

## A Review: Microwave Assisted Extraction

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### Abstract

Microwave-assisted extraction (MAE) technique involves heating solvents in contact with a sample using microwave energy in order to separate analytes from the sample matrix into the solvent. The primary benefit of MAE is its intrinsic capacity to quickly heat the sample solvent mixture. The ability of microwave radiation to penetrate and mix with a substrate allows for accurate and regulated heating. As a result, the electromagnetic energy can be specifically engineered to be transported by the microwave process to the desired location of the compounds in the substrate. The utilisation of short processing periods and energy-saving variables results in lower manufacturing costs, better product yields and consistency, and higher-quality products as compared to alternative extraction procedures. This review summarizes the basic principle involved in the MAE and its instrumentation. It also gives an overview of some of the applications of the MAE.

**Keywords:** Microwave assisted Extraction (MAE), microwave radiation, closed vessel system, open focused system

### 1. Introduction

The recent extraction methods, such as SFE, MAE, and ASE, are highly alluring since they are faster, require far less solvent, and are less harmful to the environment. For example, SFE extracts materials using carbon dioxide or modified carbon dioxide (i.e., carbon dioxide that contains a tiny quantity of a modified organic solvent). A solvent that is safe for the environment, nontoxic, and nonflammable. Moreover, modifications and changes to the supercritical fluid temperature and pressure can be made to regulate the extraction selectivity. [2]

The process of extracting chemicals or bioactive compounds from a variety of materials, including plants, herbs, and other natural sources which involves the use of heating the sample using microwave radiation during the process to facilitate the extraction of specific compounds from the solvent is called Microwave Assisted Extraction or MAE.

It involves various steps like sample preparation in which the material containing the target compound of interest is converted into fine powder in order to increase its surface area. After which suitable solvent is selected depending on the nature of the target compound. The solvent selected should be capable of dissolving the compound of interest. The sample is mixed thoroughly with the solvent to create a homogenous mixture. This mixture is then exposed to the microwave radiation which leads to heating of the mixture. The above process helps to release the target compound from the sample matrix into the solvent. The mixture is finally cooled and the extract is separated from the residual material using various separation and purification steps [16]. When compared to traditional methods of extracting compounds from diverse matrices, especially natural materials, MAE offers several advantages, such as shorter extraction times, reduced solvent use, higher extraction rates, and lower costs. The application of microwave-assisted extraction (MAE) in the extraction of natural products began in the late 1980s, and thanks to technological advancements, it has since grown in popularity and affordability. A number of sophisticated MAE instrumentations and methodologies, such as solvent-free microwave-assisted extraction (SFMAE) and pressurized microwave-assisted extraction (PMAE), are now available. [1]

## 2. Basic Principles

### 2.1. Microwave theory

In the electromagnetic spectrum, microwaves are non-ionizing electromagnetic waves with a frequency of 300 MHz to 300 GHz that fall between X-ray and infrared radiation. Microwaves are used in science today primarily for two purposes: energy vectoring and communication. The latter uses waves directly acting on materials with the capacity to partially transform the electromagnetic energy absorbed into thermal energy. The two oscillating perpendicular fields that make up microwaves are the magnetic and electric fields; the former creates the heat.

As compared to traditional heating, which works on the phenomena of conduction and convection, where a large portion of the heat energy is eventually lost to the surroundings. Whereas, MAE heating takes place in a closed system, heat is produced in a focused and selected manner with almost no heat loss to the environment. Compared to Soxhlet, this special heating method can greatly shorten the extraction period (often less than 30 minutes). [3]

The fundamental idea behind microwave heating is the direct impact of microwaves on molecules via ionic conduction and dipole rotation. Numerous applications employ these two techniques simultaneously. Ionic conduction is defined as the electrophoretic mobility of ions in the presence of an electromagnetic field.

Heat will be generated in the solution by friction from the resistance of the solution to this ion movement. Dipole rotation is the process of rotating dipoles so that they are in line with the applied field. In commercial systems, the frequency of 2450 MHz is used to induce forced molecular movement, which in turn produces heating. The dissipation factor ( $\tan \delta$ ) affects a solvent's capacity to absorb microwave radiation and transfer it to other molecules as heat. The following equation yields the dissipation factor:

$$\tan \delta = \epsilon'' / \epsilon'$$

where  $\epsilon''$  is the dielectric loss which indicates the efficiency of converting microwave energy into heat.  $\epsilon'$  is the dielectric constant which is the measure of the ability to absorb microwave energy.

