



Review

Microwave-assisted extractions of active ingredients from plants

Chung-Hung Chan^a, Rozita Yusoff^{a,*}, Gek-Cheng Ngoh^a, Fabian Wai-Lee Kung^b^a University of Malaya, Department of Chemical Engineering, Lembah Pantai, 50603 Kuala Lumpur, Wilayah Persekutuan, Malaysia^b Multimedia University, Faculty of Engineering, Jalan Multimedia, 63100 Cyber Jaya, Selangor, Malaysia

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ABSTRACT

Microwave-assisted extraction (MAE) is widely employed in the analysis and the extraction of active compounds from plants. This review summarizes the research done during the last decade on the MAE of active ingredients from plants. Advances and modifications to improve the performance of MAE are presented and discussed in detail. Modified MAE such as vacuum microwave-assisted extraction (VMAE), nitrogen-protected microwave-assisted extraction (NPMAE), ultrasonic microwave-assisted extraction (UMAE), dynamic microwave-assisted extraction (DMAE) and other advancements in MAE are also detailed in this article. In addition, the microwave extraction procedures and the important parameters influencing its performance are also included, together with the advantages and the drawbacks of each MAE techniques.

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* Corresponding author. Tel.: +603 7967 6891; fax: +603 7967 5319.

E-mail addresses: ryusoff@um.edu.my, ryusoff67@gmail.com (R. Yusoff).

1. Introduction

Solvent extraction has been intensively used for isolation of important compounds and for the qualitative and quantitative analysis in various fields such as environmental analysis, food and agricultural analysis, pharmacological drugs and herbal medicine. The latter, in particular, has attracted the interest of many researchers to replace synthetic drugs by alternative medicine since the former often results in undesirable secondary effects. One example is thiazolidinedione (TZD), which is commonly used in the treatment of diabetes mellitus type 2, was found to cause edema, abnormal water retention inside the body, and increased risk of coronary heart disease and heart attacks [1]. In contrast, active ingredients extracted from plants such as quercetin and kaempferol possess hypoglycaemic effect that can be applied to treat diabetes with minimal side effects [2,3].

Extraction represents the primary step in getting crude extract from plants. The extract obtained then has to undergo further analysis and identification of its active components. Conventional extraction techniques include soaking, maceration, water percolation, Soxhlet extraction, etc. These techniques usually required long extraction time which incorporates risk of thermal degradation of thermolabile active compounds [4].

Over the past decade, various novel extraction techniques have been introduced and investigated, most of which were claimed to be better in terms of efficiency, extraction time and solvent consumption [5–7]. The novel techniques available are microwave-assisted extractions (MAE), supercritical fluid extraction (SFE), and pressurized solvent extraction (PSE). Microwave-assisted extraction in particular has drawn significant research attention in various fields, in particular medicinal plant research, due to its special heating mechanism, moderate capital cost and its good performance under atmospheric conditions [8–10]. Microwave is an electromagnetic wave. It consists of electric field and magnetic field which oscillates perpendicularly to each others in frequency ranged from 0.3 to 300 GHz. Microwave can penetrate into certain materials and interacts with the polar components to generate heat. The heating of microwave energy acted directly on the molecules by ionic conduction and dipole rotation [8] and thus only selective and targeted materials can be heated based on their dielectric constant. The efficiency of the microwave heating depends on the dissipation factor of the material, $\tan \delta$, which measures the ability of the sample to absorb microwave energy and dissipate heat to the surrounding molecules as given by Eq. (1) [11]

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \quad (1)$$

where ε'' is the dielectric loss which indicates the efficiency of converting microwave energy into heat while ε' is the dielectric constant which measures the ability of the material to absorb microwave energy. The rate of conversion of electrical energy into thermal energy in the material is described by Eq. (2) [12]

$$P = K \cdot f \varepsilon' E^2 \tan \delta \quad (2)$$

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

Mandal et al. [11] believed that MAE has promising future in medicinal plant research and indicated that the method has potential to be developed further and possibly scaled up, despite some drawbacks associated with MAE, such as the requirement of additional clean up step to remove solvent from sample matrices and the restriction to polar solvent application only in the system [13]. Besides the fundamental closed system and open system of MAE, various modified MAE have been developed such as vac-

uum microwave-assisted extraction (VMAE), nitrogen-protected microwave-assisted extraction (NPMAE), ultrasonic microwave-assisted extraction (UMAE) and dynamic microwave-assisted extraction (DMAE). It is apparent that the technique of MAE has been continuously improved throughout the last decade where many modifications have been introduced to enhance its performance.

Other reviews emphasize on the basic information concerning the use of MAE in the extraction of active compounds from plants [11] and the relevant equipment employed for this purpose [14]. However, none of these reviews provide adequate details of the methodology for the MAE. Therefore, this review is aimed to provide an insight into MAE techniques, its advancement, equipment setup, extraction procedures, important operating conditions and the performance of the various MAE techniques. The review highlights the technology and the uniqueness of each MAE techniques with its applications and drawbacks. Interested parties can rely on the useful information presented in this review for selecting the most suitable extraction technique for their respective target compounds.

2. The development of MAE techniques

In general, MAE systems are classified into multi-mode system and focused-mode system (mono-mode). Multi-mode system allows random dispersion of microwave radiation in cavity by a mode stirrer while focused system (mono-mode) allows focused microwave radiation on a restricted zone in cavity. Usually, the multi-mode system is associated with high pressure while the mono-mode system is employed under atmospheric operating pressure. However, mono-mode system can also run at high pressure. To avoid confusion in the classification of MAE, 'closed system' and 'open system' are used to refer to the system that operates above atmospheric pressure and under atmospheric pressure, respectively [13,14]. For further understanding of the closed system and open system, schematic diagrams are illustrated in Fig. 1.

In a closed MAE system, the extractions are carried out in a sealed-vessel with different mode of microwave radiations. Extraction is normally carried out under uniform microwave heating. High working pressure and temperature of the system allow fast and efficient extraction. The pressure inside the extraction vessel is controlled in such a way that it would not exceed the working pressure of the vessel while the temperature can be regulated above the normal boiling point of the extraction solvent. Recent advancements in the closed system have led to the development of high pressure microwave-assisted extraction (HPMAE). The increase in temperature and pressure accelerates microwave-assisted extraction due to the ability of extraction solvent to absorb microwave energy [15]. Despite the fact that the closed system offers fast and efficient extraction with less solvent consumption, it is susceptible to losses of volatile compounds with limited sample throughput.

Open system is developed to counter the shortcomings of closed system such as the safety issues and it is considered more suitable for extracting thermolabile compounds. This system has higher sample throughput and more solvent can be added to the system at anytime during the process. Basically, open system operates at more mild conditions. Its advantages and disadvantages are highlighted by Luque-Garcia and Luque de Castro [14]. Open MAE system is widely used in the extraction of active compounds and is also used in analytical chemistry. This system operates at atmospheric conditions and only part of the vessel is directly exposed to the propagation of microwave radiation (mono-mode). The upper part of the vessel is connected to a reflux unit to condense any vaporized solvent. Besides that, multi-mode radiation can also be employed in open MAE system with the reflux unit.

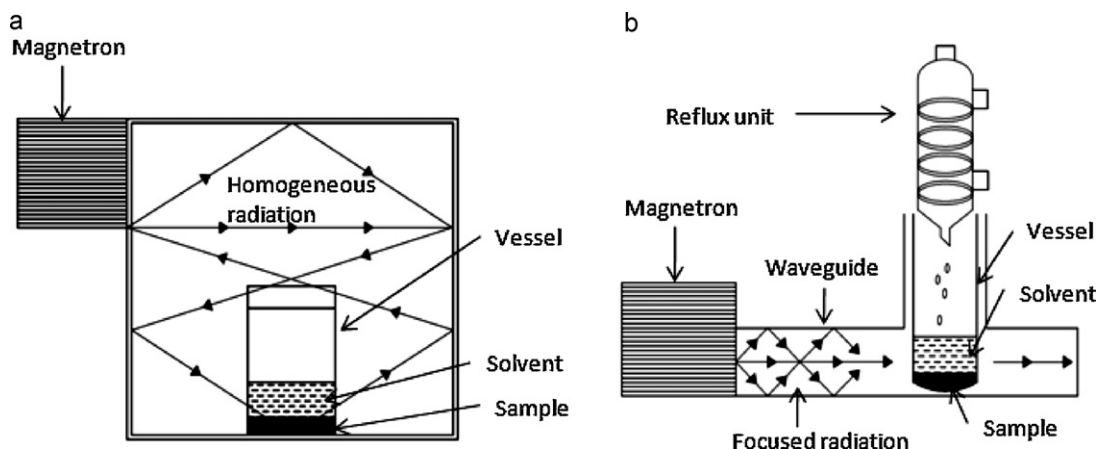


Fig. 1. (a) Closed type microwave system and (b) open type microwave system.

Permission from "microwave assisted extraction – an innovative and promising extraction tool for medicinal plant research" by Mandal et al. [11].

Poor extraction yield due to thermal degradation and oxidation of some active compounds has led to the development of more efficient MAE. These modifications are discussed hereinafter in Sections 2.1–2.5.

2.1. Nitrogen-protected microwave-assisted extraction (NPMAE)

Oxidation of the active compounds during the extraction process can be prevented by using a pressurized inert gas, such as nitrogen [16,17] and argon [18], in a closed system. Hence, oxidizable compounds under the inert condition results in a higher extraction yield. For instance, nitrogen-protected microwave-assisted extraction (NPMAE) uses nitrogen to pressurize the extraction vessel. This technique has been employed in the extraction of ascorbic acid from guava, yellow pepper, green pepper and cayenne pepper [16]. Highest extraction yield was reported in NPMAE as compared to typical MAE and Soxhlet extraction due to the protection effect exerted by nitrogen which prevents oxidation of the active compounds [16].

