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A Review on Supercritical Fluid Extraction

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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Review Article

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ABSTRACT

Industries are in search of best and cost-effective processing technologies to extract components from any material with maximum purity and quantity. The drawbacks of traditional solvent extraction methods led to the development of novel processing technologies that can be employed into any industry. This review mainly focuses on supercritical fluid extraction (SFE) process, an innovative, environmentally friendly tool for food processing techniques. Here, we are discussing about the general aspect of SFE and their recent potential commercial applications in different fields. Nowadays, this method gained lots of importance in food as well as non-food industrial applications.

Keywords: Extraction; supercritical fluid; supercritical carbon dioxide technology; purity; pharmaceuticals.

1. INTRODUCTION

"The process of obtaining specific substance from a mixture or compound by physical, chemical or mechanical means is called extraction. Physical extraction methods include maceration, distillation, etc., chemical methods include solvent extraction and supercritical fluid

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extraction while mechanical process include expeller, hydraulic press etc. Extraction is the first step for separating the desired products from the raw materials. The two phases yield during this process include feed solvent rich raffinate phase and solute rich extract phase" [1].

Extraction involves standard extraction procedures using selective solvents for the separation of active compounds of animal or plant tissue from inert components. The obtained products are intended only for oral or external use in the form of relatively impure liquids, semisolids or powders. Extraction was done continuously to obtain improved yields of drug derived from plant and animal sources. Investigation of novel techniques of extraction continued to get higher yields of the active substances from natural sources with maximum purity.

2. SOLVENT EXTRACTION

"Solvent extraction or liquid-liquid extraction refers to the partition of components of the liquid mixture by contacting with a suitable insoluble liquid solvent which dissolves one or more components" [2]. "In this method a substance is extracted from one liquid phase into another liquid phase. Liquid extraction consists of two phases: the feed solution phase and the extraction solvent as another phase. In this method, both feed and solvent forms a homogenous mixture and are separated by contacting it with one another which separates out one of the two liquids preferentially" [3]. Solvent extraction is used in processing of perfumes, ore processing, nuclear processing, production of fine organic compounds and other industries.

3. SUPER CRITICAL FLUID EXTRACTION

"The process of filter outing the extractant from any matrix using supercritical fluids as the extracting solvent is known as Supercritical Fluid Extraction (SFE). Usually, solid matrix is used, but liquid can also be used. Due to the high efficiency of SFE, this was introduced and studied extensively for separation of active compounds from herbs and other plants" [4]. "The high solvation power of supercritical fluids (SF) was first reported over a century ago" [5]. "It was first introduced in 1879 by Hannay and Hogarth. In 1969, demonstration of SFE technology for industrial applications was reported by Zosel at the Max Planck Institute for Kohlemforschung" [6].

Selective separation of the desired compounds can be achieved by supercritical CO_2 without leaving toxic residues in extracts and the thermal degradation risk of processed products. The high volatility of solvents at ambient conditions influences the solvating power near the critical points of the fluids and the sensitivity to small disturbances in temperature, pressure and modification of the solvent. Deeper penetration into solid plant matrix and more efficient and faster extraction were achieved by the favourable transport properties of fluids near their critical points.

"The high investment costs and unfamiliarity of operation restricted the commercial application of supercritical fluid technology to few products for the past three decades. The advances in process, equipment and product design and the potential for the production of high value-added products with profitable opportunities give way to more and more interest in supercritical fluid technology in different industries" [7]. Batch or continuous high-pressure equipment can be used for the extraction. Separation of desirable product is attained by the contact of the SF with the material in both cases. Expansion of the SF to atmospheric conditions due to the saturation of SF with extracted product and recovery of the solubilized product is possible in the separation vessel permitting the SF to recycle for further use.

