Supercritical Fluids: A Review of Supercritical Carbon Dioxide Application Technology in Food Processing

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Abstract

Supercritical fluids have unique properties because they offer the possibility of being manipulated, such as density, viscosity, diffusivity or surface area, through pressure and temperature control. This article provides a general and complete description of the current state of knowledge of the application of supercritical carbon dioxide and its contribution to processes within the food industry. The foundation and perspectives in the application of four techniques in which supercritical fluids are used, such as extraction, impregnation, microencapsulation, and pasteurization in food preservation processes compared to traditional processes, are reviewed and analyzed. In this review it was possible to denote that there are advantages when using supercritical carbon dioxide technology in the various processes that have its application within the food industry such as reduction in component extraction time, increase in the performance of activities antioxidants in the product, among others. However, a significant study of this type of technologies is still required since a great barrier that persists is the high operating cost when working on a larger scale.

Keywords

Supercritical fluids,CO_2 Supercritical, bioactive compounds, organoleptic properties, extraction time, diffusion time.

1. Introduction

Today, raising awareness about the connection between food and health is presented as a decisive factor in the progress of research, development and innovation within the food industry. Processes with supercritical fluids (FSC) are sustainable, environmentally friendly, profitable and offer the possibility of obtaining new products with special properties (Knez et al., 2019).

Most of the applications of supercritical fluids are related to their physical properties, which can be easily changed by controlling pressure and temperature, in particular on the possibility of varying their solvent power over a wide range. These characteristics have prompted its use in a variety of applications in the field of analytical chemistry. Perhaps the most cited handling of these compressed fluids has been in the field of encapsulation, deodorization or as extraction solvents (Ghosh & Pradhan, 2020).

Among the most frequent supercritical fluids are xenon, ethane, carbon dioxide, nitrous oxide, water, among others, which offers a range of unusual chemical possibilities (Kailasa& Wu, 2012). HeCO_2 Supercritical is becoming an important commercial and industrial solvent due to its role in chemical extraction, in addition to its low toxicity and being an environmentally friendly solvent (Cabeza et al., 2017).

The document is organized to allow an up-to-date understanding of the main features, benefits, and some applications of the$CO_2 - SC$ in processes within the food industry; In addition, the fundamentals and advances of some techniques such as extraction, impregnation, microencapsulation and pasteurization are discussed.

2 Materials and methods

A descriptive observational study was used, in addition to a systematic review of publications in digital databases of scientific and specialized information (Scopus, Science Direct, Springer, among others) and manually on the Internet through books, encyclopedias and doctoral theses. .

3 Analysis and discussion of results

Supercritical fluids

In 1879, Hannay and Hogarth introduced supercritical fluids as a solvent medium for the dissolution of solutes. However, the first commercial applications of supercritical fluids were reported in the late 1970s, with reports of coffee decaffeination (Kailasa& Wu, 2012). Since then, FSCs have become an important technique in numerous fields of engineering and technology (Belwal et al., 2020). Table 1 shows the critical temperature and pressure conditions that define the critical point of the most used solvents.

Table 1. Critical pressure and temperature conditions of the most used supercritical solvents

Compound	Formula	Critical Presion (Bar)	Critical temperature (°C)	critical density (g/ml)
Carbon dioxide	CO_2	72	31,1	0,47
Water	H_2O	214,8	374,2	0,35
Ammonia	NH_3	109,8	132,5	0,23
Argon	$With$	48,6	-122,4	0,71
Acetone	C_3H_6O	47,0	235,0	0,28
Ethanol	C_2H_5OH	72,0	243,4	0,28
methanol	CH_3OH	78,9	239,0	0,27
ethane	C_2H_6	46,0	-82,6	0,16
n-Propane	C_3H_8	47,6	32,3	0,20
n-Butane	C_4H_{10}	42,4	96,7	0,22

.Source: (G.R. Ramírez, 2017).

The term “supercritical” refers to a substance that is above its critical temperature (T_c) and its critical pressure (P_c) (Areco, 2019). At this point, the FSC behaves like a gas, acquiring characteristics that it does not have when it is in a liquid state, that is, it becomes compressible. However, its density is that of a liquid while the viscosity is that of a normal gas, which provides exceptional solvating and reactive properties (Akalın et al., 2017; Amaral et al., 2017; Espinoza et al., 2017).

Figure 1 shows the general phase diagram for a pure substance, where the temperature regions and their intersection at the triple point where the substance is in three states at the same time: solid, liquid and gaseous; beyond this point lies the supercritical area. These zones are separated by the coexistence curves of two phases (solid-gas, solid-liquid, and liquid-gas), corresponding to the sublimation, fusion, and vaporization equilibria, respectively (Perrut&Perrut, 2019).

