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Approaches to Extracting Bioactive Compounds from Spices and Herbs

(¹Vasanthkumar S. S., ²Pooja U. K. and ³Kumaresan M³)

¹Horticultural College and Research Station, TNAU, Periyakulam

²Vels Institute of Science, Technology & Advanced Studies, Pallavaram, Chennai

*Corresponding Author's email: kalaivasanth@gmail.com

The Green Chemistry movement, initiated in the 1990s, aims to reduce hazardous substances from chemical products and processes, including waste, hazard, and energy. Bioactive compounds of natural origin, such as plants, herbs, spices, agricultural by-products, and marine sources, have gained significant interest due to their potential to promote human health. Traditional extraction methods, such as solid-liquid extraction, use toxic solvents, consume high energy, and provide low selectivity and yields. Advanced techniques like supercritical fluid extraction can improve bioactivity while adhering to Green Chemistry goals.

Pressurized Liquid Extraction (PLE)

Pressurized fluid extraction (PLE) is a technique that uses pressurized solvents at high temperatures to extract substances. It offers advantages over ambient pressure and can be used with various environmentally friendly options. Subcritical water extraction (SWE, SHWE, or PHWE) is another option, which involves a pump, extraction cell, pressure valves, oven, and collection vessel. The pump introduces solvent and pushes extract out, maintaining pressure between 35 and 200 bar. Oxygen-free solvents are used to prevent oxidation and cavitation. PLE extraction systems require a collection vessel, accurate pumps, and a heating coil for dynamic extraction. High temperature and pressure conditions are essential for faster processes with lower solvent volumes. Superheated water (SWE) can be a green alternative to organic solvents in some applications. Solvent selection is crucial for extraction processes, with the most suitable solvent based on the target analytes' nature and characteristics. Environmental considerations also play a significant role, with the greenest solvent always being used. Temperature is the most influential parameter, with higher temperatures generally yielding higher extraction yields. Extraction pressure is also important, with pressures of 50-100 bar typically used to maintain the solvent liquid state. Dynamic extractions and sequential extractions can be employed for different extract compositions.

Applications of PLE to Herbs and Spices: PLE is utilized to extract various compounds from herbs and spices, including phenolic compounds, essential oils, anthraquinones, alkaloids, and pesticides. Optimization of PLE parameters and careful study of target compounds are crucial for optimal efficiency.

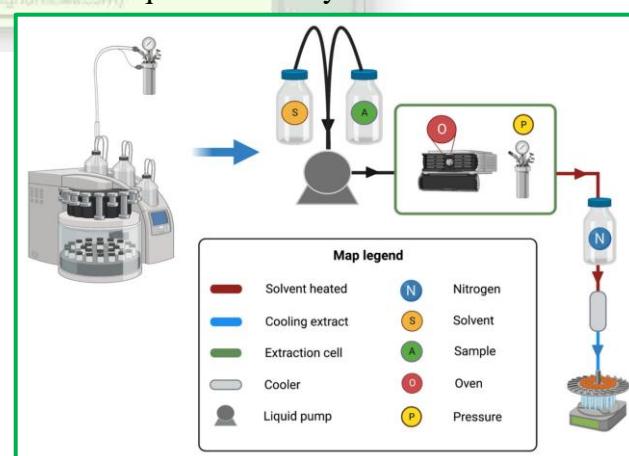


Figure 1 Pressurized Liquid Extraction

Supercritical Fluid Extraction (SFE)

Supercritical Fluid Extraction (SFE) is a method that uses solvents at temperatures and pressures above their critical points to extract bioactive compounds from natural sources. Carbon dioxide is the most commonly used solvent due to its affordability, ease of accessibility, and environmental friendliness. However, SFE has a low polarity, which can be overcome by using polar modifiers. Other solvents proposed include propane, butane, and dimethyl ether. Gas Expanded Liquid Extraction (GXL) is a clean alternative for obtaining bioactive compounds from vegetal matrixes. SFE extraction involves several parameters, including solubility of target compounds, sample amount, particle size, and use of dispersing agents. SFE can be performed in solid and liquid matrices, with equipment for solid samples comprising an extraction vessel and for liquid samples using an extraction column in countercurrent mode. In summary, SFE is a cost-effective and environmentally friendly method for extracting bioactive compounds from various herbal materials.

Applications of SFE to Herbs and Spices: SFE has been extensively studied for extracting bioactive compounds from herbs and spices, including roots, rhizomes, leaves, bark, flowers, fruits, and seeds. The content and composition of these compounds vary based on factors like location, harvesting time, cultivation practice, soil nutrients, and climatic conditions. SCCO_2 has been primarily used for non-polar bioactive compounds like fatty acids, triglycerides, phytosterols, terpenoids, tocopherols, and carotenoids.

