# Supercritical fluid technology for agrifood materials processing

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# Supercritical fluid technology for agrifood materials processing

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Supercritical fluid technology has been applied in the food area for processing and preserving food products and/or monitoring the food quality, with known advantages. The main solvent used at supercritical conditions for food applications is carbon dioxide. Some examples are presented, from the traditional decaffeination of coffee up to the micronization of vanilla, passing through innovative processes such as the extrusion of protein-based snacks and drying of beetroot. The gap between research and industries is addressed, mainly due to a lack of data about food chemical changes that may occur during some processes, as well as technical data. However, this is an area in clear expansion and probably, in the future, we will have a menu composed of meals prepared by supercritical methods.

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## Introduction

During the last 60 years, supercritical technology has been developed in laboratories and industries around the world [1]. Despite it has been applied in several areas, one of the most important areas that placed this technology on the world stage is the food area.

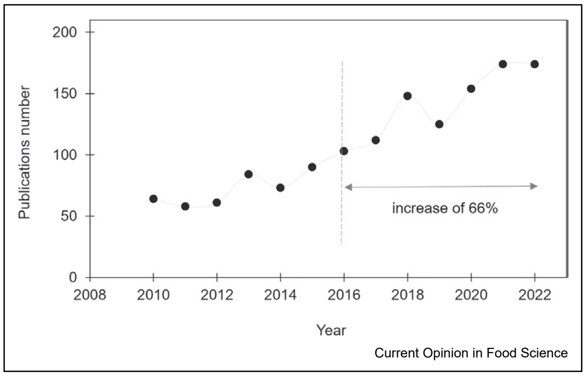
According to the Scopus database, from 2016 until now, there was an increase of 66% in the publication number (Figure 1) related to supercritical fluids and food, indicating that the food area still claims its place in this scenario. In the period from 2020 up to 2022 (October), 502 articles were published according to Scopus® using the descriptors 'supercritical fluids' and 'food', and within these results, 93% are related to extraction, while 36% studied the preservation of food. For this last number, there are overlapping results from extraction with preservation function, which explain those values.

Food processing has gained attention to guarantee food preservation on a large scale, transforming raw foods and ingredients into new products. Some food processing techniques use cutting-edge technology, while others have been practised for millennia such as brewing beer and baking leavened bread. Currently and in the future, the development of alternative technologies with reduced environmental impact, such as lower energy consumption, reduced residues, and improved quality and safety of final products, is crucial [2], as well as applying the biorefinery and circular economy concepts in the food industry [3,4].

Some sensitive raw materials such as flowers can contain valuable compounds for the food industry, and fractionation of those compounds to obtain thermolabile fractions in the first processing permits getting other important added-value compounds such as pigments and antioxidants from flowers/plants in a second stage using solvent modifiers [32]. Using residues from apple species (Bravo de Esmolfe) is also a strategy to obtain antioxidant-rich extracts with epicatechin and procyanidin B2 contents and neuroprotective activity [33]. Both approaches, fractionation or utilization of residues, are aligned with the circular economy concept, and also improve nutritional aspects of the food products, without neglecting environmental issues [2]. Fish waste can also be valorized in this concept using the same technology to extract, refine, transesteriticate, concentrate the oil, and formulate products [34].

There is an increase in the demand for a varied food supply and novel foods with different compositions, including distinct flavors, aromas, and colors, as well as textures and forms, with longer shelf life and produced by green processes. From 2015 to 2020, the U.S. Department of Agriculture (USDA) indicated that the new product introductions in the market included three food categories: beverages, snacks, sauces, and seasonings [5]. In this period, the respective consumers'

**Figure 1**




The number of publications dealing with supercritical fluids and food. The literature search was performed in Scopus, for the period of publishing data available (from 2010 up to actual) using as descriptors ‘supercritical fluids’ and ‘food’.

demands were also ‘no additives’, ‘low allergen’, and ‘free from artificial preservatives’ [5].

