Chapter 2
Fundamentals of Microwave Extraction

Priscilla C. Veggi, Julian Martinez, and M. Angela A. Meireles

2.1 Basic Principles

2.1.1 Mechanism of Microwave Extraction

The fundamentals of the microwave extraction (MAE) process are different from those of conventional methods (solid–liquid or simply extraction) because the extraction occurs as the result of changes in the cell structure caused by electromagnetic waves.

In MAE, the process acceleration and high extraction yield may be the result of a synergistic combination of two transport phenomena: heat and mass gradients working in the same direction [1]. On the other hand, in conventional extractions the mass transfer occurs from inside to the outside, although the heat transfer occurs from the outside to the inside of the substrate (Fig. 2.1). In addition, although in conventional extraction the heat is transferred from the heating medium to the interior of the sample, in MAE the heat is dissipated volumetrically inside the irradiated medium.

During the extraction process, the rate of recovery of the extract is not a linear function of time: the concentration of solute inside the solid varies, leading to a nonstationary or unsteady condition. A series of phenomenological steps must occur during the period of interaction between the solid-containing particle and the solvent effectuating the separation, including (1) penetration of the solvent into the solid matrix; (2) solubilization and/or breakdown of components; (3) transport of the solute out of the solid matrix; (4) migration of the extracted solute from the external surface of the solid into the bulk solution; (5) movement of the

P.C. Veggi (✉) • J. Martinez • M.A.A. Meireles
LASEFI/DEA/FEA (School of Food Eng.)/UNICAMP (University of Campinas),
R. Monteiro Lobato, 80, Campinas, SP 13083-862, Brazil
e-mail: pveggi@gmail.com; julian@fea.unicamp.br; meireles@fea.unicamp.br

F. Chemat and G. Cravotto (eds.), *Microwave-assisted Extraction for Bioactive Compounds: Theory and Practice*, Food Engineering Series 4,
DOI 10.1007/978-1-4614-4830-3_2,

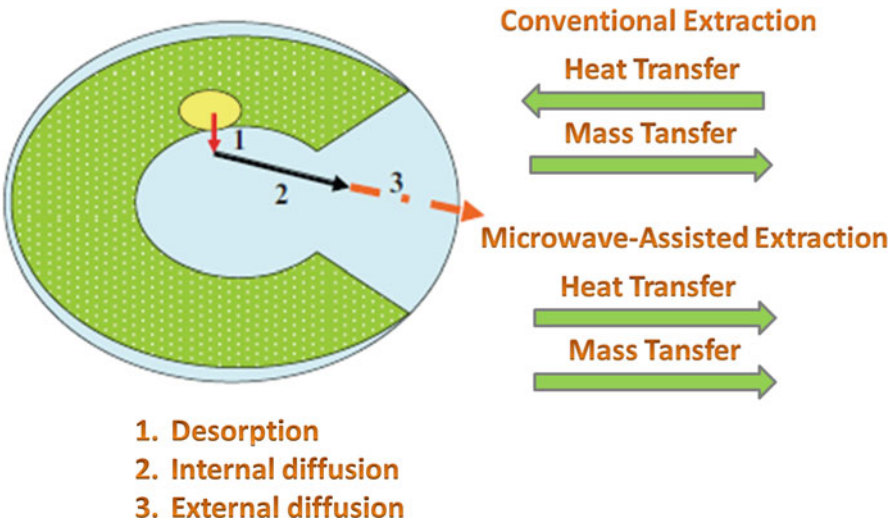

Fig. 2.1 Basic heat and mass transfer mechanisms in microwave and conventional extraction of natural products. (Adapted from Périno-Issartier et al. [2])

extract with respect to the solid; and (6) separation and discharge of the extract and solid [3].

Therefore, the solvent penetrates into the solid matrix by diffusion (effective), and the solute is dissolved until reaching a concentration limited by the characteristics of the solid. The solution containing the solute diffuses to the surface by effective diffusion. Finally, by natural or forced convection, the solution is transferred from the surface to the bulk solution (Fig. 2.2).

The extraction process takes place in three different steps: an equilibrium phase where the phenomena of solubilization and partition intervene, in which the substrate is removed from the outer surface of the particle at an approximately constant velocity. Then, this stage is followed by an intermediary transition phase to diffusion. The resistance to mass transfer begins to appear in the solid–liquid interface; in this period the mass transfer by convection and diffusion prevails. In the last phase, the solute must overcome the interactions that bind it to the matrix and diffuse into the extracting solvent. The extraction rate in this period is low, characterized by the removal of the extract through the diffusion mechanism. This point is an irreversible step of the extraction process; it is often regarded as the limiting step of the process [5].

Many forces, such as the physicochemical interactions and relationships, can be exposed during the extraction (dispersion forces, interstitial diffusion, driving forces, and chemical interactions), and the persistence and strength of these phenomena may be closely tied to the properties of the solvent (solubilization power, solubility in water, purity, polarity, etc.) [6].

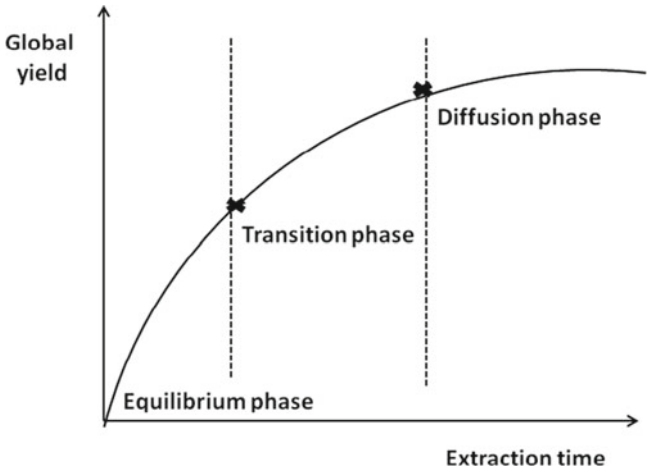

Fig. 2.2 Schematic representation of yield versus time in extraction processes. (Adapted from Raynie [4])

2.1.2 Mechanism of Microwave Heating

In the microwave heating process, energy transfer occurs by two mechanisms: dipole rotation and ionic conduction through reversals of dipoles and displacement of charged ions present in the solute and the solvent [7, 8]. In many applications these two mechanisms occur simultaneously. Ionic conduction is the electrophoretic migration of ions when an electromagnetic field is applied, and the resistance of the solution to this flow of ions results in friction that heats the solution. Dipole rotation means rearrangement of dipoles with the applied field [8].

Energy transfer is the main characteristic of microwave heating. Traditionally, in heat transfer of the conventional process, the energy is transferred to the material by convection, conduction, and radiation phenomena through the external material surface in the presence of thermal gradients. In contrast, in MAE, the microwave energy is delivered directly to materials through molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy [9].

The most important properties involved in microwave processing of a dielectric are the complex relative permittivity (ε) and the loss tangent (tan δ) [10, 11]:

$$\varepsilon = \varepsilon' - j\varepsilon'' \tag{2.1}$$

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{2.2}$$

where

$$j = \sqrt{-1} \tag{2.3}$$

Table 2.1 Physical constants and dissipation factors for solvents usually used in microwave-assisted extraction (MAE) [14, 15]

Solvent	Dieletric constant,[a] ε'	Dissipator factor tan δ ($\times 10^{-4}$)	Boiling point,[b] (°C)	Viscosity,[c] (cP)
Acetone	20.7	5,555	56	0.30
Acetronitrile	37.5		82	
Ethanol	24.3	2,500	78	0.69
Hexane	1.89		69	0.30
Methanol	32.6	6,400	65	0.54
2-Propanol	19.9	6,700	82	0.30
Water	78.3	1,570	100	0.89
Ethyl acetate	6.02	5,316	77	0.43
Hexane–acetone (1:1)			52	

[a]Determined at 20°C
[b]Determined at 101.4 kPa
[c]Determined at 25°C

The material complex permittivity is related to the ability of the material to interact with electromagnetic energy, whereas ε' is the real part, or *dielectric constant*, and ε'' is the imaginary part, or *loss factor*. The dielectric constant determines how much of the incident energy is reflected at the air–sample interface and how much enters the sample (for vacuum, $\varepsilon' = 1$); the loss factor measures the efficiency of the absorbed microwave energy to be converted into heat [12]. The loss tangent (tan δ or dielectric loss) is the most important property in microwave processing; it measures the ability of the matrix to absorb microwave energy and dissipate heat to surrounding molecules, being responsible for the efficiency of microwave heating [12, 13] As a result, a material with high loss factor and tan δ combined with a moderate value of ε' allows converting microwave energy into thermal energy.

The first factor one must consider when selecting microwave physical constants is the solvent to be used. It is important to select a solvent with high extracting power and strong interaction with the matrix and the analyte. Polar molecules and ionic solutions (typically acids) strongly absorb microwave energy because of the permanent dipole moment. On the other hand, when exposed to microwaves, nonpolar solvents such as hexane will not heat up.

