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Supercritical Fluid Extraction as a Potential Extraction Technique for the Food Industry †

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Abstract: Supercritical fluid extraction (SFE) is a non-conventional extraction technique that can be used in the food industry because it can recover both polar and non-polar compounds. This technique is carried out above the critical point of the extraction solvent, allowing for the control and manipulation of different properties such as diffusivity, viscosity, and density. This is possible due to the fluid's changes in pressure and temperature that cause variations in selectivity and power. This eco-friendly extraction technique has several advantages, including high selectivity due to changes in pressure and temperature, as well as changes in the solvent's polarity by adding cosolvents. SFE has already been used in the food industry due to the benefits of this technique and its suitability for both polar and non-polar compound extraction. The goal of this work is to compile the most recent data on SFE applications in the food industry, thereby providing insight into SFE feasibility in a large-scale process.

Keywords: green extraction technique; supercritical fluid; critical point; bioactive compounds; food application

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1. Introducing Supercritical Fluid Extraction (SFE)

Supercritical fluid extraction (SFE) is a novel technique developed in both laboratories and industries for the past 60 years [1]. This extraction technique occurs in three steps: first, a dissolving/resolving process of the analytes takes place; then, there is a sweeping of the analytes; and finally, the analytes are trapped [2]. The schematic representation of SFE is represented in **Error! Reference source not found.**A. The SFE extraction optimization plays a key role since the different parameters (e.g., temperature, pressure, time and solvent of extraction, sample size, type, and percentage of cosolvents…) directly affect the extraction yield [3]. For the optimization, two strategies can be performed: phase equilibrium strategies and experimental design statistical optimization, which is the most used. Moreover, it is mandatory to consider both phase equilibrium and mass transfer as limit stages of the process [3].

To extract the target analytes, a solvent above its critical point is used, performing as a supercritical fluid (SCF), as shown in **Error! Reference source not found.**A. A SCF is any substance above the temperature and pressure critical points, having both gas and liquid characteristics [1]. In this way, solvents have liquid-like densities while viscosity is gas-like, and diffusivity is around two orders of magnitude higher than liquids [1]. SCF can diffuse through solid matrices as gases and dissolve compounds as liquids. When SFE is applied, slight changes in temperature or pressure lead to larger changes in the fluids' density since the solvent is performing near its critical point [1]. Thus, SFE selectivity depends on the density of the solvent [1].

CO_2 is the main solvent applied in SFE since it is non-toxic, non-explosive, and chemically inert and has low viscosity, surface tension, and high diffusivity. Moreover, the extract is easy to obtain and remove, and its critical point does not negatively affect the biological components [4,5]. The critical temperature and pressure of CO_2 are moderate (31 °C and 7.38 MPa, respectively), so the operational conditions can avoid the labile compounds' degradation [5]. Even though CO_2 is ideal for the bioactive compounds' recovery [6], pure CO_2 is not suitable for the polar compounds' extraction, so adding a cosolvent is necessary to improve the solubility of the target analyte in the solvent [4]. When a cosolvent is used, it is important to consider how the critical temperature and pressure of the solvent are changed in the extraction system, causing changes in the density of CO_2 and polarity of the mixed solvent, resulting in better efficiency of the extraction process [4].

SFE is considered a green extraction technique since this process needs a lower extraction time and lower quantities of non-toxic organic solvents compared with conventional extraction techniques such as solvent extraction [2]. Moreover, by applying SFE, no solvent residues are found in the extract, and the selectivity achieved is higher than in other extraction techniques [2,3]. However, the main disadvantage of SFE is the expensive equipment compared to conventional extraction techniques [5]. In this work, the potential use of SFE in the food industry is discussed based on the current data available, considering not only the recovery yields obtained and the wide compounds application but the scaling-up process suitability.

2. Supercritical Fluid Extraction (SFE) in the Food Industry

Although SFE has been studied and applied in the past decades in other industries, the food sector has shown interest in this extraction technique since 2016 [1]. Despite the increasing interest of the food sector in SFE, there is a notable delay between research and industrial applications since there is a need to prove this technology's efficiency, validate the process, including the products in the novel foods regulations, as well as confirm the nutritional profile, toxicity, allergenic potential and presence of the contaminants in the products obtained [1].

The potential use of this novel extraction technique is represented in **Error! Reference source not found.**B. In this way, researchers have shown the potential application of this eco-friendly extraction technique in the food industry to obtain different compounds (e.g., bioactive compounds, fatty acids, pigments…). Regarding flavonoid extraction from leaves, Song et al. have applied SFE-CO_2 to *Ziziphus jujuba* Mill., under optimal conditions (52.52 °C, 27.12 MPa, 113.42 min, and 0.41 mL/min of co-solvent flow), the flavonoid content was 29.052 mg, being both kaempferol and quercetin glycosides the main compounds [6]. Another study by Frohlich et al. used SFE for antioxidant compounds and eugenol extraction in clove leaves. The optimal operation conditions were 300 bar 60 °C, obtaining 1.94% of extraction yield [7]. Fatty acids of the pulp and nu oils from *Terminalia catappa* fruits were obtained in another study by applying SFE. After an extraction run at 300 bar 60 °C, 7.4 and 61.5% of pulp and nut of extraction yield was achieved. The extract was mainly formed by unsaturated fatty acids, of which omega-3 and -6 were predominant, showing suitable physicochemical properties [8]. SFE has been also applied in edible flowers. Volatile oils from Surangi flowers were extracted with SFE-CO_2 and steam explosion. The optimal extractions conditions for SFE-CO_2 were 35.03 °C, 179 min, 142 bar, and 3 mL/min of flow rate, achieving 4.75% of overall recovery yield [9].

