

Review Article

A review of the industrial applications and scale-up of the supercritical CO₂ extraction process

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Abstract

This review examined the importance of supercritical CO₂ extraction and its industrial applications and scale-up. The need to have a sustainable development in order to tackle environmental concerns has led to the discovery of more efficient and safer alternatives to organic solvents. When a substance is pushed beyond its critical temperature and pressure, the distinction between the liquid and vapor phases vanishes, resulting in a homogenous fluid that possesses tunable physicochemical properties. Among the various candidates for supercritical applications, carbon dioxide (CO₂) stands out as the premier choice. Supercritical carbon dioxide (scCO₂) signifies the future fluid for next-generation power supply systems, operating above its critical point (31.1 °C, 73.0 bar) with its gas-like mass transfer and liquid-like density. The green-solvent properties of supercritical (scCO₂) are highly dependent on its density, pressure and temperature. Its relatively mild critical parameters (31.1 °C and 73.0 bar), combined with its non-toxicity, non-flammability, and low cost, have made the application of supercritical carbon dioxide (scCO₂) relevant in free radical polymerization. This unique behavior has revolutionized industries that previously relied on toxic organic solvents and energy-intensive thermal processes.

Keywords: Supercritical CO₂ (scCO₂), Mass transfer, Critical point temperature, Organic solvents

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INTRODUCTION

The need to have a sustainable development in order to tackle environmental concerns has led to the discovery of more efficient and safer alternatives to organic solvents [1]. Although organic solvents are effective, there are health

risks associated with them, such as volatility, toxicity and ecosystem persistence [2]. Supercritical CO₂ (ScCO₂) is known to possess some unique characteristics such as low toxicity, abundance, inertness, recyclability, exceptional diffusivity, viscosity and solvation characteristics [3]. The schematic diagram in Figure 1 illustrates the supercritical CO₂ as a reaction medium. The

application of scCO_2 in catalytic system is due to its solvent and co-reactant inert properties [4]. ScCO_2 has a critical temperature of 31.1°C and pressure of 73.8 bar.

There are three main classification phases of matter, which include: solid, liquid and gas. When a substance is pushed beyond its critical temperature and pressure, the distinction between the liquid and vapor phases vanishes, resulting in a homogenous fluid that possesses tunable physicochemical properties. Among the various candidates for supercritical applications, carbon dioxide stands out as the premier choice. The critical point of scCO_2 (31.1°C , 73 bar) makes it a suitable candidate for the next-generation power supply systems. Any small changes in temperature and pressure affect the specific enthalpy, viscosity and density of scCO_2 . The solvent power of supercritical carbon dioxide (scCO_2) is highly dependent on its density, pressure and temperature [1]. ScCO_2 relatively mild critical parameters (31.1°C , and 73 bar), combined with its non-toxicity, non-flammability, and low cost, have made the application of supercritical carbon dioxide (scCO_2) useful in free radical polymerization [2]. The use of toxic organic solvents and energy-intensive thermal processes has critically reduced due to the evolution of scCO_2 [3]. While the theoretical foundations of supercritical fluids were laid decades ago, the green solvent technology has matured significantly since the 1980s. Early commercial success in decaffeinating coffee and extracting hops has been replaced with nanotechnological applications of scCO_2 [4]. This review examined the diverse applications of scCO_2 across four key sectors, which include: energy production, industrial extraction, life

sciences, and materials engineering. In extraction processes, scCO_2 has high precision and selectivity. The green solvent property of scCO_2 can be modulated via pressure and temperature, which is useful in the isolation of both polar and moderately polar bioactives from complex matrices [5]. The usefulness of scCO_2 in the pharmaceutical industry has been documented in the area of facilitation of solvent-free purification and processing of active pharmaceutical ingredients (APIs). ScCO_2 also played a major role in the production of microencapsulation of fine particles and drug formulation without the thermal degradation associated with conventional approaches [6]. In addition to the applications of scCO_2 in pharmaceutical settings, the green solvent (scCO_2) is used to create uniform and therapeutic performance [6]. Figure 2 illustrates the diverse industrial roles of supercritical CO_2 in pharmaceutical, environmental, chemical and polymer sectors.