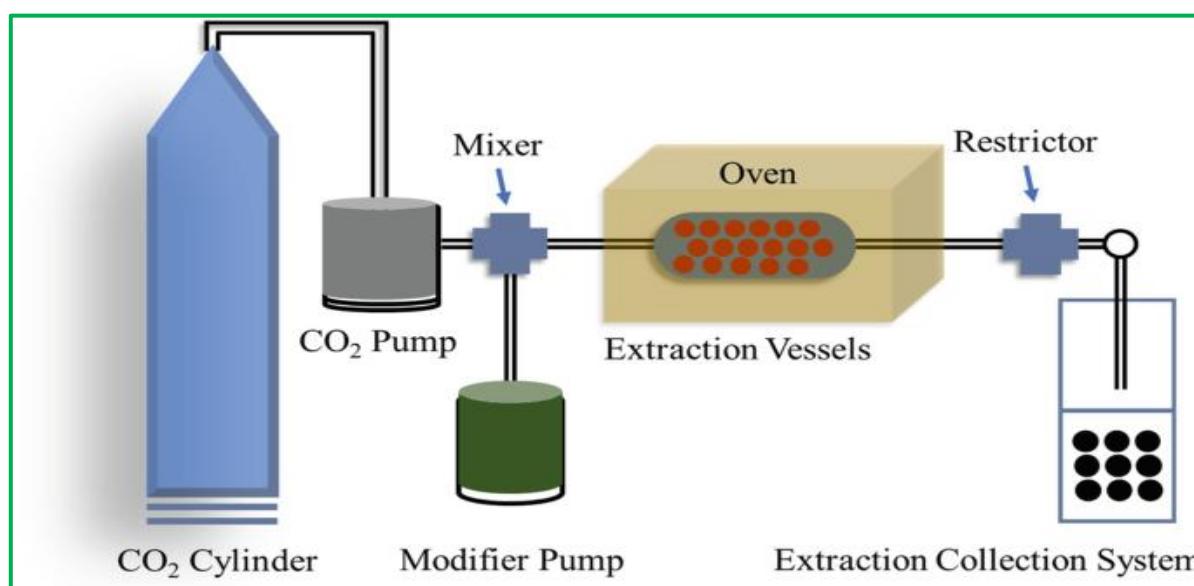


Figure 1 Supercritical Fluid Extraction

Ultrasound-Assisted Extraction (UAE)

UAE employs acoustic cavitation for fast, cost-effective extraction of compounds without degradation. This method uses power ultrasounds with frequencies ranging from 20 kHz to 100 MHz, affecting various physical and chemical processes. The choice of solvent depends on its physical properties, such as surface tension, viscosity, and vapor pressure. High surface tensions, densities, and viscosities have a higher threshold for cavitation but more harsh conditions once cavitation begins. UAE extraction processes require a liquid medium and high-energy vibrations like ultrasounds. The process is increasingly efficient in transferring knowledge into commercial development, extracting analytes in concentrated form and free from contaminants or artifacts.

Applications of UAE to Herbs and Spices: Ultrasound is gaining recognition for its potential in phyto-pharmaceutical extraction, particularly for herbal extracts, due to its mechanical effects and disruption of biological cell walls.