2.2. Vacuum microwave-assisted extraction (VMAE)

Extraction of thermal sensitive compounds using mild operating conditions i.e. low pressure and temperature can be carried out in vacuum microwave-assisted extraction (VMAE) [18,19]. This type of MAE enhances mass transfer mechanism by promoting diffusion of active compounds to the solvent via the suction pressure [19]. The risks of thermal degradation and oxidation of the active compounds can be minimized by introducing vacuum pressure, as it lowers the associated boiling temperature of the solvent. A comparison between VMAE and the typical MAE in the extraction of vitamin C from guava and green pepper; vitamin E from soybean and tea leaves showed that the increase in the extraction yield of VMAE relative to MAE in guava, green pepper, soybean and tea leaves are 53%, 145%, 20% and 60% respectively [19]. The poor yields shown by the typical MAE were claimed to be due to both thermal and oxidative degradation.

2.3. Ultrasonic microwave-assisted extraction (UMAE)

Enhancement of mass transfer mechanism in extraction can be achieved by another type of MAE known as ultrasonic microwave-assisted extraction (UMAE). Additional ultrasonic wave emitted by UMAE intensifies mass transfer mechanism as the combined microwave and ultrasonic waves provides high momentum and

energy to rupture the plant cell and elute the active compounds to the extraction solvent [20]. As a result, extraction proceeds with shorter extraction time and lower solvent consumption. UMAE has been used to extract a variety of active compounds such as lycopene from tomatoes [21], vegetable oil [22] and polysaccharides [20] from various plants. In the extraction of lycopene from tomatoes, the extraction time of UMAE was 6 min with 97.4% yield as compared to 29 min and 89.4% yield by using ultrasonic-assisted extraction (UAE). In a comparison study by Chen et al. [20] between UMAE and the conventional methods, the extraction of polysaccharides of *Inonotus obliquus* under optimal conditions of UMAE increased the yield from 2.12% to 3.25% and the purity was 73.16% as compared to 64.03% previously recorded by the traditional hot water extraction.

2.4. Dynamic microwave-assisted extraction (DMAE)

All the methods discussed so far have separate extraction step and analytical step. Both the steps work independently and required high expertise of the operator to collect and clean up the extract prior to analysis. The clean up procedure is time-consuming as it involves several steps to concentrate the extract. This can be improved by modifying the extraction process in a continuous and automatic manner and coupling on-line with analytical step. With that, dynamic microwave-assisted extraction (DMAE) has been developed where both the extraction and analytical steps can be carried out in a single step [23–29].

DMAE offers fast extraction and lower solvent consumption over conventional techniques such as reflux extraction and ultrasonic extraction as reported in the extraction of flavonoids from *Platycladus orientalis* leaves [28] and *Herba epimedii* [27]. Due to the fluidized state of extraction solvent-sample system, DMAE promotes rapid transfer of microwave energy to the extraction solvent and the sample [30]. The need of extraction cycle is eliminated and replaced by continuous extraction. Thus, the overall extraction time is reduced. Besides, the risks of analyte loss and contamination can also be minimized as the system runs continuously in a closed and automated manner. When compared to batch type MAE, continuous type MAE has the capability to extract active compounds efficiently without causing degradation [29]. The study on DMAE and typical MAE in extraction of safflower yellow from *Flos carthami* [23] and flavonoids, i.e. epimedin and icariin from *H. epimedii* [27] showed that both types of MAE accelerated the extraction and gave similar extraction yields. However, DMAE proceeded without causing an increase or decrease in pressure.

2.5. Solvent-free microwave-assisted extraction (SFME)

In view of other modified MAEs, solvent-free microwave-assisted extraction (SFME) is usually used for essential oil extraction and water can be incorporated to extract targeted compounds. SFME significantly reduces extraction time as compared to conventional methods from few hours to 20–30 min for essential oil extraction [31–33]. In the case of essential oil extraction of *Origanum glandulosum*, the extract by SFME gave the highest thymol content of 81.1% followed by typical MAE extract of 65.4% and conventional hydro-distillation (HD) extract of 41.6%. Moreover, the anti-microbial activity of thymol extracted by SFME is retained [34]. In many cases, the quality of essential oil obtained by conventional methods is affected by oxidation and hydrolyzation of active compounds due to long extraction time and high water content. Therefore, essential oil extracted by SFME is considered a better choice. For instances, the essential oils extracted by SFME from basil, garden mint and thyme are more valuable compared to those extracted by HD because higher amount of oxygenated compounds are present [35]. SFME can be improved by introducing carbonyl iron powder. The enhanced SFME can extract essential oil from *Cuminum cyminum* and *Zanthoxylum bungeanum* Maxim. in 30 min as compared to the conventional SFME of 50 min, microwave-assisted hydro-distillation of 90 min and HD of 180 min [31].

In a nutshell, the modified MAEs and the improved extraction procedures are efficient in MAE of plants if serious account is given to operating conditions and other important factors.

3. Instrumentation and equipment setup of MAE

The rapid development in MAE processes has prompted various suppliers to provide improved microwave systems and related instruments for the extraction process. Most of the microwave extractors available are laboratory-built systems based on domestic microwave oven [36,37]. Some of the commercial MAE equipments are tailored to cater for the need of specific requirements in extraction. For instance, Ultrasonic/Microwave extractor system, CW-2000 model, developed by Xin Tuo company (China) can extract lycopene from tomatoes [21]. Several commercial microwave extraction systems and the adaptation of those apparatus in MAE processes are summarized in Table 1.

The commercial microwave systems can exceed 1000 W at 100% power with maximum working temperature ranging from 200 to 300 °C. This is sufficient to extract active compounds from plants. Most of the commercial microwave systems are equipped with temperature control with monitoring system and pressure control. In the case of Ethos EX model, fiber optic sensor monitors the temperature of the system at 20 times per second and controls the microwave output in real time with non-pulse microwave heating radiation. In an open system, reflux unit is often included to control the pressure by condensing the vaporized sample mixture. Safety features of commercial microwave system include pressure relief system, explosive proof vessel and chamber door, self-sealing door and vessel cooling system.

Some modified MAE processes require additional instruments on top of the commercial system. In the case of VMAE and NPMAE, vacuum pump and nitrogen source are supplemented as shown in Fig. 2. The vacuum pump is used to provide vacuum pressure for VMAE and it is also used to remove oxygen before nitrogen is pressurized into the vessel for NPMAE. Furthermore, reflux system is installed to prevent any additional pressure built up during the extraction process. In some NPMAE, inert gas is pressurized directly inside the extraction vessel containing the sample–solvent mixture and put into close type microwave cavity. For UMAE, ultrasonic

sound transducer must be installed so that the wave can propagate directly into the extraction vessel of the focused type microwave system.

MAE can also be carried on using modified domestic oven [38–41], however this technique is less efficient compared to the commercial extractor since it does not have process monitoring system such as temperature sensor and pressure control. Besides that, some of the modified MAE processes are relatively new and require further development therefore custom made microwave system is essential to improve the performance. As in the case of DMAE, most of the instrument set up is custom made. The schematic diagram of DMAE setup developed by Chen and co-workers [23,24,27,28] is shown in Fig. 3. This system combines MAE extraction and HPLC analysis in a single step. The extraction step begins by placing the sample vessel into resonance cavity and the solvent is circulated through extraction loop. The microwave heating is activated once the solvent flow rate reaches steady state. The tuning screws in the microwave resonance are adjusted to minimize the reflected power. Once the extraction is complete, the extract is driven to the sample loop. The solvent is then mixed with the mobile phase and proceeds to the analytical step. Another type of DMAE with different equipment setup was developed by Gao et al. [29].

Commercial microwave systems are claimed to be reliable and have superior monitoring system for all kinds of microwave processes. These systems can be employed for analytical applications as well as for optimization studies of MAE. Due to the high cost of commercial microwave system, one may choose to modify domestic oven or build a custom made system. However, modifying domestic oven might not be suitable for optimization studies.

Having described the instrumentation and equipment set up, factors including the operating conditions that affect the performance of MAE will be discussed in the following section.

4. Factors influencing the performance of MAE

The efficiency of MAE strongly relies on the selection of the operating conditions and the parameters affecting the extraction mechanisms and yield. The factors that may influence the performance of MAE are solvent nature, solvent to feed ratio, extraction time, microwave power, temperature, sample characteristic, effect of stirring, etc. It is important to understand the effects and interactions of these factors on the MAE processes. Thus, this section will highlight some of the facts and guidelines regarding the selection of the operating conditions and also the interaction between the parameters. The optimum operating conditions based on various studies reported in literature are also summarized.

4.1. Solvent nature and solvent to feed ratio

The selection of suitable solvent in MAE extraction process depends on the solubility of the target analyte, solvent's penetration and interaction with sample matrix and its dielectric constant. Aqueous solution of certain organic solvent is desired for some extractions as the presence of water would improve the penetration of solvent into sample matrix and thus enhance heating efficiency [42]. Other organic solvents such as ethanol, methanol, and acetone are also found to be effective in extraction. For instance, methanol was used to extract phenolic compounds from grape skins and seeds and higher yield of polyphenols was obtained as compared to extraction using ethanol but the latter extract had stronger antioxidant properties [17]. Solvent toxicity is also evaluated in selecting suitable solvent for MAE. In the extraction of oleanolic acid from *Gymnema sylvestre*, ethanol is non-toxic thus it was selected in favor of n-butanol despite the fact that the latter has higher yield [43].