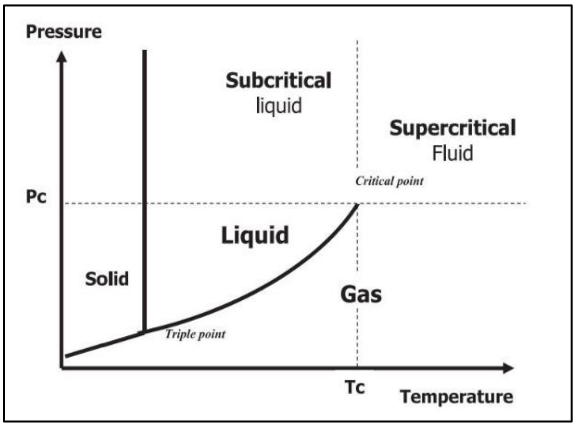

Figure 1. Solid/liquid/gas/supercritical fluid phase diagram. PC: critical pressure; Tc: critical temperature for pure compounds

Source: (Perrut&Perrut, 2019).

In the food industry, they have been used mainly for the extraction of essential oils, bioactive compounds, phenolic compounds, carotenoids, and tocopherol from various fruit and leaf extracts. Supercritical fluids are a more efficient, safe and ecological method to extract and recover flavonoids in order to study bioactive compounds (Chávez-González et al., 2020), Similarly, this technology has also been used for the removal of substances toxic chemicals, including pesticide residues, environmental contaminants, and mycotoxins from food and produce.

The physical property of enormous industrial interest of the$CO_2 - SC$ it is based on the significant variation in the solvation power, generally related to its density as a function of temperature and pressure, as seen in Figure 2 (Barberio, 2019; Kohli, 2018).

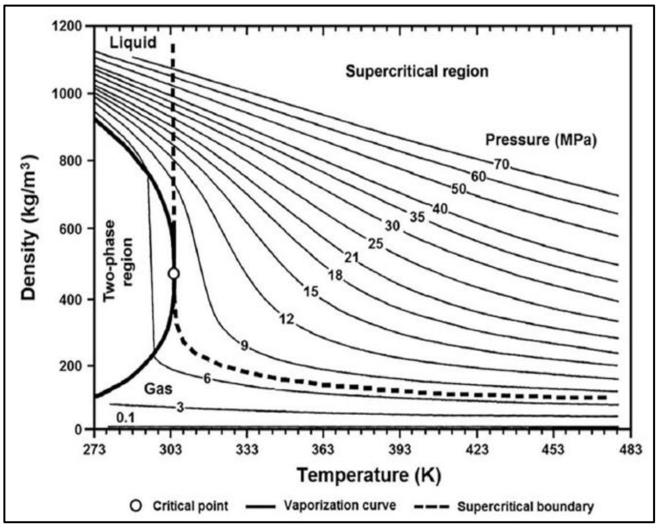

Figure 2. Density of CO_2 depending on pressure at different temperatures

Fuente: (Kohli, 2018).

The phase equilibrium, the mass transfer by convection and diffusion, and the displacement of the solution by expansion are what make up the mechanism of this process. The gas-like viscosity allows it to quickly penetrate complex structures and porous materials, like a gas, and helps dissolve the solute like a liquid. As evidenced in figure 3, this property increases with pressure in a similar way to density, however, the effect is less pronounced (Bruno et al., 2019; Essien et al., 2020; Jiménez-Sánchez et al., 2016). These properties allow adjustment of the solvent selectivity of a FSC towards a target compound, which is particularly interesting in the case of extraction (Chemat et al., 2017).

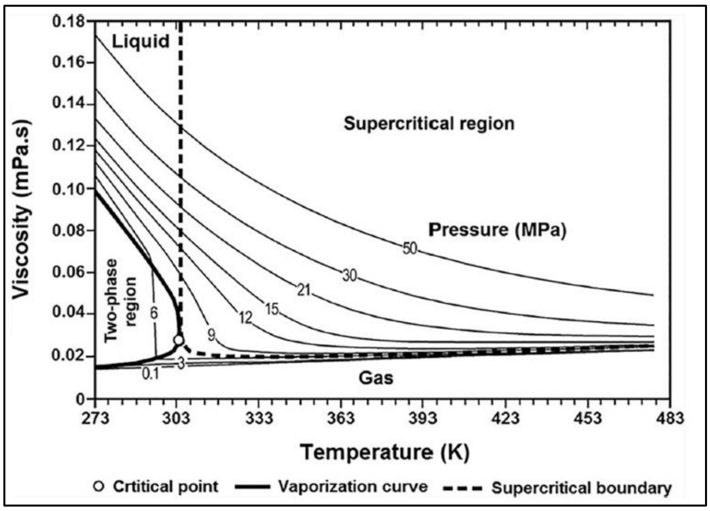

Figure 3. Variation of the viscosity of the CO_2 as a function of temperature at different pressures.