In this context, we think that supercritical fluid technology is an interesting approach to obtain food products with the required properties for the market and according to consumers’ needs by using the process of extraction to remove some additives (or their excess), and allergens, or to preserve foods without using artificial compounds.

Some authors have recently reviewed the issue of supercritical fluids applied to food processing [6,7], and recognized authors such as Professors Gerd Brunner, Michel Perrut and Jerry W. King have been highlighting the advantages of this technology for decades [1,8,9]. However, not all products have been industrially explored. In fact, there is a delay between the research and industrial applications, mainly because it is necessary to prove the technology efficiency, include products in the novel food regulations, and/or validate the process and confirm the nutritional profile of the product, its toxicity, allergenic potential, and the presence of contaminants [10,11].

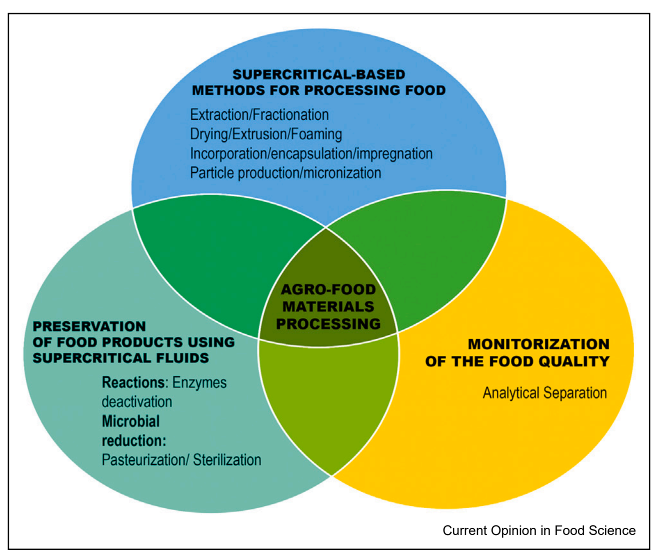
The principle of this technology is based on fluids that at pressures and temperatures above their critical values, are used as solvents. In this supercritical state, the solvent densities are the liquid-like approach, but with viscosity near normal gases, and diffusivity is around two orders of magnitude higher than liquids [8,12].

In the food area, the main solvent used to process food, using supercritical conditions, is carbon dioxide due to the inherent properties to achieve the supercritical state at a relatively low temperature and pressure, but also because it is recognized as safe (GRAS, Generally Recognized As Safe), environmentally friendly, and inexpensive. Supercritical carbon dioxide (sc$CO_2$) is also safe to handle, and because it is easy to remove (by expansion to environmental pressure), very low levels would be in foods after processing. The sc$CO_2$ can also be recycled and reused, reducing the generation of waste and environmental problems [8,12]. This solvent has desirable properties to be used in the food industry because it has high diffusion in solids and dense liquids, and it has a tuneable density by changing temperature and pressure conditions increasing the solvent solubility in the medium. The fundamentals and other physical properties of sc$CO_2$ have been widely described in the literature [12], as well as the equipment configurations [13].

Considering that the fundamentals and equipment information is accessible to researchers, we need to analyze the status of applications in the food area. Figure 2 shows the three main areas of supercritical fluid application in food. The included processing methods are extrusion/foaming, incorporation, micronization, and drying, while preservation methods include reactions and reduction of microbial populations. The third area is the monitoring of the food quality through the analytical separation of compounds by supercritical fluid chromatography and other coupled techniques such as Supercritical Fluid Extraction (SFE)–Gas Chromatography or SFE–Ion Mobility Spectrometry [14].

This article will mainly focus on supercritical-based processing techniques recently used in the food area, even though preservation and quality monitoring will be necessary for the final product. The main processes with potential to produce commercially valuable products will

**Figure 2**




Main areas of supercritical fluid application in food processing.

be discussed since they are essential to obtain food products with the desired characteristics.

## Supercritical-based methods for processing food

### Extraction

The classical extraction processes using $scCO_2$ are the decaffeination of coffee and the separation of hop volatiles for flavoring beer.