ScCO_2 has been shown to enhance the flexibility and porosity of formulations when used in combination with some polymers such as polylactic acid, polycaprolactone and polyethylene terephthalate (PET) [7]. There has been a growing interest in the use of supercritical CO_2 as a green solvent in the 18th to 21st century [8]. ScCO_2 possesses environmentally friendly characteristics and industrial applications. Applications of scCO_2 in the areas of pharmaceuticals, polymers and food processing have been documented [9]. Some of the physicochemical properties associated with ScCO_2 consist of low viscosity, high diffusivity, solvating power, non-toxicity, non-flammability and chemical inertness [10].

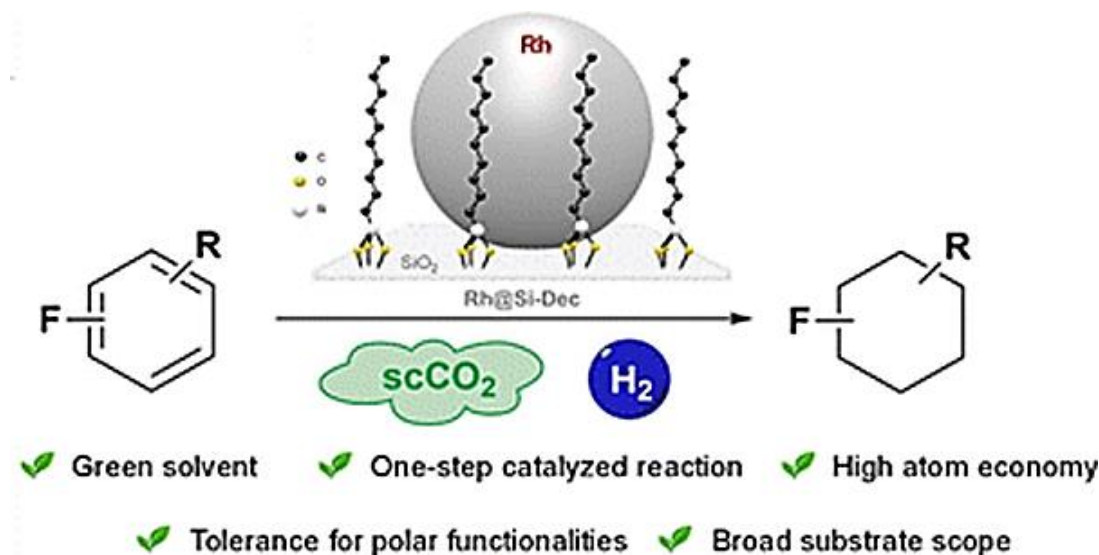


Figure 1: Schematic illustration of the reaction medium of supercritical CO_2 . Adapted from Aggarwal et al [4]