The degree of microwave absorption usually increases with the dielectric constant. In Table 2.1, the physical parameters, including dielectric constant and dissipation factors, are shown for commonly used solvents. A simple comparison between water and methanol shows that methanol has a lesser ability to obstruct the microwaves as they pass through but has a greater ability to dissipate the microwave energy into heat [8]. The higher dielectric constant of water implies a significantly lower dissipation factor, which means that the system absorbs more microwave energy than it can dissipate. This phenomenon is called *superheating*: it occurs in the presence of water in the matrix. This strong absorption provides an increase of the temperature inside the sample, leading to the rupture of cells by the in situ water. In some cases it can promote the degradation of the target compound or an "explosion" of solvent, and in

other cases it can increase the diffusivity of the target compound in the matrix [16]. Therefore, the microwave power must be sufficient to reach the boiling point of the water or other solvent, setting the separation temperature.

The second factor to be considered is the solid matrix. Its viscosity affects its ability to absorb microwave energy because it affects molecular rotation. When the molecules are "locked in position" as viscous molecules, molecular mobility is reduced, thus making it difficult for the molecules to align with the microwave field. Therefore, the heat produced by dipole rotation decreases, and considering the higher dissipation factor (δ), the higher is this factor, the faster the heat will be transferred to the solvent [11].

2.1.3 Heat Transfer in Microwave Heating

When the system is subjected solely to heating, then Eq. (2.4) can be solved by itself. Thus, the initial condition needed to determine the unique solution of Eq. (2.4) is the initial temperature of the system, given as

$$\mathrm{T(x,\ y,\ z,\ t)}\big|_{\mathrm{t=0}} = \mathrm{T_0}\left(\mathrm{x,\ y,\ z}\right) \tag{2.4}$$

The convective boundary condition at the material surfaces is given by Newton's law of cooling and is used as follows:

$$\mathrm{h}\left(\mathrm{T_a} - \mathrm{T}\big|_{n=a}\right) = \mathrm{k_t}\,\frac{\partial T}{\partial n}_{n=a} \tag{2.5}$$

And, the adiabatic boundary condition applied in the center of the substrate particles is

$$\left.\frac{\partial T}{\partial n}\right|_{n=0} = 0 \tag{2.6}$$

where n is the specific dimension, a is the boundary position, h is the convective heat transfer coefficient, $\mathrm{k_t}$ is the thermal conductivity, and $\mathrm{T_a}$ is the temperature of the surrounding air.

Considering a transient heat transfer in an infinite slab, for one-dimensional flux, the corresponding equation is

$$\frac{\partial^2 T}{\partial x^2} + \frac{q''}{k_t} = \frac{1}{\alpha}\frac{\partial T}{\partial t} \tag{2.7}$$

where x is the heat flux direction, q'' is the heat generation, $\mathrm{k_t}$ is the thermal conductivity, and α is the thermal diffusivity.

Food materials are, in general, poor electric insulators. They have ability to store and dissipate electric energy when subjected to an electromagnetic field. Microwave energy in itself is not thermal energy. The heating is a result of the electromagnetic energy generated with the dielectric properties of the material combined with the electromagnetic field applied. Dielectric properties play a critical role in determining the interaction between the electric field and the matrices [17]. The rate of conversion of electrical energy into thermal energy in the material is described by Chen et al. [18]:

$$P = K.f\,\varepsilon' E^2 \tan\delta \tag{2.8}$$

where P is the microwave power dissipation per volume unit, K is a constant, f is the frequency applied, ε' is the absolute dielectric constant of the material, E is the electric field strength, and tan δ is the dielectric loss tangent.

The distribution of the electric field depends on the geometry of the irradiated object and its dielectric properties. The depth of penetration of a wave (Dp) can also have an important role in the choice of the working frequency and depends on the thickness of the matrix being treated. The energy absorption inside the solid material causes an electric field that decreases with the distance from the material surface. The penetration depth (Dp) is the distance from the material surface where the absorbed electric field (ε) is reduced to $1/\varepsilon$ of the electric field at the surface: this corresponds to an energy loss of about 37% [19]. The penetration depth is inversely proportional to the frequency and the dielectric properties of the material, as shown by the following expression [20]:

$$Dp = \frac{c}{2\pi f'\sqrt{2\varepsilon'}\left[\sqrt{1+\tan^2\delta}-1\right]^{1/2}} \tag{2.9}$$

where c is the speed of light (m/s). This equation is approximated by the following (Eq. (2.24)), when tan $\delta << 1$, which is usually the case:

$$d = \frac{\lambda_0\sqrt{\varepsilon_r'}}{2\pi\varepsilon_r''} \tag{2.10}$$

where λ_0 is the wavelength in vacuum and d the approximate penetration depth.

The depth of penetration varies inversely with the loss factor and is even less when the product is sensitive to microwaves. If the penetration depth of the microwave is much less than the thickness of the material, only the surface is heated, and the rest of the material is heated by conduction. For transparent media, that is, a loss factor <0.01, the depth of penetration is not problematic and will dissipate the energy. The presence of a standing wave will induce the creation of "hot spots" where the power dissipated exceeds the heat transfer to cooler areas of the environment.

2.2 Heat and Mass Transfer: Balance Equations and Kinetics

Plant materials can be considered as porous media because of their similarities to solid food that can be treated as hygroscopic and capillary-porous [21]. According to Datta [22] the distinction between porous and capillary-porous is based on the presence and size of pores. Generally, porous materials have pores $\geq 10^{-7}$ m, whereas for capillary-porous materials the pores are $\leq 10^{-7}$ m. The presence of pores makes the water transport in these systems more intricate, because, in addition to the contribution of molecular diffusion, the transport within the pores is also caused by Knudsen diffusion (mean free path of molecules is long compared to the pore size), surface diffusion, and hydrodynamic flow [21].

Considering the extraction process, microwaves are generally used in two situations: (1) MAE that can be treated as a solid–solvent extraction, in which case the equations developed by Takeuchi et al. [23] can be used, and (2) solvent-free MAE (SFMAE), which can be treated as a two-step process in which in the first step, system temperature in any given location is less than that of water evaporation, and in the second step, the temperature at any given location is equal to the boiling temperature; thus, the electromagnetic energy is entirely used to evaporate the water.

2.2.1 Heat and Mass Balance Equations for Solid–Liquid MAE

The mass transfer equations for solid–liquid extraction were presented by Takeuchi et al. [23] for an isothermal process. The factors that control the extraction of a solute from a matrix using MAE are the mass transfer rate of the solute from the matrix to the solution phase and the strength of solute–matrix interactions. Although the solubility of the solute in the solvent is recurrently indicated as a limiting factor, it should not be so because the solvent-to-solid ratio is large enough to assure that the extract–solvent mixture forms an infinite diluted solution.

The rate of dissolution of a solute into the extraction solvent is controlled by the mass transfer rate of the solute from the solid matrix into the liquid. The transfer of the solute inside the solid particle occurs because of the concentration gradient in the solid–liquid interface, and it can be characterized by the effective diffusion. The equation that describes this phenomenon is based on Fick's law:

$$\frac{N_C}{A_T} = -D_{BC}\frac{dC_C}{dz} \tag{2.11}$$

where N_C is the rate of dissolution of the solute C in the solution (kg/s), A_T is the area of the solid–liquid interface (m^2), D_{BC} is the diffusivity of the solute in the solvent–inert solid (m^2/s), C_C is the concentration of solute C in the solution (kg/m^3), and z is the distance inside the porous part of the solid matrix (m). The minus sign gives a positive flux term because the gradient is negative (flow occurs down a concentration gradient, from high to low concentration).

Diffusion coefficient data are necessary to make calculations. Diffusivities may be determined experimentally or predicted. Orders of magnitude of diffusion coefficients (D_{BC}) for solids are 10^{-9} to 10^{-10} m^2/s. When concerned with impermeable porous solids with fluid-filled pores, the effective (or apparent) diffusion coefficient is used:

$$D_{CBeff} = \frac{\varepsilon}{\tau} D_{BC} \tag{2.12}$$

where ε is the void fraction or porosity of the solid and τ is the tortuousness of the pores.

On the surface of the solid particle, the transfer of the solute occurs simultaneously by molecular and turbulent transport. In this step, the mass transfer rate can be expressed by the following equation:

$$N_C = V \frac{VdC_C}{dt}\bigg|_S = A_T K_L \left(C_{CS} - C_C\right) \tag{2.13}$$

where K_L is the mass transfer coefficient (m/s), C_{CS} is the reference concentration of the solute C in the solid surface (kg/m^3), and C_C is the concentration of the solute C in the solution at time t (kg/m^3).

