted that SFM exhibited better performance in terms of various parameters such as extraction efficiency (3.51% in 23 min vs. 3.35% in 240 min), initial extraction rate (3.3772 vs. 0.1868), extraction rate constant (0.3002 vs. 0.0152), extraction capacity (3.67% vs. 3.51%), oxygenated compound content (83.93% vs. 74.81%), energy

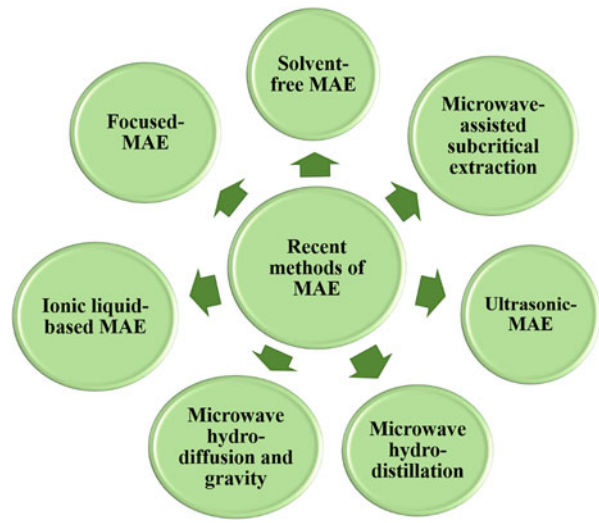

Fig. 2 Recent methods of MAE

consumption (0.22 kW h vs. 4 kW h), and environmental impact (177.87 g CO_2 vs. 3200 g CO_2). These findings demonstrated that SFM is a time-efficient, energy-saving, and eco-friendly method that has great potential as a preferable alternative to traditional methods for the extraction of essential oil from *C. camphora* leaves. Hence, SFM was suggested as it showed higher yield and volumetric mass transfer coefficient, greater proportions of oxygen compounds, lower electricity consumption, and less CO_2 emission and water waste compared to conventional hydro-distillation [77].

4.2 Focused-MAE (FMAE)

In FMAE, the sample is placed in an opened vessel and a specific area is exposed to microwave radiation. This system functions at atmospheric pressure [78], while the maximum temperature is provided by the boiling point of the extraction solvent utilized [79]. Hence, it can be used for the extraction of thermolabile components. In addition, this system is composed of a condenser that is set on the top of the vessel to avoid the loss of volatile compounds [80]. Therefore, the microwave reactor's configuration influences heat production in the reaction medium [21]. In fact, using a central composite experimental design, the extraction of betulinic acid from *Zizyphus joazeiro* was optimized by employing FMAE technology. This analysis confirms the applicability of FMAE extraction as a speedy, environmentally friendly, and effective extraction method. As per the study, the optimal temperature and duration of extraction are 70 °C and 15 min, respectively [81]. By directing microwave energy to a small region of the sample [82], FMAE attained a more efficient extraction with less energy consumption [81].

4.3 Ionic Liquid-Based MAE (ILMAE)

The merging of microwave irradiation with ionic liquids (ILs) presents an influential approach toward achieving high effectiveness and less harmful procedures. ILs are liquefied salts that retain their liquid form at low temperatures, frequently under 100 °C, and they are comprised of organic cations and organic or inorganic anions [83]. In comparison to conventional organic solvents, ILs exhibit numerous distinguishing characteristics, such as trivial vapor pressure, elevated temperature stability, low volatility, chemical stability, wide electrochemical stability window, and ionic conductivity [84]. Thus, ILs are considered as outstanding microwave absorbers. As stated by Li et al. [85], ILMAE can enhance the extraction efficiency of total biflavonoids in a shorter time and with a reduced amount of solvents, compared to conventional soxhlet extraction. In fact, according to Motlagh et al. [6], when compared to the commonly used conventional Soxhlet method, the protein yield obtained under optimized conditions using choline acetate ([Ch][Ac])-mediated water-based MAE technique (26.35%) is much higher, indicating the superiority of this approach over the Soxhlet extraction method (0.63%). The results indicated that [Ch]