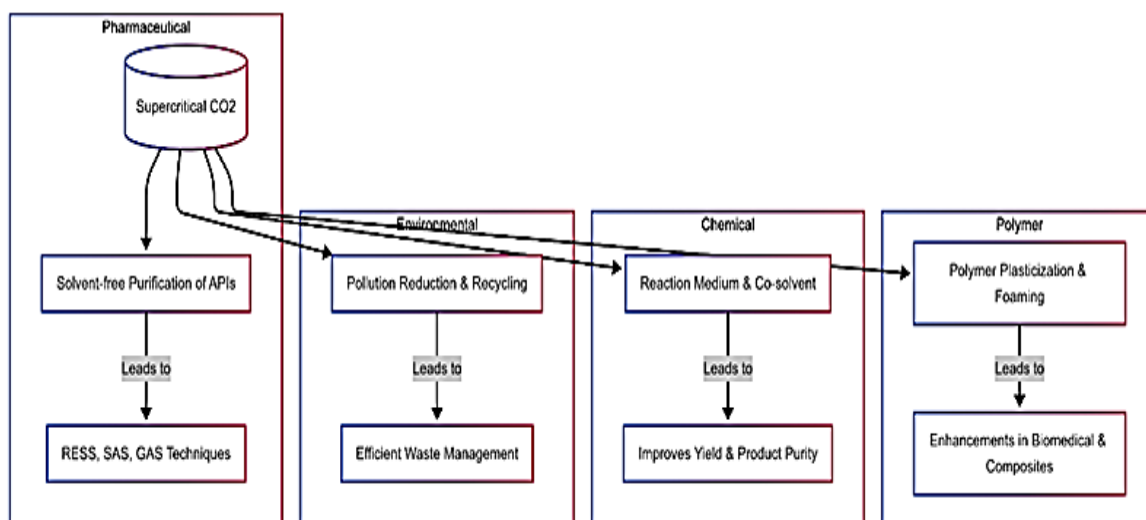


Figure 2: Industrial roles of supercritical CO₂ (scCO₂), adapted from Al-Hamimi & Turner [6]

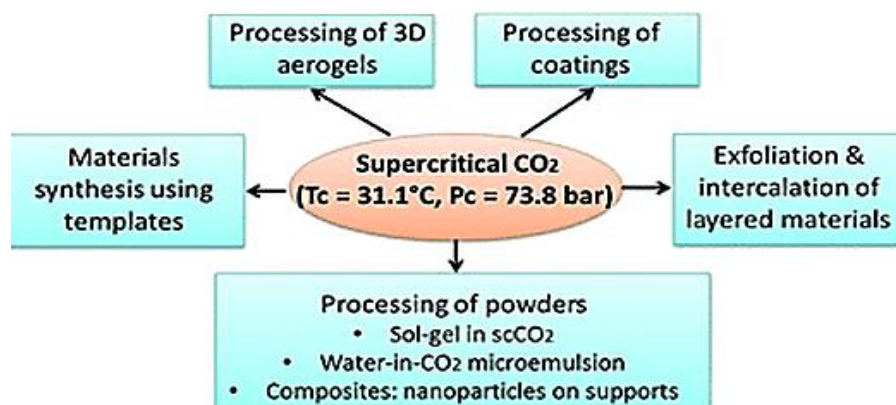


Figure 3: Applications of supercritical CO₂ in material synthesis and processing. Adapted from Arumugham *et al* [10]

In the extraction of natural products, ScCO₂ exhibited superior selectivity and efficiency, especially for thermolabile compounds in food and nutraceutical sectors. Application areas that have been documented consist of decaffeination of coffee, essential oil extraction, carotenoids, omega-3-fatty acids and polyphenols [11]. In the pharmaceutical industry, ScCO₂ is employed in drug formulation, purification and particle engineering due to the ability of ScCO₂ to function as both a solvent and anti-solvent [12]. The incorporation of ScCO₂ in drug delivery systems has supported sustained-release mechanisms and encapsulation of volatile actives in biodegradable polymers [13].

Physicochemical properties of supercritical CO₂

The transition of CO₂ into a supercritical state is triggered when there is an increase in critical temperature (31.1 °C) and pressure (73.8 bar). This results in the formation of a fluid that possesses both gas-like and liquid-like

characteristics [14]. Under supercritical conditions, the density of ScCO₂ is between 200 and 900 kg/m³ [15]. The superior performance of ScCO₂ when compared to conventional liquids in terms of viscosity and diffusivity has been recorded [15]. It possesses a low viscosity within the range of 0.02 to 0.08 mPa·s and high diffusivity of about 10⁻⁸ - 8 m²/s. Melo *et al* [15] investigated that polar compounds can be solubilized under optimized pressure and temperature conditions.