Integrating Eq. 2.14 from $t=0$ and $C_C = C_{C0}$ to $t=t$ and $C_C = C_C$, we obtain:

$$\int_{C_{C0}}^{C_C} \frac{dC_C}{C_{CS} - C_C} = \frac{Ak_L}{V} \int_{t=0}^{t} dt \tag{2.14}$$

$$\frac{C_{CS} - C_C}{C_{CS} - C_{C0}} = e^{-\left(\frac{k_L A}{V}\right)t} \tag{2.15}$$

If pure solvent is used initially, $C_{C0}=0$, and then

$$1 - \frac{C_C}{C_{CS}} = e^{-\left(\frac{k_L A}{V}\right)t} \tag{2.16}$$

$$C_C = C_{CS}\left(1 - e^{-\left(\frac{k_L A}{V}\right)t}\right) \tag{2.17}$$

2.2.2 Heat and Mass Balance Equations for SFMAE

In order to formulate the heat and mass balance, material will be considered, as suggested by Navarrete et al. [24], as a capillary-porous media that includes the insoluble solids, bound and free water, and air. Heat is generated and conducted in

the capillary-porous medium. The vapor phase forms an homogeneous system, and heat convection can be neglected. Steam is removed from the system instantaneously, that is, no diffusion or convection was considered. The evaporation of water consumed all heat generated in the system. MAE is considered to be performed in a fixed bed formed by the plant material packed inside the extraction vessel. During the extraction, system temperature will be equal to or less than the boiling temperature. So long as the temperature in a given location of the bed did not reach the boiling temperature, the general heat transfer equation or the thermal conduction equation can be used to estimate the heat transfer flux and describes the space and time behavior of the temperature field [24]:

$$\rho_S C_P \frac{\partial T}{\partial t} - \nabla \bullet \left(K_t \nabla \mathrm{T}\right) = \mathrm{P} \tag{2.18}$$

where ρ_S represents the solid material apparent density (kg m^{-3}), C_P is the specific heat capacity (J kg^{-1} K^{-1}), and K_t is the thermal conductivity (A V^{-1} m^{-1}). $T = T$ (x, y, z, t) is the absolute temperature and P = P (x, y, z, t) is the microwave energy power dissipated per volume unit; this corresponds to the heat generated by the interaction between microwaves in the plant material. Note that the parameters ρ_S, C_P and K_t should be estimated for the lumped capillary-porous media as already described. The moisture content varies during the extraction process, and these parameters vary with the moisture of the system: for MAE these parameters are not constant. Nonetheless, for other systems in which only heating is the important phenomenon, these parameters are usually taken as constants that are independent of position, time, and temperature, which simplifies the solution of the heat transfer equation. According to Navarrete et al. [24], the time-average power dissipated in a plant material per unit volume can be calculated from

$$P = \frac{1}{2}(K_t + \omega \varepsilon_o \varepsilon'')\left|E\right|^2 \tag{2.19}$$

where ω is the angular frequency of the electromagnetic wave, ε_o is the vacuum permittivity (8.8542×10^{-12} F m^{-1}), ε is the dielectric loss factor, and E is the electric field (V m^{-1}).

After the system temperature has reached the boiling temperature, the energy generated will be used for the evaporation of water. Therefore, the evaporation rate will be given by Navarrete et al. [24]:

$$\frac{\partial C_w}{\partial t} = R_w \tag{2.20}$$

where C_w (kg m^{-3}) is the water concentration per unit volume of extractor vessel and R_w is the water evaporation rate (kg s^{-1} m^{-3})

So long as water is evaporating, the rate of evaporation can be estimated from Navarrete et al. [24]:

$$R_w = \frac{P}{\lambda_w} \tag{2.21}$$

Equations 2.6 and 2.7 were proposed by Navarrete et al. [24] to describe the SFME (solvent-free microwave extraction) of Lavandin essential oil. To solve Eqs. (2.5), (2.6), and (2.7), the authors estimated the system properties using the equations of Datta [21, 22], Navarrete et al. [24], and Sihvola [25]. The specific heat of the lumped system as a function of system moisture was estimated using [21]

$$C_P = \rho_S C_{PS}\left(1-\varphi\right) + \rho_w C_{Pw} \varphi S_w + \rho_g C_{Pg} \varphi (1 - S_w) \tag{2.22}$$

where C_{Pg}, C_{PS} and C_{Pw} are the air, insoluble solid, and water specific heat, and ρ_g, ρ_S and ρ_w are the air, insoluble solid, and water densities. S_w is the amount of water in pores and is generally referred to as the water saturation; it is calculated from [21]

$$S_w = \frac{M_w (1-\varphi)\rho_S}{(1-M_w)\varphi\rho_S} \tag{2.23}$$

where M_w is the plant material moisture, which is calculated from

$$M_w = \frac{C_w V}{C_w V + \left(1 - M_{wo}\right) m_o} \tag{2.24}$$

where M_{wo} and m_o are the initial moisture content of the plant material and the mass of feed, respectively. φ is the bed porosity and is calculated using [21]

$$\varphi = 1 - \frac{\rho_B (1 - M_w)}{\rho_S} \tag{2.25}$$

where ρ_B is the bed apparent density.

2.3 Important Parameters in Microwave-Assisted Extraction and Mechanism of Action

The optimization of MAE conditions has been studied in several applications. The efficiency of the process is directly related to the operation conditions selected. Special attention should be given to usually studied parameters that may influence the performance of MAE such as solvent composition, solvent-to-feed ratio, extraction temperature and time, microwave power, and the characteristics of the matrix including its water content. Comprehension of the effects and interactions of these factors on the MAE process is significant. Thus, this topic emphasizes some of the

parameters that affect MAE, presenting guidelines regarding the selection of proper operation conditions, and also discusses the interaction between these parameters.

2.3.1 Effect of Solvent System and Solvent-to-Feed Ratio (S/F)

The most important factor that affects MAE process is solvent selection. A proper solvent choice will provide a more efficient extraction process. Solvent selection depends on the solubility of the compounds of interest, solvent penetration and its interaction with the sample matrix and its dielectric constant [26], and the mass transfer kinetics of the process [27]. The solvent should preferably have a high selectivity toward the solutes of interest excluding undesired matrix components. Another important aspect is that the optimal extraction solvents cannot be selected directly from those used in conventional extractions: it depends on the capacity of the solvent to absorb the microwave energy and consequently heat up [7, 8, 13, 28].

In general, the capacity of the solvent to absorb microwave energy is high when the solvent presents high dielectric constant and dielectric loss [27]. Solvents that are transparent to microwaves do not heat when submitted to them. Hexane is an example of microwave-transparent solvent whereas ethanol is an excellent microwave-absorbing solvent [13, 29]. Both polar and nonpolar solvents can be used in MAE, and solvents such as ethanol, methanol, and water are sufficiently polar to be heated by microwave energy [30]. In this context, the properties of the solvent can be modified when combining different solvents, which allow varying the solvent selectivity for different compounds [30]. The addition of salts to the mixture can also increase the heating rate, because besides dipole orientation the ion conductivity is the main origin of polarization and corresponds to losses to heat in dielectric heating [27]. Studies have shown that small amounts of water in the extracting solvent make possible the diffusion of water into the cells of the matrix, leading to better heating and thus facilitating the transport of compounds into the solvent at higher mass transfer rates.

In the case of volatile compounds, the addition of a solvent with relatively low dielectric properties can be used to ensure that the solvent temperature is kept lower to cool off the solutes once they are liberated into the solvent [7]. Generally, hexane is used for the extraction of volatile oils [13]. In addition, the solvent-free MAE (SFMAE) process has been designed for aromatic herbs rich in volatile oils; in this case, the moisture content within the plant matrix itself serves for extraction and no solvent is used [29, 31].

Studies have reported that ethanol or water can be added into poor microwave absorbers, such as hexane, to improve the extraction efficiency. One of the most used solvent mixtures is hexane-acetone [8], and only a small amount of water (about 10%) must be added in nonpolar solvents such as hexane, xylene, or toluene to improve the heating rate [8]. Zhou and Lui [32] evaluated different mixtures of ethanol and hexane in the extraction of solanesol from tobacco leaves; the 1:3 ratio gave the best yield. Comparing isopropanol and hexane for rice bran oil extraction,

hexane at 40°C extracted approximately 40% more oil than isopropanol. Although by increasing the temperature hexane did not extract significantly more amount of oil, isopropanol extracted about 25% more rice bran oil at 120°C [33].

Some authors studied the use of combined solvents in MAE according to the polarity of the target compounds. A methanol–water (85:15) combination proved to be a good solvent for MAE of gymnemagenin from *Gymnema sylvestre* R. Br. Higher water concentration reduced the extraction yield because high water content increases the mixture polarity to a degree where it is no longer is favorable for extraction. The same was observed by Talebi et al. [34] when extracting paclitaxel from *Taxus baccata*: a methanol–water (90:10) mixture was the best combination. Song et al. [35], extracting sweet potato leaves, found that 60–80% (v/v) ethanol concentration in water was optimal within proportions of 40% and 80% (v/v).