Table 1
Commercial available microwave system.

Company	Model	Common application	Adaptation to MAE	Features
Milestone	Ethos EX	Microwave extraction	Closed MAE, HPMAE	Close vessel, vessel cooling system, fiber optic temperature monitoring and control system, pressure control
	MicroSYNTH	Microwave synthesis	Closed MAE, NPMMAE,	Close vessel, magnetic stirrer, direct temperature and pressure control, overpressure control
CEM	MARS	Microwave extraction, digestion	Closed MAE	Close vessel, Internal temperature control and monitoring, connection-free pressure regulation
	Discover Star System	Microwave synthesis Microwave digestion	VMAE, NPMMAE Open MAE	Open vessel, temperature and pressure control Open vessel, temperature feedback control
Sineo	MAS-I, MAS-II	Microwave synthesis	Open MAE, VMAE	Open vessel, Magnetic stirrer, temperature control and monitoring
	MASTER	Microwave extraction	Closed MAE, HPMAE	Close vessel, pressure monitoring and control system, precision control of internal temperature
	MDS-8, MDS-6, MDS-10	Microwave digestion	Closed MAE, HPMAE	Close vessel, pressure monitoring and control system, precision control of internal temperature

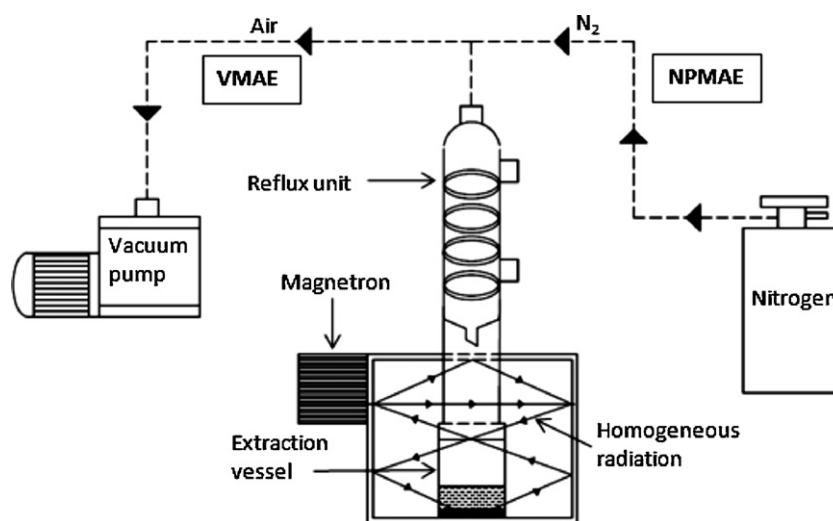


Fig. 2. Schematic diagram of VMAE and NPMMAE.

Adapted from “nitrogen-protected microwave-assisted extraction of ascorbic acid from fruit and vegetables” by Yu et al. [16].

In general, ethanol is, by far, the most used solvent and a good microwave absorber which is suitable for extracting many active compounds from plants [44].

It is important to note that the selection of a solvent for MAE can not be deduced from the conventional extraction methods as solvents that work well in conventional techniques might not be a good solvent for MAE. For example, diethyl ether that has been used extensively in solubilizing steroids from *Saxifragaceae* family is not suitable as MAE solvent [45]. However, a modifier can be added to the solvent to enhance its overall performance. Water was added as modifier to diethyl ether to enhance microwave heating efficiency in the extraction of steroids from *Rodgersia aesculifolia* Batal. [45]. In addition, ethanol or water can be added into poor microwave absorber such as hexane to improve the extraction efficiency as in the case of ginger extraction by MAE [42]. Moreover, acetone can be enhanced by adding portion of methanol in MAE of curcumin from *Curcuma longa* [46].

Room temperature ionic liquids (RTILs) are gaining attention due to their excellent solvent properties: negligible vapor pressure, wide liquid range, good thermal stability, tunable viscosity, miscibility with water and organic solvent, good solubility and extractability for various organic compounds [47]. RTILs can improve the deficiency of conventional organic solvent such as lack of molecular selectivity, unfavorable capacity and expensive recovery schemes [48]. As a result, ionic liquids have been incorporated into MAE (IL-MAE) and they have proved to be more efficient

as compared to common extraction solvents [38,47,49,50]. RTILs such as [hmim]Br [49], [bmim]Br [50] [c4min]BF₄ [38], [c6min]BF₄ [38] and [bmim]Cl [47] are recorded in the literature as extraction solvents for MAE. RTILs are preferable for precious, degradable compounds as high solvent power can accelerate the extraction and reduce the risk of over exposure to microwave heating.

Once a suitable solvent has been decided upon, its quantity to feed ratio has to be determined as it affects the extraction yield in most cases [21,51–53]. An optimum ratio of solvent to solid ratio ensures homogeneous and effective heating. Excessive solvent causes poor microwave heating as the microwave radiation would be absorbed by the solvent and additional power is required [43]. Low ratio of solvent in solid promotes mass transfer barrier as the distribution of active compounds is concentrated in certain regions which limits the movement of the compounds out of cell matrix [43]. Furthermore, the solvent and feed ratio and the vessel size exert an interactive effect in MAE as claimed by Ruan and Li [54] whereby a combination of the factors affects the efficiency of extraction especially for closed MAE system. With the same solvent to feed ratio, smaller vessel tends to generate higher internal pressure compared to larger vessel and hence it can accelerate extraction however this choice not suitable for fragile active compounds. As a summary, the ratio of solvent to solid depends on the solvent nature which is related to its ability to provide microwave heating to the sample as well as the mobility of extracted compounds in the solvent itself.

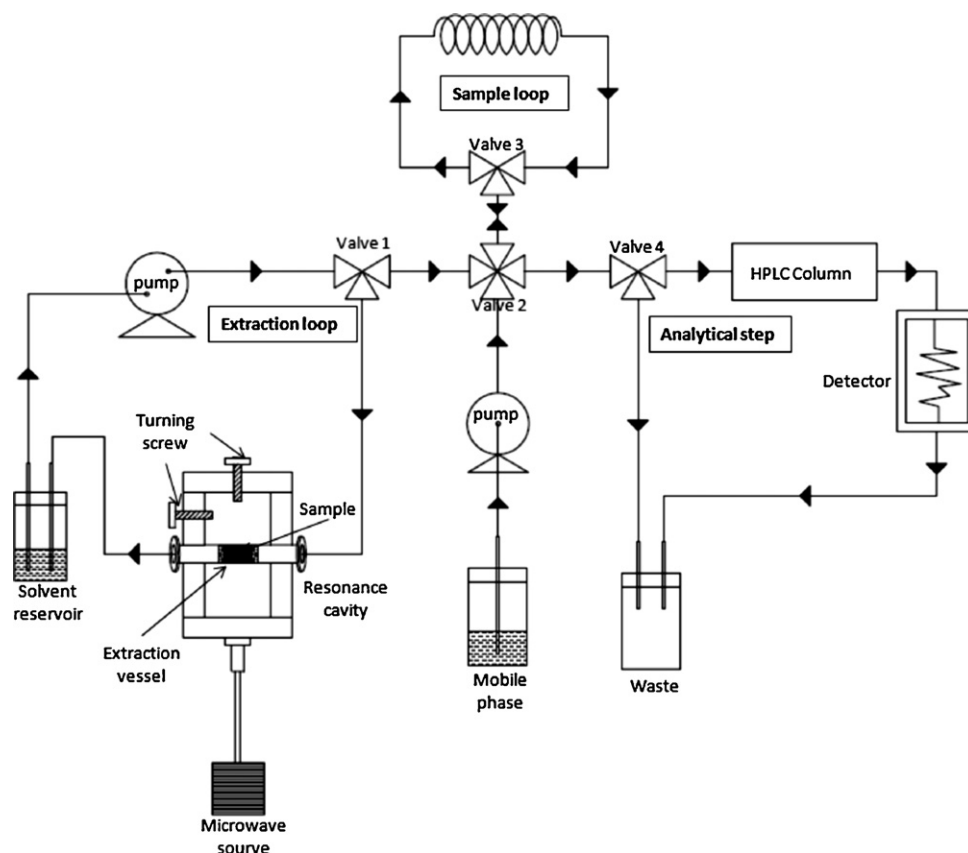


Fig. 3. Instrument setup of DMAE.

Adapted from "on-line coupling of dynamic microwave-assisted extraction with high-performance liquid chromatography for determination of andrographolide and dehydroandrographolide in *Andrographis paniculata* nees" by Chen et al. [24].

4.2. Microwave power and extraction temperature

In plant extraction, high microwave power might cause poor extraction yield due to the degradation of thermal sensible compounds. In general, the extraction yield increases proportionally with increasing microwave power up to a limit before the increase becomes insignificant or decline [43,55–57]. Microwave power provides localized heating in the sample and it acts as a driving force for MAE to destroy the plant matrix so that analyte can diffuse out and dissolve in the solvent. Increasing the power will generally improve the extraction yield and result in shorter extraction time.

As power level alone does not give sufficient information regarding the absorbed microwave energy into the extraction system, Alfaro and co-workers [42] introduced a term known as energy density, power per mass for a given unit of time, to investigate the effect of microwave power on MAE. They reported that once the plant matrix is destroyed by microwave radiation, the active compounds will be released. Higher power level does not give any contribution to the investigation of interaction between microwaves and extraction solvent with the sample.

