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Review Article

Extraction of Neutraceuticals from Plants by Microwave Assisted Extraction

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ABSTRACT

Several naturally derived food substances have now moved from territory of traditional and folklore medicines to rigorous studies aimed at identifying natural preventive therapies for disease. During chemical treatment bioactive substance can also be modified with possible negative impacts. Therefore safe and reliable extraction procedure should be improved and assessed. Because of growing interest in the extraction of bioactive compounds and nutraceuticals from plants and herbs and the search for sustainable extraction technique, MAE is fast realizing its promise as a technique that can respond to the rigorous demands this field necessitates. The traditional techniques of solvent extraction of plant materials require long extraction time and have low efficiency. Moreover, many natural products are thermally unstable and may degrade during thermal extraction. There are several novel techniques including ultrasound assisted extraction, microwave assisted extraction, supercritical fluid extraction and accelerated solvent extraction have been developed for the extraction of phytoconstituents from plants in order to shorten the extraction time, decrease the solvent consumption, increase the extraction yield and enhance the quality of extracts. MAE has risen rapidly in the latest decade, and for most applications it has proven to be effective compared to traditional extraction techniques. The studies reported prove many advantages that make MAE an excellent substitute to traditional methods such as soxhlet and solid liquid extraction, as well as other environmentally benign technologies. Results also depicted that MAE coupled with high speed counter current chromatography is highly suitable technique for extraction, separation and purification.

Key words: Microwave assisted extraction, Solid liquid extraction, Ultrasound assisted extraction, Accelerated extraction, Novel extraction technique.

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INTRODUCTION

Extraction may be defined as the process of removal of desirable soluble components from a substance, leaving out those, which are not wanted, with the aid of solvents and standardized process. Plants tissues contain chemical substances some of which provide relief and treatment in a variety of diseased conditions. In many cases the medicinal value of a natural drug, is due to single constituent. However the isolation of active agent may be an extremely difficult and expensive process. If the other constituents have no undesirable effect, the administration of the unprocessed drug or its partially purified extract may provide the desired therapeutic effect. At times the presence of other constituents may beneficially modify the effect of the active principle and hence the removal of such other constituents may not be desirable. These are the reasons why the Extracts are still used in pharmaceutical practice.[1, 2]

The various conventional extraction processes including maceration, digestion, percolation, and soxhelation are available for the extraction of the phyto-constituents from plants. However, conventional extraction technique has also significant drawbacks as the long time required for the extraction and the large amount of organic solvent wasted, which is not only expensive to dispose off but which can cause environmental pollution itself. Moreover, the conventional device is not easily automated.[3] There are several novel techniques have been developed for the extraction of phyto-constituents from plants in order to shorten the extraction time, decrease the solvent consumption, increase the extraction yield and enhance the quality of extracts.[2]

Table 1: Comparison of MAE with Soxhlet extraction technique

Prameters	MAE	Soxhlet extraction
Description	Sample is immersed in microwave absorbing solvent in a closed vesscle and irradicated with microwave energy.	Sample is placed in a glass fiber thimble and, by using a Soxhlet extractor, the sample is repeatedly percolated with condensed vapors of the solvent
Extraction time	3–30 min	3–48 hrs
Sample size	1–10 g	1–30 g
Solvent consumption	10–40 ml	30–200 ml
Investment	Moderate	Low
Advantages	• Fast and multiple extractions • Low solvent volume	• No filtration required
Drawbacks	• Elevated temperatures • Extraction solvent must be absorb microwaves • Clean up step is needed • Waiting time for the vessels to cool down.	• Long extraction times • Large solvent volumes • Clean up step is needed

One of the main advantages using MAE is the reduction of extraction time when applying microwaves. This can mainly be attributed to the difference in heating performance employed by the microwave technique and conventional heating. In conventional heating a finite period of time is needed to heat the vessel before the heat is transferred to the solution, while microwaves heat the solution directly. This keeps the temperature gradient to a minimum and accelerates the speed of heating. Additionally MAE allows for a significant reduction in organic solvent consumption as well as the possibility of running multiple samples.[3] The comparison of MAE with conventional soxhlet extraction is shown in Table 1.[4]

CHARACTERISTICS OF MICROWAVE

Microwaves lie in the electromagnetic spectrum between infrared waves and radio waves.

