Trends in Analytical Chemistry 169 (2023) 117410

Contents lists available at ScienceDirect

Trends in Analytical Chemistry

journal homepage: www.elsevier.com/locate/trac

Compressed fluids for the extraction of bioactive compounds from plants, food by-products, seaweeds and microalgae – an update from 2019 to 2023

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ARTICLE INFO

Keywords:
Algae
Bioactive compounds
Biorefinery
Compressed fluids
Food by-products
Green extraction
Microalgae
Plant
Subcritical water extraction
Supercritical fluid extraction

ABSTRACT

The search for new natural ingredients with beneficial impacts on human health is a growing trend that has arrived to answer consumer's demands. Plant-based products like herbs, fruits, vegetables, or fungi, as well as macro- and microalgae, are valuable sources of these bioactive ingredients. Moreover, the valorization of food by-products by means of environmentally friendly techniques is a hot research topic today. In this context, the use of compressed fluids-based extraction techniques, such as pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), and gas-expanded liquids (GXL) has been demonstrated to possess great potential for the recovery of natural bioactive compounds. This work is an update of our series of reviews that presents the latest and more significant advances in the use of these compressed fluids extraction techniques for the recovery of bioactive compounds from plant, algae and food by-product biomasses, covering the period from 2019 to present.

1. Introduction

The use of compressed fluids-based extraction techniques for the recovery of bioactive compounds from different natural matrices represents a hot research topic today. The present work is framed in a series of review papers starting back in 2006 that have demonstrated how the use of this group of techniques in the field of natural compounds has still not peaked [1–4]. Although there are some reasons for this observation, the main points are: i) the interest on natural bioactives to be employed in the food industry is ever increasing, with new sources, compounds and bioactivities being persistently studied [5–8], and; ii) the awareness towards the detrimental effects of the climate change means that sustainable processes are strongly preferred in every field, where the extraction of natural matrices no exception. Although the use of terms "sustainability" and "sustainable" are clearly trendy and can be found in almost every context today, more than 35 years ago the UN World Commission on Environment and Development already proposed a brilliant definition for sustainability as "the development that meets the needs of the present without compromising the ability of future generations to meet their own needs" [9]. The core of this definition is obviously closely related to the 2030 Agenda for Sustainable Development adopted by United Nations and its 17 goals for sustainable development, but also inspired to a great extent the principles of green chemistry [10]. Later on, Chemat et al. [11] introduced the principles of green extraction of natural products, stressing the need for renewable sources, alternative solvents, improvement in energy usage, and reduction of wastes.

Compressed fluids-based extraction techniques are capable of meeting these characteristics in an effort to develop truly sustainable processes for the extraction of natural bioactive compounds [12]. Within this group of techniques, diverse tools differing on the particular extraction conditions can be found, mainly pressurized liquid extraction (PLE), supercritical fluids extraction (SFE) and gas-expanded liquid extraction (GXL). Although each of these techniques are described below, all these techniques have in common a significant reduction on solvent use with respect to conventional solvent-based extractions. Moreover, they have the potential to be scaled-up and even combined in the so-called biorefinery approaches, also discussed later. Current advancements on new solvents development and application may also positively influence the application of these techniques [13]. For instance, the potential of deep eutectic solvents (DES), specifically natural deep eutectic solvents (NADES) for the extraction of natural bioactive compounds complying with the principles of Green Chemistry has been already highlighted [14]. The great variability that can be found regarding the number and physicochemical characteristics of diverse NADES [15,16] is an asset for the search of new applications of

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https://doi.org/10.1016/j.trac.2023.117410
Received 31 August 2023; Received in revised form 26 October 2023; Accepted 28 October 2023
Available online 30 October 2023

compressed fluids implementing this new kind of solvents, with the aim to further improve the sustainability of the current processes. Nevertheless, the potential of this combined use is still untapped.

On the other hand, there is a clear necessity within the food industry for natural bioactive compounds driven by the consumers' preference for natural ingredients with respect to their synthetic counterparts. In this line, the search for new bioactive compounds from a wide variety of natural sources does not stop. Among the diverse natural resources, plants are the most well-known historically. The number of species that could be used for the recovery of bioactive compounds is huge [17]. However, other less exploited natural sources, such as marine sources (seaweeds and microalgae) have raised a lot of interest in recent years [18–20]. The focus on these organisms is related to the great biodiversity still unknown and they are underused to a great extent. Interestingly, some species are able to over-produce diverse bioactive compounds when grown under specific conditions making their cultivation very attractive for natural compounds production [21]. Moreover, microalgae production is independent from the marine environment and technological improvements on photobioreactor technology are pushing forward the world biomass production [22]. Apart from those natural sources, it has been repeatedly shown how agri-food by-products and wastes generated at different stages of food production are still rich on bioactive compounds [23]. For this reason, the re-use of those by-products as a source of bioactive compounds, producing an effective valorization process is gaining interest [24].

