

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

Abstract

Supercritical fluid extraction plays a vital role across various industries, and its significance is particularly noteworthy in the food industry. This technique represents an innovative extraction method that not only increases yield but also reduces extraction time, ensuring higher quality and precise extraction of beneficial compounds from oils. Critical factors for successful SFE encompass temperature, pressure, the use of co-solvents, particle size, and moisture content. These factors allow for the selective extraction of desired components from seed oils and essential oils, ensuring top-notch quality. This approach is not only widely accepted but also aligns with environmentally sustainable practices, offering promising solutions to the challenges faced by industries seeking efficient and eco-friendly methods for extracting valuable compounds from seeds and other raw materials.

Keywords: Supercritical fluid extraction, seed oils, essential oils, spices, oil seeds

Authors

Nirav U. Joshi

Assistant Professor
College of Food Technology
Sardarkrushinagar Dantiwada Agricultural University
Sardarkrushinagar, India,
joshinirav30@gmail.com

Abhishaben M. Shingala

Assistant Professor
College of Food Technology
Sardarkrushinagar Dantiwada Agricultural University
Sardarkrushinagar, India,

Mukesh N. Dabhi

Professor and Head
Department of Processing and Food Engineering
College of Agricultural Engineering and Technology
Junagadh Agricultural University
Junagadh, India

Chandani J. Popalia

Assistant Professor
College of Food Technology
Sardarkrushinagar Dantiwada Agricultural University
Sardarkrushinagar, India,

Arjun R. Parmar

Ph.D. Scholar
Department of Processing and Food Engineering
College of Agricultural Engineering and Technology
Junagadh Agricultural University
Junagadh, India

I. INTRODUCTION

Seed oils, also known as vegetable oils or plant oils, are oils extracted from the seeds of various plants. These oils find widespread utility in cooking, industry, cosmetics, and medicine. Seed oils constitute a crucial dietary fat source, exhibiting variations in taste, nutritional makeup, and application depending on the specific plant source of extraction. Oil extraction from seeds is a crucial process with significant importance in various industries and for several reasons. Lately the researchers have been focusing on different extraction techniques of bioactive compounds of essential and seed oils and utilization of these extracted compounds from different seeds and spices to form a functional foods.

The simplest and most widely used technique for extraction of oil is solvent extraction using the petroleum based solvents like hexane, toluene, dichloromethane, chloroform etc. The cold pressing and expeller pressing is the mechanical methods for pressing the seeds or cakes to extract the oil. Problem in these methods are lower yields, energy intensive, time consuming, leaves chemical residues in extracted oil and inability of doing selective extraction of certain health promoting compounds in oils. Newly developed techniques i.e. enzyme assisted extraction, microwave assisted extraction and ultrasound assisted extraction and super critical fluid extraction solves the problems caused by traditional techniques. Among these techniques, Supercritical fluid extraction is gaining popularity as a solvent in the extraction of seed oils. In SFE, CO₂ is pressurized above its critical point, allowing it to act as both a gas and a liquid. It is a cleaner and more selective method, often used for high-value oils and in industries where solvent residue is a concern.

The present chapter focuses on the supercritical fluid extraction of oils from seeds and essentials oils from spices, different parameters affecting the supercritical fluid extraction and application in SFE in food industry.

II. SUPERCRITICAL FLUID EXTRACTION (SFE)

Supercritical Fluid Extraction (SFE) is a separation technique employed across diverse sectors, including food, pharmaceuticals, and environmental science, to extract valuable substances from solid or liquid substances. This process hinges on utilizing a supercritical fluid, usually carbon dioxide (CO₂), thanks to its mild critical point conditions, as the extraction medium.

In 1978, the initial commercial application of supercritical fluid extraction in food was carried out in Germany by Hag A.G for the purpose of decaffeinating green coffee beans. Subsequently, two years later, Carlton and United Breweries in Australia introduced a technique that utilized liquid carbon dioxide for the extraction of hop flavours. Both of these applications proved to be commercially viable and have since served as the basis for the development of various adaptations and enhancements, many of which have been implemented at an industrial level (Kazlas *et al.*, 1994). Since then SFE technique have been used in different food products.

Supercritical fluid extraction offers several advantages in terms of mild critical control point conditions i.e. low critical temperature and pressure range, use of non-toxic and non-flammable and environmentally friendly solvent, selective extractability, residue free

extraction and compatibility with sensitive compound. The most commonly used in solvent in this technique is carbon dioxide (critical conditions = 30.9 °C and 73.8 bar). Other supercritical fluid like ethane, propane and water are also used for specific applications depending on their properties and the desired extraction results.

III. BASIC PRINCIPLES OF SUPERCRITICAL FLUID EXTRACTION

A supercritical fluid is a matter that exists at conditions of temperature and pressure beyond its critical point, exhibiting characteristics of both a gas and a liquid simultaneously. In the context of SFE, CO₂ is frequently preferred due to its adaptability in transitioning to a supercritical state by manipulating pressure and temperature. In this supercritical state, CO₂ possesses a density and solvating capacity akin to that of a liquid, yet it permeates solids like a gas, rendering it an exceptional solvent for extracting a wide array of substances.

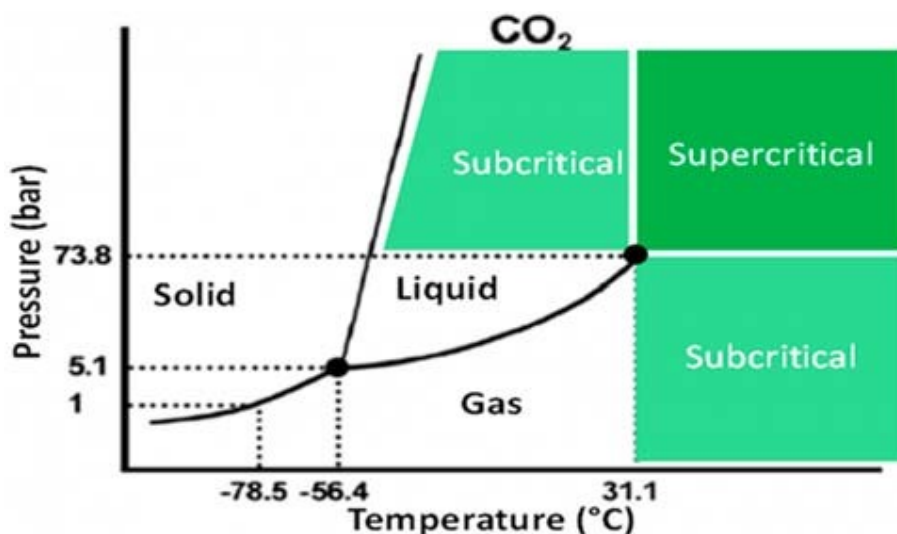


Figure 1: Diagram of Pressure-Temperature Phase of CO₂ (Gandhi *et al.*, 2017)