Ionic solutions, such as acids, and polar molecules, will firmly absorb microwave energy as the microwaves will influence their permanent dipole moment. Hexane is one example of a non-polar solvent that does not heat up when exposed to microwaves. Given that different chemicals absorb microwave radiation to varying degrees, thus the chemical components employed play a major role in heating of the surrounding media. Materials with non-homogeneous structural features or containing a homogeneous environment of distinct chemical species can be heated selectively in certain areas or components of the sample. This occurrence is referred to as superheating. [4]

## 2.2 Extraction

Despite the fact that most of the time dried plant material is used for extraction, plant cells still have microscopic moisture traces that can be heated in a microwave. Because of the microwave effect, the moisture inside the plant cell heats up and then evaporates, placing a great deal of pressure on the cell wall as the plant cell swells. The cell wall is forced from within by the pressure, stretching and eventually rupturing it. This increases the yield of phytoconstituents by facilitating the leaching of the active ingredients from the ruptured cells into the surrounding solvent. If the plant matrix is treated with solvents that have a higher heating efficiency under microwaves (a higher  $\tan \delta$  value), the outcome may potentially become more intense. The primary component of plant cell walls, cellulose, can have its ether linkages hydrolyzed by higher temperatures produced by microwave radiation and transformed into soluble fractions within one to two minutes. The increased temperature that the cell wall reaches during MAE improves cellulose's dehydration and weakens its mechanical strength, which makes it easier for solvents to reach chemicals inside the cell. [3]

### 3. Instrumentation

There are two kinds of commercially available instruments, and they work in distinct ways. The most typical method is extraction in a closed vessel with temperature and pressure controls; an alternate method employs an open extracting vessel with ambient pressure.

**Closed vessel systems.** Generally, these systems are advised for extractions under extreme conditions, such as digestion or acid mineralizations, since the solvents may be heated to temperatures approximately 100°C beyond their atmospheric boiling point. This process improves extraction efficiency as well as speed. Modern security measures, such as pressure-burst safety membranes installed on each tank, solvent vapor detectors, and high-capacity exhaust fans, mitigate the risks posed by heating extremely flammable liquids. In order to get around the non-homogeneity of the field, the cells are arranged like a household oven on a revolving carousel. The solvents can be changed, and the volume and boiling point of the solvents employed effectively determine the pressure in the vessels. By varying the microwave power, a fixed temperature can be achieved. Teflon is typically used to make the cells. The maximum power that may be delivered in closed vessel systems is between 600 and 1000 W, however the power that is selected must be carefully calibrated to prevent overheating, which could cause solute deterioration and overpressure issues. Before opening, the vessel must be brought down to room temperature. This is necessary because volatile solutes can partition into the headspace, but it can also significantly lengthen the extraction process. To completely remove the solid residue, a second filtration or centrifugation step is required. [5]

**Open focused systems.** These cells are quartz containers with a vapor condenser on top. The solvent's boiling point determines the maximum temperature, and the system operates at atmospheric pressure. In this instance, the sample is placed within the vessel and the solvent is heated and refluxed through it, resulting in a homogeneous and incredibly efficient heating process. To avoid filtering stages, the sample to be extracted can be dipped straight into the solvent or put onto a Soxhlet-style cellulose cartridge. Open cells enable the extraction of bigger samples and provide enhanced safety in sample handling when compared to closed vessel extractions. A such system is the Soxwave 3.6, which is marketed by Prolabo (Fontenay-sous-Bois, France). It has a maximum power of 250 W and runs at percentage power increments of 10 to 100%. [5]

#### 4. Methodology

The whole extraction procedure can be divided into different steps- pretreatment, extraction, isolation, and purification. Maceration, homogenization, grinding, milling, and drying (usually freeze drying to prevent flavonoid destruction) are examples of pretreatment procedures. Drying can help increase raw material yield per unit weight, extend raw material storage life, use less space, and in certain situations make processing easier. The processes of maceration, grinding, milling, and homogenization increase the surface area of contact between the solvent and the solute-containing sample. These pretreatment procedures cause cellular structures to break down, which raises the yield of the bioactive substances even more. The type and source of the sample to be extracted will determine the best extraction technique. The optimal extraction method is chosen based on its highest recovery, which means that the compounds' degradation as a result of various process stages should be as low as possible.