Although SFE seems to be a potential technique to extract different compounds that can be incorporated into several food products improving their nutritional profile, this technique can be applied to eliminate substances, e.g., oil, to reduce the fat content. Michael E. Wagner et al. extracted the oil in chips applying SFE-CO_2 at different operational conditions that ranged between 27.6–41.4 MPa, 35–80 °C and 0.5–5 g CO_2/min. Up to 100% of extraction was achieved when the highest temperature and pressure were performed [10]. The decaffeination process of coffee beans using SFE-CO_2 was studied by Lolanda De Marco et al. showing the suitability of the process since it can be performed in a single step [11]. Moreover, SFE can be applied to recover compounds or metabolites that can negatively affect the quality or/and food product safety. Hina Shanakhat et al. studied the application of SFE for the macrocyclic lactone mycotoxins recovery in maize flour. Results showed how using methanol as a solvent extract allowed 100% of these mycotoxins recovery [12].

Considering the current interest from the scientific community in the study of SFE for the compounds recovery for the food industry application, showing positive results in a laboratory-scale performance, it is necessary to continue the research considering the scaling-up process to prove the efficiency and the economic feasibility of SFE for the food industry.

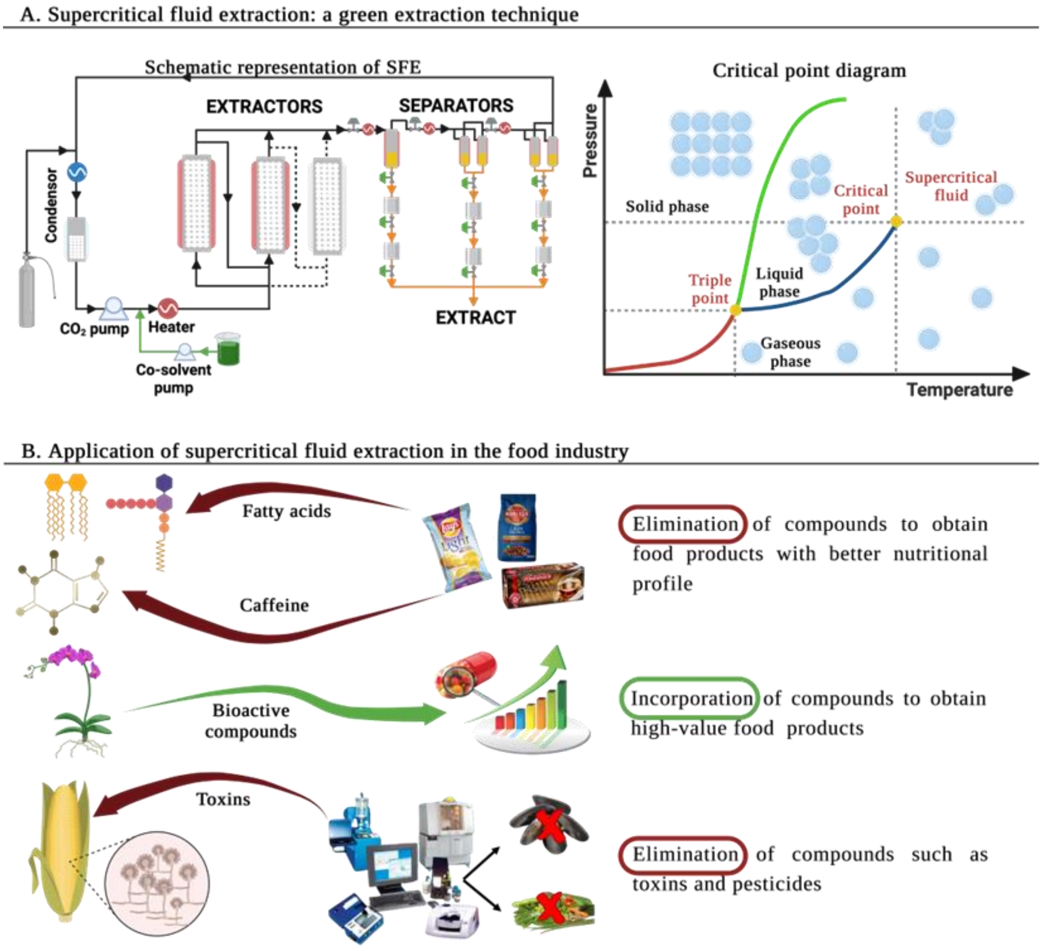

Figure 1. Supercritical fluid extraction (SFE) as a potential extraction technique in the food industry. (**A**) represents the SFE-CO_2 process and the critical point diagram of any substance. (**B**) shows different uses of SFE in the food industry including elimination and incorporation of compounds.

3. Conclusions

SFE is a green technique that has recently taken an interest in the food sector since it is characterized by its high selectivity, low solvent requirement, low extraction time, and wide range of compounds that can be extracted. In this way, several studies have established the optimal extraction conditions to release bioactive compounds, fatty acids, pigments, and other substances in different matrices, e.g., leaves, fruits, or edible flowers, showing high recovery yields. These high-value compounds can be incorporated into different food products improving their nutritional profile. However, there is a lack of data regarding the efficiency of this technology on an industrial scale applied in these high-value products. Nowadays, SFE is used in the food industry to eliminate compounds such as caffeine for decaffeinated coffee production or fatty content in low-fat. Moreover, SFE has been suggested to eliminate non-desirable substances that can be found to hurt human health, e.g., toxins. Considering the current data available, it is necessary to provide information on the efficiency of this technology on a large-scale, as well as its economic feasibility, to determine its application in the food industry.

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