Supercritical Fluid Extraction (SFE) is a method that employs a fluid phase with properties that lie between those of a gas and a liquid. At the critical point on the pressure-temperature phase diagram, the gas's liquid state becomes permanently fixed regardless of the applied pressure (as shown in Figure 1). At this juncture, the substance exhibits characteristics of both a liquid and a gas. The densities of gases surpass their critical limits as pressure increases, leading to a desirable enhancement in the solvent capabilities of liquids. This heightened solvating ability of CO₂, for example, encourages solutes to dissolve within the supercritical solvent medium. As the fluid's density shifts, so does diffusivity, promoting more efficient mass transfer between the solute and the solvent. In contrast, the viscosity values of supercritical fluids remain consistent with those of normal gases and liquids. Supercritical fluids possess an augmented solvent capacity akin to that of a liquid while also mimicking gases in their absence of surface tension.

Substances that surpass their critical temperature and pressure become pure supercritical fluids. Beyond the critical temperature, they do not undergo condensation to form a liquid but exist as a fluid (a dense gas), with their properties continuously transitioning

from gas-like to liquid-like as pressure rises at a fixed temperature (Kiran *et al.*, 2009). The selectivity of extraction can be somewhat controlled since the solvating power (influenced by density) of SCFs can be adjusted significantly by modifying pressure, temperature, or both.

Supercritical CO₂ exhibits a tendency to preferentially interact with substances that have lower molecular weights (less than 250) or possess relatively weak polar characteristics (Ravantos *et al.*, 2002). These substances include lipids, cholesterol, aldehydes, ethers, esters, and ketones. Conversely, high molecular weight compounds (greater than 400) or those with strong polar features, such as hydroxyl, carboxyl, and various sugars, polysaccharides, amino acids, proteins, phosphatides, glycosides, as well as inorganic salts, tend to have limited solubility in dense carbon dioxide. The solubility of different compounds in supercritical phase of carbon dioxide is categorized (Table 1).

Table 1: Solubility of Different Compounds in Supercritical CO₂ (Ravantos *et al.*, 2002)

Highly Soluble	Moderately Soluble	Almost Insoluble
Organic compounds of low polarity and low molecular weight (< 250)	Polar organic compounds of molecular weight lower than 400	Highly polar organic compounds of molecular weight above 400
Highly volatile substances, used for aromas and flavouring in foods	Substances with low volatility	Non-volatile substances
Thiols, pyrazines, thiazoles, acetic acid, benzaldehyde, hexanol, glycerol and acetates	Water, terpenes, oleic acid, glycerol and saturated lipids with chains of up to 12 carbons	Proteins, sugars, polysaccharides, amino acids, inorganic salts, nitrates, waxes

IV. EQUIPMENT USED IN SUPERCRITICAL FLUID EXTRACTION AND ITS COMPONENTS

A standard setup for Supercritical Fluid Extraction (SFE) comprises various essential elements, safety measures, and recent advancements in equipment. The extraction vessel or high-pressure container serves as the receptacle for the supercritical fluid and the sample material (e.g., plant matter, biomass, or other substances). It is specifically engineered to endure high pressures and temperatures. The function of this pump is to compress and circulate the supercritical fluid, usually carbon dioxide, into the extraction vessel. It maintains the required pressure and temperature conditions crucial for supercritical extraction.

Pressure control system comprises pressure transducers and control valves that work together to sustain the desired pressure within the extraction vessel. Precise pressure control is imperative for achieving consistent and reproducible outcomes. A temperature control unit is responsible for regulating the temperature inside the extraction vessel. Its purpose is to ensure that the supercritical fluid remains in its supercritical state throughout the extraction process. In specific situations, a co-solvent like ethanol or methanol may be introduced into the supercritical fluid to enhance the extraction of particular compounds. This co-solvent is typically delivered via a separate system.

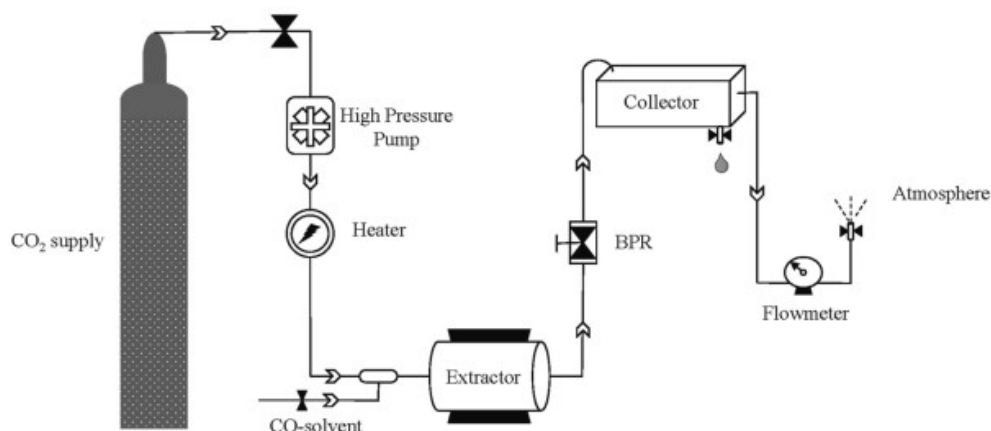


Figure 2: Flow Diagram of Supercritical Fluid Extraction System (Ahangari *et al.*, 2021)

Following the extraction process, the extracted compounds are separated from the supercritical fluid. This collection system usually incorporates a separator or fraction collector. SFE systems are equipped with safety mechanisms, including pressure relief valves, rupture discs, and pressure interlocks, designed to prevent overpressure occurrences and ensure the safety of operators. Depending on the application, analytical instruments such as gas chromatographs (GC) or mass spectrometers (MS) can be linked to the SFE setup for the real-time analysis of the extracted compounds.

