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REVIEW OF ULTRASOUND-ASSISTED EXTRACTION WITH DEEP EUTECTIC SOLVENT: A GREEN METHOD IN ESSENTIAL OIL EXTRACTION

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ABSTRACT

The search for environmentally friendly extraction methods in essential oil production has led to the development of green extractions. This review article explores the effectiveness of ultrasound-assisted extraction with deep eutectic solvents as a sustainable approach to essential oil extraction. Several studies have demonstrated that this method offers significant advantages over conventional extraction methods, including reduced energy consumption, reduced environmental impact, and improved extraction rates. The results show that deep eutectic solvent -based ultrasound-assisted extraction not only enhances extraction efficiency but also preserves the integrity of bioactive compounds, making it a viable green alternative for essential oil extraction. This method aligns with green chemistry principles by minimizing waste and energy use, positioning ultrasound-assisted extraction alongside deep eutectic solvents as a promising tool in sustainable essential oil production.

1. INTRODUCTION

The waste from fruit processing industries, comprising peels, seeds, and unused flesh, presents a global challenge due to its sheer volume. These materials are rich in valuable compounds like dietary fiber and antioxidants, offering low-cost and natural sources for various industries. Repurposing this waste through extraction, purification, and fermentation methods not only addresses environmental concerns but also aligns with the growing global emphasis on sustainable practices (Kodagoda & Marapana, 2017).

Plant food wastes might be usable and have their own market as suggested by the term “by product” (Kodagoda & Marapana, 2017). Essential oils are widely used in food, cosmetics, and medicine, with applications ranging from flavorings to insect repellents. The essential oil industry, spanning over 100 countries, has evolved since the 19th century, finding a crucial place in everyday products like fragrances and pharmaceuticals. Essential oils are recognized for their versatility and increasing importance in various sectors (Can Başer & Buchbauer, 2015)

Essential oils are complex mixtures with compositions varying from 20 to 60 natural compounds. Their extraction yield and composition vary widely and are difficult to predict because they depend on many factors such as variety, season, geographical location, ripeness of the fruit and extraction method. For essential oils from fruit peels such as bergamot, orange, and lemon, the yield ranges from 0.2% to about 0.5% (Can Başer & Buchbauer, 2015; Bora et al., 2020).

There are many different methods to extract essential oils from plants. The advanced green extraction technique is known for its short extraction time, reduced organic and toxic solvent volume, simple operation, high extraction yield and low energy consumption (Dheyab et al., 2021). Some reports show that ultrasound or deep eutectic solvents can be effective green extraction techniques for many valuable compounds related to essential oils due to its advantages.

This review article aims to provide a comprehensive understanding of the principles and performance of ultrasound-assisted extraction using deep eutectic solvents of bioactive compounds, thereby demonstrating their potential application in the exploitation and use of essential oils.

2. RESULTS AND DISCUSSION

2.1 Essential oils extraction

Essential oils, as defined by the International Organization for Standardization (ISO), are "product obtained from a natural raw material of plant origin, by steam distillation, by mechanical processes from the epicarp of citrus fruits, or by dry distillation, after separation of the aqueous phase—if any— by physical processes". Because essential oils are aromatic, volatile compounds with complex compositions, regulatory authorities in the European Union classify them as "Natural" but also as "Chemical Substances" (Can Başer & Buchbauer, 2015).

Extraction process is to separate soluble plant metabolites and obtain a complex mixture such as essential oils, alkaloids, phenolics, glycosides, terpenoids and flavonoids. Essential oils are extracted mainly from the fruit peel, leaves or flowers of plants. Essential oils from fruit or fruit by-products can be obtained by various extraction methods described in Table 1 (Clark et al., 2017; Toan et al., 2020; Suresh et al., 2021; Indriyani et al., 2023).