The three main components of any microwave system are typically the wave guide, the applicator, and the microwave source. A magnetron serves as the microwave source. A magnet is made consisting of a vacuum tube with a very negative potential cathode in the centre that emits electrons, encircled by a structured anode that creates cavities. The anode and the cavities are connected by the surrounding fields and have the desired microwave resonant frequency. A magnetron's power output can be regulated by either the magnetic field intensity or the tube current. Waveguides and transmission lines can be used to direct electromagnetic waves. These parts are employed in microwave power applications because waveguides have smaller power losses at higher frequencies, like microwaves. When the material to be heated is introduced by wall slots and the waveguide is terminated by a matched load, the waveguide itself can be utilised as the applicator for microwave heating. Since the field maxima's locations vary with time, this setup is known as a traveling wave device. Standing wave devices—where the microwaves are irradiated by specially constructed open ends of waveguides called horn antennas, or by slot arrays that cut the wall currents—are more frequently implemented in the food sector and in applications involving residential ovens. The two types of microwave ovens used namely: monomode and multimode. A frequency generated by a monomode cavity excites only one mode of resonance. The sample can be placed at the electrical field's highest point because the field's distribution is known. Numerous resonant modes can be affected by the incident wave, and the multimode cavity is sizable. The field can be made homogeneous by superimposing the modes. For homogenization, systems like revolving plates are included. The biological matrix serves as the primary extraction matrix for biological products, and it is widely recognised that the degree of analyte isolation from its matrix has a significant impact on the quality of an extraction from plant material. The importance of an analyte's recovery and the creation of efficient separation techniques for precise analysis increase with

the analyte's amount in the matrix. Two mechanisms—dipole rotation and ionic conduction—that is, the displacement of charged ions found in both the solvent and the solute and the reversal of dipoles—are involved in the energy transfer process during microwave heating. In condensed matter, energy absorption instantly results in energy redistribution between molecules and homogenous heating of the medium. The radiation frequency is correlated with the rotating motion of molecules. Weak hydrogen bonds are broken during microwave heating as a result of the molecules' dipole rotation, which also releases heat. Additionally, there occurs movement of dissolved ions, which promotes solvent penetration into the matrix and makes it easier to collect the target chemicals. A significant amount of pressure builds up inside the biomaterial during microwave processing, changing the biological tissue physical characteristics and increasing the biological matrix's porosity, which facilitates greater solvent extraction through the matrix. The microwave pressure-building impact causes swelling, which usually pushes the cells to split. As extracting pectin from orange peels, it has been found that using a microwave extraction greatly increased the degree of disintegration as compared to using a traditional approach. A mechanical shearing module, which breaks plant cell walls using physical force to hasten the breakdown of the cell's active substances into the solution, can occasionally improve the microwave extraction process. Microwaves are frequently used to extract materials directly (either with or without the use of solvents) or as a pretreatment step before final processing, which increases the yield of the finished product. [6]

## 5.Types of Microwaves assisted Extraction

5.1. Solvent based Microwave-Assisted Extraction-It was discovered that as water content increased, the majority of DES-MAE (deep eutectic solvent-based microwave-assisted extractions' heating rates dropped (and their heat capacities increased) under microwave irradiation, enabling highly efficient extraction of chemicals that are thermally sensitive. Better extraction performance was also achieved by DESs containing carboxylic acids reacting with sugar and choline chloride's hydroxyl groups, which destroyed cell walls and prevented cellulose, hemicellulose, and lignin from re-connecting through hydrogen bonds. By employing DES-MAE to extract anthraquinones from *Rheum palmatum* and adjusting extraction conditions, this was confirmed. Under ideal circumstances, DES using citric acid as the hydrogen bonding donor produced the best extraction efficiency.[14]

### 5.2. Water based Microwave assisted Extraction

Water is the primary solvent used in the extraction process of water-based microwave-assisted extraction (WMAE), an environmentally friendly and sustainable extraction method. By using microwave energy, this technique improves the extraction of target chemicals from a variety of matrices, doing away with or



minimising the requirement for organic solvents. When environmental concerns or the need to obtain extracts acceptable for use in the food and pharmaceutical industries make the use of organic solvents unattractive, WMAE is especially helpful.