Comparisons between ScCO₂ and conventional organic solvents tend to highlight some unique advantages in terms of environmental operations and safety. Unlike the conventional solvents such as ethanol, hexane, acetone and methylene chloride, which pose great risks in terms of flammability, toxicity and solvent residue, ScCO₂ is non-flammable and also does not leave residual solvent traces. In product extraction, ScCO₂ consumes less mass and also achieves a superior yield [16].

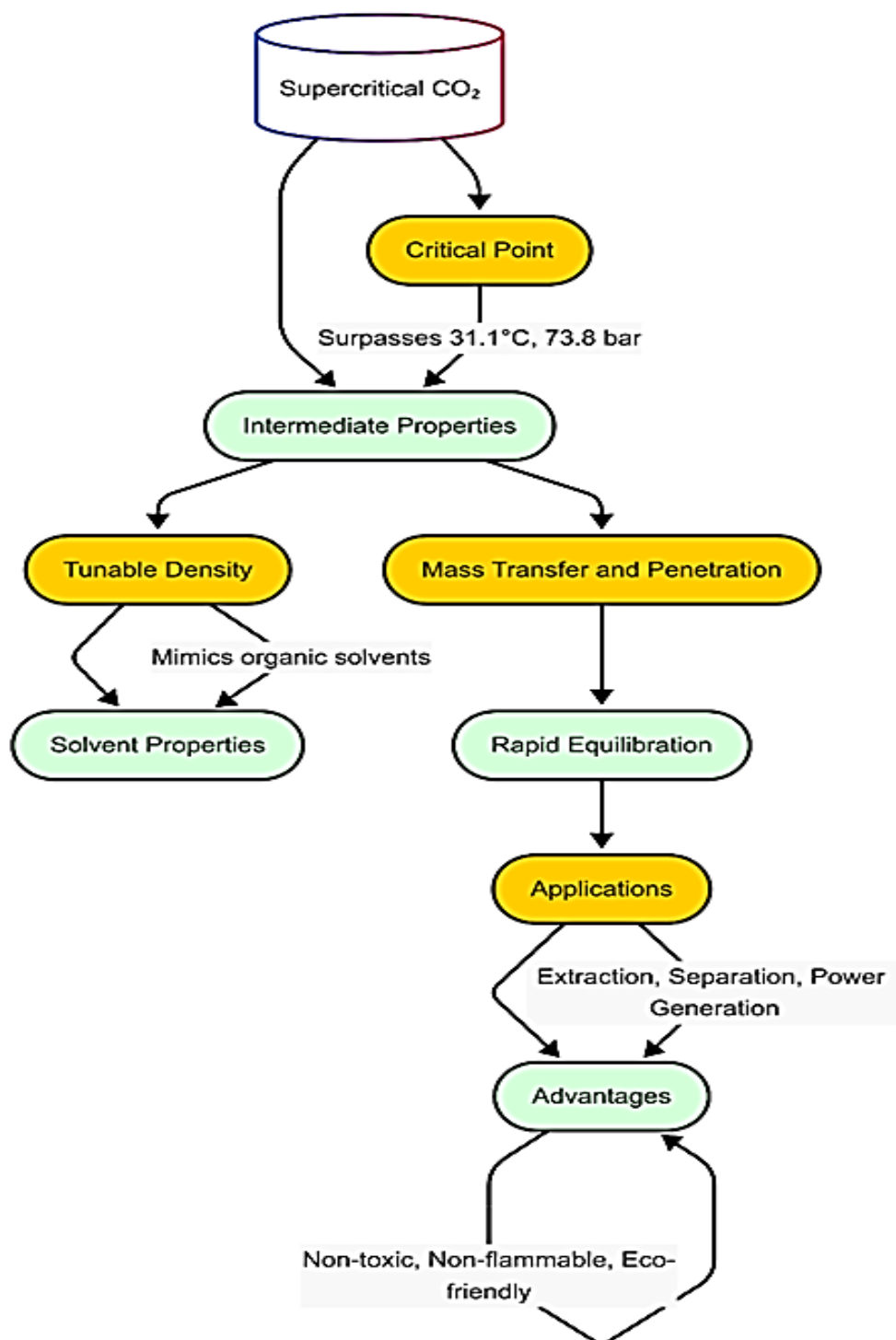


Figure 4: Overview of supercritical CO₂ properties and applications (illustrative diagram based on literature sources)