Temperature and microwave power are interrelated as high microwave power can bring up the extraction temperature of the system. Increasing the temperature causes the solvent power to increase due to a drop in viscosity and surface tension [11]. High microwave power of MAE beyond the optimum operating power reduces extraction yield as thermo sensible compounds would risk thermal degradation. At high microwave output of 1000 W in the extraction of flavonoids from *Radix astragalii* roots, Xiao and co-workers [55] found that the extraction yield decreases if the extraction temperature was higher than 110 °C due to instability of the flavonoids at those temperatures. They also claimed that over

exposure to microwave would cause the loss of flavonoids due to thermal degradation.

The choice of extraction temperature depends on the stability and extraction yield of the desired active compound. In extraction of phenolic compound from Oolong tea, Tsubaki and co-workers [58] reported that phenolic content of the extract increases with extraction temperature and the optimum temperature was found to be 170 °C. Further increase in extraction temperature gave reduced extraction yield. This confirms that the control of extraction temperature to maintain stability and to achieve high extract yield for desired active compound is important for plant extraction.

4.3. Extraction time and cycle

Apart from interactive effect on temperature, the influence of the microwave power can be extended to the extraction time. Over exposure to microwave radiation even at low temperature or low operating power was found to decrease the extraction yield due to the loss of chemical structure of the active compounds [52,53]. In order to avoid the risk of thermal degradation and oxidation, the extraction time of MAE usually varies from few minutes up to half an hour with the exception of solvent-free microwave-assisted extraction (SFME) where longer extraction time of 1 h is necessary for complete extraction of essential oil. According to Wang et al. [31], further extension of extraction time does not favor the SFME, yet the extracted hydrocarbon are subjected to a higher risk of being oxidized and hydrolyzed. The investigators continued to propose a method of mixing carbonyl iron powders (CIP) with the moist sample to absorb microwave energy of the extraction. This method can do away with pretreatment and also would effectively shorten the

extraction time and reduce water contents of the required sample [31].

If longer extraction time is required, the risk of thermal degradation can be reduced through extraction cycle. This can be manipulated by feeding fresh solvent to the residue and repeating the extraction step to ensure the completion of the extraction. Chen et al. [59] reported that two extraction cycles of 5 min each are needed to optimize the MAE of triterpenoid saponins from *Ganoderma atrum* while Yan et al. [60] discovered that 3 extraction cycles at 15 min extraction time are best for extracting astragalosides from *R. astragali*. The total number of cycles required differs from case to case and it should be justified to save the overall extraction time and the solvent consumption for the extraction.

4.4. Plant matrix characteristic

Besides the operating conditions discussed in the above sections, the characteristics of the sample also affects on the performance of MAE. The extraction sample is usually dried, powdered and sieved into fine powder prior to the extraction for optimum extraction yield. Too small particle size would cause difficulty in separating the extract from the residue and additional clean up steps may have to be employed. Moreover, fine sample treated by solvent for 90 min prior to extraction can enhance the heating efficiency of MAE, promote diffusion and improve mass transfer of active compounds to the solvent [61]. However in some of the reported cases, extended pretreatment time did not improve extraction yield as the active ingredients may have leached out from the sample matrix before extraction [62]. The dried sample matrix pretreated with water helps localized heating of microwave. As the extraction proceeds, the moisture in the sample matrix is heated up, evaporated and generated internal pressure within the cell which ruptures the cells to release the active compounds and hence the extraction yield can be improved [7]. The findings derived from the plant matrix characteristic have led to the development of SFME in essential oil extraction from several aromatic herbs. Incorporating water pretreatment in SFME, the sample would have up to 95% moisture content before extraction. The increase of the water content in plant matrix promotes hydrolyzation and reduces the risk of oxidation of active compound [31]. From the discussion presented it is clear that particle size, moisture content and solvent pretreatment have considerable effects on the sample matrix for efficient extraction.

4.5. Effect of stirring

Having discussed the influence of solvent pretreatment and fine particle sample on MAE, the emphasis of this section will be on the effect of stirring that critically affects the mass transfer process. By introducing stirring in MAE, the negative effect of low solvent to feed ratio on extraction yield can be reduced. Moreover, the mass transfer barrier created by the concentrated active compounds in a localized region due to insufficient solvent can also be minimized resulting in better extraction yield. In other words, agitation accelerates the extraction speed by accelerating desorption and dissolution of active compounds bound to the sample matrix [54]. However, the significance of this parameter is rarely explored and only a few comments and findings have been reported [32,33,63–65].

Once the influencing parameters are well understood, MAE can be performed under optimum conditions to achieve the best yield as employed by various research groups. This will be presented in the following section.

4.6. Optimum operating conditions

The determination of optimum MAE operating conditions is usually carried out through statistical optimization studies. The optimum operating conditions of MAE based on various studies reported in the open literature are summarized in Table 2.

Table 2 shows that the most widely used solvent is ethanol with concentration ranging from 40% to 100%. Water is another common extraction solvent and is also used as a pretreatment agent for solvent-free microwave-assisted extraction (SFME). As for ionic liquids, [hmim]Br has been selected as the optimum solvent for extracting several active compounds. For optimum efficiency, the volume of the solvent is normally ranged from 10 to 50 ml/g of solid samples [15,16,20,21,38,43,47,49–51,54–57,59,60,63,66–70,73–77,80–84]. The optimum extraction time can be as short as 1 min for extraction of active compound up to an hour for essential oil extraction. In some extraction studies, several extraction cycles are required to optimize the extraction yield. Referring to the data shown in Table 2, three operating extraction modes for optimizing the yield in MAE can be seen. The first mode of operation emphasizes on the power level of microwave radiation where the extraction is carried out for a fixed microwave power through pre-determined extraction time. The power of microwave employed usually ranged from 100 to 500 W to provide the impact energy to rupture the cell wall in the extraction. The second mode of operation is more focused on the extraction temperature rather than microwave power. The extraction temperature is set at the desired set point such as 50–100 °C by regulating microwave power. This mode of operation is suitable for thermo sensitive compounds. The third mode of extraction operation is to provide enough impact energy to rupture the cell wall for the extraction as well as to reduce the risk of thermal degradation during the extraction process. This can be done by two alternatives depending on the microwave system employed. The first alternative provides continuous, desired power of microwave radiation to the extraction system until it reaches the set point of the extraction temperature, where the power is regulated to maintain the temperature. The second alternative associates pulse microwave radiation at fixed power to the extraction system. This operation mode employs high microwave power of up to 1000 W to provide excellent impact energy for the extraction. From the table, the extraction yield adopted by various research teams can be expressed in mg/g or percentage. The inconsistency in the yield expression makes it impossible to conduct a direct comparison between various extraction techniques. The same applies for the mode of extraction, instrumentation and the desired ingredients in each intended extraction. The table presented is merely a compilation of the optimum operating condition of MAE in various plant extractions.

5. General MAE procedures

The extraction of active ingredients from plants involves several steps starting from the initial sample preparation followed by the extraction of active ingredients to the clean up procedure and quantification analysis. The general procedures of MAE are illustrated in Fig. 4 and will be discussed systematically.

5.1. Sample preparation

As illustrated in Fig. 4, sample preparation involves that the desired parts of the plants are oven dried or sun dried to remove the moisture. The drying temperature is kept between 40 and 60 °C [51,66,68,70,80,85,86] to avoid thermal degradation. The dried sample is then pulverized and sieved to sizes of 40–60 mesh

Table 2
Optimum operating condition of MAE in plant extraction.

