Review Article

Interprof. J. Health Sci. 2025, 23(2): IJHS-0876

Volatile Oil Extraction Using Supercritical Fluid: A Mini Review

Thanapat Songsak[1,*]

[1] Department of Pharmacognosy, College of Pharmacy, Rangsit University, Pathum Thani 12000, Thailand

*Corresponding author: E-mail: thanapat.s@rsu.ac.th

Received 19 November 2025; Revised 23 December 2025; Accepted 24 December 2025

Abstract: Volatile oils are complex mixtures of low-molecular-weight compounds responsible for the distinctive aroma of each plant species. In addition to their aroma properties, these oils exhibit a wide range of pharmacological activities, including antimicrobial, anti-inflammatory, and antioxidant effects. Supercritical fluid extraction has emerged as a highly efficient and environmentally friendly technique for obtaining volatile oils, offering multiple advantages over conventional extraction methods. This approach minimizes thermal degradation of heat-sensitive compounds, reduces the need for toxic organic solvents, and allows selective extraction of target constituents by fine-tuning operational parameters. This article reviews the fundamental principles of supercritical fluid extraction for volatile oil recovery, common supercritical solvents, and the critical factors influencing extraction efficiency, such as pressure, temperature, modifier type and concentration, extraction time, flow rate, particle size, and moisture content. Understanding and optimizing these variables is essential to maximize yield and to obtain volatile oils with desired chemical profiles.

Keywords*:* Carbon dioxide fluid, Modern extraction, Supercritical fluid, Volatile oil, Essential oil

INTRODUCTION

Volatile oils commonly known as essential oils represent a diverse and chemically complex class of secondary metabolites synthesized by plants (1). They are typically composed of a wide range of low-molecular weight organic compounds, predominantly belonging to the terpene family, including hydrocarbons such as monoterpenes (C_{10}) and sesquiterpenes (C_{15}) (2), together with their corresponding oxygenated derivatives such as alcohols, aldehydes, ketones, carboxylic acids, phenols, oxides, lactones, ethers, acetals, and esters (3). The relative proportions and structural diversity of these constituents vary extensively among plant species, genotypes, and even within different plant organs, imparting unique aromatic characteristics and biological activities. In addition to their olfactory and sensory properties, these volatile constituents play vital ecological roles in plant environment interactions, functioning in defense mechanisms, pollinator attraction, allelopathy, and communication with symbiotic organisms (4).

From an industrial and pharmacological standpoint, essential oils are among the most valuable natural products, widely utilized across diverse sectors including perfumery, cosmetics, food flavoring, pharmaceuticals, and aromatherapy. Their biological properties such as antimicrobial, antioxidant, anti-inflammatory, and insecticidal activities are closely related to their chemical composition and extraction integrity. Therefore, the method employed to extract these volatile fractions profoundly influences the yield, purity, and biological efficacy of the resulting oil (5-8).

Historically, steam distillation has been the principal technique for isolating volatile oils from aromatic plants. This process involves passing steam through plant material to volatilize essential oil components, which subsequently condense with water vapor upon cooling (9). Although simple and cost-effective, steam distillation presents significant limitations. The exposure of plant material to elevated temperatures and prolonged contact with moisture often induces hydrolytic degradation, oxidation, and rearrangement reactions, particularly among thermolabile and oxygenated compounds. These reactions not only reduce the overall yield but can also alter the characteristic chemical profile and sensory quality of the oil. For instance, certain monoterpenes may undergo isomerization or oxidation during distillation, leading to the formation of artifacts that deviate from the natural composition found in the plant (10-12).

In addition to distillation, solvent extraction methods employing organic solvents such as hexane, petroleum ether, chloroform, or ethanol have also been developed to recover volatile and semi-volatile compounds (13, 14). These techniques can extract a broader spectrum of constituents, including waxes, pigments, and non-volatile components, producing so-called "concretes" or "absolutes" after solvent removal. However, this approach introduces multiple drawbacks, including the risk of solvent residues in the final product, reduced environmental compatibility, and potential safety hazards due to solvent toxicity and flammability. Furthermore, solvent removal is an energy-intensive process that can compromise the stability of sensitive compounds (15-18). These challenges have driven the development of alternative extraction technologies that are both environmentally sustainable and capable of preserving the native chemical integrity of volatile oils.