The solvent-to-solid (feed) ratio (S/F) is an important parameter to be optimized. The solvent volume must be sufficient to guarantee that the entire sample is immersed in the solvent throughout the entire irradiation process, especially when using a matrix that will swell during the extraction [8, 13, 29].

In conventional extractions, the use of large volumes of solvent increases the extraction recovery. Studies reported that the extraction solution must not exceed 30–34% (w/v) [8]. In many applications a ratio 10:1 (ml/mg) to 20:1 (ml/mg) was found to be optimal [34, 36]. In addition, the solvent volume is an important factor to be considered because too much of the extracting solvent means more energy and time is required to condense the extraction solution in the later step and purification process. On the other hand, MAE may give lower recoveries because of nonuniform distribution and exposure to microwaves [37].

In some cases, small amounts of solvent are sufficient to extract the compounds of interest. The phenol and methylphenol extracted from oils had optimal conditions when S/F reached 2 [38]. A different behavior was observed in the MAE of artemisinin from *Aretimisia annua* L.: a higher extraction rate was achieved by a greater amount of solvent [39]. In *Ganoderma atrum*, the yield of triterpenoid saponins increased with the increase of amount of solvent until the S/F reached 25, and then it decreased rapidly [40].

2.3.2 Effect of Extraction Time and Cycle

In MAE the period of heating is another important factor to be considered. Extraction times in MAE are very short compared to conventional techniques and usually vary from a few minutes to a half-hour, avoiding possible thermal degradation and oxidation [20, 28], which is especially important for target compounds sensitive to overheating of the solute–solvent system. Overheating occurs because of the high dielectric properties of the solvent, especially ethanol and methanol, and further dilution with water that increases the heat capacity of the solvent combination [7]. Higher extraction time usually tends to increase the extraction yield. However, this increase was found to be very small with longer time [41]. Irradiation time is also

influenced by the dielectric properties of the solvent. Solvents such as water, ethanol, and methanol may heat up tremendously on longer exposure, thus risking the future of thermolabile constituents [13].

Occasionally, when longer extraction time is required, the samples are extracted in multiple steps using consecutive extraction cycles, which are also an example of the use of a larger amount of solvent and higher microwave application time [28, 42]. In this case, the fresh solvent is fed to the residue and the process is repeated to guarantee the exhaustion of the matrix. With this procedure, the extraction yield is enhanced, avoiding long heating [7, 28]. The number of process cycles will depend on the type of matrix and the solute. According to Li et al. [43], three cycles of 7 min were appropriate for MAE of triterpene saponins from yellow horn, whereas in optimization of triterpenoid saponins MAE from *Ganoderma atrum*, cycles of 5 min each were recommended [26]. Yan et al. [44] found that three extraction cycles of 5 min each are optimal for extracting astragalosides from *Radix astragali.* They also found that increasing the irradiation time from 1 to 5 min increases the extraction yield rapidly; extraction reaches its maximum at 5 min, and then the yields decreased with the extension of the irradiation time. In the case of flavonoids extraction from *R. astragali*, there was an increase in yield with time up to an exposure of 25 min and then the extraction yield started to decrease [42]. In the work of Chen et al. [26] it was observed that triterpenoid saponins yield from *Ganoderma atrum* reached its maximum at 20 min; after this time, the target compounds easily decomposed because of long exposure to high temperature. The same behavior was found by Song et al. [35].

2.3.3 Effect of Microwave Power and Extraction Temperature

Microwave power and temperature are interrelated because high microwave power can bring up the temperature of the system and result in the increase of the extraction yield until it becomes insignificant or declines [4, 42, 45]. It is known that the temperature is controlled by incident microwave power that controls the amount of energy provided to the matrix, which is converted to heat energy in the dielectric material.

At high temperatures the solvent power increases because of a drop in viscosity and surface tension, facilitating the solvent to solubilize solutes, and improving matrix wetting and penetration [13, 43, 46]. In addition, when MAE is performed in closed vessels, the temperature may reach far above the boiling point of the solvent, leading to better extraction efficiency by the desorption of solutes from actives sites in the matrix [8]. However, Routray and Orsat [7] state that the efficiency increases with the increase in temperature until an optimum temperature is reached and then starts decreasing with the further increase in temperature: this happens because the selection of ideal extraction temperature is directly linked with the stability and, therefore, with the yield of the target compound.

Microwave power is directly related to the quantity of sample and the extraction time required. However, the power provides localized heating in the sample, which

acts as a driving force for MAE to destroy the plant matrix so that the solute can diffuse out and dissolve in the solvent. Therefore, increasing the power will generally improve the extraction yield and result in shorter extraction time [4, 28]. On the other hand, high microwave power can cause poor extraction yield because of the degradation of thermally sensitive compounds. Also, rapid rupture of the cell wall takes place at a higher temperature when using higher power, and as a result impurities can also be leached out into the solvent together with the desired solute [13]. Therefore, it is important to properly select the MAE power to minimize the time needed to reach the set temperature and avoid a "bumping" phenomenon in temperature during the extraction [8]. Moreover, the overexposure to microwave radiation, even at low temperature or low operating power, was found to decrease the extraction yield because of the loss of chemical structure of the active compounds.

Knowing that power level alone does not gives sufficient information about the microwave energy absorbed into the extraction system, Alfaro et al. [47] created a term to study the effect of microwave power on MAE: energy density, defined as the microwave irradiation energy per unit of solvent volume for a given unit of time (W/ml). According to Li et al. [43], the energy density should be considered as a parameter as power level alone. In this study, the anthocyanin extraction rates from grape peel were different under the same microwave power level, extraction time, and S/F because the energy density levels were different.

Raner et al. [48] reported that variation of power from 500 to 1,000 W had no significant effect on the yield of flavonoids. The decrease in extraction yield was found at temperatures higher than 110°C because of instability of flavonoids and consequent thermal degradation [42]. In another case, higher microwave power led to thermal degradation of phenols when it was higher than 350 W (between 150 and 550 W) [35]. The temperature behavior was the same in other studies. In extracting astragalosides from *Radix astragali*, Yan et al. [44] also found that yield increased remarkably with temperature increase from 50°C to 70°C; above 70°C, the yields of astragalosides increased slowly and even decreased.

2.3.4 Effect of Contact Surface Area and Water Content

Not only the parameters already discussed but the characteristics of the sample also affect the MAE process. It is known that in a higher contact surface area the extraction efficiency increases. Also, finer particles allow improved or much deeper penetration of the microwave [49]. On the other hand, very fine particles may pose some technical problems; consequently, centrifugation or filtration is applied to prepare the matrix [13, 29]. In the preparation step the sample is grinded and homogenized to increase the contact area between the matrix and the solvent. The particle sizes are usually in the range of 100 μm to 2 mm [8]. In some cases soaking of the dried plant material in the extracting solvent before MAE has resulted in improved yield. This procedure is called pre-leaching extraction [13].

In many cases the extraction recovery is improved by the matrix moisture, which acts as a solvent. The moisture in the matrix is heated, evaporated, and generates

internal pressure in the cell, which ruptures the cell to release the solutes, hence improving the extraction yield [31]. When increasing the polarity of the solvent, water addition has a positive effect on the microwave-absorbing ability and, hence, facilitates the heating process [8, 28]. Moreover, the additional water promotes hydrolyzation, thus reducing the risk of oxidation of the compounds [41].

In extraction of astragalosides from *Radix astragali*, extraction efficiency was improved by the addition of water. The possible reason for the increased efficiency is the increase in swelling of plant material by water, which enhances the contact surface area between the plant matrix and the solvent [44].

2.3.5 Effect of Stirring

The effect of stirring is directly related to the mass transfer process in the solvent phase, which induces convection in the headspace. Therefore, equilibrium between the aqueous and vapor phases can be achieved more rapidly. The use of agitation in MAE accelerates the extraction by enhancing desorption and dissolution of active compounds bound to the sample matrix [50]. Through stirring, the drawbacks of the use of low solvent-to-solid ratio (S/F) can be minimized, together with the minimization of the mass transfer barrier created by the concentrated solute in a localized region resulting from insufficient solvent [28]. In the work by Kovács et al. [51] it is possible to observe the difference between suspensions with and without stirring. The authors found that when the suspensions were agitated with magnetic stirrers the temperature reached its maximum value within a shorter time, and the temperature differences inside individual vessels were not significant.

2.4 Comparison of Microwave-Assisted Extraction (MAE) with Other Solid–Liquid Extraction Techniques

To introduce bioactive plant extracts in pharmaceutical and cosmetic formulations, industries are looking for green and efficient extraction processes free of toxic solvents. Methodologies using biodegradable and nontoxic solvents such as water and ethanol are being developed [52].