"In recent years, SFE has received a great deal in analytical applications" [8]. "Nowadays, SFE has become an acceptable extraction technique in many areas. Extraction of active natural products from herbal and plant materials using SFE has become one of the most important application areas" [4]. This method also gained much importance in many fields, especially with the increasing public interest in herbal medicines and natural products.

4. SUPERCRITICAL FLUID

"Supercritical fluids are any substance with temperature and pressure above its critical point. It holds properties of both gas and liquid by diffusing through solids like a gas and dissolving materials like a liquid. It is having a density that analogous to a liquid, while viscosity and diffusivity are similar to a gas. Therefore, a supercritical fluid can act as a solvent similar to a liquid, but with enhanced mass transfer kinetics. The substitution of organic solvents with supercritical fluids are gaining importance in a

range of industrial and laboratory processes. The most commonly used supercritical fluids include carbon dioxide and water" [8]. The critical properties of some commonly used supercritical fluids are given in Table 1

4.1 Phase Diagram

The phase diagram for supercritical fluid is represented in Fig. 1.

4.2 Properties of Supercritical Fluid

The physical and thermal properties of supercritical fluids are in between of pure liquid and gas; therefore, known as compressible liquid or dense gas.

- Possess liquid like densities - 100 to 1000 times greater than gases
- Higher diffusivities than liquid in the range of 10^{-3} to 10^{-4} cm^2/s
- Good solvating power similar to light hydrocarbons
- Increased solubility with increasing density
- Low viscosity - 10 to 100 times less than liquid
- High miscibility of fluids with permanent gases like N_2 or H_2
- Possess high penetrating power compared to conventional solvents

Table 1. Critical properties for some components commonly used as supercritical fluids

Critical properties of various solvents [9]				
Solvent	**Molecular weight (g/mol)**	**Critical temperature (K)**	**Critical pressure MPa (atm)**	**Critical density (g/cm^3)**
Carbon dioxide (CO_2)	44.01	304.1	7.38 (72.8)	0.469
Water (H_2O)	18.01	647.1	22.06 (217.8)	0.322
Methane (CH_4)	16.04	190.4	4.60 (45.4)	0.162
Ethane (C_2H_6)	30.07	305.3	4.87 (48.1)	0.203
Propane (C_3H_8)	44.09	369.8	4.25 (41.9)	0.217
Ethylene (C_2H_4)	28.05	282.4	5.04 (49.7)	0.215
Propylene (C_3H_6)	42.08	364.9	4.60 (45.4)	0.232
Methanol (CH_3OH)	32.04	512.6	8.09 (79.8)	0.272
Ethanol (C_2H_5OH)	46.07	513.9	6.14 (60.6)	0.276
Acetone (C_3H_6O)	58.08	508.1	4.70 (46.4)	0.278

