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Research

A REVIEW ON MICROWAVE – ASSISTED EXTRACTION OF HERBAL BIOACTIVES

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Abstract:
Microwave-assisted extraction (MAE) has emerged as a powerful technique for the efficient extraction of bioactive compounds from herbal materials. This method leverages microwave energy to heat the solvent and plant matrix, leading to the rapid breakdown of plant cells and the release of valuable phytochemicals. Compared to conventional extraction methods, MAE offers several advantages, including shorter extraction times, reduced solvent consumption, and higher yields of target compounds.

The application of MAE in the extraction of herbal bioactives is particularly advantageous due to its ability to preserve the integrity and biological activity of sensitive compounds. This is achieved through the precise control of extraction parameters such as microwave power, extraction time, solvent type, and solvent-to-sample ratio. These parameters can be optimized to enhance the efficiency and selectivity of the extraction process, ensuring the maximal recovery of desired bioactives such as polyphenols, flavonoids, alkaloids, and essential oils.

Numerous studies have demonstrated the effectiveness of MAE in extracting a wide range of bioactive compounds from various medicinal plants, highlighting its potential for both research and industrial applications. The use of green solvents and the possibility of scaling up the process further underscore the sustainability and economic viability of MAE.

In conclusion, microwave-assisted extraction represents a promising and innovative approach for the extraction of herbal bioactives. Its ability to provide high-quality extracts with minimal environmental impact positions it as a valuable tool in the fields of phytochemistry, pharmacology, and natural product research. Future advancements in MAE technology and methodology will likely expand its applications and enhance its efficiency, paving the way for the development of novel herbal formulations and therapeutic agents.

Key words: Microwave-Assisted Extraction (MAE), Bioactive Compounds, Herbal Extraction, Green Solvents, Phytochemistry.

Introduction:

Extraction involves separating dissolvable substances from non-dissolvable residues using solvent(s); it can be in form of liquid or solid. There are two categories of extraction which are traditional and modern; the former includes Soxhlet, soaking, maceration, ultra-

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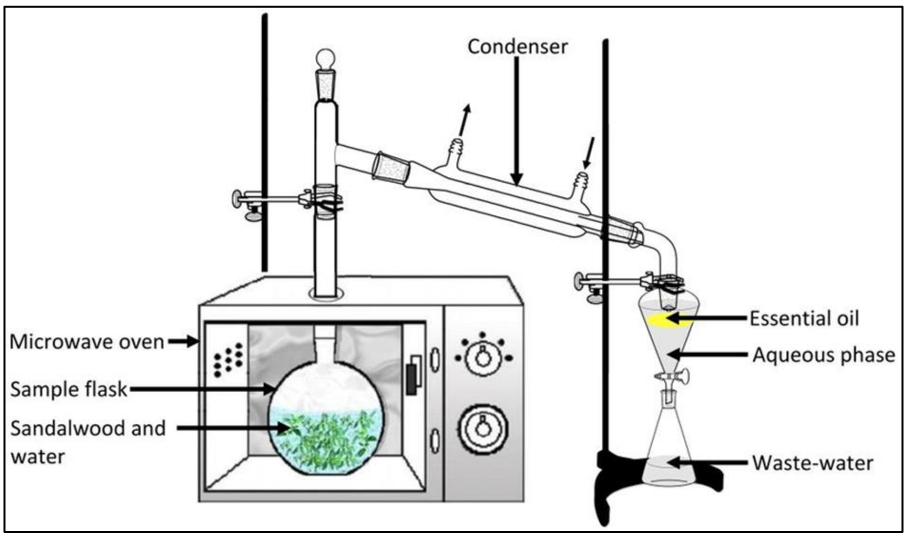
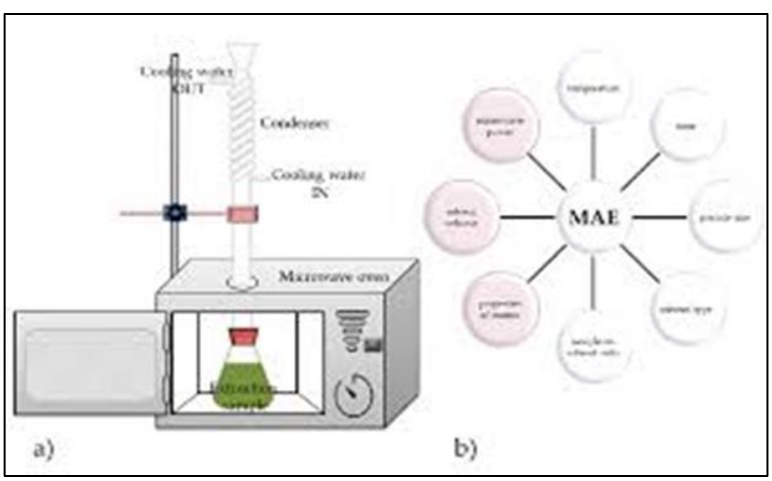
sonication, turbo-fast blending, and solvent permeation; the latter includes ultrasonic-assisted, subcritical, supercritical CO2, enzyme-assisted, pressure-assisted, and microwave-assisted methods. The traditional methods are mainly associated with an extended time of extraction, destruction of heat-sensitive bioactive compounds, and enormous consumption of solvents. It is then important to explore modern methods of extraction to overcome the setbacks associated with the traditional methods. Out of all the modern methods of extraction, microwave-assisted extraction (MAE) has received the greatest attention due to its reduced consumption of solvent, shorter operation time, reproducibility, improved recovery yield, good selectivity, and reduced sample manipulation. Gedye et al. and Giguere et al. were groups that first described the usage of microwave energy in 1986, it was employed in organic synthesis; microwave energy was also employed in the extraction of biological samples for analyzing organic compounds. [1]