- They have wavelengths between 0.01 and 1 meter, and operate in a frequency range between 0.3 and 300 GHz.
- Compared to conventional heating, microwave heating enhances process and results in significant energy saving. This is primarily because microwaves heat up just the sample and not the apparatus, and therefore energy consumption is less.
- Microwave heating has been found to be up to 50% more efficient, compared to conventional heating methods.
- The energy in a microwave photon (0.037 kcal/mol) is very low, relative to the typical energy required to break a molecular bond (80-120 kcal/mol). Therefore microwave excitation of molecules does not affect the structure of an organic molecule, and the interaction is purely kinetic.
- Reaction and conducted through microwaves are cleaner and envoi mentally friendly than conventional heating methods.[5]
- Microwaves will transfer energy in 10^{-9} seconds with each cycle of electromagnetic energy. This means that energy transfers at a faster rate than the molecules can relax; this leads to enhancement interaction rates and product yields.[6]
- Microwave radiation can be focused directly on to the sample, heating is more efficient and thus homogeneity and re extraction (MAE) offers a rapid delivery of energy to a total volume of solvent and reproducibility improve greatly.[7]

THE HISTORY OF MAE

The use of microwave radiation as a method of heating is over five decades old. Microwave technology originated in 1946, when Dr. Percy Le Baron Spencer, while conducting laboratory tests for a new vacuum tube called a magnetron, accidentally discovered that a candy bar in his pocket melted on exposure to microwave radiation. Dr. Spencer developed the idea further and established that microwaves could be used as a method of heating. Subsequently, he designed the first microwave oven for domestic use in 1947. Although microwave energy has great potential for rapidly heating materials, microwave ovens have only recently appeared in analytical laboratories. Applications of microwave-assisted techniques in other fields of analytical chemistry, such as sample drying, moisture measurements, chromogenic reactions, speciation and nebulization of sample solutions can be found in a recent review by Jin *et al*. From digestion procedures, the step to extraction procedures is not far. Even so, it would take more than 10 years before the first publication on extractions appeared. In the food technology area, Greenway and Kometa extracted vitamins from foodstuffs. Today MAE has become relatively mature and some standard methods have been published.[8]

PRINCIPLES AND MECHANISMS

MAE offers a rapid delivery of energy to a total volume of solvent and solid matrix with subsequent heating of the solvent and solid matrix, efficiently and homogenously. On exposure to an oscillating electromagnetic field of appropriate frequency, polar molecules try to follow the field and align themselves in phase with the field. However, owing to inter-molecular forces, polar molecules experience inertia and are unable to follow the field. This results in the random motion of particles, and this random interaction generates heat. Dipolar polarization can generate heat by either one or both the following mechanisms.[5]

- Interaction between polar solvent molecules such as water, methanol and ethanol
- Interaction between polar solute molecules such as ammonia and formic acid.

The key requirement for dipolar polarisation is that the frequency range of the oscillating field should be appropriate to enable adequate inter-particle interaction. If the frequency range is very high, inter-molecular forces will stop the motion of a polar molecule before it tries to follow the field, resulting in inadequate inter-particle interaction. On the other hand, if the frequency range is low, the polar molecule gets sufficient time to align itself in phase with the field. Hence, no random interaction takes place between the adjoining particles. Consequently, microwaves can heat a whole material to penetration depth simultaneously. Furthermore, the migration of dissolved ions increased solvent penetration into the matrix and thus facilitated the release of the chemicals Figure 1.[5]

INSTRUMENTATION

There are two types of commercially available MAE systems; closed extraction vessels under controlled pressure and temperature, and focused microwave ovens at atmospheric pressure.[2,4,7]

CLOSED-VESSEL MICROWAVE ASSISTED EXTRACTION SYSTEM

The closed MAE system is generally used for extraction under drastic condition such as high extraction temperature. Today MAE equipment designed for laboratory purposes is safe to work with and offers the user various ways to control the extraction process. Commercial systems used for closed-vessel MAE consist of a magnetron tube, an oven where the extraction vessels are set upon a turntable, monitoring devices for controlling the temperature and pressure, and a number of electronic power components.

The extraction process starts with loading of the sample into the extraction vessel, followed by solvent addition and closing of the vessel. Microwave radiation is applied and a pre-extraction step is initiated in order to heat the solvent to the set values. Normally the heating takes less than 2 min. The sample is further irradiated and extracted for a certain time (static extraction step), usually in the range of 10–30 min. When the extraction is concluded the samples are allowed to cool down to a temperature reasonable to handle. Prior to analysis the addition of an internal standard and/or a clean step might be have needed.

FOCUSED MICROWAVE-ASSISTED EXTRACTION SYSTEM (FMAE)

FMAE was performed at atmospheric pressure at a frequency of 2450 MHz using a Soxhlet apparatus with programmable heating power. Powdered air-dried material was placed in a quartz-extraction vessel with solvent. After extraction, the vessel was allowed to cool to room temperature extracts were centrifuged and the supernatant removed and evaporated to dryness under vacuum. The apparatus used in FMAE is shown in Figure 2.