[Ac]-based MAE of proteins from *Nannochloropsis oceanica is* superior to the conventional method of Soxhlet methods, making it a highly recommended innovative approach for protein separation. The findings of this investigation had the potential to aid in identifying and utilizing important biochemical compounds from microalgae through IL-based MAE, leading to the development of new and enhanced bioproduct technologies. Furthermore, the major obstacle in astaxanthin extraction is the effective disruption of the thick and resistant cell walls of *Haematococcus pluvialis*. However, the utilization of biocompatible protic ionic liquids-based microwave-assisted liquid-solid extraction (PILs-MALSE) has resolved this issue in the study of Fan et al. [7]. One of the protic ionic liquids, ethanolammonium caproate (EAC), has the ability to dissolve mannan, which is one of the key components of the cell walls of *Haematococcus pluvialis*. Fan et al. [7] elucidated that compared to traditional extraction techniques, the PILs-MALSE method is more efficient for extracting astaxanthin. In addition, the effectiveness of MAE combined with protic ionic liquids (PILs) in obtaining phycobiliproteins was assessed by Rodrigues et al. [24]. The most efficient solvent was a combination of 2-hydroxyethylammonium acetate (2-HEAA) and 2-hydroxyethylammonium formate (2-HEAF), using a process conducted at 62 W power and a ratio of 10 mL/g. These authors found that MAE using PILs could be effective for the extraction of phycobiliproteins with concentrations of 33 g L^{-1}, 0.84 g L^{-1}, and 0.41 g L^{-1} of allophycocyanin, phycocyanin, and phycoerythrin, respectively. Guo et al. [86] successfully utilized ILMAE to extract 6-, 8-, and 10-gingerols and 6-, 8-, and 10-shogaols from ginger. The highest extraction yields of gingerols and shogaols were obtained using 1-decyl-3-methylimidazolium bromide [C10MIM]Br. The efficiency of extraction of these compounds was greatly influenced by the alkyl chain length and anions of cations. Compared to methanol-based MAE (MMAE), ILMAE not only produced higher extraction yields but also had a shorter extraction time. The same tendency was observed when 1-octyl-3-methylimidazolium acetate [Omim][OAc] was added to the water as the extracting solvent. This IL has been demonstrated to enhance lipid extraction ability when exposed to microwave irradiation. [Omim][OAc] at 2.5% allowed the extraction of 19.2% of lipids [87]. Despite many accomplishments, there is still insufficient knowledge about the precise mechanism linking microwaves, ILs, and nanostructures or polymers [83].

4.4 Ultrasonic MAE (UMAE)

The concurrent utilization of ultrasonic and microwave extraction methods led to a notably greater quantity of bioactive compounds in comparison to the traditional decoction extraction techniques, highlighting the synergistic effects of these novel approaches (Kwansang et al. 2022). As reported by Sun et al. [12], the

UMAE approach, developed for the extraction of polysaccharides from *Camptotheca acuminata* fruit (CAFP), yielded higher amounts in a shorter duration than traditional hot water extraction (HWE) techniques. The CAFP yield obtained via UMAE was 6.81%, which is 1.04 times greater than the yield from HWE. Furthermore, the UMAE approach required a shorter extraction time of 20 min compared to HWE, which necessitated 120 min of extraction. In this line, Zheng et al. [88] indicated that the extraction yield of polysaccharides from *Trametes orientalis* was 7.52%. In addition, the investigation of Zhang et al. [13] revealed that the research results showed that UMAE had a greater degree of damage to the cell wall of *Dictyophora indusiata* polysaccharides (DPs) and better antioxidant capacity. The UMAE method had the highest polysaccharides yield, which was related to the conformational stretching and degradation avoidance of DPs in the higher molecular weight components under the simultaneous action of microwave and ultrasonic. In the same line, according to Shen et al. [89], *Panax notoginseng* polysaccharides (PNPS) were extracted using UMAE, and RSM was utilized to optimize the extraction parameters. The ideal extraction conditions were identified as 10 min ultrasonic duration, 50 W ultrasonic power, 4 min microwave duration, and 540 W microwave power, which led to a PNPS extraction rate of 11.03%. Characterization of PNPS using SEM, FTIR, and UV-Vis indicated that the UMAE method did not cause any degradation to the polysaccharides.

The findings of Xu et al. [90] suggested that UMAE resulted in a greater yield of pectin compared to conventional heating. The ideal parameters for UMAE were identified as an extraction temperature of 86 °C, an extraction time of 29 min, and a solid-liquid ratio of 1:48 (w/v), resulting in a maximum pectin yield of 21.5%. The study of Lu et al. [91] aimed to optimize the extraction of various degrees of polymerized oligosaccharides from lotus seeds using UMAE through RSM. The results demonstrated that the optimal UMAE conditions for lotus seed oligosaccharides were determined to be an extraction time of 325 s, a liquid-solid ratio of 10.00 mL/g, ultrasonic power of 300.46 W, and microwave power of 250 W. These conditions resulted in a 76.59% increase in the yield of total oligosaccharides, with a 17.47% increase in trisaccharides and a 27.21% increase in tetrasaccharides. Additionally, the extraction time was significantly reduced compared to traditional hot water, ultrasonic-assisted, and MAE methods. Regarding oil extraction, Wang et al. [92] found that the utilization of UMAE resulted in higher oil yield and greater superoxide radical scavenging activity of white pepper compared to MAE and UAE, indicating its superior efficiency as an extraction method. In terms of the specific components extracted, UMAE generally yielded more monoterpenes and sesquiterpenes than MAE and UAE. Therefore, UMAE has the potential to become a prominent