The different biological activities of essential oils may be due to the characteristics of their chemical composition and lead to their different applications. Many studies have been widely published in recent years showing the different effects of plant part, plant origin and extraction method on oil yield and the main components of essential oils (Budiarto & Sholikin, 2022)

The most common and simple method to obtain essential oils from citrus peel is hydrodistillation/steam distillation or mechanical pressing. The steam distillation method has a higher yield of peel essential oil recovery than the cold pressing method, specifically 3 times higher when extracting essential oil from Citrus aurantifolia and 2 times higher when extracting essential oil from Citrus limonia (Eman et al., 1998)

Table 1: Summary of essential oil extraction methods

Method	Principle	Advantages
1. Conventional methods		
Cold expression or cold pressing	The essential oil glands in the peel and epidermis are broken down under physical impact to obtain an aqueous emulsion. The	The oldest method, mainly for the production of citrus essential oils. Get food grade essential oils

	essential oil is recovered after centrifuging the mixture.	
Solvent extraction	The material is placed into a tank containing solvents such as alcohol, hexane, ethanol, petroleum ether and methanol and a liquid mixture containing essential oils along with other compounds is obtained. Essential oils are recovered after filtering and distillation.	Can be used to extract heat unstable essential oils Inexpensive. Hot solvents can be used to speed up the process.
Distillation	During distillation, the material is exposed to boiling water (hydro-distillation) or steam (steam distillation) to release essential oils through evaporation and condensation. Essential oils are recovered after separation from water.	A cheap and common extraction. Get food grade essential oils.
Supercritical extraction	The solid material is placed in a vessel into which supercritical fluid is added at a specific flow rate until the appropriate extraction conditions are achieved.	A clean and efficient extraction Produce high quality essential oils
2. Novel "green" methods		
Microwave-assisted extraction	Based on the direct impact of microwaves on the molecules of the material leading to friction and heating of the solution, while increasing the permeability and solubility of target compounds in the solvent	Simplicity and reasonable cost. High extraction efficiency, short extraction time and high selectivity.
Controlled pressure drop process	Based on thermomechanical processing by allowing the product to rapidly transition from high vapor pressure to vacuum pressure.	High quality, quick and standard method of extracting essential oils.
Ultrasound-assisted extraction	Based on the production and destruction of microscopic bubbles using ultrasonic waves. As the bubbles increase in size, they collapse violently and cause mechanical forces that lead to cell membrane damage and essential oil separation.	Minimal effect on extractable compounds. Effective with solvents that are widely recognized as safe. High yield, fast extraction speed and shortened extraction time.

Sharma, Chand, and Sharma (2019) compared the effectiveness of the method of extracting essential oil from the Kinnow peel by hydrodistillation and centrifugation after grinding. Although the hydrodistillation takes longer than the physical method (91 minutes compared to 25 minutes), the extraction efficiency is much higher (0.910% compared to 0.179%). At the same time, the essential oil from hydrodistilled extract is yellow color while the other is reddish brown. Although the quality of both extracted essential oils is similar, the steam distillation method gives a higher yield than the supercritical carbon dioxide method (Kasuan et al., 2013).

Distillation extraction process usually takes about

1-10 hours (Preedy, 2016) or 3 - 8 hours (Kasuan et al., 2013), so impurities can form due to high temperatures or water acidity leading to differences in the composition of the extracted volatile oils. During steam distillation, the extraction rate decreases as the extraction temperature and time increase. The quality of the oil in terms of aromatic structure and physical color is also degraded by high temperatures or prolonged heating (Kasuan et al., 2013)

Some disadvantages of conventional extraction techniques include high temperatures and long distillation times or using too much solvent and wasting time during the solvent extraction process. While conventional extraction methods are cost-effective and easy to perform, new extraction methods have been shown to be more effective in separating essential oils from plant material and can also provide high quality essential oils.

2.2 Ultrasound-assisted extraction

Figure 1 showed the principle of acoustic cavitation. Ultrasound consists of sound waves with high frequencies that exceed human hearing (16–18 kHz) and below the microwave frequencies (up to 10 MHz). It converts electrical energy into mechanical energy through a transducer, creating vibrations in the liquid that cause acoustic cavitation. The collapse of this nucleus can be symmetric (shock waves) or asymmetric (microscopic rays causing cell damage). Cavitation can be steady (higher frequency) or transient (lower frequency) (Dias et al., 2021; Medina-Torres et al., 2017).