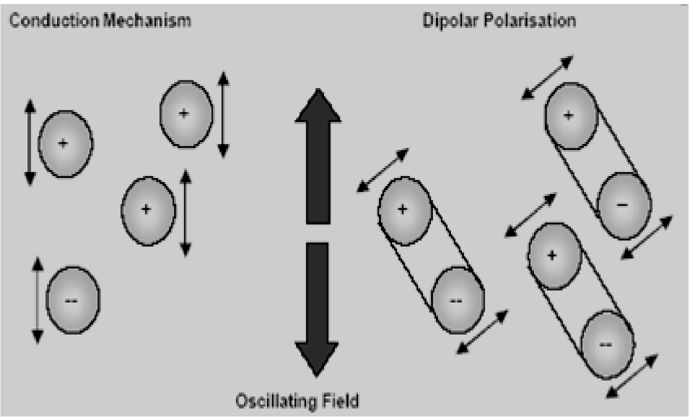

Figure 1: Methods of heating by microwave radiation.

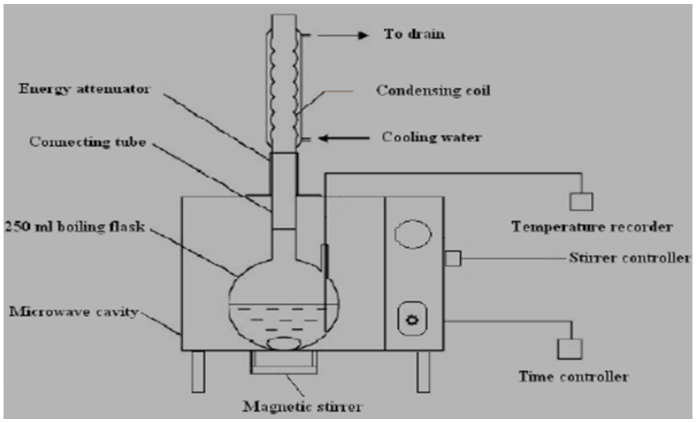

Figure 2: Apparatus for focused microwave assisted extraction.

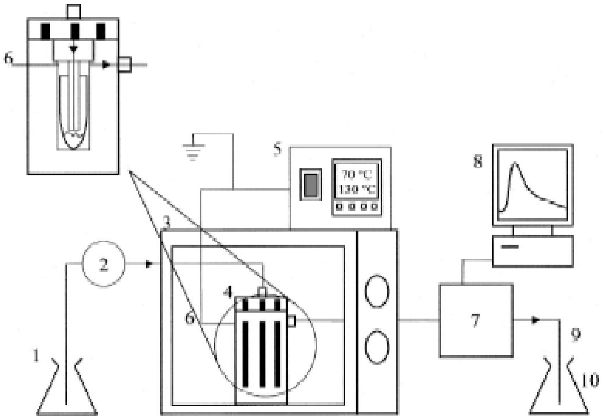

Figure 3: Apparatus for dynamic microwave assisted extraction.

1-Solvent, 2-Pump, 3-Microwave oven, 4-Extraction cell, 5-Temperature set point controller, 6-Thermocouple, 7-Fluorescence detector, 8-regestering device, 9-Ristrictor, 10-Extractor.

Table 2: Application of Microwave assisted extraction to natural product.

Name of compound	Matrix	Extraction condition	References
Vicine	*Vicia faba*	Methanol: water (1:1); 30S; 1140W	10
Terpenes	*Vitis vinifera*	Dichloro methane; 475W; 10 Min.; 90 °C	11
Essential oil	*Monarda fistulosa*	Hexane	12
Volatile oil	*Mentha piperita*	Hexane, Alkanes	13
Volatile oil	*Thuja occidentalis*	Hexane, Alkanes	13
Essential oil	Rosemary and Peppermint leaves	Hexane, Carbon tetra chloride, Toluene, 750W; 60S	14
Carotenoids	*Capsicum annuum*	Acetone, ethanol, Dioxanel; 50W; 120S	15
Taxanes	*Taxus* Species	Ethanol;54S; 85 °C	16
Withanolides	*Lochroma gesnerioides*	Methanol; 25W; 40S	17
Cocaine, Benzoylecgonine	*Erythroxylum coca*	Methanol; 125W; 30S	7
Ginsenoside	Ginseng	Methanol; 300W; 2450MHz; 30S; 72.2°C	18
Essential oil	Rose hip seed	Hexane; 30Min; 40°C	19
Para Cymene, Thymoquinone	*Nigella sativa*	Distilled water; 850W; 10Min.	20
Zingiberene	*Zingiber officinale*	Microwave absorption media (ICP, GP, ACP); 85W; 30Min; 100°C	9
Alphapinene, Betapinene, Camphene	*Illicium verum*	Microwave absorption media (ICP, GP, ACP); 85W; 30Min; 100°C	9
Artimisinin, Artimisinic acid	*Artimisia annua*	Water, Ethanol, Toluene; 15 Min.60°C	21