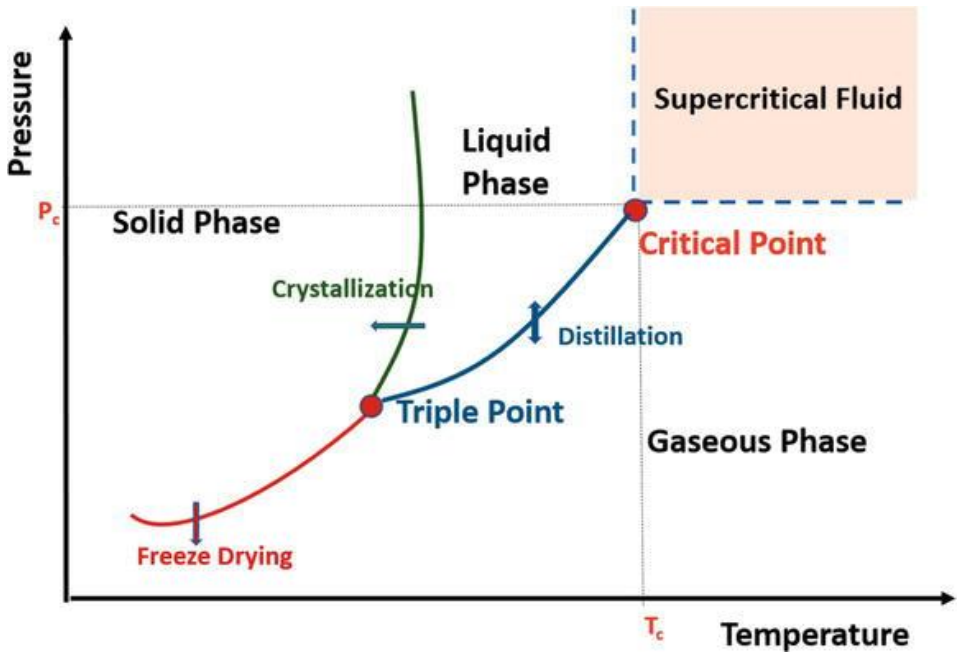

Fig. 1. Phase diagram of supercritical fluid
(Source: Intechopen, 2022)

4.3 Carbon Dioxide

"For the development of a SFE process, proper selection of SFs is very important and number of compounds can be used as solvents in this technique" [7]. "Among various supercritical fluids used for extraction, supercritical CO_2 (SC-CO_2) is the most widely used one since it is non-toxic, non-flammable, non-corrosive, tasteless and odourless, inert and supercritical operation at low pressures and near room temperature is easy to handle" [10]. "Its high degree of purity, cheapness and availability in bulk quantities are another benefit" [11]. "Carbon dioxide ensure minimal alteration of the bioactive compounds and to preserve functional and therapeutic properties" [12]. Use of gaseous CO_2 at room temperature and pressure ensure very simple recovery of compound and solvent-free extracts. FDA (U.S. Food and Drug Administration) and EFSA (European Food Safety Authority) were acknowledged this molecule as "Generally Recognized As Safe" (GRAS) and environmental friendly.

4.4 Other Super Critical Fluids

"Extraction of highly polar compounds from a matrix is less effective because of the least polar nature of the Carbon Dioxide" [13]. "Polar molecules are poorly soluble in SC-CO_2 and hence are not extractable" [14]. "For this reason, the use of other solvent compounds is needed in order to enhance solubility and the selectivity of the process and they must be added only in small quantities" [7]. "This phenomenon is known as co-solvent effect attributed to various components acting as solubility enhancers and is called co-solvent effect. Cosolvents or modifiers include hexane, methanol, ethanol, isopropanol and dichloromethane. The lower toxicity and miscibility of ethanol in CO_2 surges its application as a co-solvent in SFE , but safety and environment considerations due to some unfavourable properties limited its usage" [15,16]. Other co-solvents used are Nitrous Oxide which is similar in solvating and separations properties to CO_2 and alkanes better solvent characteristics for non-polar solutes.

5. PRINCIPLE

"In supercritical carbon dioxide technology (SC-CO_2 technology) destruction of microorganisms is achieved by utilizing pressure in combination with carbon dioxide without affecting the nutritional content and organoleptic attributes by an alternative for pasteurization of bioactive compounds in food and medicine" [17]. "The solubility of the target compound is the driving force for any extraction process which depends on the interactions between the solvent and solute. Reduction of extraction time, less consumption of organic solvents; being suitable for thermo-sensitive substance, production of cleaner extracts and environmental benignity make this method as a superior alternative technique for extraction of bioactive species from natural produces" [18].

"By employing pressure and temperature above the critical point of a compound, mixture or element attain solvating properties of supercritical fluids which becomes the basis for SFE process. Modification of extractability of SF by proper controlling of SFE parameters enable application of this process from food to pesticide research. For extraction of non-polar compounds CO_2 is the preferred one, but the addition of a miscible polar compound such as ethanol as modifier increases the polarity of SC-CO_2 technology" [19]. "Low concentration of undesired compounds in the extracts is obtained by the selectivity of the SFE process" [20]. Besides, SC-CO_2 becomes gaseous after depressurization and can be easily eliminated from a flow system.