Microwaves are electromagnetic fields in the frequency range 300 MHz to 300 GHz or between wavelengths of 1 cm and 1m. These electromagnetic waves made up of two oscillating perpendicular fields: electrical field and magnetic field. Microwaves are used as information carriers or as energy vectors. This second application is the direct action of waves on material which is able to absorb a part of electromagnetic energy and to transform it into heat. The most commonly used frequency for commercial microwave instruments is



2450 MHz, which corresponds to an energy output of 600- 700 Watts.1 At this frequency, the electric field swings the orientation of water molecules 2.45 x 109 times every second and the chaos inherent to the system opposes the synchrony of the oscillation with whether it is plants or microbes, exists as pressurized liquid extraction, vertical (turbo) extraction, supercritical fluid extraction.3,4 The first step in the process of obtaining secondary metabolites from biogenic materials is to release them from the matrix

Extraction Techniques

1. Supercritical fluid extraction (SFE): This is a green technique for extracting compounds from plants that employ supercritical fluids, such as carbon dioxide, as "Microwave extraction solvent. SFE offers several advantages over conventional extraction techniques because of the physicochemical properties of supercritical solvents, which include improved transport characteristics that result in quicker extraction rates. The high-quality extracts generated by SFE make it a hopeful method in the domains of food, pharmaceuticals, and cosmetics.

Principle:

1.Critical Point:

- A substance reaches its supercritical state when the temperature and pressure exceed the critical point, beyond which distinct liquid and gas phases do not exist.
- In the supercritical state, the fluid has gas-like diffusivity and viscosity, along with liquid-like density, enabling it to penetrate materials like a gas and dissolve substances like a liquid.

Mechanism:

1. Formation of Supercritical Fluid:

- A fluid (commonly carbon dioxide, CO_2) is compressed and heated beyond its critical temperature and pressure to become supercritical.
- For CO_2, this occurs at a critical temperature of **31.1°C** and critical pressure of **7.38 MPa**.

2. Contact with Feed Material:

- The supercritical fluid is passed through the material containing the target compounds (e.g., plants, seeds, or other matrices).

3. Dissolution and Extraction:

- The supercritical fluid penetrates the material, dissolving the target compounds due to its high density and solvating power.[3]

that of the field. Thus creating an intense heat that can escalate as quickly as several degrees per second (estimated as 100C/s at 4.9 GHz). Most of the bulk of the biomass, irrespective of

by means of extraction.5 Due to the often very complex composition of the material and the minute amounts of some of the constituents present, the choice of extraction method is of great importance.[2]

2. Ultrasound-assisted extraction (UAE): This is a fast, cost-effective, and scalable technique for extracting compounds from plant resources. The process involves using ultrasound waves, which enhance the mass transport of bio-active constituents from the plant material to the solvent media through cavitation, mechanical agitation, and thermology.

Principle:

UAE is based on the application of high-frequency sound waves (usually between 20 kHz and 100 kHz) to a liquid or solvent that contains the material to be extracted. These sound waves generate mechanical energy, leading to several key phenomena that facilitate the extraction process.

Mechanism:

Cavitation:

- Ultrasonic waves create microscopic bubbles in the liquid. These bubbles rapidly grow and collapse, producing localized high pressure and temperature.
- The intense energy released during cavitation disrupts the cell walls of the material, enhancing the release of intracellular compounds into the solvent.[4]

Pressurized liquid extraction (PLE): Pressurized liquid extraction (PLE) is an automated method of extracting compounds from various materials using traditional solvents. PLEoperates under constant pressure and controlled parameters, including temperature, extraction time, and number of cycles. As the final extract is collected, it is automatically filtered, simplifying the process,and making it more efficient.