Fuente: (Kohli, 2018).

The low critical point of CO_2 ($T_c = 31.1$°C; $P_c = 72\ bar$) allows the development of low temperature processes for thermosensitive products (G. R. Ramírez, 2017); In addition, being a gas at room temperature and pressure, solvent-free products can be obtained (Belizón et al., 2018; Viganó et al., 2020). Compared to other solvents frequently used within the food industry, theCO_2 It has some characteristics that make it ideal for use in extraction with supercritical fluids (EFS):

- Dissolves nonpolar or slightly nonpolar compounds.
- Insolubility for molecules such as proteins, polysaccharides, sugars and mineral salts.
- Low pigment solubility.
- It does not leave residues at the end of the treatment (thanks to its complete evaporation in atmospheric conditions) (Becerra, 2018).

As could be noted in the study by (Fornereto Soldan et al., 2021), they showed that the increase in temperature and the addition of ethanol increased the yields of EFS extractions, demonstrating that it could be an interesting technique to replace traditional extractions and minimize industrial waste, with yields on occasions like or even higher than those reported by other authors.

The miscibility of the polar components in theCO_2 it is limited due to a low polarity aspect, which reduces its ability to extract high molecular weight or high polarity compounds. This problem can be avoided by adding a solvent modifier, or cosolvent (ethanol being the most commonly used), which increases the solubility of polar compounds and, in turn, reduces the selectivity of the process by increasing the number of extractable compounds (Mazzutti et al., 2020).

Examples of applications in the food industry are the extraction of cholesterol and other lipids from egg yolk; fractionation of milk fat; extraction of lipids and cholesterol from meat and meat products; of fish; extraction of natural colorants from various food products; extraction, refining and fractioning of vegetable oils and fats; among other applications (Cabeza et al., 2017).

Supercritical fluid extraction

Conventional extraction techniques are generally used to extract biologically active compounds from plant sources. However, these techniques have some drawbacks, such as a long extraction time, high solvent and energy consumption, and low selectivity (Machado et al., 2017).

On the other hand, unconventional techniques such as ultrasound-assisted extraction (UAE), pressurized liquid extraction (ELP) and supercritical carbon dioxide extraction ($CO_2 - SC$) are considered alternatives as they require less energy and provide a safe and high-quality product/extract (Chemat et al., 2020).

Compared with traditional extraction methods using organic solvents, the$CO_2 - SC$ it has many advantages because it is non-toxic and has non-flammable intermediate products; Furthermore, it is efficient since it can produce high purity oils with high yields, and it satisfies the growing market demands for “natural” products. Said extracts have shown potency as antibacterial, antiproliferative and antioxidant agents in comparison with their counterpart obtained with organic solvents (Essien et al., 2020; Zizovic et al., 2018).

The EFS is divided into two macroscopic steps: extraction and separation of the solute from the solvent (Chemat et al., 2020). The low temperature and the absence of oxygen during extraction preserve bioactive compounds and make this technology appropriate for thermally sensitive components, such as polyunsaturated fatty acids. Also, theCO_2 it can be easily separated from the extract by changing the operating conditions below the critical point. In addition to this, supercritical extraction is a flexible method for the fractionation of solutes by varying temperature and pressure or adding a cosolvent (Gustinelli et al., 2018; Ivanovs&Blumberga, 2017).

Although theCO_2 supercritical has extraction limitations for polar compounds, it is a very suitable solvent for lipophilic compounds and other non-polar compounds (Semenoglou et al., 2021); Such is the case of the work by Melo-Guerrero et al., 2020, they compared the composition and antioxidant activity of chamomile essential oil obtained by supercritical fluid extraction and steam distillation. They determined that supercritical fluid extraction (SFS) offers considerable advantages over traditional methods. Chamomile essential oil was extracted in less time usingCO_2 supercritical and the extraction yield was higher.

Although the cost of supercritical fluid technology has continuously decreased in recent decades, the operating costs remain high, which is the main drawback for implementing EFS. As (Ballesteros-Vivas et al., 2019), provide, in addition to the expensive equipment, the optimization of processes in EFS continues to be a challenge for each process. Although, by covering these drawbacks, SFE can provide vaporizable transport properties that allow faster and selectively efficient processes, compared to liquid extraction, so it should be considered as a suitable extraction alternative.