6. SUPERCRITICAL FLUID EXTRACTION PROCESS

The SFE system must contain a pump for pumping the CO_2, a pressure vessel to contain the sample, a pressure maintenance arrangement in the system, a collecting vessel and heating and cooling mechanism. Pumping of liquid to a heating zone causes heating of the liquid which is further converted to supercritical conditions. After reaching the extraction vessel, the material to be extracted is dissolved in the SF by rapidly diffuses into the solid matrix. Low pressure separator swept the dissolved material from the extraction cell where the extracted material settles out. After the completion of extraction, cooling, recompressing and recycling or discharging of CO_2 can be done. The operating principle of semi batch supercritical fluid extraction is represented in Fig. 2.

6.1 Pumps

Carbon dioxide is usually pumped as a liquid, usually below 5°C and a pressure of about 50 bars. As the solvent is almost incompressible, it

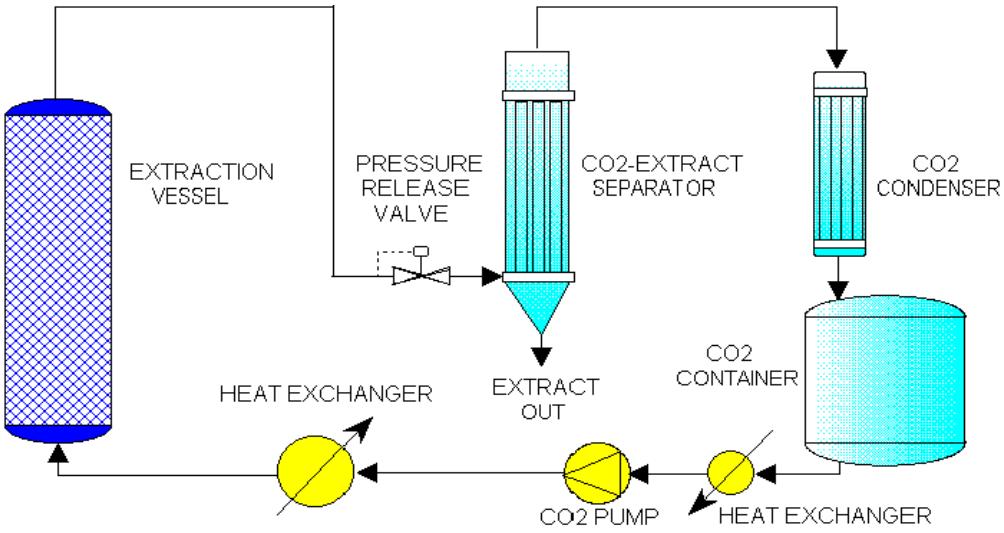

Fig. 2. Operating principle of semi batch supercritical fluid extraction
(Source: ResearchGate, 2000)

is pumped as a liquid. Much of the pump stroke is used for compressing the fluid rather than pumping due to supercritical nature of the fluid. Reciprocating CO_2 pumps or syringe pumps are used for small-scale extractions, while larger scale extractions use diaphragm pumps. Both pump heads and CO_2 require cooling for the efficient working of the pump.

6.2 Pressure Vessels

"Pressure vessels of different types vessels are available from simple tubing to more sophisticated purpose-built cells with quick release fittings. The minimum pressure requirement is 74 bars, but less than 350 bar is preferred for most extractions. In case of vegetable oil extraction, higher pressures of 800 bar are sometimes required for complete miscibility of the two phases" [21]. A heating mechanism is equipped with the vessel. For small vessels, heating system can be placed inside an oven, while larger vessels use oil or electrically heated jacket. If rubber seals are used on the vessel, proper care must be taken to avoid dissolution of CO_2 in the rubber which causing swelling and rupture of rubber on depressurization.