Over the past few decades, a variety of innovative extraction techniques have been introduced to address these issues, including microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), pressurized liquid extraction (PLE), and supercritical fluid extraction (SFE). These methods share common goals shorter extraction times, higher selectivity, reduced solvent consumption, and enhanced preservation of bioactive constituents (19). Among these, SFE has emerged as the most promising technique, particularly when employing supercritical carbon dioxide ($scCO_2$) as the extraction medium (20, 21). The advantages of $scCO_2$ arise from its unique physicochemical properties that combine gas-like diffusivity with liquid-like solvating power, enabling efficient mass transfer and selective solubilization of target compounds under relatively mild operating conditions (22). Despite its numerous advantages, SFE also presents several limitations that must be considered. The primary constraint is the high initial capital investment associated with SFE equipment, which requires specialized high-pressure vessels, pumps, and control systems, leading to substantially higher costs compared with conventional extraction techniques. In addition, operational complexity and the need for skilled personnel can limit its widespread adoption, particularly at small or artisanal production scales. The relatively low polarity of $scCO_2$ also restricts its ability to efficiently solubilize highly polar compounds unless co-solvents are added, which may complicate downstream processing and reduce the simplicity of solvent-free recovery. Furthermore, scale-up of SFE processes can be technically challenging, as mass transfer, pressure control, and extraction kinetics may differ significantly between laboratory and industrial scales. These economic and technical considerations continue to represent important barriers to the broader industrial implementation of SFE, despite its recognized benefits in terms of product quality and environmental sustainability (23).

$ScCO_2$ extraction has gained prominence due to its green chemistry attributes and adaptability across a wide range of plant materials (22). Its low critical temperature (31 °C) and moderate critical pressure (73.9 bar) allow extraction of volatile oils without compromising thermolabile constituents (24, 25). Unlike traditional organic solvents, CO_2 is non-toxic, non-flammable, inert, inexpensive, and easily separated from the extract through depressurization, leaving no solvent residues (20).

Its solvating strength can be modulated by fine adjustments of pressure and temperature, enabling selective extraction of compounds based on polarity and molecular weight. When a higher degree of polarity is required for instance, to extract oxygenated monoterpenes or phenolic constituents small amounts of co-solvents such as ethanol or methanol can be introduced to modify the solvent's polarity (20).

$ScCO_2$ has been successfully utilized in the extraction of essential oils from a broad spectrum of botanicals, including *Citrus grandis* (26), *Coriandrum sativum* (27), *Eugenia involucrata* (28), *Foeniculum vulgare* (27, 29), *Mentha pulegium* (27), *Ocimum basilicum* (30), *Perilla frutescens* (31), *Santolina chamaecyparisus* (27), *Satureja fruticosa* (27), *Satureja montana* (27), and *Thymus vulgaris* (27). Comparative studies consistently indicate that SFE produces oils with superior quality, higher yields of thermolabile constituents, and enhanced concentrations of target compounds compared with conventional extraction methods like Soxhlet extraction, solvent maceration, or steam distillation (32). The superior efficiency of SFE arises from its ability to operate under finely controlled conditions that prevent chemical degradation and minimize contamination, making it an ideal approach for producing pharmaceutical and food grade essential oils (33).

Principles of supercritical fluid extraction

A supercritical fluid is a phase of matter existing above its critical temperature and pressure, where the distinction between liquid and gas phases disappears. In this state, the substance exhibits intermediate properties high density akin to a liquid, yet low viscosity and high diffusivity similar to a gas (22). These characteristics allow supercritical fluids to penetrate solid matrices more effectively and dissolve target solutes more efficiently than conventional solvents. The tunability of solvent density by adjusting pressure and temperature provides an additional level of control over extraction selectivity (23).

Various substances can exist in a supercritical state and serve as extraction solvents, including ethylene, nitrous oxide, propane, sulfur hexafluoride, methanol, water, ammonia, and *n*-pentane, each characterized by distinct critical constants (temperature, pressure, and density), as shown in Table 1. However, CO_2 dominates approximately 90% of all SFE applications due to its ideal balance of critical parameters, low cost, safety, and ease of recovery.