Author	MAE	Plant (active ingredients)	Instrument	Operating conditions	Extraction yield
Liazyd [63]	Closed MAE	Grape skins (anthocyanins)	Ethos 1600 microwave extractor, Milestone	40% methanol, 12.5 ml/g, 500 W, 5 min, 100 °C ^f	1.858 mg/g ^c
Yang [66]	Closed MAE	Purple corn (anthocyanins)	NJL07-3 microwave extractor, Jiequan microwave equipment, 1100 W	15 M HCl: 95% ethanol in 15:85 ratio, 20 ml/g, 555 W, 19 min ^d	1.851 mg/g ^c
Yang [67]	Closed MAE	<i>G. Sibiricum</i> Linne, whole plants (corilagin and geraniin)	Microwave extractor, Sineo	Water, 40 ml/g, 500 W, 20 min, 33 °C ^f	6.79 mg/g (corilagin), 19.82 mg/g (geraniin) ^c
Kong [68]	Closed MAE	Pigeonpea leaves (ajaninstilbene acid and pinostrobin)	MARS-II microwave unit, CEM, 1000 W	80% ethanol, 30 ml/g, 300 W, 1 min (2 min total), 2 extraction cycles, 65 °C ^f	18.8 mg/g (ajaninstilbene acid), 3.5 mg/g (pinostrobin) ^c
Inoue [69]	Closed MAE	<i>Citrus unshiu</i> (hesperidin)	MicroSYNTH labstation, Milestone, 1000 W	70% ethanol, 10 ml/g, adjusted power, 8 min, 140 °C ^e	47.7 mg/g ^c
Zheng [51]	Closed MAE	Milk thistle seed (silymarin)	Ethos-1 advanced multimode microwave extraction system, Milestone, 1600 W	82% ethanol, 38 ml/g, adjusted power, 60 min, 112 °C ^e	56.67 mg/g ^c
Hayat [70]	Closed MAE	Citrus mandarin peels (phenolic acids)	CW-2000 microwave extractor, Xintuo Technology, 800 W	66% methanol, 16 ml/g, 152 W, 49 s ^d	3779.37 µg/g ^c
Xiao [55]	Closed MAE	<i>Radix astragali</i> roots (flavonoids)	Ethos ⁺ T microwave digestion, Milestone,	90% ethanol, 25 ml/g, 1000 W, 25 min (50 min total), 2 extraction cycles, 110 °C ^f	1.19 mg/g ^c
Chen [59]	Closed MAE	<i>Ganoderma atrum</i> (triterpenoid saponins)	MDS 3003 microwave extractor, Shanghai Xinyi company	95% ethanol, 25 ml/g, 800 W, 5 min (10 min total), 2 extraction cycles, 78 °C ^f	0.97% ^a
Ballard [71]	Closed MAE	Peanut skins (phenolic compound)	MES-1000 microwave extraction system, 950 W	30% ethanol, 25 ml/g, 855 W, 30 s	144 mg/g ^c
Japon-Lujan [72]	Closed MAE	Olive leaves (oleuropein)	Microdigest 301, Prolabo, 200 W	80% ethanol, 8 ml/g, 200 W, 8 min ^d	95% ^b
Li [73]	Open MAE	<i>Tribulus terrestris</i> (steroidal saponins)	MAS-II microwave oven, Sineo, 900 W	70% ethanol, 20 ml/g, 500 W, 5 min ^d	91.30% ^b
Li [74]	Open MAE	<i>Eucommia ulmoides</i> oliv. (geniposidic and chlorogenic acid)	Commercial microwave oven from LG electrical equipment, 700 W	80% ethanol (geniposidic acid), 20% ethanol (chlorogenic acid), 20 ml/g, 350 W, 30–40 s ^d	75.6–83.2% (geniposidic acid), 77.4–86.3% (chlorogenic acid) ^b
Lu [45]	Open MAE	<i>Rodgersia aesculifolia</i> Batal, rhizomes (steroids)	Commercial microwave oven from LG electrical equipment, 800 W	80% ethyl ether, 2 ml/g, 320 W, 40 s ^d	6.35% ^a
Hu [75]	Open MAE	<i>Radix Bupleuri</i> roots (saikosaponins)	Custom made	47–50% ethanol, 30 ml/g, 360–400 W, 6 min, 73 °C ^f	95.05–96.91% ^b
Yan [60]	Open MAE	<i>Radix astragali</i> roots (astragalosides I–IV)	MARS-II microwave unit, CEM, 1000 W	80% ethanol, 25 ml/g, 700 W, 5 min (15 min total), 3 extraction cycles, 70 °C ^f	0.788 mg/g ^c
Wang [76]	Open MAE	<i>Potentilla anserina</i> rhizomes (polysaccharides)	NJC 03-2 microwave experiment equipment, Jiequan microwave equipment, 800 W	water, 15 ml/g, 370 W, 77 min, 63 °C ^f	13.33% ^a
Mandal [43]	Open MAE	<i>Gymnema sylvestre</i> leaves (oleanolic acid)	CATAR microwave extractor, Catalyst Systems, 700 W	90% ethanol, 25 ml/g, 500 W, 8 min ^d	0.76% ^a
Wang [77]	Open MAE	Apple pomace (pectin)	NJL07-3 microwave extractor, Jiequan microwave equipment, 1100 W	95% ethanol, 15 ml/g, 500 W, 21 min ^d	15.75% ^a
Li [78]	Open MAE	Defatted residue of yellow horn (triterpene saponins)	MAS-II microwave oven, Sineo, 900 W	40% ethanol, 30 ml/g, 900 W, 7 min, 3 extraction cycles, 50 °C ^f	11.62% ^a
Chen [28]	DMAE	<i>Platycladus orientalis</i> leaves (flavonoids)	Custom made	80% methanol, 1 ml/min (10 mg), 80 W, 5 min ^d	1.72% ^a
Chen [24]	DMAE	<i>Andrographis paniculata</i> nees (andrographolide and dehydroandrographolide)	Custom made	60% methanol, 1 ml/min (10 mg), 80 W, 6 min ^d	1.13% (andrographolide), 0.65% (dehydroandrographolide) ^a
Du [49]	IL-MAE	<i>Corydalis saxicola</i> Bunting (Dehydrocavidine)	MAS-I microwave oven, Sineo, 1000 W	1.5 M [hmim]Br, 20 ml/g, adjusted power, 10 min, 70 °C ^e	9.45 mg/g ^c
Lucchesi [79]	SFME	<i>Elletaria cardamomum</i> seeds (essential oil)	'DryDist' microwave laboratory oven, Milestone, 1000 W	Sample moisture in dry basis: 67%, 390 W, 75 min, 100 °C ^f	2.70% ^a
Lucchesi [35]	SFME	Basil, garden mint, thyme (essential oil)	'DryDist' microwave laboratory oven, Milestone, 1000 W	Sample moisture in wet basis: 90% (basil), 95% (mint), 80% (thyme), 500 W, 30 min, 100 °C ^f	0.029% (basil), 0.095% (garden mint), 0.16% (thyme) ^a
Wang [15]	HPMAE	Panax ginseng (ginsenosides)	WRT-C microwave system, Meicheng Technology,	70% ethanol, 40 ml/g, adjusted power, 10 min, 109–112 °C, 400 kPa ^e	43.32 mg/g ^c

Table 2 (Continued)

Author	MAE	Plant (active ingredients)	Instrument	Operating conditions	Extraction yield
Yu [16]	NPMAE	Guava, green pepper, yellow pepper and cayenne pepper (ascorbic acid)	Microwave system, Lingjiang Technology Company, 1000 W	0.25% metaphosphoric acid, 10 ml/g, 400 W, 10 min, pressurized nitrogen at atmospheric pressure ^d	2.0 mg/g (guava), 1.4 mg/g (yellow pepper), 0.4 mg/g (green pepper), 1.0 mg/g (cayenne pepper) ^c
Chen [20]	UMAE	<i>Inonotus obliquus</i> (polysaccharides)	CW-2000 UMAE apparatus, Shanghai Xintuo, 800 W	Water, 20 ml/g, 90 W, 19 min, ultrasonic wave (50 W, 40 kHz) ^d	3.25% ^a
Zhang [21]	UMAE	Tomatoes (lycopene)	CW-2000 UMAE apparatus, Shanghai Xintuo, 800 W	Ethyl acetate, 11 ml/g, 98 W, 6 min, ultrasonic wave (50 W, 40 kHz) ^d	97.40% ^b
Xiao [19]	VMAE	Guava, green pepper (vitamin C)	MAS-I microwave oven, Sineo, 1000 W	1 M acetic acid, 10 ml/g, adjusted power, 4 min (guava) and 2 min (green pepper), 70 °C (guava) and 80 °C (green pepper), vacuum pressure at 40 kPa ^e	1.383 mg/g (guava), 0.343 mg/g (green pepper) ^c
Xiao [19]	VMAE	Soybean, tea leaves (vitamin E: γ -tocopherol and α -tocopherol)	MAS-I microwave oven, Sineo, 1000 W	Ethanol, 10 ml/g, adjusted power, 20 min (tea leaves) and 10 min (soybean), 50 °C (tea leaves) and 80 °C (soybean), vacuum pressure at 40 kPa ^e	γ -Tocopherol: 1.07 mg/100 g (tea leaves); 9.07 mg/100 g (soybean), α -tocopherol: 5.51 mg/100 g (tea leaves); 2.79 mg/100 g (soybean) ^c

^a Yield (%) = mass of extracted active compound \times 100/mass of sample.

^b Yield (%) = mass of extracted active compound \times 100/mass of total active compound content in the sample.

^c Yield (mg/g or μ g/g) = mass of extracted compound/mass of sample.

^d Extraction operation mode 1.

^e Extraction operation mode 2.

^f Extraction operation mode 3.

numbers [15,17,24,27,51,54,60,66–68,70,81,87] to promote efficient extraction when exposed to solvent.

In certain cases, sample pretreatment prior to extraction is conducted to enhance the extraction process. This is done by soaking the samples in an extraction solvent such as water and methanol. As previously briefed in the case of solvent-free microwave-assisted extraction (SFME), the moisture of the sample matrix resulted from water pretreatment is responsible for the extraction of the active ingredients [31,34,79]. In other cases, some undesired components of the samples were removed by pretreatment with an organic solvent i.e. petroleum ether [24,27,78]. This can be done by soaking the sample overnight to remove the lipids portion and chlorophyll.

5.2. Extraction procedures

After sample preparation and pretreatment, the sample is subjected to extraction. For modified MAE techniques, additional instruments are required. In the case of open microwave system, reflux unit is initiated and the condensing tube is connected to the extraction vessel inside the microwave cavity. As for UMAE, ultrasonic transducer is needed and is normally set to a power of 50 W and frequency of 40 kHz [20,21]. In the case of VMAE, the condensing tube is kept under vacuum to 40 kPa [19]. In NPMAE, inert gas can be pressurized through the condensing tube in which the vessel is vacuumed. After proper setup, the sample is then irradiated under specific operating conditions. As in HPMAE of ginseng, the pressure of the vessel is allowed to reach up to 400 kPa [15].

5.3. Clean up procedures

Once the extraction is complete, the extract is filtered and collected. To prevent losses of active compounds, the extract may be filtered and the vessel may be washed with the extraction solvent. The collected extract is centrifuged at a speed ranging from 400 up to 8000 rpm [20,40,41,43,75,84,88,89] from 5 to 15 min [20,40,41,75,84,88,89] and filtered through 0.45 μ m membrane [41,74,84,88] prior to HPLC analysis. For some thermo stable and non-volatile active components, the extract is usually concentrated

by a rotary evaporator at 45–65 °C [51,75] under reduced pressure and filtered prior to quantification analysis.

The information presented in this section is intended as a reference and guidelines for MAE of active compound in plants. For better understanding and appreciation of the information shared, comparison study on the MAEs and other extraction techniques are discussed in the proceeding section.