eco-friendly method for extracting essential oil from *P. nigrum* due to its maximum extraction yields and short extraction time. Furthermore, Yu et al. [93] employed the UMAE method to extract polyphenols, flavonoids, triterpenoids, and vitamin C from *Clinacanthus nutans*. The optimized conditions for the extraction process included the use of distilled water, a solid-liquid ratio of 1:55 g/mL, an irradiation power of 90 W, and an extraction cycle lasting 75 s. With the previously described conditions, the extraction yields of polyphenols, flavonoids, triterpenoids, and vitamin C were found to be 8.893, 25.936, 16.789, and 0.166 mg/g, respectively. These findings suggest that UMAE is a highly efficient method for the extraction of bioactive substances from *Clinacanthus nutans*. Overall, these findings suggest that UMAE has the potential to be a more efficient and effective technique for bioactive compounds extraction as compared to traditional methods.

4.5 Microwave Hydro-distillation (MHD)

The mechanism of MHD produces heat by absorbing microwave radiation from the plant material, resulting in the evaporation of essential oil components [21]. The condensed vapor is then collected as a liquid that consists of essential oil. Newer research has examined the possibility of MHD for extracting essential oils from diverse aromatic plants [94–97]. Elyemni et al. [97] compared the efficiency of two extraction methods, microwave-assisted hydro-distillation (MHD) and Clevenger hydro-distillation (CH), for obtaining essential oils from *Rosmarinus officinalis* L. MAH only requires 20 min to obtain the same yield of essential oils that takes CH 180 min. Furthermore, the quality of the essential oil was enhanced by an increase of 1.14% in oxygenates. Additionally, as reported by Megawati et al. [95], the utilization of microwave-assisted hydro-distillation (MHD) for the extraction of mace essential oil was proven to be more effective than hydro-distillation (HD). MHD resulted in 8.62% essential oils in just 42 min, whereas HD only produced 7.03% in 73 min. In addition, MHD consumed less energy (756 kJ) compared to HD (1095 kJ). As the power input is increased, a higher yield of essential oil is obtained. At 300, 600, and 800 W within 10 min, yields obtained were 2.68, 4.56, and 5.41%, respectively, while at 20 min, yields obtained were 5.13, 7.39, and 6.83%. The findings of Mollaei et al. [25] devoted that MHD may be a useful technique for extracting essential oil from *F. angulata* due to its ability to decrease the distillation duration, minimize energy usage, and enhance biological properties when compared to the HD method.

4.6 Microwave Hydro-diffusion and Gravity (MHG)

The MHG apparatus is essentially a microwave unit that operates similarly to a standard commercial model. It utilizes a combination of microwave radiation and the force of gravity at ambient pressure to perform extraction from fresh plant material [98]. As described

by De Castro and Peinado et al. [54], this method involved the introducing of a matrix into a reactor inside a microwave oven. Microwaves trigger the heating of the water present in the matrix, leading to the elimination of cells that contain essential oil. The hydro-diffusion process took place, wherein both the essential oil and internal water of the matrix were released from the plant's interior to its exterior. To condense the distillate, a cooling system situated outside the microwave oven is employed and then collected under gravity. In addition, MHG extraction has demonstrated optimal outcomes in diverse applications aimed at extracting active compounds, including antioxidant molecules from aromatic plants such as *Cuminum cyminum*, *Cytisus scoparius*, *Brassica rapa*, *Quercus robur*, and *Pleurotus ostreatus* [17, 26, 99]. As detailed by Benmoussa et al. [99], the use of MHG resulted in a higher yield of essential oil (1.579%) in a shorter time (16 min vs. 150 min required for HD) as compared to conventional hydro-distillation (HD) (1.550%). Additionally, the MHG technique requires less electricity, emits less carbon dioxide, and generates less wastewater. Examination via GC-MS affirmed that the quality of cumin essential oils procured by MHG and HD were comparable. According to this investigation, the effectiveness of MHG and ethanolic solid-liquid extraction methods were compared using various plant sources, that is, *Cytisus scoparius*, *Brassica rapa*, *Quercus robur*, and *Pleurotus ostreatus* [17] (Table 2). The results illustrated that MHG technology is suitable for generating extracts with interesting antioxidant characteristics.

4.7 Microwave-Assisted Subcritical Extraction (MASE)

By combining microwave-assisted and subcritical water extraction (MASE), Moirangthem et al. [11] found that anthocyanins could be extracted from straw with an 85.8% efficiency when exposed to a temperature of 90 °C for 5 min. This combination was also observed to have superior antioxidant activity as compared to a conventional methanol extract. Both the straw and bran's microwave extracts did not exhibit any noticeable cytotoxicity on Jurkat cells in vitro.