The role of supercritical CO₂ in extraction of bioactive compounds

Due to the physicochemical characteristics and environmental benefits, scCO₂ is used in the extraction of natural products and bioactive compounds across different industrial sectors

such as food, cosmetics, pharmaceuticals and nutraceuticals [17]. Figure 5 represents a schematic illustration of the supercritical fluid extraction (SFE) process utilizing supercritical CO₂ with ethanol, which serves as a cosolvent in the bioactive compound extraction.

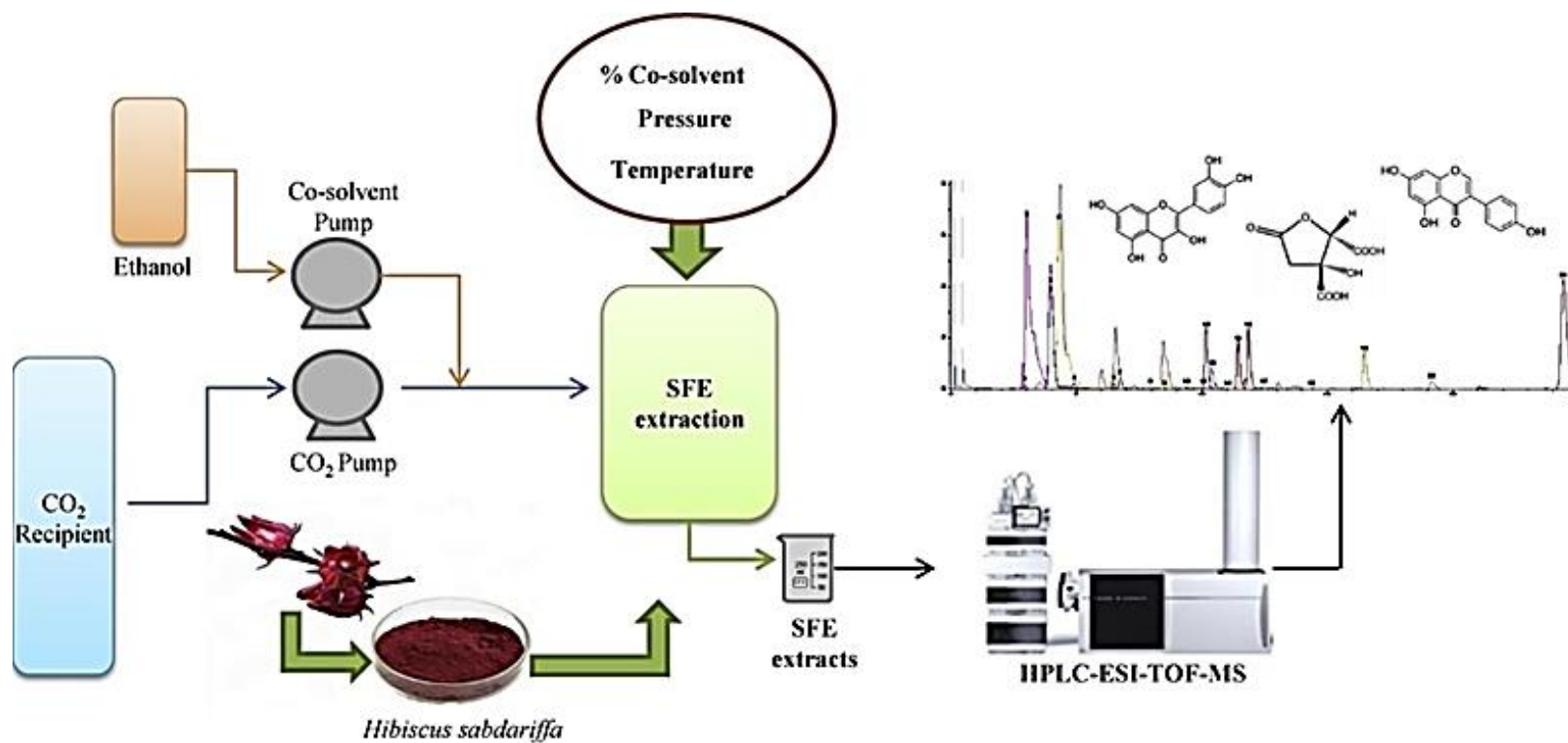


Figure 5: Supercritical fluid extraction process for natural product extraction using ScCO₂ and co-solvents. Adapted from Bezerra *et al* [18]

Applications of supercritical CO₂ (scCO₂)

Energy and power generation

Redefining efficiency: The supercritical CO₂ power cycle

The quest for higher thermal efficiency has led the power industry to shift away from steam and move toward scCO₂ [19]. ScCO₂ systems possess higher pressure than gas turbine cycles. These high pressures and densities associated with scCO₂ enable them to deliver a high energy conversion efficiency [20]. The ScCO₂ Brayton cycle operates at about 25 MPa, while the steam cycles that involve phase changes operate around 16 to 25 MPa. This enables the scCO₂ to be placed at a higher level than the advanced steam setup. Turbomachinery for scCO₂ is a fraction of the size of steam equivalent, leading to smaller plant footprints and lower capital costs. Gas turbine (Brayton) cycles using air work at much lower pressures, typically around 1 to 2 MPa [21].

Recuperation is central to this design, which involves the use of waste heat from the turbine to preheat the fluid before it enters the main heater. This enables the cycle to minimize fuel consumption. The effectiveness of the recompression variant of this cycle helps to reduce heat loss and also maintain the properties of the fluid [22].

Next-generation nuclear reactors