6. Comparison between microwave-assisted extraction (MAE) and other extraction techniques

Many reports [8,13,90,91] on the application and performances of microwave-assisted extraction suggested that MAE is a good and reliable method in sample extraction. In the extraction of active compounds from plant, MAE was reported to be more efficient compared to other conventional extraction methods such as Soxhlet extraction (Soxhlet), heat reflux extraction (HRE), ultrasonic-assisted extraction (UAE), and maceration (ME). A comparison between MAE and conventional techniques at their respective optimized conditions are shown in Table 3.

From Table 3, it is obvious that the extraction yield of MAE is higher and required shorter extraction time when compared with other extraction techniques [59]. Attributed to its localized heating mechanism, MAE can be completed in just a few minutes as in the extraction of astragalosides from *R. astragali* roots whereas it normally takes a few hours for conventional extraction [60]. The advantage of MAE is further supported by the MAE of coumarin and o-coumaric acid from *Melilotus officinalis* (L.) Pallas [93], in which 10 min of MAE is sufficient as compared to 60 min by ultrasonic-assisted extraction (UAE). Reduction in extraction time from several hours to 3 min was also observed when applying MAE instead of hot solvent extraction (HSE) in the extraction of antioxidant from *Folium eriobotryae* [83]. Other than that, the ability of MAE in reducing sample preparation time and solvent consumption were also reported in the extraction of volatile organic acids from tobacco leaves [80].

The distinct advantages of MAE have turned it into a reliable extraction method with high stability and reproducibility

Table 3
Comparison on the extraction yield between MAE and conventional techniques.

References		Extraction method	Yield
Chen [59]	<i>Ganoderma atrum</i> (triterpenoid saponins)	MAE: 95% ethanol, 25 ml/g, 800 W, 5 min (10 min total), 2 extraction cycles, 78 °C	5.11% ^a
		SFE: CO ₂ + ethanol, 30 l/h (80 g sample), 25 MPa, 55 °C HRE: 95% ethanol, 25 ml/g, 1 h, 95 °C	1.52% ^a 2.22% ^a
Yan [60]	<i>Radix astragalii</i> roots (4 astragalosides)	UAE: 95% ethanol, 25 ml/g, 15 min, room temperature, ultrasonic bath	1.72% ^a
		MAE: 80% ethanol, 25 ml/g, 700 W, 5 min (15 min total), 3 extraction cycles, 70 °C	AG I: 0.788 mg/g AG II: 0.351 mg/g AG III: 0.206 mg/g AG IV: 0.278 mg/g ^b
		Soxhlet: 80% ethanol, 20 ml/g, 4 h, 90 °C	AG I: 0.770 mg/g AG II: 0.347 mg/g AG III: 0.193 mg/g AG IV: 0.242 mg/g ^b
		HRE: 80% ethanol, 20 ml/g, 1 h, 90 °C	AG I: 0.761 mg/g AG II: 0.352 mg/g AG III: 0.203 mg/g AG IV: 0.257 mg/g ^b
		UAE: 80% ethanol, 20 ml/g, 40 min, ultrasonic bath	AG I: 0.519 mg/g AG II: 0.302 mg/g AG III: 0.19 mg/g AG IV: 0.225 mg/g ^b
Li [78]	Defatted residue of yellow horn (<i>triterpene saponins</i>)	ME: 80% ethanol, 20 ml/g, 12 h	AG I: 0.411 mg/g AG II: 0.299 mg/g AG III: 0.166 mg/g AG IV: 0.206 mg/g ^b
		MAE: 40% ethanol, 30 ml/g, 900 W, 7 min, 3 extraction cycles, 50 °C UAE: 40% ethanol, 30 ml/g, 60 min, 3 extraction cycle, 50 °C, ultrasonic bath HRE: 40% ethanol, 30 ml/g, 90 min, 3 extraction cycles, 50 °C	11.62% ^a 6.78% ^a 10.82% ^a
Zhang [92]	<i>Macleaya cordata</i> (Willd) R. Br. Fruits (<i>sanguinarine</i> and <i>chelerythrine</i>)	MAE: 0.1 M HCl, 100 ml/g, 280 W, 5 min	17.10 mg/g (<i>sanguinarine</i>) 7.09 mg/g (<i>chelerythrine</i>) ^b
		UAE: 0.1 M HCl, 100 ml/g, 30 min, ultrasonic bath	10.74 mg/g (<i>sanguinarine</i>) 5.61 mg/g (<i>chelerythrine</i>) ^b
		ME: 0.1 M HCl, 100 ml/g, 30 min, 100 °C	16.87 mg/g (<i>sanguinarine</i>) 7.31 mg/g (<i>chelerythrine</i>) ^b

^a Yield (%) = mass of extracted active compound × 100/mass of sample.

^b Yield (mg/g) = mass of extracted compound/mass of sample.

Table 4
Summary of development of MAE techniques.

	Standard MAE	DMAE	NPMAE	VMAE	UMAE	SFME
Capital cost	Medium	High	Medium	Medium	High	Medium
Operating cost	Medium	Low	High	High	Medium	Low
Availability of equipment	Commercially available	Laboratory setup	Laboratory setup	Laboratory setup	Commercially available	Commercially available
Additional instrumentation	–	Dynamic closed system	Vacuum pump and source of inert gas	Vacuum pump	Ultrasonic transducer setup	–
Features	Typical microwave-assisted extraction	Continuous operation, able to couple on-line with analytical procedure	Inert condition	Vacuum condition (low pressure and temperature)	Impacted by ultrasonic wave additionally	No solvent added
Targeted active compounds	Thermo stable compound	Variety of compounds	Oxidizable compound	Oxidizable and thermal sensible compound	Low diffusion and hardly extracted compound	Essential oil
Advantages	Fast extraction	Accelerate extraction step, reduce solvent consumption, saved analytical time, effectively extract degradable compounds	Prevent oxidation of active compound during MAE	Prevent oxidation and thermal degradation of active compounds	Improve mass transfer mechanism, low solvent consumption	Higher yield and shorter extraction time for essential oil extraction
Drawbacks and limitations	Risk of thermal degradation, partitioning of analyte into headspace for HPMAE	Expensive setup, low throughput, problematic sample feeding and withdrawal	Longer extraction step	Longer extraction step, poor extraction yield for stable compounds	Additional setup	Possibility of oxidation and hydrolyzation of active compounds

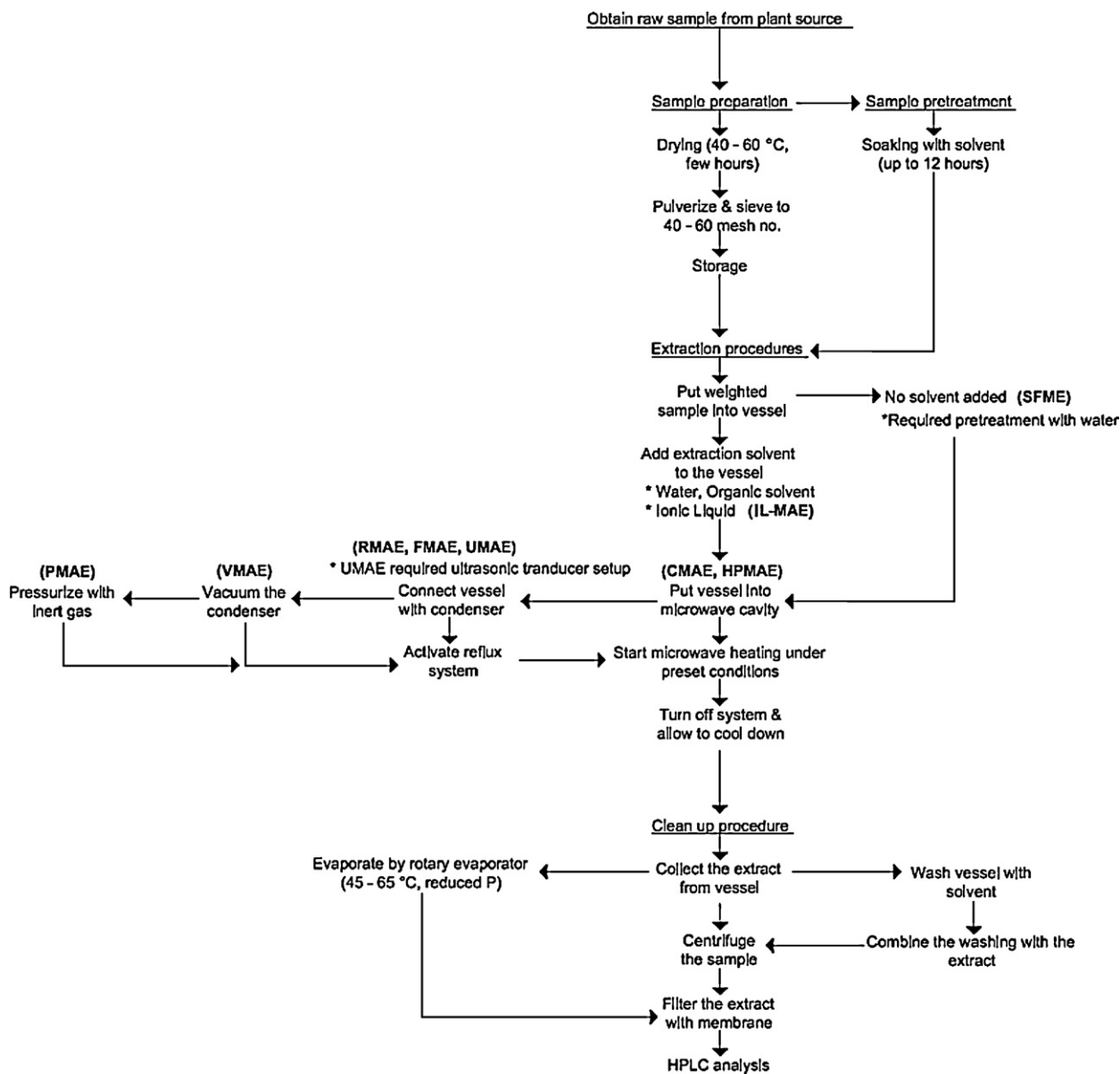


Fig. 4. Schematic procedures of MAE.