The traditional techniques of solvent extraction of plant materials are based on the correct choice of solvents and the use of heat or/and agitation to increase the solubility of the desired compounds and improve the mass transfer. Soxhlet extraction is the most common and is still used as a standard in all cases [53]. As a result of several secondary metabolites, the development of high performance and rapid extraction methods is an absolute necessity [54]. The new extraction techniques with shortened extraction time, reduced solvent consumption, increased pollution prevention, and with special care for thermolabile constituents have gained attention. In the many published papers comparing MAE with other advanced and conventional extraction methods, MAE has been accepted as a potential and powerful alternative for the extraction of organic compounds from plant materials [55].

The ideal extraction technology depends on the type of compound to be extracted, whereas the extraction method efficiency is based on the highest recovery, especially of the effective constituents, the shortest processing time, the lowest production cost, and use of minimum organic solvent [56]. There have been numerous reviews and research on the advances of different extraction techniques, comparing their results. In the extraction of bioactive compounds from plants, MAE was reported to be more efficient compared to conventional techniques such as Soxhlet and advanced methods of extraction including ultrasound-assisted extraction (UAE), pressurized liquid extractions (PLE), and supercritical fluid extraction (SFE), which have emerged as energy-saving technologies. Over the years the procedures based on MAE have replaced some conventional extraction methods and have been adopted over decades in laboratories and industry.

In addition, the progress in microwave extraction gave rise to other categories of techniques to improve its performance: (1) microwave-assisted distillation (MAD) for the isolation of essential oils from herbs and spices [57]; (2) microwave hydrodiffusion and gravity (MHG), a combination of microwave heating and distillation at atmospheric pressure that requires less energy and no solvent and simply combines microwaves and earth gravity at atmospheric pressure [58]; (3) vacuum microwave hydrodistillation (VMHD), which uses pressures between 100 and 200 mbar to evaporate the azeotropic mixture of water–oil from the biological matrix [59]; (4) microwave-integrated Soxhlet extraction (MIS), a combination of microwave heating and Soxhlet [60]; and (5) solvent-free microwave extraction (SFME), based on the combination of microwave heating and distillation, which is performed at atmospheric pressure [61]. If these techniques are explored scientifically, they can be proven to be efficient extraction technologies for ensuring the quality of herbal medicines worldwide [13].

As already mentioned, MAE is increasingly employed in the extraction of natural products as an alternative to traditional techniques of extraction for several reasons: reduced extraction time, reduced solvent consumption, and less environmental pollution as a result of increased efficiency and clean transfer of energy to the matrix; improved extraction yield and product quality, because materials can be rapidly heated, and often processed at lower temperatures; up to 70% energy saving compared to conventional energy forms from the high energy densities and the direct absorption of energy by the materials; compact systems, as small as 20% of the size of conventional systems; and selective energy absorption resulting from the dielectric properties of the material and applicator design [52, 55, 62].

On the other hand, some disadvantages can also be mentioned: additional filtration or centrifugation is necessary to remove the solid residue after the process; the efficiency of microwaves can be poor when the target compounds or solvents are nonpolar, or when they are volatile; and the use of high temperatures that can lead to degradation of heat-sensitive bioactive compounds [63].

Considering these advantages and drawbacks of MAE compared to other techniques, a discussion on MAE performance compared to conventional and advanced techniques as Soxhlet, SFE, UAE, and PLE is appropriate. Table 2.2 presents their advantages and drawbacks; and Table 2.3 shows studies comparing the extraction technologies and their respective optimization.

Table 2.2 Comparison of traditional and advanced extraction techniques for analytical-scale extraction (adapted from Ref. 8 and 64)

	Extraction technique				
	Soxhlet	Microwave-assisted extraction (MAE)	Ultrasound-assisted extraction (UAE)	Supercritical fluid extraction (SFE)	Pressurized solvent extraction (PLE)
Brief description	Sample is placed in a glass fiber thimble and by using a Soxhlet extractor, the sample is repeatedly percolated with recondensed vapors of the solvent	Sample is immersed in a microwave-absorbing solvent in a closed vessel and irradiated with microwave energy	Sample is immersed in solvent in a vessel and submitted to ultrasonic using US probe or US bath	Sample is loaded in a high-pressure vessel and extracted with supercritical fluid (most commonly carbon dioxide at pressures of 150–450 bar and temperatures of 40°–150°C). The analytes are collected in a small volume of solvent, in a separator or onto a solid-phase trap, which is rinsed with solvent in a subsequent step	Sample and solvent are heated and pressurized in an extraction vessel; when the extraction is finished, the extract is automatically transferred into a vial
Extraction time	3–48 h	3–30 min	10–60 min	10–60 min	5–30 min
Sample size	1–30 g	1–10 g	1–30 g	1–5 g	1–30 g
Solvent use	100–500 ml	10–40 ml	30–200 ml	2–5 ml (solid trap) 5–20 (liquid trap)	10–100 ml
Investment	Low	Moderate	Low	High	High
Advantages	Easy to handle, no filtration necessary, high matrix capacity	Fast and multiple extraction, easy to handle, moderate solvent consumption, elevated temperatures	Easy to use, multiple extractions	Fast extraction, low solvent consumption, concentration of the extract, no filtration necessary, possible high selectivity, low temperatures, no use of toxic solvents, automated systems	Fast extraction, no filtration necessary, low solvent consumption, elevated temperature, automated systems
Drawbacks	Long extraction time, large solvent volume, cleanup step is needed	Extraction solvent must absorb microwave energy, filtration step required, waiting time for the vessels to cool down	Large solvent volume, filtration step required, repeated extractions may be required	Many parameters to optimize, especially analyte collection	Possible degradation of thermolabile analytes, cleanup step is needed

Table 2.3 Comparison on the extraction yield between MAE and other techniques

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
Sweetgrass leaves (*Hierochloe odorata* L.)	MAE: Pw = 200 W; s = acetone; S/F = 10; T = 80 C; P = P_{atm}; t = 15 min; one-step extraction	5,8-Dihydroxycoumarin (0.42% db[a]) 5-Hydroxy-8-*O*-β D-glucopyranosyl-benzopyranone (0.11% db[a])	[65]
	SFE: Two-step: (1) = 35 MPa; T = 40 C (2) = P = 25Mpa; T = 40 C S = ethanol (20%); t = 2 h; v = 0.5 l/min	5,8-Dihydroxycoumarin (0.49% db[a]) 5-hydroxy- 8-*O*-β - D-glucopyranosyl-benzopyranone (0.06% db[a])	
	Soxhlet: S/F = 50; s = acetone; t = 6 h	5,8-Dihydroxycoumarin (0.46% db[a]) 5-Hydroxy-8-*O*-β -D-glucopyranosyl-benzopyranone (0.08% db[a])	
Artemisia annua L.	MAE: Pw = 650 W; s = solvent oil; S/F = 15; T = ambient; t = 12 min	Artemisinin (92.1% db[a])	[66]
	SFE: P = 30 MPa; s = CO_2; S/F = 6; T = 35 C; t = 2.5 h	Artemisinin (33.2% db[a])	
	Soxhlet: s = Solvent oil; S/F = 11.67; T = 35 C; t = 6 h	Artemisinin (60.4% db[a])	
Licorice roots (*Glycyrrhizia glaubra*)	MAE: Pw = 700 W; s = ethanol; S/F = 10; T = 85 –90 C; t = 4 min	Glycyrrhizic acid –GA (2.26%[a])	[67]
	US: s = ethanol; S/F = 10; t = 20.5 h	Glycyrrhizic acid –GA (2.26%[a])	
	Soxhlet: s = ethanol; S/F = 10; t = 10 h	Glycyrrhizic acid –GA (2.5%[a])	

Green tea leaves	MAE: Pw = 700 W; s = ethanol: water (1:1 v/v); S/F = 20; T = 20 C; t = 4 min	Tea polyphenols (30%[a]) Tea caffeine (4%[a])	[36]
	UAE: s = ethanol: water (1:1 v/v); S/F =; T = 20 C–40 C; t = 90 min	Tea polyphenols (28%[a]) Tea caffeine (3.6%[a])	
	Heat reflux extraction: s = ethanol: water (1:1 v/v); S/F =; T = 85 C; t = 45 min	Tea polyphenols (28%[a]) Tea caffeine (3.6%[a])	
Grapefruit	MAE: Pw = 0.9 kW; s = water; S/F = 30; T = 20 C; t = 6 min	Pectin (27.81% db[a])	[68]
	UAE: s = water; S/F = 30; T = 70 C; t = 25 min	Pectin (17.92% db[a])	
	UAE + MAE: Pw = 0.45 kW; S/F = 30; t = 30 min (UAE) and 10 min (MAE)	Pectin (31.88% db[a])	
	Heat batch: s = water; S/F = 30; T = 90 C; t = 90 min	Pectin (19.16% db[a])	
Ganoderma atrum	MAE: s = ethanol: water (9.5: 0.5 v/v); S/F = 25; T = 90 C; t = 5 min	Global yield (5.11% db[a])	[69]
	UAE: s = ethanol: water (9.5: 0.5 v/v); S/F = 25; t = 30 min; f = 33 kHz	Global yield (1.72% db[a])	
	SFE: P = 25 MPa; T = 55 C; s = CO_2 + ethanol; t = 3 h	Global yield (1.52% db[a])	
	Shaking: s = ethanol: water (9.5: 0.5 v/v); t = 3 h	Global yield (2.58% db[a])	
	HRE: s = ethanol (9.5: 0.5 v/v); S/F = 25; T = 95 C; t = 1 h	Global yield (2.22% db[a])	