From the $scCO_2$ caffeine extraction process, caffeine and decaffeinated coffee are two products obtained in one single process being a great advantage. Since caffeine extraction is a well-known process, De Marco et al. [15] studied the environmental impacts of this process through a life-cycle assessment approach. To decaffeinate 1 kg of the suggested blend, the emissions to air, water, and soil were considered in all stages of production. This study reveals that the steps that mostly affect the environment are the agricultural stage, transportation, and caffeine extraction. To improve efficiency in the decaffeinated coffee production chain, the authors have proposed a reduction of fertilizer amount of 20% and the partial substitution of energy source [15]. This study justifies the SFE as a suitable process to obtain decaffeinated coffee in the coffee production chain.

While different studies still have been developed using different solvents and mixtures, such as $scCO_2$, $scCO_2$+ ethanol, $scCO_2$+ ethyl acetate, compressed propane [16], or sulphr hexafluoride and dimethyl ether [17], in food industries, the $scCO_2$ is already the chosen solvent to obtain the hop extracts (concentrated extract of α-acids and essential oils) for beer brewing. The addition of enriched extracts contributes to beer's bitterness, aroma, foam, and microbiological stability; the recommendation of use is based on the α-acids' concentration reducing by 10 or 20%, compared with hop pellets [18].

Another interesting and recent application of $scCO_2$ extraction in the food area is related to entomophagy. The demand for protein promotes the consumption of edible insects, and it has recently drawn the attention of food industries, with a growing practice of producing insect-based food. The composition of protein (60%) and fat (40%) is difficult product conservation through oxidation. Therefore, the defatted protein by $scCO_2$ extraction is a solution to improve the shelf life of this product and to increase the protein yield [19], also answering the demand for protein-based food products and considering the full use of raw material in the context of the biorefinery [20].

Uzel [21] used the concept of 'slow food' (from the movement of slow food [35]) to develop functional bread with reishi (*G. lucidum*) extracts obtained by $scCO_2$ and subcritical water extraction (SWE), in different experiments, joining new technologies in food production and marketing strategies. Different compounds were extracted, such as ß-glucans, polysaccharides, and triterpenoids using SWE, and ganoderic acids and alcohols were obtained using $scCO_2$. Extracts were added into a bread recipe in 0.4% (w/weight of solids), a hundred consumers ingested this functional bread over 21 days, and 87% answered positively about the sensory properties and health effects such as improvement of digestion, regulation of cholesterol levels, and strengthening of the immune system and metabolism.

### Drying

$ScCO_2$ drying is another process of interest in the food industry since the structure of vegetables can be maintained during the water removal process [22]. Zambon et al. [23] studied the $scCO_2$ drying and microbial inactivation of apple slices simultaneously. Compared with air-drying and freeze-drying, the $scCO_2$ drying process presented relevant results competing with the freeze-drying process and improving the quality of a final product by reduction of some microorganisms (namely vegetative bacteria, yeasts, and molds). These authors also studied the drying of strawberry slices in mild conditions (at 10 MPa and 40 °C up to 6 h), where the process reduced 98% of initial moisture content, reducing yeasts and molds, but presented a limited inactivation power toward total mesophilic bacteria [24].

The potential of simultaneous $scCO_2$ drying with the stabilization of the product highlights the innovation of the process with great importance in the food industry.

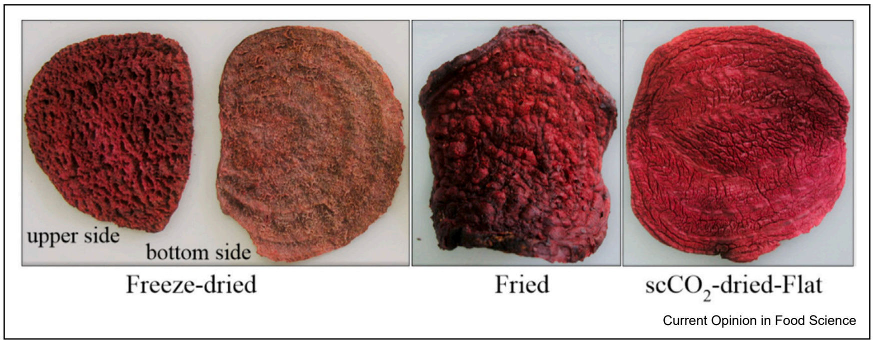
Tomic et al. [25] have recently compared the sensory quality and acceptance of dried ready-to-eat beetroot snacks prepared with different drying methods. The $scCO_2$ beetroot snacks were prepared at 10 MPa and 40 °C for 14 h and received the score 'very good' in the acceptability sensory analysis for nonprecooked $scCO_2$-dried samples, which is similar to fried beetroot according to 60% of tested consumers.