6.3 Pressure Maintenance

Maintenance of pressure in the system must be carried out from the pump right through the pressure vessel. A simple restrictor can be used in smaller systems to maintain pressure at different flow rates either as a capillary tube cut to length, or a needle valve. Backpressure regulator is used in larger systems in which a spring, compressed air, or electronically driven valve employed for upstream pressure maintenance. During pressure maintenance adiabatic expansion of the CO_2 occur which results in significant cooling, to avoid this heating must be supplied whichever method is used. Presence of water or other extracted material in the sample causes problems related to freezing of these in the restrictor or valve and initiation blockages.

6.4 Collection

A vessel with lower pressure than extraction vessel is used for collecting the supercritical solvent. The pressure causes sharp variation in the density and dissolving power of supercritical fluids, which further initiate precipitation of material due to much lower solubility in the lower density CO_2. Fractionation of the dissolved material is possible by using a series of vessels at reducing pressure. The CO_2 can be recycled or depressurized to atmospheric pressure and vented. The pressure is usually dropped to atmospheric pressure in case of analytical SFE, but nowadays, for trapping the precipitated components the gaseous carbon dioxide is bubbled through the solvent.

6.5 Heating and Cooling

This is an important aspect consist of cooling the fluid before pumping to maintain liquid conditions, and heating after pressurization.

Heating must be provided to block excessive cooling when the fluid is expanded into the separator. Preheating the fluid in a length of tubing inside the oven containing the extraction cell is sufficient for analytical purposes, where electrical or heating with a hair dryer is enough in the restrictor. The thermodynamic properties of the SF can be used for calculating energy requirement during each stage of the process for larger systems.

6.6 Extraction/Separation Methods

Several methods are available for the separation of solute from supercritical fluid. The three basic concepts that can be used to accomplish this include

- The solvent capability of the SF changes by changing the pressure or temperature.
- Washing solute out of the SF using a solvent that can strip the solute from the SF.
- Separation of multiple solutes within the SF using a packed column.

The simplest and easiest method of removal use the concept of changing the pressure of the SF so that fluid is no longer exist in supercritical condition or the solute become insoluble in the SF. In this method the solute/solvent combination passed to a restriction valve and then to a collection vessel. The pressure inside the collection vessel was reduced to room pressure, which cause change of state from supercritical to initial gas, thus the solvent released into the collection vessel. The collection vessel consists of a solvent to receive the solute for performing further analysis. Keeping the vessel in empty condition during other times causes crystallization of the solute upon leaving the SF.

Another method consists of changing the temperature of SF to remove the solute. Temperature change may cause the fluid to leave the supercritical region by maintaining the pressure of the fluid. Here the pressurized fluid can be recirculated without having to repressurize it which could save money. This method was utilized by Lancas et al. [22] to remove their respective solutes from the SF. Temperature drop of the solvent cause removal of the solute until the solute is no longer soluble and precipitates from the SF and referred as a cryogenic trap. After completion of the extraction, depressurization of the system takes place and the extract can be removed from the sample chamber.

Washing is the other method used for the removal of the solute from SF. In this method, constant pressure should be maintained as like cryogen gap without having to shut the process down. Decaffeination of green coffee beans to remove the caffeine from SC-CO_2 is the best example of this type of removal [23].

7. ADVANTAGES AND DISADVANTAGES

7.1 Advantages

- Environmental improvement and reduced product contamination.
- Rapid process completed within 10 to 60 minutes.
- Supercritical fluid can be separated from analyte by simply releasing pressure and complete separation of solvent from extract and raffinate is possible.
- Recovery of solvent and analytes are simple and easy.
- Elimination of organic solvents which reduces risk of storage.
- Suitable for extraction and purification of compounds having low volatility present in solid or liquid.
- Continuous process and low handling cost.
- Efficient and versatile method

7.2 Disadvantages

- Lak of standard extraction procedure
- Difficulties in extracting polar compounds
- Inefficiency in cleaning up

8. APPLICATIONS

Application of SFE is gaining much importance in different areas. These include food, natural products, pharmaceuticals, environmental applications etc. It is based on the observation that when any gas compressed above a critical point, its dissolution power will increase [24]. In this review, recent research works conducted in SFE from last five years are mentioning below.