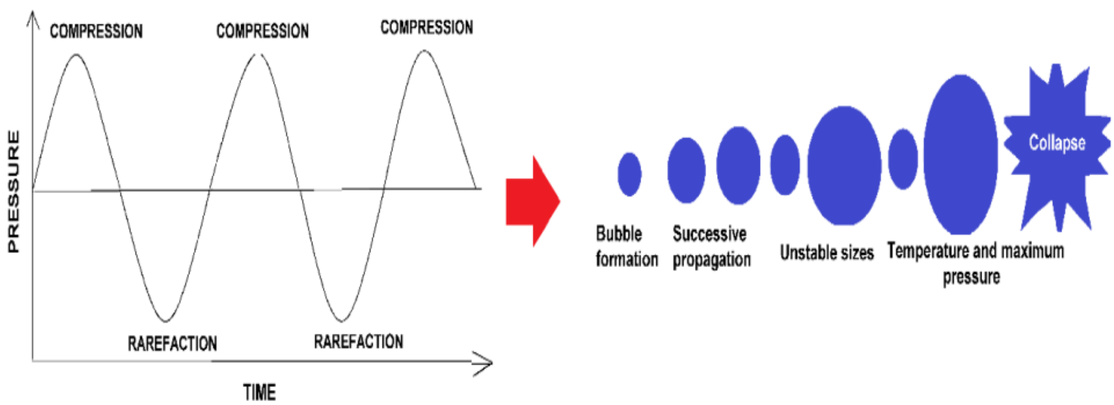
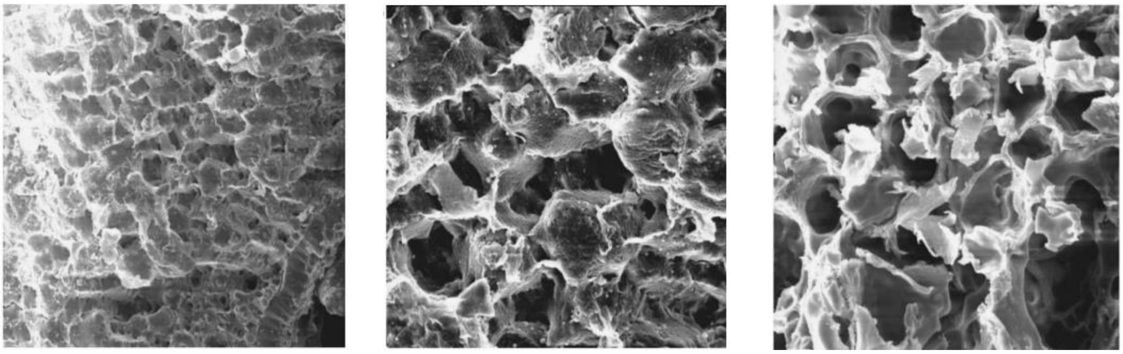


Figure 1. The principle of acoustic cavitation (Medina-Torres et al., 2017)

Ultrasonic irradiation can be direct or indirect through an ultrasonic bath system, with direct contact providing significantly higher power. Ultrasound waves with main characteristics such as length, amplitude, frequency, speed, power and intensity are applied in the processes of emulsification, crystallization and compound extraction. Ultrasound-assisted extraction replaces toxic solvents, reduces energy consumption, shortens processing time and temperature, and is especially useful in extracting compounds that are susceptible to thermal decomposition such as essential oils (Soria & Villamiel, 2010; Shirsath et al., 2012; Medina-Torres et al., 2017; Clark et al., 2017).

Ultrasound-assisted extraction is mainly due to acoustic cavitation, as ultrasound waves penetrate the liquid medium causing damage to the plant wall, enhancing solvent penetration and facilitating the release of compounds. The mass transfer process takes place in two stages: the first stage requires disruption of the layer containing the herbal tissue, while the next stage involves mass transfer from the herbal plant to a solution controlled by kinematics (Mirdehghan et al., 2021).