(continued)

Table 2.3 (continued)

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
Yellow horn (*Xanthoceras sorbifolia* Bunge.)	MAE: Pw = 900 W; s = ethanol:water (40: 60 v/v); S/F = 30; T = 50 C; t = 7 min × 3 cycles	Global yield (11.62% db[a])	[43]
	UAE: Pw = 250 W; s = ethanol:water (40: 60 v/v); S/F = 30; T = 50 C; t = 60 min × 3 cycles	Global yield (6.78% db[a])	
	HRE: Pw = 800 W; s = ethanol: water (40: 60 v/v); S/F = 30; T = 50 C; t = 90 min × 3 cycles	Global yield (10.82% db[a])	
Turmeric plant (*Curcuma longa* L.).	MAE: Pw = 60 W; s = acetone; S/F = 3; T = 50 C; t = 5 min	Curcumin (90.47% db[a])	[70]
	UAE: Pw = 150 W; s = acetone; S/F = 3; T = 21 C; t = 5 min	Curcumin (71.42% db[a])	
	Soxhlet: s = acetone; S/F = 5; t = 8 h	Curcumin (2.10% db[a])	
	SFE: P = 30 MPa; s = CO_2 + ethanol (10%); T = 50 C; t = 240 min; v = 5 ml/min	Curcumin (69.36% db[a])	
Silybum marianum (L.) (milk thistle)	MAE: Pw = 600 W; s = ethanol: water (80:20 v/v); S/F = 25; t = 2 min × 6 cycles	Silybinin (1.37% db[a])	[56]
	Soxhlet: s = ethanol: water (80:20 v/v); S/F = 100; t = 12 h	Silybinin (1.09% db[a])	
	Stirring: s = ethanol: water (80:20 v/v); S/F = 100; t = 24 h	Silybinin (0.48% db[a])	
	Maceration: s = ethanol: water (80:20 v/v); S/F = 100; t = 24 h	Silybinin (0.36% db[a])	

Coriandrum sativum	MAE: Pw = 200 W; s = ethanol: water (50:50 v/v); S/F = 20; T = 50 C, t = 18 min	Phenolics content (0.082% db[b])	[71]
	UAE: s = ethanol: water (50:50 v/v), S/F = 10; t = 30 min	Phenolics content (0.041% db[b])	
Cinnamomum zeylanicum	MAE: Pw = 200 W; s = ethanol: water (50:50 v/v); S/F = 20; T = 50 C, t = 18 min	Phenolics content (1.679% db[b])	[71]
	UAE: s = ethanol: water (50:50 v/v), S/F = 10; t = 30 min	Phenolics content (0.506% db[b])	
Cuminum cyminum	MAE: Pw = 200 W; T = 50 C, s = ethanol: water (50:50 v/v); S/F = 20; t = 18 min	Phenolics content (1.159% db[b])	[71]
	UAE: s = ethanol: water (50:50 v/v), S/F = 10; t = 30 min	Phenolics content (0.290% db[b])	
Crocus sativus	MAE: Pw = 200 W; T = 50 C, s = ethanol: water (50:50 v/v); S/F = 20; t = 18 min	Phenolics content (2.939% db[b])	[71]
	UAE: s = ethanol: water (50:50 v/v), S/F = 10; t = 30 min	Phenolics content (0.500% db[b])	

(continued)

Table 2.3 (continued)

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
Sea buckthorn	MHG:	Isorhamnetin 3-*O*-rutinoside (0.123% db[a])	[2]
(*Hippophae rhamnoides)*	Pw = 400 W; t = 15 min; h = 57%	Isorhamnetin 3-*O*-glucoside (0.097% db[a])	
		Quercetin 3-*O*-Glucoside (0.025% db[a])	
		Isorhamnetin (0.00084% db[a])	
	Agitated:	Isorhamnetin 3-*O*-rutinoside (0.187% db[a])	
	s = methanol: water (80:20 v/v); S/F = 10; t = 8 min	Isorhamnetin 3-*O*-glucoside (0.162% db[a])	
		Quercetin 3-*O*-Glucoside (0.016% db[a])	
		Isorhamnetin (0.00064% db[a])	
Cranberry press cake	MAE: s = ethanol; S/F = 5.7; T = 125 C; t = 10 min	Quercetin (0.1272% db[a])	[72]
	Stirring: s = ethanol; S/F = 5; t = 2 h	Quercetin (0.1537% db[a])	
Morinda citriflora (roots)	MAE: Pw = 720 W; s = ethanol: water (80:20 v/v); S/F = 100; T = 60 C; t = 15 min	Global yield (95.91% db[a])	[73]
	UAE: s = ethanol; S/F = 100; T = 60 C; t = 60 min	Global yield (62.23% db[a])	
	Maceration: s = ethanol; S/F = 100; t = 3 days	Global yield (63.33% db[a])	
	Soxhlet: s = ethanol; S/F = 100; T = 100 C; t = 4 h	Global yield (97.74% db[a])	

Soybean germ	MAE: S/F = 17.5; T = 120 C; t = 0.5 h	Global yield (16.5% wb[a])	[74]
	MAE + UAE: Pw = 60 W (UAE) and 100 W (MAE); S/F = 5; T = 45 C; t = 1 h	Global yield (14.1% wb[a])	
	Soxhlet: s = hexane; S/F = 6.67; t = 4 h	Global yield (8.65% wb)	
Lavandula angustifolia Mill., Lamiaceae (lavender flowers)	MASD: Pw = 500 W; s = water; S/F = 4; t = 10 min	Monoterpenes (3.54% db[a]) Oxygenated monoterpenes (78.29% db[a]) Sesquiterpenes (2.77% db[a]) Global yield (8.86% db[a])	[75]
	SD: s = water; S/F = 4; t = 90 min	Monoterpenes (4.92% db[a]) Oxygenated monoterpenes (75.14% db[a]) Sesquiterpenes (2.87% db[a]) Global yield (8.75% db[a])	
Caraway (*Carum carvi* L.)	MDG: Pw = 100 W; t = 45 min	Global yield (2.59% db[a]) Carvone (67.59% db[a]) Limonene (30.10% db[a])	[76]
	Hydrodistillation: S/F = 5; t = 300 min	Global yield (2.54% db[a]) Carvone (66.89% db[a]) Limonene (30.30% db[a])	
Tomato	MAE: Pw = 100 W; s = methanol; S/F = 50; t = 45 min	Total phenolic contents (0.646% db[b])	[77]
	Shaker: s = ethanol: water (60: 40 v/v); S/F = 50; T = 45 C; r = 400 rpm; t = 15 h	Total phenolic contents (0.603% db[b])	

(continued)

Table 2.3 (continued)

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
Foeniculum vulgare Miller (seeds)	MWHD: Pw = 300 W; s = water; S/F = 2; T = 100 C; t = 200 s	Global yield (1.14% db[a])	[78]
	HD: Pw = 300 W; s = water; S/F = 8; t = 319 s; T = 100 C; r = 50 rpm	Global yield (0.265% db[a])	
Iochroma gesnerioides (leaves)	MAE: Pw = 25 W; s = methanol; S/F = 50; t = 40 s	Withaferin A (0.48% db[a]) Iochromolide (0.85% db[a]) Withacnistin (0.39% db[a])	[79]
	Soxhlet: (1) s = water; S/F = 6; t = 15 min (2) s = methanol; S/F = 100; t = 6 h	Withaferin A (0.41% db[a]) Iochromolide (0.81% db[a]) Withacnistin (0.38% db[a])	
Xanthoceras sorbifolia Bunge. (yellow horn)	MAE: Pw = 900 W; s = ethanol: water (40: 60 v/v); S/F = 30; T = 50 C; t = 7 min × 3 cycles	Triterpene saponins (11.62% wb[a])	[43]
	UAE: Pw = 250 W; s = ethanol: water (40: 60 v/v); S/F = 30; T = 50 C; t = 60 min × 3 cycles	Triterpene saponins (6.78%[a])	
	Reflux: Pw = 800 W; s = ethanol: water (40: 60 v/v); S/F = 30; T = 50 C; t = 90 min × 3 cycles	Triterpene saponins (10.82%[a])	
Ocimum Basilicum L. (basil)	SFME: Pw = 500 W; T = 100 C; t = 30 min	Eugenol (43.2% wb[a]) Linalool (25.3% wb[a]) Global yield (0.029% wb[a])	[80]
	HD: s = water; S/F = 12; T = 100 C; t = 4.5 h	Eugenol (11.0% wb[a]) Linalool (39.1% wb[a]) Global yield (0.028% wb[a])	