The advanced nuclear designs, such as the Integral Fast Reactor (IFR) and lead-cooled Fast Reactors (LFR), are increasingly used together with scCO₂. This is because of the stability associated with scCO₂ under radiation. The high density of the fluid allows for efficient heat removal in smaller volumes, a critical factor for the economics of small modular reactors (SMRs) [23,24].

Solar thermal integration

Concentrated Solar Power (CSP) faces a bottleneck in conversion efficiency. By integrating scCO₂ cycles, CSP plants operate at higher temperatures (700 °C to 800 °C), approaching the Carnot limit more closely than ever before. Argonne National Laboratory, USA, was the first to develop the concept of scCO₂ cycles for both sodium and lead-cooled fast reactors [10]. The closed-loop nature of these cycles is also a major asset in arid regions where CSP is viable, but water is scarce.

Properties and examples of supercritical fluid

Industrial process applications of scCO₂

Supercritical fluid extraction (SFE)

The extractive nature of scCO₂ technology is based on adjusting the temperature and pressure for the selective extraction process of specific compounds. The liquid-like density and gas-like diffusivity of scCO₂ enable it to dissolve solutes actively and infiltrate matrices with high efficiency. An increase in temperature and pressure leads to an increase in fluid density, which in turn increases the solubility of the specific compound [25]. At low pressure (< 150 bars), scCO₂ behaves like a non-polar solvent [26,27]. However, a co-solvent like ethanol is added to the non-polar nature of supercritical fluid to enable it to dissolve hydrophilic substances like sugars, proteins, and minerals. As pressure rises (> 300 bar), its polarity and density increase, allowing it to solubilize heavier and more complex molecules [26,28]. This technology is ubiquitous in the food industry. It is the standard for decaffeinating coffee and tea, because it removes caffeine without stripping away flavor notes or leaving chemical residues. Similarly, the extraction of hops for brewing and nutraceuticals from algae relies on SFE for product purity [29].