Table 1. Critical constants of selected solvents employed in SFE (34)

Compounds	Critical temperature		Critical pressure		Critical density (g/mL)
	K	°C	MPa	Bar (atm)	
Ethylene	283.0	9.9	5.12	51.2 (50.5)	0.23
Carbon dioxide	304.1	31	7.39	73.9 (72.9)	0.47
Nitrous oxide	309.6	36.5	7.26	72.6 (71.7)	0.46
Propane	369.8	96.7	4.26	42.6 (42.0)	0.22
Sulfur hexafluoride	318.8	45.7	3.76	37.6 (37.1)	0.75
Methanol	513.4	240.3	7.99	79.9 (78.9)	0.27
Water	637.0	363.9	22.1	221.0 (218.1)	0.32
Ammonia	405.4	132.3	11.3	113.0 (111.5)	0.24
n-Pentane	469.8	196.7	3.37	33.7 (33.3)	0.23

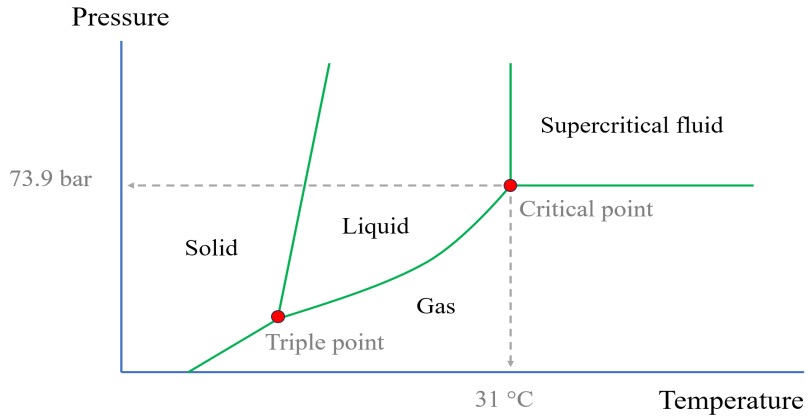

Figure 1. A phase diagram for CO_2

The phase diagram of CO_2 delineates the transitions among its solid, liquid, gaseous, and supercritical states as a function of temperature and pressure. When operating conditions exceed 31 °C and 73.9 bar, CO_2 becomes supercritical, forming a dense fluid capable of dissolving nonpolar and moderately polar solutes (Figure 1). Minor adjustments in temperature and pressure can drastically alter the solvent density and solvating strength, permitting precise tuning of extraction selectivity for different classes of volatile compounds. This versatility makes SFE particularly suitable for essential oils, where chemical diversity and volatility require highly controlled extraction parameters.

A typical SFE system consists of a CO_2 reservoir connected to a high-pressure pump, often combined with a modifier reservoir for co-solvent addition, which together deliver pressurized fluid to the extraction vessel containing the raw material. The extraction vessel is equipped with pressure and temperature control to maintain supercritical conditions. Downstream, the extract-laden fluid passes through one or more separators, where controlled depressurization and temperature adjustment enable selective precipitation of target compounds. Pressure regulators, valves, and gauges are integrated throughout the system to ensure stable operation, precise control of extraction parameters, and safe recovery of both the extract and recycled CO_2 (35).

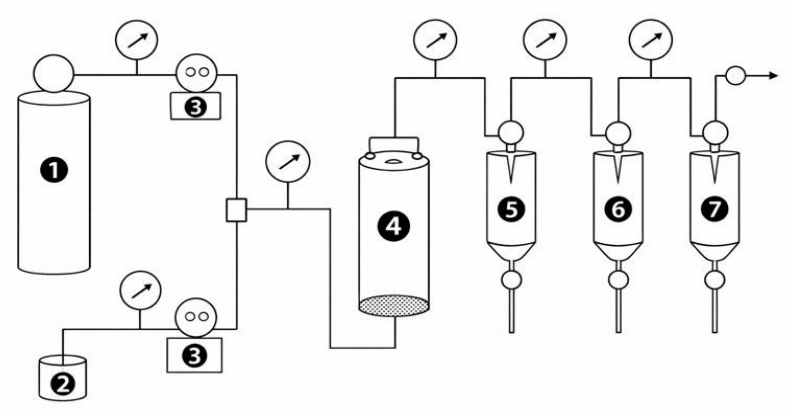

Figure 2. Schematic representation of a SFE system. ❶ denotes the carbon dioxide reservoir, ❷ represents the co-solvent reservoir, ❸ indicates the pump, ❹corresponds to the extraction vessel, while ❺, ❻, and ❼ refer to separator I, separator II, and separator III, respectively (35).