Considering these results, clearly $scCO_2$ drying has crucial advantages in what relates to nutritional value and bioactive compound preservation. Figure 3 shows the dried beetroot snacks prepared by $scCO_2$, presenting the characteristic magenta color with a better appearance than the fried sample.

### Extrusion/foaming

Chauvet et al. [26] reviewed the extrusion method assisted by $scCO_2$ for the microcellular foaming of polymers. Since $scCO_2$ is soluble in several molten polymers, acting as a temporary plasticizer, and allowing a decrease of the temperature during the process, this overcomes the requirement of formation of single phase in the

**Figure 3**




Dried beetroot samples were prepared with different methods, published by MDPI, 2021 [25].

batch foaming. The $scCO_2$ has advantages as a blowing agent mainly because, in the final product, there is no residue compared with the processes that use carbonated salts, and the final product has a porous 3D structure.

A healthy porous snack with up to 60% (wt) of whey protein was prepared using supercritical fluid extrusion, without any chemical modifications. Expansion operation was performed below whey protein denaturation temperature, which prevents hard texture, creating a uniform expanded structure [27], and being an interesting advantage of this technique from the authors' point of view.

### Impregnation

The incorporation of bioactive compounds has also been reviewed by some authors [28], however, most of the applications related to incorporation or impregnation are based on a polymeric structure to carry on bioactive compounds, which means this is valid for food applications and not necessarily for edible products. Antioxidants, antimicrobial agents, and so on are incorporated into matrices such as polylactic acid (PLA), polyethylene terephthalate (PET)/polypropylene (PP), cellulose acetate, chitosan, and others [28] to develop films and active food packaging.

Some natural based-polymeric films can also be edible when used in foods such as cheese or fruits and vegetables. Films based on alginate can produce antimicrobial packaging being an alternative to enhance food safety [29]. Another important example of impregnation is polysaccharide-based aerogels that were impregnated with vitamin $D_3$, whose stability was tested under 2–8 °C. Since this vitamin is thermosensitive, the impregnation was conducted at low temperatures by using a subcritical $CO_2$ condition, despite higher loadings being found at higher temperatures. After 5 weeks, 65–78% of vitamin $D_3$ remained in alginate-based aerogel particles [30], showing the efficiency of the applied method.

### Micronization

Vanillin is a bioactive compound obtained from vanilla orchid pods (*Vanilla planifolia*) and used as a flavoring agent in food with antioxidant and antimicrobial properties. With the global demand for vanillin estimated at roughly 20,000 tons per year, the processing should guarantee no residues in the final product, which can be obtained by the $scCO_2$ micronization method, reducing the organic solvents used in conventional methods. The Rapid Expansion of Supercritical Solutions method may uniformly prepare vanillin microparticles, with a size one hundred times lower compared with commercial vanillin (from 700 μm to 3–26 μm), and no changes were found in the morphology or crystallinity of particles [31].

## Challenges and current trends

The preservation of food products by using $scCO_2$ was already proved in scientific literature, by using pasteurization, sterilization, or enzyme deactivation [9,13]. Meanwhile, some juice processing can induce alterations in chemical composition and nutritional values, which may be preserved when treated with $scCO_2$ [13]. Clearly, in all processes, all alterations depend on food composition and interactions with $scCO_2$, and on medium conditions, including water, temperature, and exposure time.