V. FACTORS AFFECTING SUPERCRITICAL FLUID EXTRACTION

The crucial factors governing the Supercritical Fluid Extraction (SFE) of seed oils include temperature, pressure, the rate of CO₂ flow, seed particle size, the potential addition of co-solvents, and the duration of the extraction process.

Temperature regulation in supercritical fluid extraction (SFE) is achieved through a thermostatic bath or chamber, and in larger-scale operations, by employing concentric fluid heat exchange tubing. Maintaining precise temperature control is critical during the extraction of seed oils due to their sensitivity to temperature fluctuations. Temperature variations can impact both the solubility of seed oils and the prevention of oxidative reactions caused by elevated temperatures. Typical temperature ranges commonly applied for the extraction of various seed oils typically span from 40°C to 80°C (Ahmaad *et al.*, 2019). The solubility of many seed oil components in CO₂ also increases as temperature increases. Increasing the temperature decreases the CO₂ density, resulting in the decrease of the solubility of oil in SC-CO₂ up to the cross-over point of the seed oil yield versus extraction pressure as various isotherms, due to raise of the vapor pressure of the oil (Montanes *et al.*, 2018).

The pressure of the supercritical fluid also affects the efficiency of the extraction process. Higher pressures can increase the solubility of the oil in the supercritical fluid, but too high of pressure can cause the oil to degrade or change its chemical properties. General pressure in SFE ranges from 100-400 bar (Fornanri *et al.*, 2012).

Products parameters such as particle size and moisture content also play critical role in extraction. The size of the particles in the oil being extracted can affect the extraction efficiency. Smaller particles have a larger surface area and are more easily extracted using supercritical fluids. The moisture content of the oil can also affect the efficiency of the extraction process. Oils with high moisture content may require additional processing steps to remove the water before extraction.

Other than these parameters, the flow rate of the supercritical fluid can impact the extraction efficiency. High flow rates may result in lower extraction efficiency due to limited contact time between the fluid and the oil. Co-solvents, such as ethanol or methanol, can be added to the supercritical fluid to improve the solubility of the oil. The type and amount of co-solvent used can affect the extraction efficiency and the quality of the extracted oil. The duration of the extraction process can impact the extraction efficiency. Longer extraction times may result in higher extraction yields but can also result in the extraction of unwanted compounds, such as chlorophyll.

VI. APPLICATIONS OF SUPERCRITICAL FLUID EXTRACTION

Supercritical fluid extraction finds vast application in different industries i.e food industry, pharmaceutical industry, healthcare and nutraceutical industry, cosmetics and isolation and analytical systems and environmental analysis (Ahmad *et al.*, 2019). Supercritical Fluid Extraction (SFE) is a versatile method widely used in the food industry. It serves to extract natural flavours and fragrances from a variety of sources, including spices, herbs, and botanicals. SFE stands out as the preferred technique for decaffeinating coffee and tea, utilizing supercritical carbon dioxide to selectively remove caffeine from coffee beans or tea leaves while preserving other flavour components (Gandhi *et al.*, 2018). SFE holds significant value in the extraction of oils from seeds, nuts, and seafood, yielding top-quality edible oils like olive oil without resorting to chemical solvents. SFE is employed to extract bioactive compounds, encompassing antioxidants and polyphenols, from fruits, vegetables, and plant materials. These extracted compounds serve as functional ingredients in food products and dietary supplements.

The extraction of different seed oils by supercritical fluid extraction is shown in Table 2. To ensure the safety and quality of food items, SFE is utilized for the elimination of contaminants like pesticides, mycotoxins, and unwanted flavours. Additionally, SFE is employed to derive essential oils from aromatic plants, enhancing the flavour and aroma of food products. This method is also applied to extract lipids from food sources like fish oils and lipid-rich microorganisms, finding use in dietary supplements and fortifying food items. Furthermore, SFE is employed to extract nutraceutical components such as vitamins, phytosterols, and fatty acids from food sources, facilitating their incorporation into functional foods and dietary supplements.

VII. CONCLUSION

Supercritical fluid extraction offers many roles in different industries. Particularly for food industry, the techniques serve as a novel extractive technique which enhances yield with shorter extraction time and greater quality with control extraction of desired health benefitting compounds from extracted oils. The critical factors in SFE includes temperature,

pressure use of co solvent and particle size and moisture content through which we can selectively extract the desired quality of extracted seed oils and essential oils. It can also be coupled with other techniques like microwave, ultrasound and enzyme assisted extraction to enhance product quality. It is also widely accepted and environmentally sustainable method which offers promising solutions to the challenges faced by industries seeking efficient and sustainable methods for extracting valuable compounds from seeds and other raw materials.

Table 2: Review of Different Oils extracted through Supercritical Fluid Extraction

Sr. No.	Product	Material Condition	Process conditions				Maximum yield (%w/w) in SFE at Optimized Condition	Other Methods	Reference
			Flow Rate	Temperature	Pressure	Co-solvent			
1.	Almond (<i>Amygdalus scoparia</i>)	0.25-0.3 mm	0.6 g/min	40-80°C	20-40 MPa	Ethanol (5% and 10%)	42.35% (10% EtOH, for 40°C, 40 MPa Pressure and 0.6 g/min)	55.26% (PLE)	Balvardi <i>et al.</i> (2015)
	Almond	-	10-30 kg/h	35-50°C	350-550 bar	-	54.5% (powder), 29.8% (milled), 15.1% (broken), 5.5% (whole)	-	Leo <i>et al.</i> (2005)
	Peach almond (<i>Prunus persica</i>)	35% w/w 882 and 3360 µm	3.3 and 10 g/min	40°C	150 and 250 bar	-	19.08% (250 bar pressure, 880 µm particle size, 10 g/min flow rate after 420 min ET)	-	Mezzomo <i>et al.</i> (2009)
	Peach almond (<i>Prunus persica</i>)	26.1% d.b. 985 µm	8.3 g/min	30-50°C	100-300 bar	Ethanol (2% and 5%)	24% (50 °C and 300 bar, 5% EtOH)	24% (Maceration) 21% (Soxhlet)	Mezzomo <i>et al.</i> (2010)
	Chanar almond (<i>Geoffroea decorticans</i>)	0.82 mm	3.62 g/min	40 and 60°C	20-40 MPa	-	40% (60°C and 40 MPa)	49.6% (Soxhlet)	Salinas <i>et al.</i> (2020)
	Baru almond (<i>Dipteryx alata</i>)	<0.85 mm	3 ml/min	50-70°C	10-30 MPa	Water (0.3-10%) Ethanol (0.3-	31.06% (50°C, 30 MPa and 0.3% Ethanol)	4.61% (Cold Press)	Peixoto <i>et al.</i> (2022)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