[49,63,73,87,94,95] suitable to be used in analytical chemistry where precision and repeatability of analytical result are valued most. In the investigation on the extraction of anthocyanins from grape skins, MAE has successfully quantified the limit that failed to be quantified by the conventional techniques [63]. The high efficiency of MAE is owing to the uniqueness of microwave heating and its interaction with the extraction system enhancing mass transfer. To verify this fact, scanning electron microscopy (SEM) was employed by several groups to study the mechanism of MAE [41,56,84]. The results confirmed that microwave radiation has destructive effects on extraction sample matrix and the rapid extraction occurred when the active compounds elute and dissolve in solvent once the cell is ruptured. Most importantly, the therapeutic values of the extracted active compounds can be preserved [67].

From the economic aspect, MAE is feasible as it requires moderate cost of equipment setup and is much cheaper as compared to non-conventional extraction methods such as SFE. Moreover, MAE has low risks and no major safety issues as most extractions are generally carried out under atmospheric condition. On the other hand, there are some drawbacks and limitations associated with MAE. As

discussed earlier, non-polar solvent should normally be discouraged as they are poor absorbers for microwave heating. In other circumstances, applications of non-polar solvent cannot be avoided in MAE as the solubility of extract of interest is higher as compared to polar solvents. The contradicting facts have clearly generated some difficulties in selecting solvents for MAE. However, many polarity associated problems can be overcome by adding modifiers into non-polar solvents to enhance the microwave absorbing capacity of the solvent [42,95]. Pretreatment with a polar solvent prior to extraction would also help the situation [46].

Another disadvantage of MAE is associated with its low selectivity as it is heavily dependent on the solvent nature and the extraction temperature. Hence, fractionation by liquid-liquid extraction is often carried out to acquire pure active compounds after plant extraction especially for medicinal purposes. From the comparison of the two non-conventional techniques, SFE offers higher selectivity than MAE and the system can also fractionate the extract during extraction process by regulating operating conditions. However, SFE requires expensive setup and severe operating conditions. It is also more favorable in extracting non-polar com-

pounds [96]. Another drawback of MAE is that additional time for clean up steps is required and the extracted active compounds may be lost during the procedures. To accommodate for this problem, DMAE has been developed to shorten the overall extraction process with quantification analysis that operates in a continuous manner.

Despite the disadvantages associated with MAE, its advantages are overwhelming. In general, MAE techniques are excellent in terms of its extraction efficiency, technique stability and reproducibility and also the ability to retain the functional values of extracted active compounds.

7. Guidelines for selecting MAE techniques

The brief guidelines for selecting suitable techniques i.e. DMAE, NPMAE, CMAE, SFME, UMAE are listed in this section. The summary of the development of these extraction techniques are tabulated in Table 4 and the applications, advantages and drawbacks for each technique are also presented.

Standard MAE is commonly employed either in open or closed systems to extract thermo stable compounds. For extraction of degradable active compounds, there are various modified MAE techniques that are suitable for the application. DMAE is suitable to extract degradable compounds that require multiple extraction cycles as the technique performs under mild conditions and in continuous manner. This technique promotes a fast and efficient analytical step, as it can be on-line coupled with HPLC analysis system. The drawbacks of this technique are that it has low throughput, inconvenient feeding and removal of sample and residue as well as requirement of additional equipment setup such as pumps and valves. Besides, for highly fragile compounds which pose high risks of oxidation and thermal degradation, VMAE is suitable as the extraction is carried out in vacuum condition and at low temperature. The extraction usually requires longer extraction time due to the mild condition. Alternatively extraction of thermal degradable compounds can also be achieved through NPMAE. It gives faster extraction than VMAE but requires additional extraction step. On the other hand, SFME is more preferable to be used in essential oil extraction and it is more efficient than the traditional HD method. In some circumstances in which the associated active compounds have low diffusion and difficult to be extracted, UMAE can be employed as it improves the mass transfer mechanism and reduces the extraction time. This technique can provide high activation energy or the impact energy required for the extraction to proceed. However, for other extraction cases, additional ultrasonic wave might not give higher yields due to the fact that microwave radiation alone is sufficient to overcome the activation energy of the extraction.

8. Final remarks

Microwave-assisted extraction (MAE) is a feasible extraction technique. Various MAE techniques have improved the performance in plant extraction by coupling with respective modifications. Information collected from various reports and articles were reviewed and presented as useful guidelines in this article. Hence, suitable MAE techniques with specific operating conditions can be employed for plant extraction as the performance of MAE depends on the proper selection of extraction techniques, equipment setup and the extraction procedure with optimized operating parameters. As a concluding remark, MAE system is considered a promising technique for plant extraction.

References

- [1] A. Benbow, M. Stewart, G. Yeoman, *BMJ* 322 (2001) 236.
- [2] X.K. Fang, J. Gao, D.N. Zhu, *Life Sci.* 82 (2008) 615.