Principle:

High pressure (typically 500–3000 psi) is applied to keep the solvent in its liquid state even at elevated temperatures. This allows for efficient extraction without the solvent vaporizing. Elevated temperatures (50°C–200°C) increase the solvent's extraction efficiency by: Enhancing the solubility of the target compounds

Mechanism:

1. **Preparation**: The material containing the target fluid is loaded into the extraction chamber.

2.**Pressurization**: A pump injects the pressurizing medium into the chamber, increasing pressure and sometimes temperature to desired levels.

3.**Extraction**: The fluid separates from the source material due to the applied pressure and is carried by the medium to the outlet.

4.**Separation and Collection**: The extracted fluid is filtered and collected for further use. Residual solids or waste are removed from the chamber.

5.**Release and Reset**: The chamber is depressurized and prepared for the next cycle.[5]

3. Enzyme assisted extraction (EAE): This is a method that utilizes hydrolytic enzymes to dismantle cell walls and other constituents, facilitating the more efficient retrieval of metabolites from plant material. This technique offers environmental sustainability and cost-effectiveness, presenting a promising advancement over traditional and contemporary extraction approaches

Principle:

Enzymes act on specific substrates (e.g., cell wall polysaccharides, proteins, or lipids) to break down cellular barriers and release target compounds such as polyphenols, proteins, lipids, or pigments. Enzymes catalyze biochemical reactions by lowering the activation energy, enhancing the breakdown of complex molecules under mild conditions (temperature, pH).

Mechanism:

1.Cell Wall Disruption:

- Enzymes such as cellulases, hemicellulases, pectinases, and proteases hydrolyze cell wall components (cellulose, hemicellulose, pectin, or proteins).
- This weakens or breaks the structural integrity of the cell wall, making it easier to extract intracellular compounds.[6]

5.Microwave-assisted extraction (MAE): This is a technique that utilizes the power of microwaves to stimulate the movement of liquids 'molecules, allowing for efficient extraction of target components. Compared to traditional extraction methods, MAE offers benefits such as shorter extraction times, reduced solvent costs, and increased automation. MAE heats both the solvent and the material.[9]

Principle:

Even though dried plant material is used for extraction in most cases, but still plant cells contain minute microscopic traces of moisture that serves as the target for microwave heating. The moisture when heated up inside the plant cell due to microwave effect, evaporates and generates tremendous pressure on the cell wall due to swelling of the plant cell. The pressure pushes the cell wall from inside, stretching and ultimately rupturing it, which facilitates leaching out of the active constituents from the rupture's cells to the surrounding solvent thus improving the yield of phytoconstituents. This phenomenon can even be more intensified if the plant matrix is impregnated with solvents with higher heating efficiency under microwave.

Secondary metabolites of plant origin are obtained by MAE because microwave energy has a rapid application in the matrix of the plant material. The energy is assimilated by the extract and the plant material, which is absorbed by the substances present in the plant material, having a greater affinity for polar molecules, for example, water. The internal temperature of the vegetable matrix increases, which causes overheating, which then causes vaporization. This phenomenon causes the rupture of plasma membranes and cell walls.[7]

Mechanism:

The secondary metabolites are distributed in the plant in different places. Some are found in the cytoplasm or the cell walls. Favorable location of the secondary metabolites will facilitate the transfer of secondary metabolites to the solvent and *vice versa* from the solvent to the plant material; this allows for a more successful extraction.

The difference between MAE and conventional extraction methods (Soxhlet extraction and heat flow extraction) lies in the fact that conventional methods rely on a permeation and solubilization process to obtain the intracellular compounds from the plant material.

Microwave heating heats the entire volume of the sample of plant material from the inside. This is unlike what happens with conventional heating, in which the heat comes from the outside and where contact with a hot surface is required to conduct heat. In addition, the water contained in the plant cells in situ is also heated with microwave radiation.

To understand the effect of microwave energy in obtaining secondary metabolites from plant material, microscopic analyzes have been performed. These tools have been used to demonstrate the structural changes produced in plant samples after the application of microwaves. An example of this is the work carried out with the *Erigeron breviscapus* plant, where a comparative analysis of samples processed and not treated by microwaves was carried out to isolate scutellarin. SEM was used to carry out this analysis, demonstrating that the samples treated with microwaves presented damage to the integrity of the *E.breviscapus* tissue, causing the release of compounds of interest in the extract.[8]

Instruments:

There are two types of commercially available MAE systems: closed extraction vessels and focused microwave ovens. The former performs extraction under controlled pressure and temperature. The latter is also named as focused microwave assisted Soxhlet or solvent extraction (FMASE), in which only a part of the extraction vessel containing the sample is irradiated with microwave. However ,both the above-mentioned systems are available as multimode and single- mode or focused system.[9] A multimode system allows random dispersion of microwave radiation within the microwave cavity, so every zone in the cavity and sample it contains is evenly irradiated . Single mode or focused systems allows focused microwave radiation on are stricted zone where the sample is subjected to a much stronger electric field than in the previous case. Even a modified multimode domestic microwave oven operates as an open vessel extraction system. Principle elements of a microwave device Both multi- mode and focused microwave devices comprise four major components:

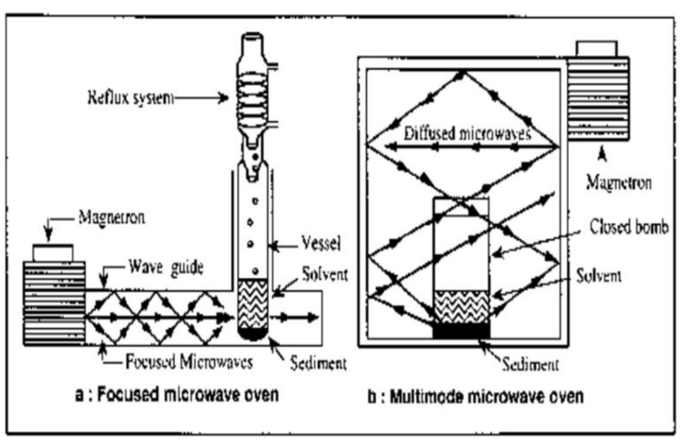
(a) Microwave generator: magnetron, which generates microwave energy

(b) Wave guide: which is used to propagate the microwave from the source to the microwave cavity

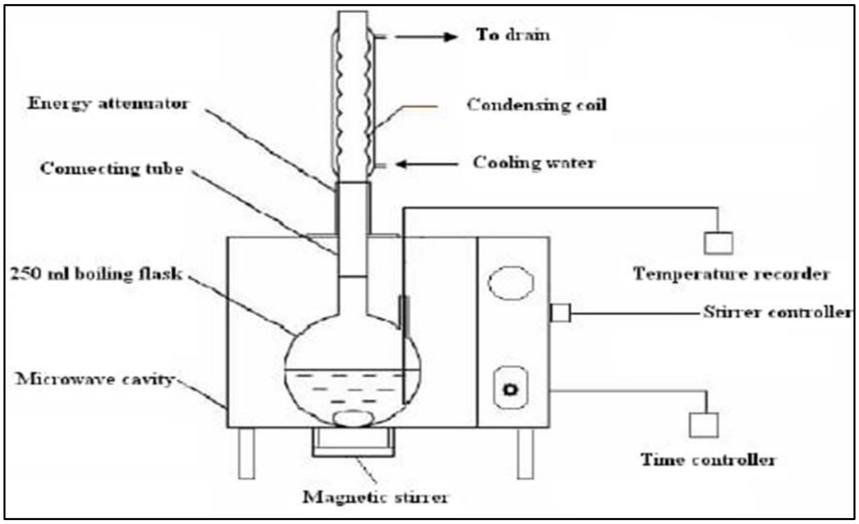
(c) The applicator: where the sample is placed

(d) Circulator: this allows the microwave to move only in the
forward direction.[10]

However, the applicator in case of multi- mode system can be a closed cavity inside which microwaves are randomly dispersed. Uniform distribution of microwave energy insidethe cavity can be achieved by using beam reflectors or turn table that makes heating of the sample independent of the position. In focused microwave systems, the extraction vessel is however kept directly in a microwave waveguide and that acts as the applicator. The bottom few inches of the vessel are directly exposed to the microwaves, whereas the upper region of the vessel remains cool as glass is transparent to microwaves and hence does not get heated up in the process. This results in an effective condensing mechanism inherent in the design .[6]



Microwave oven of extraction



Microwave assisted extraction of rubber seed oil

Summary:

Microwave-assisted extraction (MAE) has emerged as a powerful, eco-friendly, and efficient technique for the extraction of herbal bioactives. Its advantages, such as reduced extraction time, lower solvent usage, and high extraction yields, position it as a valuable alternative to conventional extraction methods. However, challenges such as thermal degradation of heat-sensitive compounds, scalability issues, and the need for optimization across diverse herbal matrices must be addressed.

With ongoing advancements in technology, such as the development of green solvents, hybrid extraction methods, and AI-driven process optimization, MAE has significant potential for widespread adoption in various industries, including pharmaceuticals, nutraceuticals, and cosmetics. By integrating sustainability and innovation, MAE can contribute to the efficient utilization of natural resources while supporting the growing demand for high-quality plant-based products. In conclusion, MAE represents a forward-looking solution that aligns with modern principles of green chemistry and process intensification, making it an essential tool for the future of herbal bioactive extraction.

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