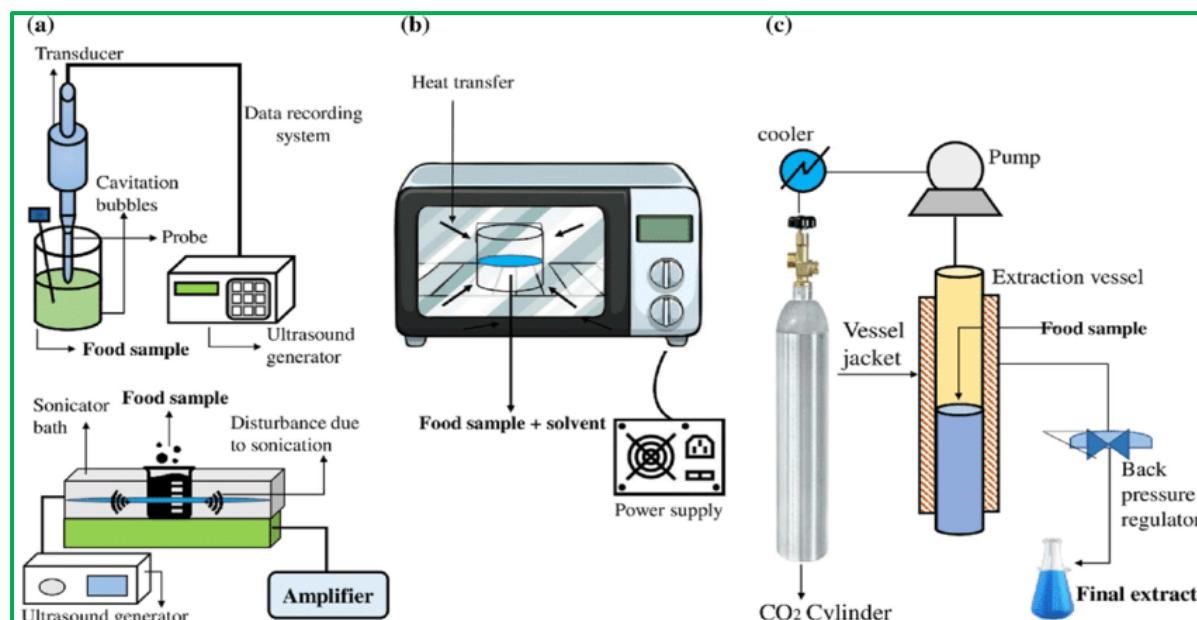


Figure 2 Ultrasound-Assisted Extraction

Microwave-Assisted Extraction (MAE)

Microwave-assisted extraction (MAE) is a method that uses microwave radiation to heat solvents, promoting the transfer of target compounds from the sample matrix into the solvent. This technique allows for fast extractions with minimal solvent needed, avoiding degradation of thermolabile compounds and providing green extracts from herbal resources. MAE was first described in 1986 and has since been applied to biological samples. The solvent is crucial in MAE extraction, with higher dielectric constants resulting in faster heating rates. A combination of solvents can be used, with thermolabile compounds preferring solvents with low dielectric properties. MAE techniques involve homogenizing and mixing samples with a solvent, and the suspension is irradiated at a frequency greater than 2000 MHz for short periods. Closed-system microwave heating has also been developed. There are two approaches to applying microwave energy: bulk heating and selective heating.

Applications of MAE to Herbs and Spices: MAE has gained popularity for extracting bioactive compounds from herbs and species due to its high-quality final extracts, particularly phenolic compounds, essential oils, alkaloids, and saponins, which are particularly interesting.

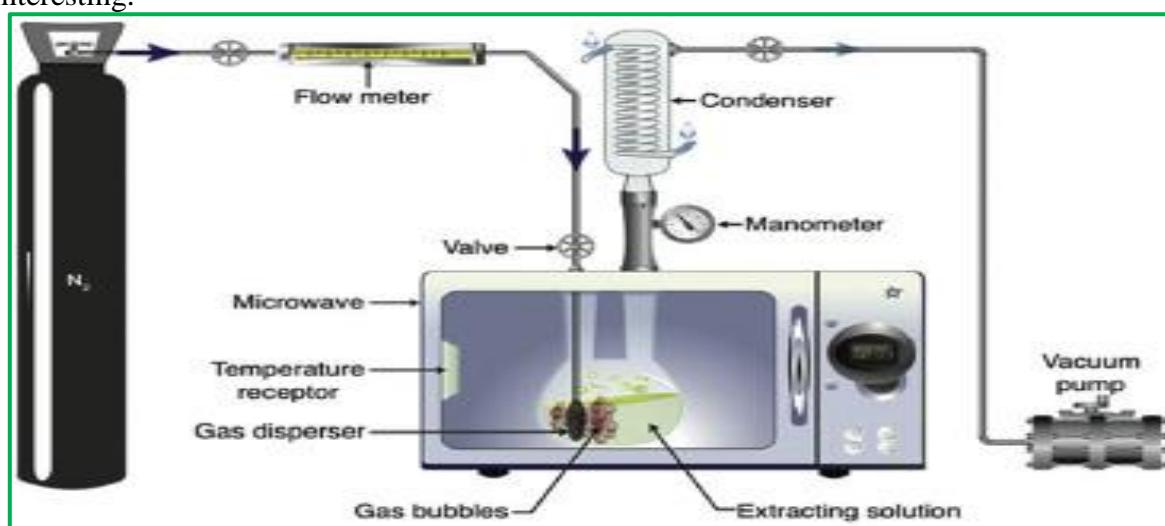


Figure 3 Microwave-Assisted Extraction

Enzyme-Assisted Extraction (EAE)

Enzymes are increasingly used to extract bioactives from plant materials due to their environmental and regioselectivity properties. They catalyze hydrolysis reactions of cell wall components, weakening these structures and allowing for the recovery of bioactive compounds. The composition of the plant material's cell wall is crucial for selecting the most appropriate enzyme, with parameters like charge, reaction temperature, reaction time, and pH being important. Enzymes can be used alone or combined with advanced extraction techniques like PLE or SFE, as cell damage increases solvent penetration and mass transfer rate.