Mentha crispa L. (garden mint)	SFME: Pw = 500 W; T = 100 C; t = 30 min HD: s = water; S/F = 12; T = 100 C; t = 4.5 h	Limonene (9.7% wb[a]) Carvone (64.9% wb[a]) Global yield (0.095% wb[a]) Limonene (20.2% wb[a]) Carvone (52.3% wb[a]) Global yield (0.095% wb[a])	[80]
Thymus vulgaris L. (thyme)	SFME: Pw = 500 W; T = 100 C; t = 30 min HD: s = water; S/F = 12; T = 100 C; t = 4.5 h	γ-Terpinene (17.1% wb[a]) Eugenol (51.0% wb[a]) Global yield (0.160% wb[a]) γ-Terpinene (22.8% wb[a]) Eugenol (40.5% wb[a]) Global yield (0.161% wb[a])	[80]
Elletaria cardamomum L. (cardamom)	SFME: Pw = 390 W; T = 100 C; h = 67%; t = 75 min HD: s = water; S/F = 10; T = 100 C; t = 6 h	Global yield (2.70% db[a]) 26.23 1,8-Cineole 5.29 Linalool 2.60 Terpin-4-ol 3.88 α-Terpineol 3.63 Linalyl acetate 45.45 α -Terpinyl acetate	[81]
Gymnema sylvestre R. Br.	MAE: Pw = 280 W; s = methanol: water (85: 15 v/v); S/F = 25; t = 6 min Reflux: s = methanol: water (85: 15 v/v); S/F = 100; T = 95 C; t = 6 h Maceration: s = methanol: water (85: 15 v/v); S/F = 100; t = 24 h Stirring: s = methanol: water (85: 15 v/v); S/F = 100; t = 24 h	Gymnemagenin (4.3% db[a]) Gymnemagenin (3.3% db[a]) Gymnemagenin (1.7% db[a]) Gymnemagenin (2.2% db[a])	[82]

(continued)

Table 2.3 (continued)

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
(*Melilotus officinalis* (L.) Pallas) (yellow sweet clover)	MAE: Pw = 100 W; s = water: ethanol (50: 50 v/v); S/F = 20; T = 50 C; t = 5 min × 2 cycles USAE: s = water: ethanol (50: 50 v/v); S/F = 20; t = 60 min Soxhlet: s = ethanol: water (95: 5 v/v); S/F = 16.67; t = 8 h	Coumarin (0.3978% db) *O*-Coumaric acid (0.1257% db[a]) Melilotic acid (0.9052% db[a]) Coumarin (0.3569% db[a]) *O*-Coumaric acid (0.1269% db[a]) Melilotic acid (0.8092% db[a]) Coumarin (0.2156% db[a]) *O*-Coumaric acid (0.0708% db[a]) Melilotic acid (0.6314% db[a])	[83]
Salvia miltiorrhiza Bunge. (dried root)	MAE: s = ethanol: water (95: 5 v/v); S/F = 10; T = 80 C; t = 2 min Reflux: s = ethanol: water (95: 5 v/v); S/F = 10; t = 45 min UAE: s = ethanol: water (95: 5 v/v); S/F = 10; t = 75 min Soxhlet: s = ethanol: water (95: 5 v/v); S/F = 10; t = 95 min	Tanshinone IIA (0.29% db[a]) Cryptotanshinone (0.23% db[a]) Tanshinone I (0.11% db[a]) Tanshinone IIA (0.25% db[a]) Cryptotanshinone (0.24% db[a]) Tanshinone I (0.11% db[a]) Tanshinone IIA (0.28% db[a]) Cryptotanshinone (0.25% db[a]) Tanshinone I (0.10% db[a]) Tanshinone IIA (0.33% db[a]) Cryptotanshinone (0.25% db[a]) Tanshinone I (0.12% db[a])	[84]

Radix astragali (dried root)	MAE: Pw = 700 W; s = ethanol: water (80:20 v/v); S/F = 25; T = 70 C; t = 5 min × 3 cycles Soxhlet: s = ethanol: water (80:20 v/v); S/F = 20; T = 90 C; t = 4 h Reflux: s = ethanol: water (80:20 v/v); S/F = 20; T = 90 C; t = 1 h UAE: Pw = 100 W; s = ethanol: water (80:20 v/v); S/F = 20; t = 40 min Maceration: s = ethanol: water (80:20 v/v); S/F = 20; t = 12 h	Astragalosides I (0.0788% db[a]) Astragaloside II (0.0351% db[a]) Astragaloside III (0.0206% db[a]) Astragaloside IV (0.0278% db[a]) Astragalosides I (0.770% db[a]) Astragaloside II (0.347% db[a]) Astragaloside III (0.193% db[a]) Astragaloside IV (0.242% db[a]) Astragalosides I (0.761% db[a]) Astragaloside II (0.352% db[a]) Astragaloside III (0.203% db[a]) Astragaloside IV (0.257% db[a]) Astragalosides I (0.519% db[a]) Astragaloside II (0.302% db[a]) Astragaloside III (0.190% db[a]) Astragaloside IV (0.225% db[a]) Astragalosides I (0.411% db[a]) Astragaloside II (0.299% db[a]) Astragaloside III (0.166% db[a]) Astragaloside IV (0.206% db[a])	[44]
Sweet potato [*Ipomoea batatas* (L.) Lam.]	MAE: Pw = 123 W; s = ethanol: water (53: 47 v/v); S/F = 25; t = 2 min CSE: s = ethanol: water (60: 40 v/v); S/F = 30; t = 120 min	Total phenolics (6.115% db[b]) Total phenolics (5.969% db[b])	[35]

(continued)

Table 2.3 (continued)

Plant material	Operational conditions: type of solvent(s), solvent to feed ratio (S/F), temperature (T), pressure (P), time (t), raw material moisture content (h), rotation (r), frequency (f), power (Pw), flow rate (v), power to feed ratio (P/F), humidity (h)	Bioactive compound extracted and extraction yield (dry basis, db; wet basis, wb)	References
Tobacco leaves	MAE: Pw = 700 W; s = hexane: ethanol (1: 3 v/v) + NaOH (0.05 mol/l); S/F = 10; t = 40 min	Solanesol (0.91% db[a])	[32]
	HRE: s = hexane: ethanol (1: 3 v/v) + NaOH (0.02 mol/l); S/F = 10; T = 60 C; t = 180 min	Solanesol (0.87% db[a])	
Lavandula angustifolia Mill., Lamiaceae (lavender flowers)	MSD: Pw = 200 W; v = 8 g/min; t = 6 min	1,8-Cineole (14.40% db[a]) Linalool (42.52% db[a]) Global yield (2.7% db[a])	[85]
	SD: t = 30 min	1,8-Cineole (13.71% db[a]) Linalool (40.43% db[a]) Global yield (2.7% db[a])	
Radix astragali (root of *Astragalus*; Huangqi)	MAE: s = ethanol: water (95: 5 v/v); S/F = 25; T = 110 C; t = 25 min × 2 cycles	Flavonoids (0.1190%[a])	[42]
	Soxhlet: s = methanol; S/F = 25; T = 85 C; t = 4 h	Flavonoids (0.1292%[a])	
	UAE: s = methanol; S/F = 20;T = 60 C; t = 30 min × 2 cycles	Flavonoids (0.0736%[a])	
	HRE: s = ethanol: water (90: 10 v/v); S/F = 25; T = 75 C; t = 2 h × 2 cycles	Flavonoids (0.0934%[a])	

Yellow onion	VMHG: Pw = 500 W; P = 700 mbar; P/F = 1 W/g; T = 81 C; t = 26 min; h = 84.5%	Quercetin (0.662%db[a]) Global yield (3.18% db[a])	[86]
	MHG: P = 1 bar; T = 100 C; t = 23 min; h = 84.5%	Quercetin (0.283%db[a])	
	CSE: s = methanol: water (80: 20 v/v); S/F = 10; r = 8,000 rpm; t = 5 min	Quercetin (0.890% db[a])	

HRE heat reflux extraction, *MHG* microwave hydrodiffusion and gravity, *MASD* microwave-accelerated steam distillation, *SD* steam distillation
SFME solvent-free microwave extraction, *MDG* microwave dry-diffusion and gravity, *MWHD* microwave-assisted hydrodistillation
HD hydrodistillation, *USAE* Soxhlet extraction, ultrasound-assisted extraction, *MSD* microwave steam distillation
VMHG vacuum microwave hydrodiffusion and gravity, *CSE* conventional solvent extraction
[a]Yield (%) = g compound per 100 g sample
[b]Yield (%) = g gallic acid equivalent (GAE) per 100 g mass of sample
Adapted from Eskilsson and Björklund [8]; Chemat et al. [64]

2.4.1 MAE Versus Soxhlet

Soxhlet is the typical technique and the main reference for evaluating the performance of other solid–liquid extraction methods as it has long been one of the most used solid–liquid extraction techniques. In Soxhlet extraction the solid material containing the solutes is placed inside a thimble holder, which is connected to a flask containing the extraction solvent, and submitted to reflux. After this process, the extract is concentrated by evaporation of the solvent [87]. This method has a large dependence on plant characteristics and particle size, as the internal diffusion may be the limiting step during extraction, and extraction and evaporation temperatures affect the quality of the final products [31].