By applying microwave energy to the water-containing sample, WMAE causes the matrix's internal pressure to rise and the sample to heat up selectively. This procedure increases the target chemicals' solubility in water, which quickens their release. Compared to conventional procedures, the quick heating and effective energy transfer result in shorter extraction times and higher extraction yields.

### 5.3. Ionic Liquid Microwave-Assisted Extraction (ILMAE)

Ionic Liquid Microwave-Assisted Extraction (ILMAE) is a sophisticated extraction method that optimises the extraction of bioactive chemicals from various matrices by fusing the concepts of microwave energy with ionic liquids (ILs). Ionic liquids, which are tunable and have low volatility, are used as substitute solvents. Microwave energy speeds up the extraction process, making it more efficient than using conventional techniques.

An ionic liquid is employed as the extraction solvent in ILMAE. Ionic liquids are a special class of salts that have melting points lower than 100°C. They have tunable physicochemical features, low vapour pressure, and good solubility for a wide range of chemicals. Target chemicals can be extracted quickly and selectively when ionic liquids and microwave energy are combined. The ionic liquid is heated by microwave radiation, which encourages analytes to dissolve from the sample matrix. This method works particularly well for extracting substances with different polarity.

### 5.4. Solid-Phase Microwave-Assisted Extraction (SPMAE)

The novel extraction method known as Solid-Phase Microwave-Assisted Extraction (SPMAE) combines the benefits of microwave energy with the ideas of solid-phase extraction (SPE). By using a solid support material, this technique is intended to extract analytes from solid matrices quickly and effectively. For the extraction of different chemicals, including bioactive molecules, from solid materials, SPMAE is especially helpful.

In SPMAE, the sample is mixed with a solid support material and microwave energy is applied to improve the extraction process. Target analytes are released from the solid matrix more quickly when the solid support is heated by the microwave radiation. Selecting the right solid support material is essential because it affects the extraction's efficiency and selectivity. Solid supports that are frequently employed include silica, polymers with molecular imprints, and different adsorbents.

### 5.5 Pressurized Microwave-Assisted Extraction (PMAE)

The concepts of microwave-assisted extraction (MAE) are combined with pressurised circumstances to create the enhanced extraction technique known as pressurised microwave- assisted extraction (PMAE). This technique applies microwave energy and high pressure to the sample matrix to improve the speed and efficiency of extraction procedures. The sample is subjected to pressurised microwave radiation in PMAE. Target chemical extraction from the sample matrix is accelerated by the use of microwaves and high pressure. The extraction temperature is raised in the pressurised environment, which helps break down matrices or cell walls and encourages the release of analytes into the extraction solvent.

### 5.6 Ultrasound-Microwave-Assisted Extraction (UMAE)

The hybrid extraction method known as ultrasound-microwave-assisted extraction (UMAE) combines the ideas of microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE). This synergistic method applies microwave and ultrasonic energy to the sample matrix at the same time in an attempt to improve extraction efficiency.

The sample is simultaneously exposed to microwave radiation and ultrasonic waves in UMAE. These energies work together to speed the extraction of target molecules from the sample matrix by improving mass transfer, disrupting cells, and promoting solute diffusion. The mechanical rupture of cell structures is facilitated by ultrasonic waves, whereas microwave energy offers targeted and fast heating.