Supercritical Solvent Impregnation

The prevention of contamination and spoilage of food is achieved using food additives, that is, substances intentionally inserted into food to provide some technological function, which can range from preserving the product to providing specific characteristics such as flavor, color and texture (Carocho et al., 2018; Santos et al., 2021). Supercritical fluid processes, such as rapid supercritical solution expansion (ERSS), supercritical antisolvent (SAS), supercritical solvent impregnation (ISS), antisupercritical solvent precipitation, and supercritical fluid emulsion extraction (SEFS) have emerged as interesting techniques within the food industry (Gönen& Gupta, 2020).

Supercritical solvent (ISS) impregnation is a promising technique with great potential for the preparation of novel polymer systems to add antibacterial properties (Zizovic, 2020). The technical applications of impregnation are applied to modify the properties of substances through the physical or chemical binding/adsorption of the impregnated to a material or surface (Weidner, 2018).

Although processes such as extrusion, solvent casting or melt blending have traditionally been used, with ISS, and with carbon dioxide impregnation, the disadvantages of slow diffusion processes and process times are overcome. long in the case of impregnation from liquid solutions, or low yield in the case of impregnation from gaseous phases (Cejudo Bastante et al., 2019; Knez et al., 2019; Milovanovic et al., 2018).

In a typical process, this technique is based on the use of a non-toxic and inert medium, commonly a saturated mixture ofCO_2, as a solvent to incorporate the active compound into the polymeric matrix. Due to the high diffusivity, low viscosity, and high solubility of theCO_2 for a variety of solutes and substrates, it allows the impregnation of solid matrices of a wide range of materials, being able to cause swelling/plasticization of the polymer, resulting in the homogeneous impregnation of the polymer (García-Casas et al., 2018; Lee & Soh, 2020).

This method is gaining more and more attention in the field of the food industry, mainly for both biodegradable and non-biodegradable synthetic materials (Milovanovic et al., 2018; Villegas et al., 2017). Given its versatility, by means of supercritical impregnation, different types of organic solutes can be incorporated, mainly hydrophobic and thermosensitive compounds (such as medicines or essential oils) in natural and synthetic polymer numbers based on the effect provided by theCO_2 (Mosquera Ruiz, 2021).

Such is the case of (Bastante et al., 2021), they fabricated independent resistant films composed of nano fibrillated cellulose (CNF) and mango leaf extract (EHM) by supercritical solvent impregnation and compared it with films prepared by conventional casting methodology. with solvent. The comparison shows a clear benefit of the innovative ISS in terms of bioactive properties. In fact, the antioxidant and antimicrobial activities are enhanced in the films made by CO_2-assisted impregnation by CNF with EHM.

Temperature has opposite effects on impregnation. On the one hand, the diffusivity of the solute increases with temperature; on the other hand, the sorption of CO2 in the matrix tends to decrease (Ganan et al., 2020). As in the case of Franco et al., 2019, in said project, the authors studied the adsorption of α-tocopherol (TOC) in polymeric films using supercritical impregnation, considering firstly monolayer polypropylene (PP) and polyethylene (PET) and then PET/PP multilayer films. In this work they demonstrated that supercritical impregnation is an effective method to overcome the main limitations associated with the production of active packaging with conventional techniques. This result can be attributed to the low temperature used and the high diffusivity of the$CO_2 - SC$.

Supercritical carbon dioxide assisted impregnation ($CO_2 - SC$) has been proposed as a simple and effective alternative to incorporate non-polar additives in polar matrices (M. E. V. Ramírez et al., 2017). However, more research is needed to implement the use of this type of technology in other applications within the food industry (Ganañ et al., 2020).

Microencapsulated

Microencapsulation allows the preservation of bioactive compounds by improving their thermal, chemical, and photographic stability in the medium to protect them from extreme pH, light, or oxygen. For food applications, there are many challenges in developing suitable and efficient microencapsulation systems (Dhakal& He, 2020).

Currently, the development of new microencapsulation techniques using supercritical fluids has emerged, since it offers valuable advantages, such as control of particle size, size distribution and morphology, compared to conventional techniques (Franca-Oliveira et al. al., 2021; Tarone et al., 2020; Temelli, 2018); that is, depending on the role played by the$CO_2 - SC$ In encapsulation techniques, it can be classified as solvent, antisolvent, solute, cosolvent, extractor, among others (Klettenhammer et al., 2020).