Decaffeination of tea or coffee [25], extraction of flavors from herbs [26], extraction of fats and oils [27], extraction of sugars from bamboo [28], extraction of antioxidants such as vitamin E and C [29], extraction of aromas from different juices [30], de-acidification of oil [31] and extraction of tocopherols from vegetable oils [32] are the major fields where SFE technique is used. Other important applications of SFE are such as extraction of hops [33], extraction of bioactive

compounds [34-36], extraction of free amino acids [37], and the separation of spices and essential oils [38].

Ferrentino et al. [39] applied SFE for the extraction of phenolics compounds from freeze-dried apple pomace. Campone et al. [40] employed SFE for the recovery of flavonoids from brown onion peels. The extract obtained under optimized conditions (10 MPa, 40°C and 85% ethanol) presented high content of quercetin and quercetin derivatives. Apple seed oil was extracted from apple seed under conditions such as pressure: 300, 500, 750, 1000 and 1300 bar and temperature 43, 53 and 63°C [41]. Green coffee beans were used for extracting oil using 0-5.7% ethanol as co-solvent under optimized conditions (200–400 bar and 40–60°C) by Bitencourt et al. [42]. Jokic et al. [43] separated oxygenated monoterpenes, α-humulene, viridiflorol and manool from sage leaves under 100–300 bar pressure and 40–60°C temperature. Total phenolic compounds and total flavonoids were collected from radish leaves using pressure: 300 and 400 bar, temperature: 35, 40, and 50°C, CO_2 flow rate: 10 g/min and co-solvent: ethanol [44].

Extraction of flavonoids from *Odontonema strictum* leaves were carried out by Ouedraogo et al. [45] under 200 and 250 bar, 55–65°C, CO_2 flow: 15 g/min and 95% ethanol. Pimentel-Moral et al. [46] conducted extraction of phenolic compounds from *Hibiscus sabdariffa* using pressure: 150–350 bar, temperature: 40 to 60°C, Co-solvent: ethanol 7–15% and total flow: 25 g/min). Tocopherol was extracted from Quinoa under optimized conditions of pressure: 200–400 bar and temperature: 40–60°C [47]. Different studies have been carried out to extract β-carotene and lycopene from tomato processing waste stream [48]. Extraction of cholesterol and some other fats without removing polar fats that is liable for sensory characteristics [49]. Pourmortazavi et al. [50] done extraction of cholesterol, lipids and fats at optimum particle size and moisture content. Alvarez et al. [51] studied the effectiveness of SFE and the use of ethanol as a co-solvent for the recovery of phytochemicals with antioxidant capacity from soybean oil extraction by-products (soybean expellers).

Catharanthus roseus, a rich source of alkaloids, from which two dimeric alkaloids were extracted that are extensively used as antineoplastic drugs vinblastine and vincristine in pharmacy [52]. Sánchez-Camargo et al. [53] optimized the SFE conditions for the isolation of carotenoids from mango peel using a Box–Behnken design. Roselló-Soto et al. [54] investigated the effect of SFE pressure (10–40 MPa) on the recovery of oil from “horchata” by-products. Lima et al. [55] has been used the extract of mango peel as a natural antioxidant in sunflower oil to control lipid oxidation; the optimal conditions were 25 MPa, 60°C and ethanol in water (15%) by using a Box–Behnken design.