It is a general and well-established technique, which surpasses in performance other conventional extraction techniques except, in a limited field of applications, the extraction of thermolabile compounds. Furthermore, it presents other disadvantages such as poor extraction of lipids, long operation time, high solvent consumption, and operation at the solvent's boiling point [88]. The advantages of this method include no requirement of a filtration step after leaching and the displacement of transfer equilibrium by repeatedly bringing fresh solvent into contact with the solid matrix [31, 88].

Studies show that MAE allows the reduction of time and solvent consumption, as well as improvement in global yield. Kaufmann et al. [89], extracting whitanolides from *Lochroma gesneroides*, showed a drastic reduction in solvent usage (5 vs. 100 ml) and in extraction time (40 s vs. 6 h). Another study concluded that the same quantity and quality of tanshiones from *Salvia miltiorrhiza* Bunge was obtained with 2 min of MAE and 90 min of Soxhlet [90]. Higher yield was obtained when extracting artemisinin from *Artemisia annua* L. by MAE; in 12 min, 92.1% of artemisinin was recuperated by MAE whereas several hours were needed by Soxhlet to reach only about 60% extraction efficiency [66].

2.4.2 MAE Versus Supercritical Fluid Extraction (SFE)

For green extraction, the use of SFE is very attractive because the solute is easily recovered and the solvent can be recycled by the simple manipulation of parameters such temperature and/or pressure. Supercritical fluids present liquid-like densities, whereas their viscosity is near that of normal gases and their diffusivity is about two orders of magnitude higher than in typical liquids [91]. Carbon dioxide (CO_2) is the most used solvent in SFE because it is safe, nontoxic, and generally available at a reasonable cost. However, even at high densities, CO_2 has a limited ability to dissolve highly polar compounds. The addition of modifiers to CO_2 can improve the extraction efficiency by increasing the solubility of the solute in the solvent.

The ease of tuning the operating conditions to increase the solvation power makes this technology a good option for the selective recovery of several types of

substances. This combination of properties makes SFE an important process in the food, pharmaceutical, and cosmetic industries because it is possible to fabricate products without toxic residues, with no degradation of active principles, and with high purity. Thus, SFE can be a fast, efficient, and clean method for the extraction of natural products from vegetable matrices [92].

Compared to SFE, MAE has a disadvantage, because cleanup is usually needed for this relatively selective technique [8, 63]. However, method development is often more complex in SFE and additionally sample throughput is not as high as in MAE [8]. Furthermore, the efficiency of MAE can be poor when either the target compounds or solvents are nonpolar, or when they are volatile. According to Stalikas [93], drying of the samples can be avoided for sample preparation with MAE, whereas samples are usually dried before SFE.

From the economic point of view, MAE is feasible as it requires moderate cost for equipment setup [63] and is much cheaper as compared to SFE. Moreover, MAE has low risks and no major safety issues as most extractions are generally carried out under atmospheric condition [28].

Several studies compared SFE and MAE. Hao et al. [66] extracted artemisinin from *Artemisia annua* L. by MAE, Soxhlet, and SFE. They found that MAE saves much time (12 min) and gives a high extraction rate (92.1%); SFE gives the lightest extract color but the lowest extraction yield while several hours were needed for Soxhlet. The same results were found by Grigonis et al. [64] comparing MAE with SFE and Soxhlet. The MAE gave the most concentrated extract with 8.15% of 5,8-dihydroxycoumarin (extract yield, 0.42%) from sweet grass. In addition, only 5 min gave the highest yield of triterpenoid saponins (0.968%), whereas SFE and UAE required several hours or even more than 10 h and gave a lower yield [40].

2.4.3 MAE Versus Ultrasound-Assisted Extraction (UAE)

Ultrasound-assisted extraction (UAE) in the food industry has been the subject of research and development; its emergence as a green novel technology has also attracted attention to its role in environmental sustainability [94]. Ultrasound has been used in various processes of the chemical and food industries; it is a rapid technique, consumes small amounts of fossil energy, and allows reducing solvent consumption, thus resulting in a more pure product and higher yields.

The principle of high-power ultrasound has been attributed to the acoustic cavitation phenomenon that appears when high-intensity acoustic waves are generated in a fluid [95]. The extraction mechanism involves two types of physical phenomena: diffusion through the cell walls and washing out the cell content once the walls are broken [96]. Ultrasound waves modify their physical and chemical properties after their interaction with subjected plant material, and their cavitational effects facilitate the release of extractable compounds and enhance mass transport by disrupting the plant cell walls [94, 97, 98].

Developments in ultrasound technology and its potential benefits have triggered interest in the application of power ultrasound on a wider range of chemistry processing [99].

The combination of sonication and microwaves was studied for extraction of lipids from vegetables and microalgae sources. Ultrasonication alone, microwave irradiation alone, or a combination of both techniques gave excellent extraction efficiencies in term of yield and time, with a tenfold reduction in the time needed with conventional methods, and increase of yields from 50% to 500% [74]. MAE possessed higher efficiency (11.62%) for the extraction of triterpene saponins from yellow horn (*Xanthoceras sorbifolia* Bunge.) compared with UAE (6.78%) and reflux extraction (10.82%) [43].

2.4.4 MAE Versus Pressurized Liquid Extraction (PLE)

Pressurized liquid extraction (PLE), also referred to as pressurized solvent extraction (PSE) and accelerated solvent extraction (ASE), is now well accepted as an alternative to Soxhlet extraction [100] and has been successfully used to isolate antioxidants from plants [101], such as thermolabile anthocyanins from jabuticaba (*Myrciaria cauliflora*) [102].

The use of the PLE technique is an attractive alternative because it allows fast extraction and reduced solvent consumption [102]. This technique allows the use of solvents or solvent mixtures with different polarities under high pressures (up to 20 MPa), keeping the extraction solvent in the liquid state [103], and temperatures ranging from room temperature up to 200°C [104].

The pressurized solvent at a determined temperature is pumped into an extraction vessel containing the sample matrix. Using high temperature accelerates the extraction process by increasing the solubility of the analytes in the solvent and thus increasing the kinetic rate of desorption of the solute from the sample matrix; this occurs because the pressurized solvent remains in the liquid state well above its boiling point, allowing high-temperature extraction [103]. Considerable increase in the mass transfer rates results from the decrease of viscosity and superficial tension of the solvent.

Moreover, the use of high temperatures, which on the one hand increases extraction rates, on the other hand may lead to degradation of thermolabile compounds [105]. PLE uses liquid solvents; therefore, its basic principle is considered similar to those of classic extraction. Partly because these newer technologies are automated and the solvents are under "superheated" conditions (the effect of microwaves in MAE or elevated temperature or pressure in PLE), they are more user friendly, much quicker, and require significantly less organic solvent [49].

Although in PLE the filtration step is "included" in the process, in MAE a cleanup step is often needed. MAE is considered an easy technique, and compared to SFE and PLE, it is less expensive [8].

Although good recovery rates were obtained with both extraction methods, MAE provided advantages with regard to sample handling, cost, analysis time, and solvent consumption.

2.5 Conclusion

There has been much research and many advances in development in the microwave-assisted extraction of a number of plant compounds. This chapter showed the phenomena of mass and heat transfer of the MAE process as well the parameters that influence MAE extraction of bioactive compounds. Therefore, optimized operating parameters can improve MAE performance. Also, MAE is better or comparable with other techniques. As a concluding remark, the MAE system is considered a promising technique for plant extraction because of its use of different physical and chemical phenomena compared to those in conventional extractions.

Acknowledgments Priscilla C. Veggi thanks Fundação de Amparo a Pesquisa do Estado de São Paulo (FAPESP) for Ph.D. assistantships (2008/10986-2). The authors thank FAPESP (2009/17234-9) and CNPq (302778/2007-1) for financial support.

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Microwave-assisted Extraction for Bioactive Compounds
Theory and Practice
(Eds.)F. Chemat; G. Cravotto
2013, XII, 238 p. 72 illus., 38 illus. in color., Hardcover
ISBN: 978-1-4614-4829-7