						10%) Water+Ethanol (5%)			
2.	Beech (<i>Pongamia pinnata</i> L.)	0.5-1.00 mm	5-15 g/min	60-100°C	200-300 bar	Ethanol (5% and 10%)	36% (60 °C, 333 bar, 1.0 mm, 7 g/min and 9 % Ethanol in 250 min)	-	Suryavanshi and Mohanty (2018)
3.	Canola (<i>Brassica campestris</i>)	12.7-42.5%	2.5 g/min	35-75°C	20.7-62 MPa	-	7.1% (30%, 75°C and 62 MPa)	-	Dunford and Temelli (1997)
	Canola	1.64% 473.9 µm	70 g/min	40-60°C	300-500 bar	Ethanol (2.3% and 10%)	10.3% (40°C, 300 bar and 10% Ethanol in 240 min)	21.06% (Soxhlet)	Li <i>et al.</i> (2010)
	Canola	250 µm	70 g/min	50°C	400 bar	Ethanol (4.7%)	33.61%	45.78% (Soxhlet)	Khattab <i>et al.</i> (2009)
4.	Carrot	0.25-0.5 mm	0.5-2 L/min	40-70°C	27.6-55.1 MPa	Canola oil (2.5% and 5%)	2.69% (70°C, 55.1 MPa, 2 L/min and 5% Canola oil)	2.46% (Soxhlet)	Sun and Temelli (2006)
5.	Celery	210-490 µm	1.1- 3 kg/h	45-55°C	100-200 bar	-	21.9% (210 µm, 1.1 kg/h, 45°C and 150 bar)	-	Papamichail <i>et al.</i> (2000)
6.	Chia	800 µm	8 mL/min	70°C	28 MPa	Acetone (2,6 and 10%)	33.90% (8 ml/min, 70°C, 28 MPa and 10% Acetone after 5 hr)	33.90% (Soxhlet)	Dabrowski <i>et al.</i> (2018)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

	Chia (<i>Salvia hispanica</i> L.)	5.3% d.b.	4 mL/min	40-80°C	220-340 bar	-	30.7% (45°C, 335 bar and 100-400 µm using 24 s grinding time)	35.5% (Soxhlet)	Ishak <i>et al.</i> (2021)
	Chia	0.54 mm	1.8 g/min	40-80°C	136-408 bar	-	7.18% (80°C and 408 bar)	-	Uribe <i>et al.</i> (2011)
7.	Coconut	-	10 g/min	30-60°C	200-300 bar	-	28.84% (60°C, 300 Bar, 1 h)	20.75% (Cold Press) 30.23% (Soxhlet)	Aytac (2021)
8.	Coffee	4.0% d.b. 0.273 µm	1.5-2.2 g/min	40-60°C	20-50 MPa	Ethanol (5-10%) isopropanol(5-10%) Ethyl lactate (5-10%)	10.4% (40°C, 50 MPa and 10% Ethanol)	12% (Soxhlet)	Coelho <i>et al.</i> (2020)
9.	Cottonseed	0.25-0.6 mm	0.2 L/min	60-80°C	350-550 MPa	-	43.16% (80°C, 550 MPa)	40% (Soxhlet)	Bhattacharjee <i>et al.</i> (2007)
	Cottonseed	9.5% 0.25 mm		80°C	48.3 MPa	Ethanol (5%) isopropanol (5%)	33% (80°C, 48.3 MPa and 5% Ethanol)	-	Kuk and Hron (1994)
10.	Eucalyptus	12.3% 400 µm	2 L/min	40-80°C	10-50 MPa	-	4.78% (70°C, 40 MPa and 120 min)	3.8% (HD) 36.3% (Soxhlet)	Zhao <i>et al.</i> (2014)
11.	Fennel	18-100 mesh	0.033-0.43 g/s	30-57°C	100-300 bar	-	4.2% (35°C, 80 MPa and	-	Hatami <i>et al.</i> (2018)
12.	Flaxseed	6.2% 0.25-1 mm	1-3 L/min	50-70°C	35-55 MPa	-	28.1% (70°C, 55 MPa and 180 min)	38% (Soxhlet)	Bozan and Temelli (2002)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

	Flaxseed (<i>Linum usitatissimum</i> L.)	40 mesh	20 g/min	50°C	350 bar		32.16%	36.26% (Soxhlet) 35.73% (Solvent)	Chauhan <i>et al.</i> (2015)
	Flaxseed	3.4% <0.85 mm	2-4 g/min	50-70°C	30-50 MPa	-	26.7% (70°C, 50 MPa, 4 g/min and 22 min)	32% (Soxhlet)	Ozkal (2009)
13.	Gourd seed	0.36 mm	-	25-35°C	18-20 MPa	-	40% (40°C, 19 MPa and 130 min)	43.5% (Solvent)	Bernardo-Gil <i>et al.</i> (2009)
14.	Grapeseed	10-60 mesh	0.4 mL/min	35-40°C	30-40 MPa	Ethanol (10%)	6.2% (20-40 mesh, 40°C, 40 MPa and w/o 10% Ethanol)	10.6% (Solvent)	Cao and Ito (2003)
	Grapeseed	0.5 mm	6 g/min	50°C	50 MPa	-	13.9% (50°C, 50 MPa and 1.5 h)	16.1% (Soxhlet)	De Souza <i>et al.</i> (2020)
	Grapeseed	0.75 mm	0.17 g/s	40°C	160-200 bar	-	16.5% (40°C and 180 bar)	-	Passos <i>et al.</i> (2009)
15.	Jojoba	2.1% 0.21-2.36 mm	0.5-2 mL/min	70-90°C	200-600 bar	Hexane (0-10%)	52.2% (70°, 300 bar and 5% Hexane)	51.8% (Soxhlet)	Salgin <i>et al.</i> (2004)
16.	Linseed	1-4 mm	2 L/min	25°C	41.37-57.85 MPa	Ethanol (0-1 mL)	40.95% (57.85 MPa, 54 extraction time and 0.8 mL Ethanol)	41.28% (Soxhlet)	Ivanov <i>et al.</i> (2012)
17.	Mango	0.28 mm	6.7 g/min	40-60°C	25-35 MPa	Ethanol (5-15%)	6.25% (60°C, 30 MPa and 15% Ethanol)	-	Sanchez-camargo <i>et al.</i> (2019)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