Precision cleaning and drying

ScCO₂ is useful as a cleaning solvent when compared to water and other solvents, especially in industries that involve microscopic contamination [30]. The low viscosity of scCO₂ allows it to flow into nano-scale crevices and to enhance the dissolution of organic contaminants through a decompression process [31, 32]. The transition from supercritical fluid to gas involves no liquid-vapor interface, and there is also no surface tension to damage delicate microstructures during drying. This lack of capillary stress is vital for aerogel production [33]. If a wet gel is dried via evaporation, the surface tension collapses the pore structure. Supercritical drying bypasses this collapse, and thus preserves the silica network of the material [34,35].

Pharmaceutical and food processing

In natural products for drug manufacturing, purity is an important factor to be put into consideration. Traditional solvent extraction often leaves behind traces of residues that require extensive downstream processing to remove [36-38]. In supercritical fluid extraction

(SFE), a cosolvent like ethanol is usually selected because it is economically produced in pure form and added to the carbon dioxide to enhance the solubility of polar molecules [39,40].

Particle engineering

Particle size, temperature, pressure and concentration affect scCO₂ extraction processes [39]. Particle size has an influence on the mass transfer and accessibility of the solvent to the solute components [41]. Due to the poor solubility of some drugs, scCO₂ is useful in enhancing the solubility of these drugs using some combination techniques such as Rapid Expansion of Supercritical Solutions (RESS) [41,42].

Food systems

In the food sector, the focus remains on natural processing. ScCO₂ is used to fractionate fats, separate cholesterol from dairy, and extract antioxidants from plant matter without using "chemical" solvents that could endanger consumers. ScCO₂ is used in the lipid industry to concentrate Omega-3 fatty acids from fish oil, and methylglyoxal (MGO) from honey [43].

Material science applications

Polymer synthesis and modification

Supercritical CO₂ has found a niche in polymer chemistry as a "green" reaction medium. It allows for surfactant-free polymerization, where the polymer precipitates out of the CO₂ as a clean powder. Furthermore, scCO₂ has the tendency to lower the melting point and density of polymers [44]. This allows thermally sensitive polymers to be processed at lower temperatures. These polymers exhibit excellent water solubility, strong electrostatic interactions, and notable swelling behavior, which enable their applications as sensors and water treatment agents [44,45].

Foaming

ScCO₂ has replaced traditional chemical blowing agents due to their unique properties such as non-toxicity, non-flammability and low cost [46]. The use of scCO₂ as a physical blowing agent has been recorded [47]. In this process, the polymer is saturated with scCO₂ and the saturated polymer undergoes some cooling, which enables the scCO₂ to stay dissolved in the polymer matrix [47]. This "microcellular foaming" creates materials that are lighter and stronger

than traditional foams, without using ozone-depleting chlorofluorocarbons (CFCs) [48].

Chemical recycling

A rapidly emerging field is the use of scCO₂ in textile and plastic waste recycling. For example, polyethylene terephthalate (PET) plastic (used in water bottles) is broken down to its monomer building blocks using scCO₂ as a solvent. ScCO₂ has the capacity to enable selective recovery of metals and dyes, thus contributing to circular economy practices [49]. This offers a pathway to a clean environment, thus turning waste plastics back into virgin-quality material, without posing significant contamination risks [50].

CONCLUSION

The usefulness of scCO₂ in the pharmaceuticals has facilitated solvent-free purification and processing of active pharmaceutical ingredients (APIs). ScCO₂ has also played a major role in the production of fine particle microencapsulation and drug formulation without the thermal degradation associated with conventional approaches.

DECLARATIONS

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Ethical approval

None provided.

Use of Artificial intelligence/Large language models

We also declare that we use Generative artificial intelligence (AI) and AI-assisted technologies in proofreading the manuscript.

Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Conflict of interest

No conflict of interest is associated with this work.

Contributions of authors

We declare that this work was done by the author(s) named in this article, and all liabilities pertaining to claims relating to the content of this article will be borne by the authors

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