Caffeine was extracted from Guayusa leaves on study conducted by Cadena-Carrera et al. [56] under optimized parameters (150, 200 and 250 bar, 45, 60 and 75°C and CO_2 mass flow: 8.3 g/min). Germacrene was obtained using SFE technique from leaves of *Piper klotzschianum* by Lima *et al*. (2019) (180, 200 and 220 bar, 40, 60 and 80°C and Co-solvents: methanol 1%, ethanol 3% and isopropanol 5%) [57]. Natolino and Da Porto [58] used pomegranate seed oil for fatty acid extraction at pressure: 240, 280 and 320 bar, temperature: 40, 50 and 60°C and CO_2 flow rate: 133.3 g/min.

Devani et al. [59] conducted a study on rotten onion waste using SFE to evaluate the oleoresin extraction yield, sulfur content and pyruvate content through a central composite rotatable design through evaluating the effects of pressure (15–45 MPa), temperature (50–90°C), time (30–150 min) and particle size (0.4–1.2 mm). Hatami et al. [60] obtained tocols from passion fruit by-products by proposing a process based on the integration of SFE and supercritical adsorption (SESA). Salinas et al. [61] optimized SFE to extract oil from chanar almonds, a residue from “arrope” production. Using the optimum conditions such as pressure: 250, 300 and 350 bar, temperature: 40, 50 and 60°C, CO_2 flow rate: 3, 5 and 6 g/min and particle size: 0.2–0.4 mm raspberry seed oil was extracted from raspberry seeds by Pavlic et al. [62].

The selective enrichment of terpenoids in the different olive leaves extracts were done by innovative supercritical CO_2 fractionation process based on the online coupling of supercritical fluid extraction (SFE) and dynamic adsorption/desorption by using different commercial adsorbents such as silica gel, zeolite, and aluminium oxide under operating at 30 MPa and 60°C [63]. Luis et al. [64] extracted phenolic compounds from mango seed kernels and sugar using supercritical CO_2 and EtOH as an extraction solvent and evaluated the effect of

extraction pressure (11–21 MPa), temperature (40–60°C) and co-solvent contribution (5–15% w/w EtOH) on extraction yield, oxidative stability of sunflower edible oil, total phenolics content, total flavonoids content, and DPPH radical assay. A study was conducted by Thibault et al. [65] to selectively extract bioactive compounds and pigments from rosemary using supercritical fluid extraction by optimisation using supercritical fluid extraction online coupled with a supercritical fluid chromatography (SFE-SFC) system.

Mari et al. [66] evaluated the effects of SFE on the recovery of bioactive and antioxidant compounds using *Coccomyxa onubensis*, by studying variables such as extraction yield, lutein purity and recovery, total phenols and antioxidant capacity using a Box–Behnken design based on a response surface methodology along with the overall extraction curve fitted to a spline linear model. A study aims to explore the optimal preparation technology of fucoxanthin from *Undaria pinnatifida* stems using supercritical carbon dioxide methods and provides approaches for the extraction and preparation of bioactive compounds from a waste seaweed part [67].

Shi et al. [68] used uric acid as the model compound of nitrogenous components in chicken manure to investigate the transformation mechanism of nitrogen during the supercritical water gasification (SCWG) process. Mateusz et al. [69] determined the capacity of residues of alfalfa and goldenrod after supercritical CO_2 extraction to carry micronutrients and energy production against raw plant materials.

9. CONCLUSION

Supercritical Fluid Extraction (SFE), an innovative extraction technique that is gaining momentum in the past two decades. In general, supercritical carbon dioxide technology is widely used. It is used for the decaffeination of coffee, extraction of bioactive compounds from natural sources and by-products, extraction of fats and oils, separation of colour, flavour and essential oils from herbs or spices, extraction of antioxidants etc. This versatile method can be employed in food industry, pharmaceuticals, cosmetics, analytical procedures and environmental analysis. Therefore, supercritical fluid technologies are very much relevant with its wide application and it is foreseen that the technologies will be expanded in the next decade.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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