Applications of EAE to Herbs and Spices: Ethylamine-based extraction (EAE) is a method for extracting essential oils and oleoresins from herbs and spices. Enzymes like cellulases, hemicellulases, and pectinases are used based on their action on cell wall components and spice composition. Other factors like pH, reaction time, spice/enzyme ratio, and temperature are adjusted for optimal reaction conditions. EAE has been used to increase essential oil yields in black pepper and cardamom, gingerol recovery, and vanilla extract. Enzymatic pretreatment is also used to enhance extraction yield and biological activities.

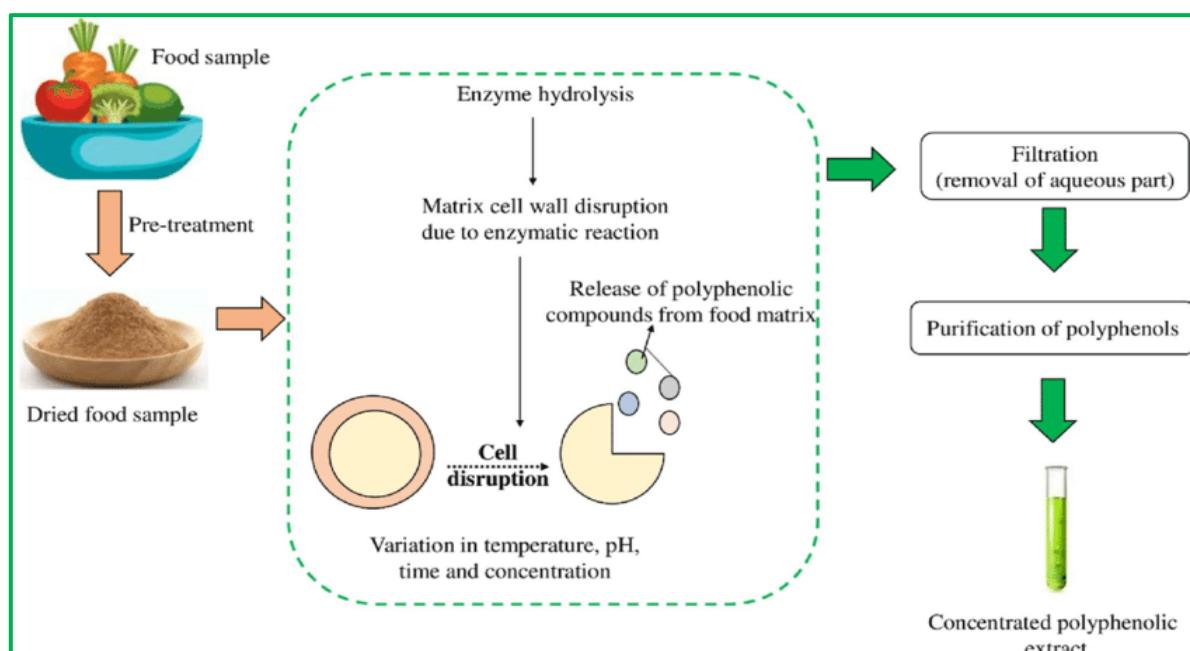


Figure 4 Enzyme-Assisted Extraction

Integration of Different Extraction Techniques

Nowadays, obtaining high-quality, bioactive products while improving efficiency and reducing costs is crucial. Integrating extraction techniques into the same process can achieve effectiveness, greenness, and sustainability. This approach allows for purification of components from different raw materials and sequential extraction of compounds from the same sample. Important integrated novel extraction processes since 2012 are presented.

Ultrasound–Microwave-Assisted Extraction (UMAE): Ultrasound-microwave-assisted extraction (UMAE), also known as ultrasonic-microwave synergistic extraction (UMSE), is a technique that combines ultrasound and microwaves to extract bioactive compounds from herbs. It has been successful in extracting alkaloids from *Picrasma quassioides*. The optimal extraction process involves selecting the correct solvent, solvent/solid ratio, and extraction time. Ionic liquids (ILs) are also used in UMAE technology, with the length of the alkyl chain affecting the rate and the concentration affecting the yield. The optimum parameters for IL-

UMAE include a 0.5 mol/l solution, 15 ml/g solvent, 500W microwave irradiation, and 8 minutes of extraction time.