Factors affecting volatile oil extraction by SFE

Several physicochemical and operational parameters govern the efficacy, selectivity, and chemical profile of volatile oils obtained by $scCO_2$ extraction. A detailed understanding of these parameters is essential for optimizing extraction protocols to maximize yield, maintain chemical integrity, and tailor the composition of the resulting oil to specific industrial or pharmacological purposes.

Pressure and temperature

Pressure primarily dictates solvent density and solvation capacity. Higher pressures typically increase the density of $scCO_2$, enhancing the solubility of nonpolar compounds and improving extraction yield. However, excessive pressure can result in the co-extraction of undesired components, such as waxes or pigments, which may complicate downstream processing. Temperature exerts a dual and sometimes opposing effect: while increasing temperature reduces solvent density (potentially lowering solubility), it simultaneously elevates the vapor pressure of solutes, facilitating their desorption and diffusion from the plant matrix. The balance between these effects depends on the physicochemical nature of the solutes and must be empirically optimized (23, 35, 36).

Modifiers (co-solvents)

The inclusion of a polar modifier typically ethanol, methanol, or water can substantially broaden the solvating range of $scCO_2$ by increasing its polarity (20, 23). For example, ethanol has been used as a co-solvent for the extraction of volatile oil from *Lavandula hybrida* (37), while methanol has been used as co-solvent for the extraction of volatile oil from *Descurainia sophia* (38). This facilitates the extraction of oxygenated and phenolic constituents that would otherwise remain poorly soluble. However, an excessive modifier concentration may adversely alter the phase behavior of the system or reduce selectivity (23, 39).

Extraction time

The duration of extraction influences both total yield and compositional balance. Shorter extraction periods favor highly volatile compounds such as monoterpenes, whereas prolonged extraction enhances recovery of heavier, less volatile constituents like sesquiterpenes and oxygenated derivatives. Thus, extraction time should be optimized to balance yield, quality, and process economics (23, 40).

Flow rate of supercritical fluid

The flow rate of the supercritical solvent determines solvent residence time and mass transfer kinetics. Lower flow rates promote higher solute–solvent interaction and more efficient extraction per unit of CO_2, while higher flow rates shorten extraction time but may compromise efficiency (36, 41, 42). As observed in the extraction of volatile oil from *Coriandrum sativum*, a flow rate of 0.79 kg/h resulted in a higher extraction yield than flow rates of 1.10 and 1.59 kg/h (43).

Particle size of plant material

The size of plant particles directly affects diffusion and solvent accessibility. Reducing particle size increases surface area and enhances mass transfer; however, excessively fine powders can lead to packing issues, channeling, and uneven solvent flow. Optimal particle sizes typically range between 0.3–1.0 mm, though this varies by plant species (23, 44, 45). For example, a mean particle size of 0.4 mm for *Thymus vulgaris*, compared with 0.6 and 0.8 mm, showed the highest extraction rate for volatile oil extraction (46). However, the optimal particle size differed among individual volatile oil components in *Coriandrum sativum* (43).

Moisture content

Moisture acts as a polar modifier, influencing the partitioning behavior of solutes. Moderate water content facilitates the extraction of polar constituents by increasing system polarity, but excessive moisture reduces solubility of lipophilic compounds due to phase separation into aqueous and supercritical phases. The matrix's moisture level must be lowered to below 15% prior to treatment (23). Hence, controlling moisture content within an optimal range is crucial to maximize both selectivity and yield (47, 48).

CONCLUSION

SFE, particularly with $scCO_2$, represents a highly efficient, tunable, and environmentally sustainable method for volatile oil recovery from plant materials. By carefully adjusting parameters such as pressure, temperature, co-solvent type, extraction time, flow rate, particle size, and moisture content, it is possible to maximize yield while preserving the chemical integrity of target compounds. The flexibility of SFE makes it applicable across a wide range of botanical sources, and its environmentally benign nature positions it as a preferred technology for producing high-quality volatile oils in both research and industrial contexts. Future developments in process modeling, scale-up, and co-solvent strategies are expected to further enhance the efficiency and selectivity of this technique.

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