In addition, Yang et al. [27] used MASE to extract steviol glycosides from *Stevia rebaudiana* (Bertoni). The results depicted that the yields of major steviol glycoside, including rebaudioside A and stevioside, and rebaudioside C were comparable to those obtained by the conventional extraction method that used 70% ethanol under sonication for 45 min, within just 1 min of reaching subcritical water conditions at 140 °C. This method can be a cost-effective alternative for producing high-purity steviol glycoside sweeteners. Moreover, Cai et al. [28] attempted to enhance oil extraction efficiency by utilizing seed pretreatments, such as microwave assistance, to increase subcritical extraction fluid penetration without affecting the physicochemical properties of the phytochemicals in oilseeds. These authors established that using

Table 2
MAE methods for bioactive compounds extraction

Methods	Compounds	Plants	References
Solvent-free MAE	TPC/TFC Essential oil TPC Essential oil	*Lagenaria siceraria* fruit *Coleus aromaticus* *Centella asiatica L.* *Cinnamomum longepaniculatum* leaves	[22] [16] [1] [15]
FMAE (Focused MAE)	Betulinic acid Sesamol	*Zizyphus joazeiro* bark Sesame seed	[81] [23]
ILMAE (Ionic liquid-based MAE)	Lipids and eicosapentaenoic acid Flavonoids Quercetin Heneicos-1-ene Phycobiliproteins Amentoflavone/hinokiflavone Astaxanthin	*Nannochloropsis oceanica* Passion fruit and mango leaves *Nothopanax scutellarium* leaves Coriander foliage *Arthrospira platensis* *Selaginella sinensis* *Haematococcus pluvialis*	[101] [102] [103] [104] [24] [105] [7]
Ultrasonic MAE	Polysaccharides Caffeic and ferulic acids Polysaccharides α-mangostin Polysaccharides	*Trametes orientalis* *Clinacanthus nutans* *Camptotheca acuminata* fruits *Garcinia mangostana* *Pericarp* *Dictyophora indusiata*	[88] [106] [12] [107] [13]
Microwave hydro-distillation (MHD)	Essential oils	*Rosmarinus officinalis* L. Clove (*Syzgium aromaticum*) stem *Myristicae arillus* *Ferulago angulate* *Pelargonium graveolens*	[97] [96] [95] [25] [94]
Microwave hydro-diffusion and gravity (MHG)	Essential oil Phenolic compounds Carotenoid/phenolic/lipid/protein contents	*Cuminum cyminum* L. Blackberries (*Rubus* spp.) *Cytisus scoparius;Brassica rapa; Quercus robur; Pleurotus ostreatus*	[99] [26] [17]
Microwave-assisted subcritical extraction	Anthocyanins Steviol glycosides Oil Berberine hydrochloride	*Manipur black* rice *Stevia rebaudiana* leaves Tigernut *Berberis aristata* roots	[11] [27] [28] [100]

microwaving as a pretreatment prior to subcritical extraction is an uncomplicated technique to improve oil output while producing high-quality oil. In fact, Cai et al. [28] employed subcritical n-butane extraction with the aid of microwave pretreatment to extract tigernut oil from tigernut meal. Microwaving (560 W, 6 min) substantially increased subcritical extraction efficiency. The most oil was obtained by subcritical extraction of tigernut oil at a temperature of 52 °C for 32 min after three extraction cycles, with a

liquid-solid ratio of 3.62 kg/(kg of tigernut meal), resulting in a maximum yield (24.736%) of tigernut oil. The oil's high-quality attributes, including a ratio of unsaturated to saturated fatty acids of 4.68 UFA/SFA, low acid value (3.30 mg KOH/g oil), low peroxide value (0.28 meq.kg^{-1}), and a predominance of oleic acid, were identified. Additionally, an optimization of the conditions for extracting berberine from *Berberis aristata* roots by microwave-assisted subcritical water extraction (MASCW) was conducted by Manikyam et al. [100]. This method was employed to extract berberine at temperatures ranging from 110 to 170 °C using various combinations of five subcritical parameters. The experimental data concentration of berberine (223.82 μg/mL) was found to be significantly correlated under specific subcritical parameters. This new hybrid extraction technique can be an eco-friendly option for producing high-purity compounds [27].

5 Conclusion

The MAE has minimized energy, time, and solvent consumption, which makes it a sustainable technology. Furthermore, it has been coupled with other extraction techniques such as ultrasonic, hydrodistillation, and subcritical extraction. These combinations had considerably reduced energy and time, and improved bioactive compound yields. The versatility of MAE in the recovery of bioactive and nutraceuticals from various kinds of vegetal materials could be applied in advanced practical applications in food and pharmaceutical fields.

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