## 6. Application of microwave assisted extraction in various fields

6.1. Extraction of phenolics from Potato- Potatoes (*Solanum tuberosum*) are the fourth most widely cultivated crop in the world, behind rice, wheat, and maize. Most potatoes are peeled before being processed as raw materials for the food and starch industries. By refluxing, MAE was shown to be a more effective and efficient extraction method than traditional solvent extraction. The MAE parameters were tuned at 60% ethanol, 80°C, 2 minutes, and a solid-to-solvent ratio of 1:40 (g/mL) using an orthogonal array design. The bulk of the phenolics are shown by the fact that the entire potato had a higher individual phenolic acid concentration and overall phenolic content than the peeled flesh. Compared to the same sample extracted using the CE method ( $9.6 \pm 0.2$  mg GAE/g DW), the total phenolic content ( $10.3 \pm 0.3$  mg GAE /g DW) for the MAE showed a substantial increase (10.4%). Compared to the standard solvent extraction method (1:50, g/mL), the MAE extraction method used a smaller volume of solvent (1:40, g/mL).[7]

6.2. Increased amount of polysaccharides obtained by microwave-assisted extraction method with antioxidant, antiradical, and antimicrobial activities-



The polysaccharide obtained by MAE (131.0  $\mu\text{mol}$  Trolox equiv./g) from *C. quadrangularis* was shown to have significantly better ORAC (oxygen radical absorbance capacity) values than that obtained by hot-water extraction (107.7  $\mu\text{mol}$  Trolox equiv./g) in an experiment. At all utilized concentrations, the okra polysaccharide extracted by MAE was found to have a higher ferric reducing antioxidant power than that obtained by hot water extraction and PWE. By altering the structural structure of the extracted polysaccharides by loosening the cell wall matrix and splitting the parenchyma cells, the use of microwave radiation lowers their molecular weight and increases the extraction yield and antioxidant activity. According to results of another experiment, the polysaccharide extracted by MAE exhibited greater antibacterial activity than that produced by HWE. *S. aureus*, *E. coli*, *B. pumilus*, and *B. subtilis* had minimum inhibitory concentration values of 0.25, 0.025, 0.01, and 0.5 mg/mL, respectively.[8]

6.3. Microwave-Assisted Extraction and Ultrasound-Assisted Extraction of Bioactive Compounds from Grape Pomace-A by-product of the industrial processing of grape juice, grape pomace has potential applications as a source of bioactive substances. Using an acidic aqueous solution containing 2% citric acid as a solvent and microwave-assisted extraction at powers of 600, 800, and 1,000 W and durations of 5, 7, and 10 min, the phenolic compounds from grape pomace were isolated. In contrast to the exhaustive extraction with methanol acidified solution, the results of the MAE method of extraction showed that the contents of total phenolic compounds and antioxidant activity by ABTS and DPPH increased with time. The optimal extraction condition was found to be a microwave at 1,000 W for 10 min, during which time 45% of the anthocyanins were recovered.[9]

#### 6.4. Application of MAE in food industry-

When compared to a traditional solvent extraction method, the extracted oil with higher tocopherol and total phenolic concentrations was obtained using MAE. Hu et al. conducted another study which found that the application of MAE, in combination with a 1:1 v/v ethanol and n-hexane solvent, 360 W microwave power, and 7.5 mL solvent per gram of sample, improved the quality of silkworm pupal oil. Comparing the extracted oil to the Soxhlet solvent extraction method, the results demonstrated that the extracted oil had stronger antioxidant activities, higher oxidation stability, and higher total phenolic contents. The study's scanning electron microscope image viewing also showed that the sample's cell structure could be broken down by microwaves, which improved the oil's release during extraction. The quality of the extracted product and food preservation are thought to have improved recently with the use of MAE.[10]

#### 6.5. Extraction of pectic polysaccharide from jackfruit waste-

Utilizing cutting-edge methods like pulsed electric field (PEF) in conjunction with microwave treatment, pectin—a valuable polysaccharide found in jackfruit waste—can be effectively recovered. Using polynomial regression models to improve the combined extraction processes led to a notable increase in pectin production (18.24%) and a decrease in energy consumption (0.0986 kW-h). This aids in the extraction of pectin from jackfruit, supporting sustainable resource use and addressing environmental issues with waste disposal.[11]

#### 6.6. Extraction of Carotenoids from Pumpkin Peel and Pulp-

Comparing innovative green extraction techniques to traditional extraction methods, the total carotenoids content, total phenolic content, and antioxidant activity of pumpkin peel and pulp extracts were dramatically increased. These techniques used ultrasound and microwave-assisted extraction using maize oil as a solvent. When compared to conventional extraction, the extracts from microwave-assisted extraction demonstrated nearly twice as much suppression of 2,2-Diphenyl-1-picrylhydrazyl (DPPH) free radicals, suggesting more antioxidant potential. Innovative green extraction methods yielded pumpkin carotenoids extracts with higher oxidative stability in corn oil, as indicated by the protection factor. This suggests that the food, pharmaceutical, and cosmetic industries may find use for these extracts as natural colorants and antioxidants.[12]