18.	Proso Millet bran (<i>Panicum miliaceum</i> L.)	7-8% 290-1200 μm	2-10 ml/ml/h	40-60°C	300-500 bar	-	7% (60°C and 400 bar)	-	Devittori <i>et al.</i> (2000)
19.	Moringa	3.5-4.6%	2.2-2.8 mL/min	40-70°C	40-80 MPa	Ethanol (1 mL)	39.6% (57°C and 80 MPa)	-	Belo <i>et al.</i> (2019)
	Moringa	15.2% 250 μm	2 L/min	40-80°C	30-50 MPa	-	6.34% (60°C, 50 MPa and 120 min dynamic time)	9.27% (Soxhlet)	Zhao and Zhang (2013)
20.	Nutmeg	< 1 mm	1 mL/min	40-50°C	20.7- 41.4 MPa	-	33.7% (40°C, 41.4 MPa and < 1 mm sample size)	-	Al-Rawi <i>et al.</i> (2011)
	Nutmeg	0.556- 2.117 mm	2.78×10^{-5} m^3/s - 13.9 $\times 10^{-5}$ m^3/s	40-50°C	10-20 MPa	-	5% (50°C, 15 MPa, 8.33×10^{-5} m^3/s and 0.556 mm)	-	Machmudah <i>et al.</i> (2006)
21.	Oat bran	75-750 μm (<250 μm and >250 μm)	-	35-80°C	14-55 MPa	-	4% (35°C, 55 MPa, 5 min static time, 5 min dynamic time and <250 μm)	7.4% (Soxhlet)	Fernández- Acosta <i>et al.</i> (2019)
22.	Okra	0.519 mm	6 kg/h	40-60°C	150-450 bar	-	15.9% (50°C and 450 bar)	16.3% (Solvent)	Andras <i>et al.</i> (2005)
23.	Olive	28%	50 g/min	40-60°C	200-300 bar	-	29.01% (40°C, 250 bar and 180 min ET)	33% (Soxhlet)	Belbaki <i>et al.</i> (2017)
24.	Palm	1-2 mm	-	51°C	19.8 MPa	Ethanol (0-100 mL)	3.67 % (51°C, 19.8 MPa and 100 ml Ethanol)	-	Krishnaih <i>et al.</i> (2012)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

25.	Peach	250-350 μm	0.69-2.37 g/min	40-51°C	15-19.8 MPa	Ethanol (2.5-5%)	32% (51°C, 19.8 MPa and 5% Ethanol)	-	Sanchez-Vicente <i>et al.</i> (2009)
26.	Peanut	425 μm	3 mL/min	40-70°C	10-30 MPa	Ethanol (5%)	15.47% (70°C, 30 MPa and 5% Ethanol)	36.28%	Putra <i>et al.</i> (2018)
27.	Pennyroyal	9% d.b. 0.3-0.7 mm	1.6 kg/h	40-50°C	90-100 bar	-	2.43% (50°C, 100 bar and 0.3 mm)		Reis-Vasco <i>et al.</i> (1999)
28.	Pepper	60 mesh	0.14-0.77 g/min	30-50°C	150-300 bar	-	2.05% (50°C, 300 bar and 0.77 g/min)		Ferreira <i>et al.</i> (1999)
	Pepper	175 μm	1.1-3.0 kg/h	40°C	90-150 bar	-	13% (40°C, 100 bar and 3 kg/h)		Perakis <i>et al.</i> (2005)
29.	Pomegranate	0.3-0.9 mm	6.6-23.4 L/h	33.2-66.8°C	13.2-46.8 MPa	-	14.02% (60°C, 40 MPa and 20 L/h)		Liu <i>et al.</i> (2009)
	Pomegranate	40 mesh	-	40-60°C	200-350 MPa	Water, Hexane and Ethanol (9-18 mL/100g)	3.39% (40°C, 275 atm and 18 Ethanol mL/100g)		Abbasi <i>et al.</i> (2008)
30.	Quinoa	1.0-1.4 mm	10.5-27 L/h	30-130°C	12.5-28.5 MPa	-	45.63% (75°C, 28.5 MPa and 55 min ET)		Przygoda and Wejnerowska (2015)
	Quinoa	0.05-0.80 mm	10.5-27 L/h	40-80°C	18-30 MPa	Methanol and Ethanol (1.3-20%)	6.3% (40°C, 25 MPa, 80 min and 20% Ethanol: Methanol 1:1 w/w)		Wejnerowska and Ciaciuch (2018)