Different extraction methods produced distinguishable physical changes in the plant material. For example, the spongy texture of the Cardamomum seed core after the ultrasound process indicated that the essential oil has been purified; the glandular wall then appear to be damaged and empty following hydrodistillation (Figure 2). So, the change in material structure through ultrasonic extraction was effective after just 10 minutes at room temperature, compared to conventional hydrodistillation methods that required higher temperatures of about 100 °C and took about 4 hours (Sereshti et al., 2012).



(a) without treatment (b) after Ultrasound-assisted c) after Hydrodistillation

Figure 2. Morphological photomicrograph of Cardamomum in essential oil extraction (Sereshti et al., 2012).

The application of ultrasonic pretreatment method (500 W, 20 KHz at room temperature) in extracting essential oil from Citrus sinensis by-product helped increase extraction efficiency by 33% and reduced extraction time by 33% compared to conventional hydrodistillation. The essential oil had a pleasant aroma and higher antioxidant and antibacterial property. In particular, ethanol extract contained a full range of active compounds with excellent antioxidant properties (Sandhu et al., 2021).

Several reports have been published on the successful extraction of a variety of bioactive compounds, including essential oils, using ultrasound-assisted extraction. Compared with the Soxhlet method, ultrasound-assisted extraction for Fennel essential oil with water-ethanol was more economical and environmentally friendly (Mirdehghan Ashkezari et al., 2021). Although quality of olive oil enrichment with rosemary essential oil was similar, the ultrasound-assisted maceration process significantly reduced processing time from hours or days to just a few minutes compared to the traditional maceration (Hasnia et al., 2018). Scanning electron microscopy confirmed the foaming effect on the surface of Tahitian lemon (Citrus latifolia Tan.) pomace seeds after extracting biological compounds such as polyphenols, hesperidin, narirutin,… using the ultrasound-assisted extraction (Oliveira et al., 2022).

The ultrasonic essential oil extraction process has been effectively applied to 4 species of Schisandra collected in the Qinling mountains, in which the extraction efficiency was quite high at 7.51%, 6.91%, 6.11% and 5.88% respectively for Schisandra grandiflora, Schisandra rubriflora, Schisandra sphenanthera and Schisandra propinqua (Wang et al., 2018).

The ultrasonic extraction process of essential oil was found to be an effective, low-cost and high-quality method for four natural products from citrus waste including essential oils containing D-limonene, pectin, hesperidin and beta-carotene. The composition and yield of essential oils depended on the ripeness of the citrus tree. The citrus essential oil yield and composition varied depending on the ripeness of the fruit (Tsitsagi et al., 2021).

Besides factors such as sample pretreatment, solvent type, solute/solvent ratio, temperature and extraction time, the characteristics of the ultrasound system have a great influence on efficiency and yield of phenolic compound extraction. For example, operation equipment mode (continuous or pulsed manner), ultrasound system (ultrasonic bath or system with a coupled probe), frequency and amplitude (Medina-Torres et al., 2017). Their effectiveness is influenced by various parameters, such as sonication time, temperature, solvent composition, size of the chopped plant, and ultrasound power (Mirdehghan Ashkezari et al., 2021).

2.3 Deep eutectic solvent (DES) extraction

The current emphasis in chemistry is on developing environmentally friendly technologies. Not only the methods, but also the extractants have evolved to greener ones. DES, a class of "green" solvents, has attracted attention because of their unique properties. DES, a solid-state mixture of proton acceptors (quaternary ammonium, tetraalkylammonium or phosphonium salts) and proton donors (acids, alcohols, amines or carbohydrates) connected by hydrogen bonds, has a lower melting point than its components, which remain liquid at low temperatures. Natural DES, derived from natural compounds in the solid state, offers advantages such as ease of preparation, biodegradation, and use of renewable, non-toxic compounds. Natural DES was applied in electrochemistry, organic synthesis, and the extraction of bioactive plant metabolites for potential use in the food and pharmaceutical industries without the need for their removal from the final product (Cakir et al., 2018; Palos-Hernández et al., 2022; Cajnko et al., 2023).