- [3] A. Andrade Cetto, H. Wiedenfeld, M.C. Revilla, I.A. Sergio, *J. Ethnopharmacol.* 72 (2000) 129.
- [4] M.D. Luque de Castro, L.E. Garcia-Ayuso, *Anal. Chim. Acta* 369 (1998) 1.
- [5] B. Ondruschka, J. Asghari, *Chimia* 60 (2006) 321.
- [6] M. Herrero, J.A. Mendiola, A. Cifuentes, E. Ibáñez, *J. Chromatogr. A* 1217 (2010) 2495.
- [7] L. Wang, C.L. Weller, *Trends Food Sci. Technol.* 17 (2006) 300.
- [8] C. Sparr Eskilsson, E. Björklund, *J. Chromatogr. A* 902 (2000) 227.
- [9] H. Li, G. Li, Z. Zhang, *Chin. J. Anal. Chem.* 31 (2003) 1261.
- [10] B. Howard, *Am. Lab.* 27 (1995) 24.
- [11] V. Mandal, Y. Mohan, S. Hemalatha, *Pharmacogn. Rev.* 1 (2007) 7.
- [12] M. Chen, E.J. Siochi, T.C. Ward, J.E. McGrath, *Polym. Eng. Sci.* 33 (1993) 1092.
- [13] J.R. Dean, G. Xiong, *TrAC: Trends Anal. Chem.* 19 (2000) 553.
- [14] J.L. Luque-García, M.D. Luque de Castro, *TrAC: Trends Anal. Chem.* 22 (2003) 90.
- [15] Y. Wang, J. You, Y. Yu, C. Qu, H. Zhang, L. Ding, H. Zhang, X. Li, *Food Chem.* 110 (2008) 161.
- [16] Y. Yu, B. Chen, Y. Chen, M. Xie, H. Duan, Y. Li, G. Duan, *J. Sep. Sci.* 32 (2009) 4227.
- [17] A.A. Casazza, B. Aliakbarian, S. Mantegna, G. Cravotto, P. Perego, *J. Food Eng.* 100 (2010) 50.
- [18] V. Pasquet, J.R. Chérouvrier, F. Farhat, V. Thiéry, J.M. Piot, J.B. Bérard, R. Kaas, B. Serive, T. Patrice, J.P. Cadoret, L. Picot, *Process Biochem.* 46 (2011) 59.
- [19] X.H. Xiao, J.X. Wang, G. Wang, J.Y. Wang, G.K. Li, *J. Chromatogr. A* 1216 (2009) 8867.
- [20] Y. Chen, X. Gu, S. Huang, J. Li, X. Wang, J. Tang, *Int. J. Biol. Macromol.* 46 (2010) 429.
- [21] L. Zhang, Z. Liu, *Ultrason. Sonochem.* 15 (2008) 731.
- [22] G. Cravotto, L. Boffa, S. Mantegna, P. Perego, M. Avogadro, P. Cintas, *Ultrason. Sonochem.* 15 (2008) 898.
- [23] L.G. Chen, L. Ding, H. Zhang, J. Li, Y. Wang, X. Wang, C. Qu, H. Zhang, *Anal. Chim. Acta* 580 (2006) 75.
- [24] L. Chen, H. Jin, L. Ding, H. Zhang, X. Wang, Z. Wang, J. Li, C. Qu, Y. Wang, H. Zhang, *J. Chromatogr. A* 1140 (2007) 71.
- [25] J. You, H. Zhang, L. Ding, T. Xiao, H. Zhang, D. Song, *Chem. Res. Chin. Univ.* 23 (2007) 148.
- [26] D. Boldor, A. Kanitkar, B.G. Terigar, C. Leonard, M. Lima, G.A. Breitenbeck, *Environ. Sci. Technol.* 44 (2010) 4019.
- [27] L. Chen, H. Jin, L. Ding, H. Zhang, J. Li, C. Qu, H. Zhang, *Sep. Purif. Technol.* 59 (2008) 50.
- [28] L. Chen, L. Ding, A. Yu, R. Yang, X. Wang, J. Li, H. Jin, H. Zhang, *Anal. Chim. Acta* 596 (2007) 164.
- [29] M. Gao, B.Z. Song, C.Z. Liu, *Biochem. Eng. J.* 32 (2006) 79.
- [30] M. Ramil Criado, S. Pombo da Torre, I. Rodríguez Pereira, R. Cela Torrijos, *J. Chromatogr. A* 1024 (2004) 155.
- [31] Z. Wang, L. Ding, T. Li, X. Zhou, L. Wang, H. Zhang, L. Liu, Y. Li, Z. Liu, H. Wang, H. Zeng, H. He, *J. Chromatogr. A* 1102 (2006) 11.
- [32] C. Deng, Y. Mao, N. Yao, X. Zhang, *Anal. Chim. Acta* 575 (2006) 120.
- [33] C. Deng, J. Ji, N. Li, Y. Yu, G. Duan, X. Zhang, *J. Chromatogr. A* 1117 (2006) 115.
- [34] M. Bendahou, A. Muselli, M. Grignon-Dubois, M. Benyoucef, J.M. Desjobert, A.F. Bernardini, J. Costa, *Food Chem.* 106 (2008) 132.
- [35] M.E. Luccchesi, F. Chemat, J. Smadja, *J. Chromatogr. A* 1043 (2004) 323.
- [36] L.G. Croteau, M.H. Akhtar, J.M.R. Belanger, J.R.J. Pare, *J. Liq. Chromatogr.* 17 (1994) 2971.
- [37] K. Ganzler, I. Szinai, A. Salgo, *J. Chromatogr.* 520 (1990) 257.
- [38] Y. Lu, W. Ma, R. Hu, X. Dai, Y. Pan, *J. Chromatogr. A* 1208 (2008) 42.
- [39] F. Amarni, H. Kadi, *Innov. Food Sci. Emerg. Technol.* 11 (2010) 322.
- [40] F. Hu, C. Deng, Y. Liu, X. Zhang, *Talanta* 77 (2009) 1299.
- [41] B. Zhang, R. Yang, C.Z. Liu, *Sep. Purif. Technol.* 62 (2008) 480.
- [42] M.J. Alfaro, J.M.R. Belanger, F.C. Padilla, J.R.J. Pare, *Food Res. Int.* 36 (2003) 499.
- [43] V. Mandal, S.C. Mandal, *Biochem. Eng. J.* 50 (2010) 63.
- [44] H.Y. Zhou, C.Z. Liu, *J. Chromatogr. A* 1129 (2006) 135.
- [45] Y. Lu, X.F. Yue, Z.Q. Zhang, X.X. Li, K. Wang, *Chromatographia* 66 (2007) 443.
- [46] V. Mandal, Y. Mohan, S. Hemalatha, *J. Pharm. Biomed. Anal.* 46 (2008) 322.
- [47] F.Y. Du, X.H. Xiao, G.K. Li, *J. Chromatogr. A* 1140 (2007) 56.
- [48] A.B. de Haan, *Tsinghua Sci. Technol.* 11 (2006) 171.
- [49] F.Y. Du, X.H. Xiao, P.P. Xu, G.K. Li, *Acta Chromatogr.* 22 (2010) 459.
- [50] F.Y. Du, X.H. Xiao, X.J. Luo, G.K. Li, *Talanta* 78 (2009) 1177.
- [51] X. Zheng, X. Wang, Y. Lan, J. Shi, S.J. Xue, C. Liu, *Sep. Purif. Technol.* 70 (2009) 34.
- [52] J. Hao, W. Han, S. Huang, B. Xue, X. Deng, *Sep. Purif. Technol.* 28 (2002) 191.
- [53] J. Wang, J. Zhang, X. Wang, B. Zhao, Y. Wu, J. Yao, *Int. J. Biol. Macromol.* 45 (2009) 483.
- [54] G.H. Ruan, G.K. Li, *J. Chromatogr. B* 850 (2007) 241.
- [55] W. Xiao, L. Han, B. Shi, *Sep. Purif. Technol.* 62 (2008) 614.
- [56] S. Chemat, H. Ait-Amar, A. Lagha, D.C. Esveld, *Chem. Eng. Process.* 44 (2005) 1320.
- [57] J.H. Kwon, J.M.R. Belanger, J.R.J. Pare, V.A. Yaylayan, *Food Res. Int.* 36 (2003) 491.
- [58] S. Tsubaki, M. Sakamoto, J. Azuma, *Food Chem.* 123 (2010) 1255.
- [59] Y. Chen, M.Y. Xie, X.F. Gong, *J. Food Eng.* 81 (2007) 162.
- [60] M.M. Yan, W. Liu, Y.J. Fu, Y.G. Zu, C.Y. Chen, M. Luo, *Food Chem.* 119 (2010) 1663.
- [61] X. Pan, G. Niu, H. Liu, *Chem. Eng. Process.* 42 (2003) 129.
- [62] X. Pan, G. Niu, H. Liu, *J. Chromatogr. A* 922 (2001) 371.

- [63] A. Liazid, R.F. Guerrero, E. Cantos, M. Palma, C.G. Barroso, *Food Chem.* 124 (2011) 1238.
- [64] A.M.M. Sousa, V.D. Alves, S. Morais, C. Delerue-Matos, M.P. Gonçalves, *Biore-sour. Technol.* 101 (2010) 3258.
- [65] C. Deng, N. Yao, B. Wang, X. Zhang, *J. Chromatogr. A* 1103 (2006) 15.
- [66] Z. Yang, W. Zhai, *Innov. Food Sci. Emerg. Technol.* 11 (2010) 470.
- [67] Y.C. Yang, J. Li, Y.G. Zu, Y.J. Fu, M. Luo, N. Wu, X.L. Liu, *Food Chem.* 122 (2010) 373.
- [68] Y. Kong, Y.G. Zu, Y.J. Fu, W. Liu, F.R. Chang, J. Li, Y.H. Chen, S. Zhang, C.B. Gu, *J. Food Compos. Anal.* 23 (2010) 382.
- [69] T. Inoue, S. Tsubaki, K. Ogawa, K. Onishi, J. Azuma, *Food Chem.* 123 (2010) 542.
- [70] K. Hayat, S. Hussain, S. Abbas, U. Farooq, B. Ding, S. Xia, C. Jia, X. Zhang, W. Xia, *Sep. Purif. Technol.* 70 (2009) 63.
- [71] T.S. Ballard, P. Mallikarjunan, K. Zhou, S. O'Keefe, *Food Chem.* 120 (2010) 1185.
- [72] R. Japon-Lujan, J.M. Luque-Rodriguez, M.D. Luque De Castro, *Anal. Bioanal. Chem.* 385 (2006) 753.
- [73] T. Li, Z. Zhang, L. Zhang, X. Huang, J. Lin, G. Chen, *J. Sep. Sci.* 32 (2009) 4167.
- [74] H. Li, B. Chen, Z. Zhang, S. Yao, *Talanta* 63 (2004) 659.
- [75] Z. Hu, M. Cai, H.H. Liang, *Sep. Purif. Technol.* 61 (2008) 266.
- [76] J. Wang, J. Zhang, B. Zhao, X. Wang, Y. Wu, J. Yao, *Carbohydr. Polym.* 80 (2010) 84.
- [77] S. Wang, F. Chen, J. Wu, Z. Wang, X. Liao, X. Hu, *J. Food Eng.* 78 (2007) 693.
- [78] J. Li, Y.G. Zu, Y.J. Fu, Y.C. Yang, S.M. Li, Z.N. Li, M. Wink, *Innov. Food Sci. Emerg. Technol.* 11 (2010) 637.
- [79] M.E. Lucchesi, J. Smadja, S. Bradshaw, W. Louw, F. Chemat, *J. Food Eng.* 79 (2007) 1079.
- [80] X. Zhu, Q. Su, J. Cai, J. Yang, *Anal. Chim. Acta* 579 (2006) 88.
- [81] H. Teng, K. Ghafoor, Y.H. Choi, *J. Korean Soc. Appl. Biol. Chem.* 52 (2009) 694.
- [82] X. Pan, H. Liu, G. Jia, Y.Y. Shu, *Biochem. Eng. J.* 5 (2000) 173.
- [83] Z. Chen, L. Zhang, G. Chen, *J. Chromatogr. A* 1193 (2008) 178.
- [84] M. Gao, W. Huang, M. RoyChowdhury, C. Liu, *Anal. Chim. Acta* 591 (2007) 161.
- [85] N. Sutivisedsak, H.N. Cheng, J.L. Willett, W.C. Lesch, R.R. Tangsrud, A. Biswas, *Food Res. Int.* 43 (2010) 516.
- [86] C. Sun, Y. Xie, H. Liu, *Chin. J. Chem. Eng.* 15 (2007) 474.
- [87] V. Romarís-Hortas, A. Moreda-Piñeiro, P. Bermejo-Barrera, *Talanta* 79 (2009) 947.
- [88] C. Sun, H. Liu, *Anal. Chim. Acta* 612 (2008) 160.
- [89] H.Y. Zhou, C.Z. Liu, *J. Chromatogr. B* 835 (2006) 119.
- [90] L. Sanchez-Prado, C. Garcia-Jares, M. Llompert, *J. Chromatogr. A* 1217 (2010) 2390.
- [91] J.L. Luque-Garcia, M.D. Luque de Castro, *Talanta* 64 (2004) 571.
- [92] F. Zhang, B. Chen, S. Xiao, S. Yao, *Sep. Purif. Technol.* 42 (2005) 283.
- [93] E. Martino, I. Ramaiola, M. Urbano, F. Bracco, S. Collina, *J. Chromatogr. A* 1125 (2006) 147.
- [94] D. Sterbová, D. Matejíček, J. Vlček, V. Kubán, *Anal. Chim. Acta* 513 (2004) 435.
- [95] S. Elkhori, J.R.J. Pare, J.M.R. Belanger, E. Perez, *J. Food Eng.* 79 (2007) 1110.
- [96] M. Zougagh, M. Valcárcel, A. Ríos, *TrAC: Trends Anal. Chem.* 23 (2004) 399.