SUPERCRITICAL FLUID EXTRACTION OF OILS FROM DIFFERENT BIOLOGICAL MATERIALS

31.	Raspberry	3.25-5.13% 200-400 μm	0.2-0.4 kg/h	40-60°C	250-350 bar	-	18.29% (50°C, 350 bar, 0.4 kg/h and Willamette var.)		Maric <i>et al.</i> (2020)
32.	Rice bran	0.5% 202 μm	10 g/min	30-50°C	10-30 MPa	-	39% (30°C and 30 MPa)		Jesus <i>et al.</i> (2010)
33.	Rosehip	0.556-2.112 mm	2-4 mL/min	40-80°C	150-450 bar	-	15.76% (80°C, 300 bar and 3 mL/min)		Machmudah <i>et al.</i> (2007)
34.	Sage	0.3-0.8 mm	0.72-1.32 kg/h	40-50°C	90-100 bar	-	1.87% (40°C, 90 bar, 0.8 mm and 1.32 kg/h)		Langa <i>et al.</i> (2009)
35.	Sesame	2.1% 0.72 mm	3 cm^3/min	40-60°C	19-25 MPa	-	35% (40°C, 25 MPa and 510 min ET)		Corso <i>et al.</i> (2010)
36.	Soybean	-	0.8-1.8 L/min	40-60°C	200-300 bar	-	19.5% (40°C and 300 bar)		Dobarganes <i>et al.</i> (2002)
37.	Sunflower	0.23-2.18 mm	1-6 cm^3/min	40-80°C	20-60 MPa	-	88.8% (0.23 mm, 40°C, 40 MPa and 4 cm^3/min)		Salgin <i>et al.</i> (2006)
38.	Tea seed	7%	1 mL/min	60-80°C	300-400 bar	Ethanol (7.5-15%)	31.6% (60°C, 350 bar and 15% Ethanol)		Rajaei <i>et al.</i> (2005)
39.	Thyme	0.84-6.00 mm	2 mL/min	40-60°C	80-120 bar	-	0.70% (40°C, 100 bar and 90 min)	1.47% (SD)	Sonsuzer <i>et al.</i> (2004)
40.	Turmeric	6.93% 0.2-0.73 mm	5-15 g/min	40-60°C	20-40 MPa	Ethanol (0-15%)	5.5% (53.72°C, 25.54 MPa, 13.6 g/min, 0.54 mm and 14.67% Ethanol)	5.96% (Soxhlet)	Priyanka and Khanam (2018)

REFERENCES

- [1] Ahmad, T., Masoodi, F.A., Rather, S.A., Wani, S.M. and Gull, A., 2019. Supercritical fluid extraction: A review. *J. Biol. Chem. Chron*, 5(1), pp.114-122.
- [2] Balvardi, M., Mendiola, J.A., Castro-Gómez, P., Fontecha, J., Rezaei, K. and Ibáñez, E., 2015. Development of pressurized extraction processes for oil recovery from wild almond (*Amygdalus scoparia*). *Journal of the American Oil Chemists' Society*, 92, pp.1503-1511.
- [3] Dunford, N.T. and Temelli, F., 1997. Extraction conditions and moisture content of canola flakes as related to lipid composition of supercritical CO₂ extracts. *Journal of food science*, 62(1), pp.155-159.
- [4] Gandhi, K., Arora, S. and Kumar, A., 2017. Industrial applications of supercritical fluid extraction: a review. *Int. J. Chem. Stud*, 5(3), pp.336-340.
- [5] Leo, L., Rescio, L., Ciurlia, L. and Zacheo, G., 2005. Supercritical carbon dioxide extraction of oil and α -tocopherol from almond seeds. *Journal of the Science of Food and Agriculture*, 85(13), pp.2167-2174.
- [6] Mezzomo, N., Martínez, J. and Ferreira, S.R., 2009. Supercritical fluid extraction of peach (*Prunus persica*) almond oil: kinetics, mathematical modeling and scale-up. *The Journal of Supercritical Fluids*, 51(1), pp.10-16.
- [7] Mezzomo, N., Mileo, B.R., Friedrich, M.T., Martínez, J. and Ferreira, S.R., 2010. Supercritical fluid extraction of peach (*Prunus persica*) almond oil: process yield and extract composition. *Bioresource Technology*, 101(14), pp.5622-5632.
- [8] Peixoto, V.O.D.S., de Oliveira Silva, L., Castelo-Branco, V.N. and Torres, A.G., 2022. Baru (*Dipteryx alata* Vogel) Oil Extraction by Supercritical-CO₂: Improved Composition by Using Water as Cosolvent. *Journal of Oleo Science*, 71(2), pp.201-213.
- [9] Raventós, M., Duarte, S. and Alarcón, R., 2002. Application and possibilities of supercritical CO₂ extraction in food processing industry: an overview. *Food Science and Technology International*, 8(5), pp.269-284.
- [10] Salinas, F., Vardanega, R., Espinosa-Álvarez, C., Jimenez, D., Munoz, W.B., Ruiz-Domínguez, M.C., Meireles, M.A.A. and Cerezal-Mezquita, P., 2020. Supercritical fluid extraction of chañar (*Geoffroea decorticans*) almond oil: Global yield, kinetics and oil characterization. *The Journal of Supercritical Fluids*, 161, p.104824.
- [11] Suryawanshi, B. and Mohanty, B., 2018. Modeling and optimization: Supercritical CO₂ extraction of *Pongamia pinnata* (L.) seed oil. *Journal of Environmental Chemical Engineering*, 6(2), pp.2660-2673.
- [12] Dunford, N.T. and Temelli, F., 1997. Extraction conditions and moisture content of canola flakes as related to lipid composition of supercritical CO₂ extracts. *Journal of food science*, 62(1), pp.155-159.
- [13] Li, H., Wu, J., Rempel, C.B. and Thiyam, U., 2010. Effect of operating parameters on oil and phenolic extraction using supercritical CO₂. *Journal of the American Oil Chemists' Society*, 87(9), pp.1081-1089.
- [14] Khattab, R.Y. and Zeitoun, M.A., 2013. Quality evaluation of flaxseed oil obtained by different extraction techniques. *LWT-Food Science and Technology*, 53(1), pp.338-345.
- [15] Sun, M. and Temelli, F., 2006. Supercritical carbon dioxide extraction of carotenoids from carrot using canola oil as a continuous co-solvent. *The Journal of supercritical fluids*, 37(3), pp.397-408.
- [16] Papamichail, I., Louli, V. and Magoulas, K., 2000. Supercritical fluid extraction of celery seed oil. *The Journal of Supercritical Fluids*, 18(3), pp.213-226.
- [17] Dąbrowski, G., Konopka, I. and Czaplicki, S., 2018. Supercritical CO₂ extraction in chia oils production: impact of process duration and co-solvent addition. *Food science and biotechnology*, 27, pp.677-686.
- [18] Ishak, I., Hussain, N., Coorey, R. and Abd Ghani, M., 2021. Optimization and characterization of chia seed (*Salvia hispanica* L.) oil extraction using supercritical carbon dioxide. *Journal of CO₂ Utilization*, 45, p.101430.
- [19] Uribe, J.A.R., Perez, J.I.N., Kauil, H.C., Rubio, G.R. and Alcocer, C.G., 2011. Extraction of oil from chia seeds with supercritical CO₂. *The Journal of Supercritical Fluids*, 56(2), pp.174-178.
- [20] Aytac, E., 2022. Comparison of extraction methods of virgin coconut oil: cold press, soxhlet and supercritical fluid extraction. *Separation Science and Technology*, 57(3), pp.426-432.
- [21] Coelho, J.P., Filipe, R.M., Robalo, M.P., Boyadzhieva, S., Cholakov, G.S. and Stateva, R.P., 2020. Supercritical CO₂ extraction of spent coffee grounds. Influence of co-solvents and characterization of the extracts. *The Journal of Supercritical Fluids*, 161, p.104825.
- [22] Bhattacharjee, P., Singhal, R.S. and Tiwari, S.R., 2007. Supercritical carbon dioxide extraction of cottonseed oil. *Journal of Food Engineering*, 79(3), pp.892-898.
- [23] Kuk, M.S. and Hron, R.J., 1994. Supercritical carbon dioxide extraction of cottonseed with co-solvents. *Journal of the American Oil Chemists' Society*, 71, pp.1353-1356.