The most common particle formation method is the supercritical antisolvent (SAS) process, theCO_2 as an anti-solvent to rapidly remove organic solvent from a phase containing the active ingredient and coating material. In a typical operation, the$CO_2 -$

SC is introduced into the solution by expanding it. This leads to a reduction in the bulk density of the solvent and causes a reduction in the solute solubility in the expanded solution (Lee & Soh, 2020; Olguin Rojas, 2019).

Supercritical fluid emulsion extraction (SEEF) is a novel encapsulation technology that combines conventional emulsion processes with the unique properties of supercritical fluids (Prieto et al., 2017); for example, the author Mihalcea et al., 2018 valued the functional potential of sea buckthorn carotenoids extracted by the method$CO_2 - SC$ and microencapsulated by emulsion. The authors found satisfactory parameters of color and antioxidant activity, suggesting a lower rate of carotenoid degradation and sequentially of activity in the samples with microencapsulated powder.

It is important to review techniques for the encapsulation of bioactive compounds, because they provide better protection of the active ingredient against degrading factors. In a recent work, Cruz et al., 2020 used the encapsulation technique through the extraction of emulsions to conclude that the analyzes showed that they did not present a degradation during encapsulation, allowing the conservation of the extract for food and pharmaceutical applications.

Pasteurization

Food processing and preservation aims to preserve the nutritional properties and extend the shelf life of food by inactivating pathogenic and spoilage microorganisms (Anukiruthika et al., 2020; Tang et al., 2021). Traditional thermal pasteurization (TP) treatments can cause adverse changes in food quality, including detrimental effects on physicochemical properties and heat-sensitive nutrients such as vitamins, and undesirable changes in color, flavor, and texture, negatively affecting consumer acceptance (Bertolini et al., 2020; Putnik et al., 2019).

Non-thermal food processing appears as an interesting alternative for the food industry due to the greater nutrient retention and minimal sensory changes in processed products (Amaral et al., 2017). Microorganisms are destroyed and enzymes can be inactivated in liquids, such as juices, for example, by continuous flow of$CO_2 - SC$ along flow paths. Contact between the flows can be achieved with countercurrent columns, vessel agitation (shaking or mixing), or with a membrane containing minute pores in which the flows contact each other in a non-dispersive manner. The process does not negatively affect the properties of the liquid, such as taste, aroma and nutritional content (Kohli, 2018).

FSC pasteurization is carried out more efficiently in continuous mode than in static mode. Supercritical carbon dioxide technology (technology$CO_2 - SC$) uses pressure in combination with carbon dioxide to destroy microorganisms without affecting the nutritional content, organoleptic attributes, being a promising alternative for the pasteurization of bioactive compounds in food and medicines (Amaral et al., 2017; Jiménez-Sánchez et al., 2016). FSCs have also been used for the inactivation of microorganisms due to the ability of theCO_2 to penetrate the cell wall. The various mechanisms by which theCO_2 Supercritical inactivates microorganisms are pH alteration, protein denaturation and physical alteration of the cell wall (Anukiruthika et al., 2020).

Therefore, the$CO_2 - SC$ It is considered an effective tool for the pasteurization and inactivation of enzymes in food and biological systems, since it takes advantage of the bactericidal properties, being able to inactivate a wide range of microorganisms (Bertolini et al., 2020; Cabeza et al., 2017); an example being the work of (Marszałek et al., 2019), where it was found that the $CO_2 - SC$ had a significant influence on the polyphenol oxidase (PPO) and peroxidase (POD) activities, whereas a greater reduction in the residual activities of both enzymes was observed when the pressure was increased. $CO_2 - SC$. In addition, the results were compared with other non-thermal technology such as High-Pressure Processing (HPP), which is also based on the ability of pressure to inactivate enzymes, showing similar results.

Microorganisms are destroyed and enzymes can be inactivated in liquids such as juices, so the process does not negatively affect organoleptic properties (Kohli, 2018). These changes in the protein structure can have a positive impact on its binding properties, allergenicity, and techno functional properties. These types of alternatives, like heat treatments, can induce the unfolding, aggregation and/or flocculation of proteins.

4 Conclusions

The reviewed and updated scientific literature has allowed us to know the advances and some trends, which has shown that CO2-SC presents advantages within the processes compared to traditional methods in the food industry. Among the advantages found we have: shorter extraction times, diffusion and greater selectivity or, providing greater antioxidant capacities to the product on which it works.

It has been mentioned that, to increase the efficiency of the process, improve the quality of the final product and even obtain various products in the food line, CO2-SC can be supplemented by incorporating a cosolvent, ethanol being the most widely used. However, additional studies are needed for the use of these techniques in the industrialization of products in food systems.

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