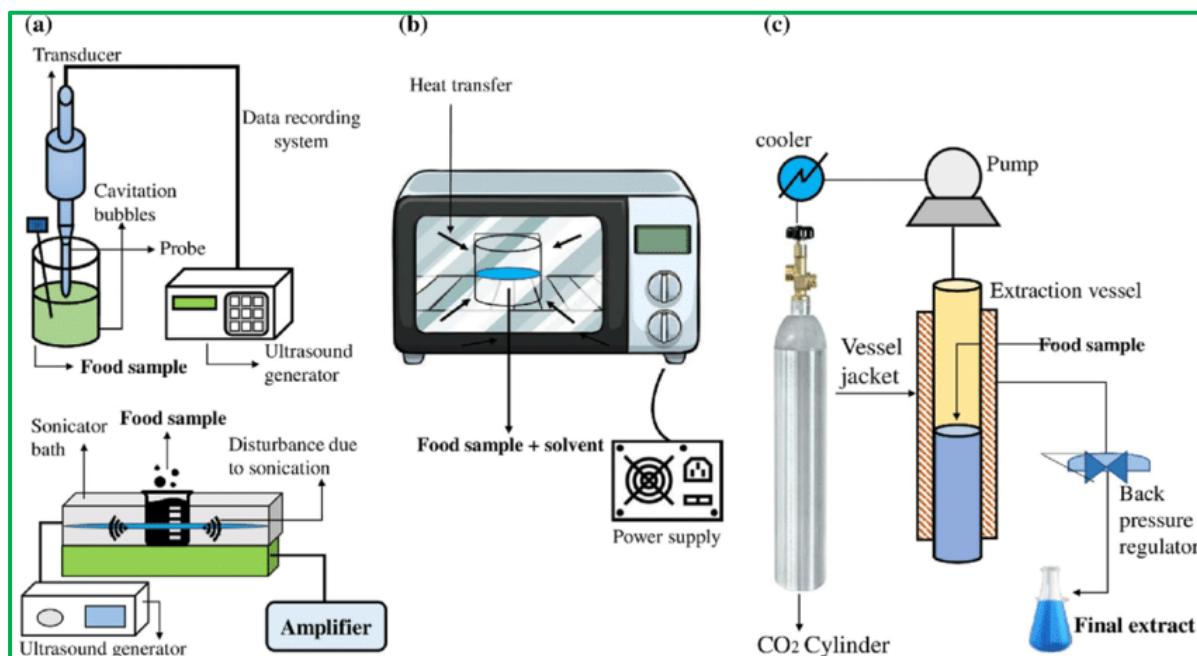


Figure 5 Ultrasound–Microwave-Assisted Extraction

Enzyme-Based–Ultrasound–Microwave-Assisted Extraction (EUMAE): The combination of EAE and UMAE is utilized to extract compounds like orcinol glucoside from *Curculigo orchioides* Gaertn. The process involves incubating the powdered sample with an enzyme solution, optimizing the UMAE extraction, and resolving the residue in ethanol/water at 50°C for 15 minutes at 400W.

Supercritical Fluid Extraction–Pressurized Fluid Extraction (SFE–PLE): SFE and PLE are essential extraction techniques for bioactive isolation, and their combination in sequential or in situ configurations has potential for future applications. Osorio-Tobón et al. (2014) developed a home-built equipment for sequential extraction of curcuminoids from *Curcuma longa* L rhizomes, optimizing the PLE process using temperature and pressure. Golmakani et al. (2014) developed a modified SFE apparatus for PLE extraction of antioxidant compounds of *Scutellaria pinnatifida*, optimizing the process for maximum extraction yield, total phenolic, total flavonoid, and antioxidant activity.

Supercritical Fluid Extraction Assisted by Ultrasound (SFE–UAE): The study evaluated the benefits of an integrated process for extracting capsaicinoids and phenolic compounds from *Capsicum frutescens* L. using an ultrasound-assisted SFE unit. Results showed no significant differences between the two techniques at short times and low US power. However, a positive effect on extraction yield was achieved when higher power was applied (360W). The application of ultrasound to the SFE had a positive effect, with the extraction yield obtained by SFE-UAE being 35% higher than SFE alone.

Conclusion

This article explores Green Chemistry techniques for extracting valuable compounds from herbs and spices, including PLE, SFE, UAE, MAE, EAE, and combined processes. It uses LCA to assess environmental impact and costs, and discusses the use of enzymes, disruption methods, and intensification methods for sustainable production.

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