- [24] Zhao, S. and Zhang, D., 2014. Supercritical CO₂ extraction of Eucalyptus leaves oil and comparison with Soxhlet extraction and hydro-distillation methods. *Separation and Purification Technology*, 133, pp.443-451.
- [25] Hatami, T., Johner, J.C.F. and Meireles, M.A.A., 2018. Extraction and fractionation of fennel using supercritical fluid extraction assisted by cold pressing. *Industrial crops and products*, 123, pp.661-666.
- [26] Bozan, B. and Temelli, F., 2002. Supercritical CO₂ extraction of flaxseed. *Journal of the American Oil Chemists' Society*, 79(3), pp.231-235.
- [27] Chauhan, R., Chester, K., Khan, Y., Tamboli, E.T. and Ahmad, S., 2015. Characterization of *Linum usitatissimum* L. oil obtained from different extraction technique and in vitro antioxidant potential of supercritical fluid extract. *Journal of pharmacy & bioallied sciences*, 7(4), p.284.
- [28] Ozkal, S.G., 2009. Response surface analysis and modeling of flaxseed oil yield in supercritical carbon dioxide. *Journal of the American Oil Chemists' Society*, 86, pp.1129-1135.
- [29] Bernardo-Gil, M.G., Casquilho, M., Esquivel, M.M. and Ribeiro, M.A., 2009. Supercritical fluid extraction of fig leaf gourd seeds oil: Fatty acids composition and extraction kinetics. *The Journal of Supercritical Fluids*, 49(1), pp.32-36.
- [30] Cao, X. and Ito, Y., 2003. Supercritical fluid extraction of grape seed oil and subsequent separation of free fatty acids by high-speed counter-current chromatography. *Journal of Chromatography A*, 1021(1-2), pp.117-124.
- [31] De Souza, R.D.C, Machado, B.A.S., Barreto, G.D.A., Leal, I.L., Anjos, J.P.D. and Umsza-Guez, M.A., 2020. Effect of experimental parameters on the extraction of grape seed oil obtained by low pressure and supercritical fluid extraction. *Molecules*, 25(7), p.1634.
- [32] Passos, C.P., Silva, R.M., Da Silva, F.A., Coimbra, M.A. and Silva, C.M., 2010. Supercritical fluid extraction of grape seed (*Vitis vinifera* L.) oil. Effect of the operating conditions upon oil composition and antioxidant capacity. *Chemical Engineering Journal*, 160(2), pp.634-640.
- [33] Salgin, U., Çalimli, A. and Zühtü Uysal, B., 2004. Supercritical fluid extraction of jojoba oil. *Journal of the American Oil Chemists' Society*, 81, pp.293-296.
- [34] Ivanov, D.S., Čolović, R.R., Lević, J.D. and Sredanović, S.A., 2012. Optimization of supercritical fluid extraction of linseed oil using RSM. *European Journal of Lipid Science and Technology*, 114(7), pp.807-815.
- [35] Sanchez-Camargo, A.D.P., Gutierrez, L.F., Vargas, S.M., Martinez-Correa, H.A., Parada-Alfonso, F. and Narvaez-Cuenca, C.E., 2019. Valorisation of mango peel: Proximate composition, supercritical fluid extraction of carotenoids, and application as an antioxidant additive for an edible oil. *The Journal of Supercritical Fluids*, 152, p.104574.
- [36] Devittori, C., Gumy, D., Kusy, A., Colarow, L., Bertoli, C. and Lambelet, P., 2000. Supercritical fluid extraction of oil from millet bran. *Journal of the American Oil Chemists' Society*, 77, pp.573-579.
- [37] Belo, Y.N., Al-Hamimi, S., Chimuka, L. and Turner, C., 2019. Ultrahigh-pressure supercritical fluid extraction and chromatography of *Moringa oleifera* and *Moringa peregrina* seed lipids. *Analytical and bioanalytical chemistry*, 411, pp.3685-3693.
- [38] Zhao, S. and Zhang, D., 2014. Supercritical CO₂ extraction of Eucalyptus leaves oil and comparison with Soxhlet extraction and hydro-distillation methods. *Separation and Purification Technology*, 133, pp.443-451.
- [39] Al-Rawi, S.S., Ibrahim, A.H., Majid, A.S.A., Majid, A.M.A. and Ab Kadir, M.O., 2013. Comparison of yields and quality of nutmeg butter obtained by extraction of nutmeg rind by soxhlet and supercritical carbon dioxide (SC-CO₂). *Journal of food engineering*, 119(3), pp.595-601.
- [40] Machmudah, S., Sulaswatty, A., Sasaki, M., Goto, M. and Hirose, T., 2006. Supercritical CO₂ extraction of nutmeg oil: Experiments and modeling. *The Journal of supercritical fluids*, 39(1), pp.30-39.
- [41] Fernández-Acosta, K., Salmeron, I., Chavez-Flores, D., Perez-Reyes, I., Ramos, V., Ngadi, M., Kwofie, E.M. and Perez-Vega, S., 2019. Evaluation of different variables on the supercritical CO₂ extraction of oat (*Avena sativa* L.) oil; main fatty acids, polyphenols, and antioxidant content. *Journal of cereal science*, 88, pp.118-124.
- [42] Andras, C.D., Simandi, B., Örsi, F., Lambrou, C., Missopolinou-Tatala, D., Panayiotou, C., Domokos, J. and Doleschall, F., 2005. Supercritical carbon dioxide extraction of okra (*Hibiscus esculentus* L) seeds. *Journal of the Science of Food and Agriculture*, 85(8), pp.1415-1419.
- [43] Belbaki, A., Louaer, W. and Meniai, A.H., 2017. Supercritical CO₂ extraction of oil from Crushed Algerian olives. *The Journal of Supercritical Fluids*, 130, pp.165-171.
- [44] Krishnaiah, D., Bono, A., Sarbatly, R. and Fadhilah, S., 2012. Supercritical Fluid Extraction of Palm Kernel Oil from Palm Kernel. *American Journal of Food Technology*, 7(3), pp.168-172.