Antioxidant	*Hierochloe odoratol*	Acetone, Ethylacetate, Acetone: Hexane (1:1)	22
Essential oil	*Cuminum cyminum Zanthoxylum bungeanum*	Solvent free microwave exytraction, 30 Min., 85 W	9
Furanocoumarins	*Pastinaca sativa*	Methanol 80%; Power 40%; 30Min.	23
Polyphenols & Caffine	*Green tea leaves*	Ethanol; 8min.; Liquid /Solid ratio 10:1	24
Trans-resveratrol	*Rhizma polygoni cuspidati*	Ionic liquid; 10Min.;60°C	25
Notoginseng saponin	*Panax notoginseng*	Water saturated n- butanol; 4min.; 50°C; 125 W	26
Glycyrrhizic acid	Licorice root	Ethanol 40-60%; 5Min.; 85-90°C	27
Sanguinarine, Chelerythrine	*Macleaya cordata*	Hydrochloric acid: aquous solution; 5Min.	28
Campothecin	*Nothapodytes foetida*	Methanol 90%; 7 Min.100 W.	29
Cocaine	*Erythroxylum species*	Water & methanol; 125W: 30 Sec.	30
Carvon, Limonene, Piperitotenone	*Lippia alba*	Water: 800W: 30Min.	31
Coumarin	*Melilotus officinalis*	Ethanol 50%; 5Min; 50°C	32
Scutellarin	*Erigeron breviscapus*	Ethanol & water; 40Min.; 80°C	33
Ginsenosides	*Panax ginseng*	Ethanol 70%; 15Min.; 60°C	34
Artimisinin	*Artemisia annua*	Water: acetonitrile (40:60v/v), 40Min.; 700W: 50°C	35
Solanesol	Tobacco	Methanol; 700 W; 60°C 12 min.	36

DYNAMIC MAE SYSTEM

Ericsson and Colmsjo (2000) introduced a dynamic MAE system, which was demonstrated to yield extract equivalent to yield of extract from Soxhlet extraction, but in much shorter time. The dynamic microwave extractor consists of a solvent delivery system, a microwave oven, an extraction cell, a temperature set point controller with type K thermocouple, HPLC fluorescence detector and fused-silica restrictor. The apparatus used in dynamic MAE is shown in Figure 3.

APPLICATION OF MAE

MAE has been used to extract nutraceuticals from plants such as essential oil, lipids, dietary supplements etc**.** MAE can extract neutraceutical products from plant sources in a fast manner than conventional solid liquid extractions. A higher extraction yield can be achieved in a shorter extraction time using MAE.The main advantages of MAE over Soxhlet extraction are associated with the drastic reduction of solvent consumption (5 V 100ml) and extraction time (40S V 6h). It was also find that the presence of water in solvent of methanol had a beneficial effect and allowed faster extractions than with organic solvent alone.[2]

Solvent free microwave extraction (SFME), a novel method used to extract essential oils from plant materials, has been developed in the recent years. SFME is a green technology, because essential oil can be extracted by this method without addition of any solvent. For fresh materials, SFME can be used to extract essential oil directly. For dried materials, however, it was necessary to moisten the samples before extraction, which made the process complex and time consuming. To promote the process of extraction of essential oil from dried plant materials, Microwave absorption media (MAM). Iron carbonyl powder (ICP), Graphite powder (GP), Active carbon powder (ACP) are all well-known microwave absorbers.[9] The various application of MAE illustrated in Table 2.

CONCLUSION

MAE has risen rapidly in the latest decade. The major benefits are decreased extraction times reduced solvent consumption and increased sample throughput. Although careful method development may generate some extraction selectivity, there is a need for additional clean up after completed extraction. For some application only a filtration step is needed, whereas for others solid-phase extraction or additional liquid-liquid extraction steps have to be performed to be able to use the final analytical technique. Compared to super critical fluid extraction (SFE) this is a disadvantage, since clean up is usually not needed for this relatively selective technique. By considering economical and practical aspects, MAE is a strong competitor to other recent sample preparation techniques. The companies that have manufactured MAE plants, especially the smaller, are receiving inquiries from industries worldwide. Much of the demand for the use of MAE will come from consumers who may be willing to pay a little extra for products that were produced without the use of solvents. The future of MAE in research look bright. With so many new applications under consideration and funding available for any technology that promises to be clean, there will be increased research activity for many years to next century. It seems that MAE is now over the hump and that it is rapidly developing into extraction method of choice for the 21 century.

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CONFLICT OF INTEREST

The author declares for no conflict of interest inherent in his submission.

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