Nowadays, the integration of those processes is a great innovation, since it can reduce time, equipment, energy, and consequently costs. Several combinations can be possible (extraction–drying, micronization–impregnation, drying–sterilization, etc), even though analyzing the food quality online using analytical separation with $scCO_2$, such as the analysis of pesticides in foods to avoid chronic diseases, for example.

Since this technology has a strong knowledge basis promoted by the academic sector, the next steps of the technology development will be the proof of the nutritional values of those food products processed by $scCO_2$, and to identify what group of food effectively has no chemical and nutritional changes, to be then produced in large scale. Authors such as Uzel [21] and Tomic et al. [25] have visualized the future by trying to understand the acceptability of consumers, which is one of the final steps for including a food product in the market.

## Final remarks

Nowadays, the technology is already valorized by some important characteristics of the $scCO_2$ process, such as using one raw material to obtain a variety of final products, such as caffeine and decaffeinated coffee obtained in one single $scCO_2$ extraction process; integration and combination of processes taking advantage of extraction–microbial inactivation or drying–sterilization in a single equipment/unit, such as fruits and vegetable snack preparation; and stabilization of thermolabile or photosensitive compounds during the process, such as vitamins and antioxidant extraction or incorporation in edible matrices.

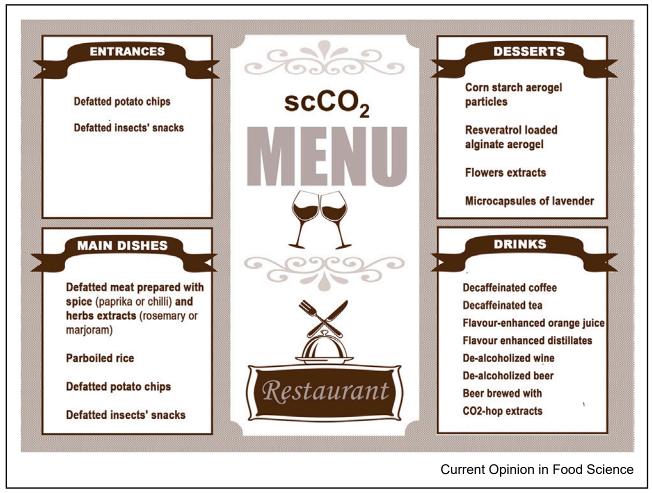
Despite the literature presents cases of the economical evaluation of supercritical technology [36,37], most of them are for extraction and not for different processes based on this technology that should also be evaluated. Optimization procedures and scaling-up are also bottlenecks for some processes based on this technology since most of them are static or semicontinuous processes. Moreover, there is a lack of process parameters in the literature to construct a clear opinion of the future of this technology, despite being clear the advantages of this technology when applied to food processing.

The authors consider that to complete the way of $scCO_2$ food process beyond the existing market, at least three steps are needed:

i) Confirm the chemical changes during the $scCO_2$ process for a group of food or specific food, and identify the nutritional value of those food products to validate the process;
ii) Make an economic evaluation of food processes using $scCO_2$ (process other than extraction) to guarantee some competitivity with the established processes in industries, as well as a life-cycle analysis for other processes;
iii) Identify the consumer acceptability of the food processed by $scCO_2$, avoiding misunderstandings about the technology, and highlighting the green process concept.

Some of these issues were already pointed out by Jerry King in 2014 [1], and most of them do not have answers. Nonetheless, there is continuous growth of this technology, and in the future, we probably will choose, in a restaurant menu, the $scCO_2$-processed foods to compose our meals (Figure 4).

**Figure 4**




Supercritical fluid technology-based menu (Based on Brunner, 2004).

This article had the intention to point out the current state of supercritical fluid technology (considering the recent years) applied to food processing, also envisioning the future for this technology. At this moment, we already have a quite completed menu based on $scCO_2$ processing food to improve our gastronomic experiences under the label 'food produced by green process', in a sustainable way.

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## Data Availability

Data will be made available on request.

## Conflict of interest statement

The authors declare that they have no competing interests.

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- •• of outstanding interest.


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