- [45] Sánchez-Vicente, Y., Cabañas, A., Renuncio, J.A. and Pando, C., 2009. Supercritical fluid extraction of peach (*Prunus persica*) seed oil using carbon dioxide and ethanol. *The Journal of Supercritical Fluids*, 49(2), pp.167-173.
- [46] Putra, N.R., Rizkiyah, D.N., Zaini, A.S., Yunus, M.A.C., Machmudah, S., Idham, Z.B. and Hazwan Ruslan, M.S., 2018. Effect of particle size on yield extract and antioxidant activity of peanut skin using modified supercritical carbon dioxide and soxhlet extraction. *Journal of Food Processing and Preservation*, 42(8), p.e13689.
- [47] Reis-Vasco, E.M.C., Coelho, J.A.P. and Palavra, A.M.F., 1999. Comparison of pennyroyal oils obtained by supercritical CO₂ extraction and hydrodistillation. *Flavour and Fragrance Journal*, 14(3), pp.156-160.
- [48] Ferreira, S.R., Nikolov, Z.L., Doraiswamy, L.K., Meireles, M.A.A. and Petenate, A.J., 1999. Supercritical fluid extraction of black pepper (*Piper nigrum* L.) essential oil. *The Journal of Supercritical Fluids*, 14(3), pp.235-245.
- [49] Perakis, C., Louli, V. and Magoulas, K., 2005. Supercritical fluid extraction of black pepper oil. *Journal of Food Engineering*, 71(4), pp.386-393.
- [50] Liu, G., Xu, X., Hao, Q. and Gao, Y., 2009. Supercritical CO₂ extraction optimization of pomegranate (*Punica granatum* L.) seed oil using response surface methodology. *LWT-Food Science and Technology*, 42(9), pp.1491-1495.
- [51] Abbasi, H., Rezaei, K. and Rashidi, L., 2008. Extraction of essential oils from the seeds of pomegranate using organic solvents and supercritical CO₂. *Journal of the American Oil Chemists' Society*, 85(1), pp.83-89.
- [52] Przygoda, K. and Wejnerowska, G., 2015. Extraction of tocopherol-enriched oils from Quinoa seeds by supercritical fluid extraction. *Industrial Crops and Products*, 63, pp.41-47.
- [53] Wejnerowska, G. and Ciaciuch, A., 2018. Optimisation of oil extraction from quinoa seeds with supercritical carbon dioxide with co-solvents. *Czech Journal of Food Sciences*, 36(1), pp.81-87.
- [54] Marić, B., Pavlič, B., Čolović, D., Abramović, B., Zeković, Z., Bodroža-Solarov, M., Ilić, N. and Teslić, N., 2020. Recovery of high-content ω -3 fatty acid oil from raspberry (*Rubus idaeus* L.) seeds: Chemical composition and functional quality. *LWT*, 130, p.109627.
- [55] Jesus, S.P., Grimaldi, R. and Hense, H., 2010. Recovery of γ -oryzanol from rice bran oil byproduct using supercritical fluid extraction. *The journal of supercritical fluids*, 55(1), pp.149-155.
- [56] Machmudah, S., Kawahito, Y., Sasaki, M. and Goto, M., 2007. Supercritical CO₂ extraction of rosehip seed oil: Fatty acids composition and process optimization. *The Journal of supercritical fluids*, 41(3), pp.421-428.
- [57] Langa, E., Della Porta, G., Palavra, A.M.F., Urieta, J.S. and Mainar, A.M., 2009. Supercritical fluid extraction of Spanish sage essential oil: Optimization of the process parameters and modelling. *The Journal of Supercritical Fluids*, 49(2), pp.174-181.
- [58] Corso, M.P., Fagundes-Klen, M.R., Silva, E.A., Cardozo Filho, L., Santos, J.N., Freitas, L.S. and Dariva, C., 2010. Extraction of sesame seed (*Sesamum indicum* L.) oil using compressed propane and supercritical carbon dioxide. *The Journal of Supercritical Fluids*, 52(1), pp.56-61.
- [59] Dobarganes Nodar, M., Molero Gómez, A. and Martínez De La Ossa, E., 2002. Characterisation and process development of supercritical fluid extraction of soybean oil. *Food science and technology international*, 8(6), pp.337-342.
- [60] Salgın, U., Döker, O. and Çalimli, A., 2006. Extraction of sunflower oil with supercritical CO₂: Experiments and modeling. *The Journal of Supercritical Fluids*, 38(3), pp.326-331.
- [61] Rajaei, A., Barzegar, M. and Yamini, Y., 2005. Supercritical fluid extraction of tea seed oil and its comparison with solvent extraction. *European Food Research and Technology*, 220, pp.401-405.
- [62] Sonsuzer, S., Sahin, S.E.R.P.İ.L. and Yilmaz, L.E.V.E.N.T., 2004. Optimization of supercritical CO₂ extraction of *Thymbra spicata* oil. *The Journal of supercritical fluids*, 30(2), pp.189-199.
- [63] Priyanka and Khanam, S., 2018. Influence of operating parameters on supercritical fluid extraction of essential oil from turmeric root. *Journal of Cleaner Production*, 188, pp.816-824.