The essential oil trade is increasingly subject to legal regulations focusing on safety aspects (Can Başer & Buchbauer, 2015). The choice of extraction method and solvent plays an important role in determining the quality and quantity of the final product. If alcohol is a solvent, then it is considered "food grade" and safe for consumption. Essential oils extracted with solvents may contain residue, making them unsuitable for food applications (Suresh et al., 2021).

Careful consideration of factors such as selectivity, solubility, cost, and safety is important in selecting solvents for extraction. Water is widely used in the pharmaceutical, agricultural and food industries due to its compliance with Food and Drug Administration (FDA) requirements. However, water effectiveness is limited to polar compounds, with little effect on nonpolar compounds (Clark et al., 2017). Although water is the most common solvent in traditional herbal processing, in some cases some active ingredients in plants are insoluble in water. Aibinu et al. (2007) showed that some active ingredients in lime have high antibacterial/antifungal effects but will not escape unless the lime is combined with a specific solvent.

Other solvents include acetone, methanol, ethanol, ethyl acetate, and propanol. While highly polar solvents such as methanol can dissolve specific compounds, toxicity concerns have led to a preference for safer alternatives such as ethanol (Indriyani et al., 2023). In fact, research on extracting limonene - the main ingredient in lime peel essential oil showed the difference between n-hexane and ethanol solvent compared to water in the two methods of maceration and Soxhlet extraction. In the maceration extraction, using n-hexane gave the highest extraction efficiency (40.03%), followed by distilled water (33.10%) and ethanol (30.92%). Meanwhile, the yield of limonene obtained when extracted by the Soxhlet method using n-hexane is higher than that of ethanol (45.87% versus 42.75%), while distilled water has limitations for this method (Hasibuan et al., 2021). Although hexane was a common solvent for obtaining limonene, the main component of orange peel essential oil, it is considered toxic to health and the environment (Ibáñez et al., 2020)

Choline chloride, widely used due to its affordability and biodegradability, affects the melting point of DES based on the hydrogen bond donor ratio (Dheyab et al., 2021). The synthesis of DES, such as the combination of choline chloride and urea, has been explored since 2003, with variations involving choline chloride and carboxylic acids such as maleic acid, citric acid, acetic acid, malic acid or fructose, sucrose, glucose, trehalose and water. DES demonstrates the ability to extract bioactive compounds from a variety of plants including phenolics, flavonoids, isoflavonoids, terpenoids, alkaloids, anthocyanins, anthraquinones and polysaccharides (Shirsath et al., 2012).

Wu et al. (2021) studied the effects of 19 DES on the separation of terpenoids from citrus essential oils. The results showed that Tetrabutylammonium Chloride:1,3-Butanedio with a molar ratio of 1:2 has high extraction efficiency and is considered a potential solvent to separate valuable terpenoids during subsequent essential oil processing. Using DES as choline chloride: oxalic acid (1:1) in microwave-assisted hydrolysis promised to be a green, effective, fast and clean method to separate essential oils from Litsea cubeba (Lour.) Pers. fruits. The extracted essential oils were very different in composition and the ratio of substances with higher antioxidant and lower antifungal activities than some other methods. While the traditional hydrodistillation method required about 35 minutes to boil the raw material mixture to release essential oil, the time of microwave-assisted hydrodistillation with DES was shortened to only about 3.12 minutes. Besides, thanks to the characteristics and functions of DES, the distillation process according to microwave-assisted hydrodistillation with DES gave an extraction efficiency nearly 1.99 times higher than microwave-assisted hydrodistillation after 30 minutes of extraction (Guo et al., 2021).

3. CONCLUSION

In summary, the literature reviewed emphasizes the transformative impact of innovative methods in essential oil extraction, especially ultrasound-assisted extraction with DES. The findings from various literatures demonstrate that ultrasound-assisted extraction with DES consistently outperforms conventional methods, offering a sustainable and efficient alternative for obtaining essential oils from plant materials.

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