#### 6.7. Extraction of bioactive compounds from eggplant peel by-product

The microwave-assisted extraction of bioactive chemicals from eggplant peel was achieved by the application of a three-level, five-factor central composite design. By adjusting the microwave power, extraction time, liquid–solid ratio, ethanol concentration, and solvent pH, the extraction yields, total phenolic content (TPC), total flavonoid content (TFC), total anthocyanin content (TAC), and DPPH radical scavenging activity, expressed as IC<sub>50</sub>, were all improved. Reduced ethanol concentrations and liquid-solid ratios were used in tandem with a significant ( $p < .01$ ) increase in microwave power and an increase in extraction yield, TAC, TPC, and TFC. When power, extraction time, liquid-solid ratio, ethanol concentration, and solvent pH were 269.82 W, 7.98 min, 5.01 ml/g, 73.49%, and 3.06%, respectively, the maximum predicted extraction yield (3.27%), TPC (1,049.84  $\mu\text{g GAE/ml}$ ), TFC (130.40  $\mu\text{g QE/ml}$ ), and TAC (6.99 mg/L) and the minimum predicted IC<sub>50</sub> (0.52 ml/mg) were all simultaneously obtained. In general, microwave conditions that are optimized can be used to effectively extract bioactive components from eggplant peels.[13]

## 6.8. Microwave-assisted extraction of pharmaceuticals, personal care products and industrial contaminants in the environment

Microwave-assisted extraction (MAE) is a low-impact and highly effective technique that preserves high yields while consuming less solvent and extracting analytes from environmental samples. The benefits of MAE include lower energy and trash production. Compared to other extraction approaches, MAE performs more similarly and is more efficient. It is also easier to apply. Using MAE to extract different chemical families of emerging contaminants from environmental samples has proven to be successful. MAE has been widely used to extract developing contaminants, like industrial compounds, medicines, and personal care items, with extraction yields that are on par with or higher than those of traditional methods like Soxhlet extraction.[17]

6.9. Jonagold cultivar apple peels can be efficiently used in a microwave-assisted method using green solvents and an inert atmosphere to extract high added value chemicals like antioxidants, dietary supplements, and cosmetics. Green solvents, harsh operating conditions, and microwave-assisted procedures are used to optimize the extraction process so that mass transfer is increased without sacrificing the biomolecules' activity. Response surface modeling was used in the study to optimize the process parameters, and the resultant extract had excellent antiradical capabilities together with high antioxidant yields. After extraction, the solid residue also showed increased carbon content and calorific value, indicating its suitability for energy recovery. Thus, MAE offers a great deal of promise for both the extraction of valuable chemicals from apple skins and the potential energy source potential of the remaining solid.[18]Peppermint leaves are an excellent source of bioactive substances like terpenoids and polyphenols, which may find use in food, medicine, and cosmetic products. Peppermint polyphenols and terpenoids have been successfully recovered from the plant at the same time using microwave-assisted extraction (MAE), which has increased antioxidant activity and produced high extraction yields. The Provincial Secretariat for Science and Technological Development, Vojvodina Autonomous Province, Serbia, provided assistance for the study's findings. This suggests that MAE enhanced antioxidant capability and produced excellent extraction yields [19]. For the extraction of curcumin and antioxidants from turmeric, Natural Deep Eutectic Solvents (NADES) present a viable green solvent alternative. These solvents may find use in the food, pharmaceutical, and cosmetics industries, among other industries. Shorter processing times, less solvent usage, and possible scalability are only a few benefits of the promising effective recovery of bioactive components from turmeric using microwave-assisted extraction (MAE) employing NADES.NADES are a cost-effective and environmentally beneficial method for extracting

polyphenolic chemicals, with the potential to be used as food additives and in other applications due to their stability, low toxicity, and biocompatibility.[20]

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