In this review, we combine all the above-mentioned aspects, i.e., the search for natural bioactive components and the use of compressed fluids for their recovery from natural sources, including plants, seaweeds, microalgae and agri-food by-products, with the aim to provide an updated overview of this research field. Following our previous work [4], the most relevant applications (from 2019 to present) related to this field are described and discussed, grouped by the extraction tool employed. Sample preparation approaches in which these techniques are used in the frame of an analytical methodology are out of the scope of the present review. Besides, a general overview of each technique is provided as well as the possibilities of their coupling into biorefinery approaches. Finally, the future needs in the field as well as the more plausible future trends are critically identified and assessed.

2. Pressurized liquid extraction (PLE)

2.1. General aspects

PLE or accelerated solvent extraction (ASE) is one of the emergent compressed fluid extraction techniques with a higher impact in food applications. A clear sign of this is the high number of studies that have used this extraction technique in recent years for a wide variety of food matrices (Table 1). In PLE, the extraction is carried out using solvents at high temperatures (up to 200 °C) maintained in their liquid state thanks to the application of high pressures, typically 3.5–20 MPa. The use of these high temperatures and pressures (always below the corresponding critical point) in PLE produces modifications in the physical properties of the solvent that favor the solubility and the mass transfer rate of the analytes into the solvent during the extraction. This is due to a decrease in viscosity and surface tension of the solvent under PLE conditions in comparison to atmospheric conditions, which improves the penetration of the solvent into the matrix and favors the contact between the solvent and the extractable compounds. As the temperature rises, always keeping the liquid state, the viscosity and the surface tension of the solvent are reduced and, thus, faster extraction processes and maximized extraction yields are achieved at the highest temperatures [2,25,26]. Nonetheless, it is important to take into consideration that high temperatures maintained for a long time can produce the destabilization of many labile analytes. Consequently, both the temperature and the extraction time have a high impact on the quality of the obtained extract. The extraction time establishes the time that the solvent remains in contact with the matrix, and therefore, with the compounds of interest. It can be controlled to produce the full exhaustion of the sample. In this sense, the extraction can be done in one cycle or several consecutive static cycles, where fresh solvent is introduced in the PLE instrument to replace the saturated solvent. There is also the possibility of performing a dynamic extraction procedure, in which fresh solvent is being flowed through the extraction cell during the extraction time. Nevertheless, this latter approach is by far less used.

Besides temperature, time, and pressure, another parameter that hugely affects the extraction selectivity is the extraction solvent. Nowadays, the most commonly used solvents in PLE are those considered environmentally friendly, mainly water and ethanol, or mixtures of them, to reduce the negative impact that other organic solvents cause in the environment. When water is exclusively used as solvent, the technique is referred to as subcritical water extraction (SWE), superheated water extraction (SHWE), or pressurized hot water extraction (PHWE). In this case, besides the reduction of viscosity and surface tension, other physical parameters are altered in water, mainly the dielectric constant (ε). At atmospheric conditions, water presents a very high ε (ε = 80); however, at high temperatures and pressures, the dielectric constant of water is significantly reduced (ε = 27 at 250 °C and 5 MPa) which means that the polarity can be controlled by the temperature and pressure applied in the process [27,28]. Therefore, water under SWE has similar behavior to some organic solvents at atmospheric conditions (ε methanol = 33; ε ethanol = 24, at 25 °C), which means that water could potentially dissolve molecules medium and low polarity at SWE conditions, becoming an alternative to organic solvents [29].

2.2. Recent applications for the extraction of plants, seaweeds, microalgae and food by-products

In recent years, the use of green extraction techniques for the recovery of bioactives from food by-products or wastes has greatly increased. In particular, considering food applications of PLE, the number of publications that have used PLE for this purpose has overcome, by far, the rest of matrices (Table 1). The large number of works dedicated to the use of food by-products is closely related to the interest in producing waste-to-value products reducing the environmental impact and achieving a circular economy.

Among the bioactive ingredients obtained by PLE from these by-products, phenolic compound families are the type of compounds that have attracted the highest attention. As mentioned above, the extraction solvent is the main parameter that controls the selectivity of the extracted compounds during the PLE extraction. Grape by-products obtained from winemaking are very complex matrices, containing a great variety of phenolic compounds families. This means that the PLE conditions for the recovery of these compounds have to be carefully optimized. In this sense, in a recent study, the evaluation of the best extraction solvent for the recovery of phenolic compounds, especially procyanidins, from grape pomace with a type 2 diabetes mellitus enzymes inhibition effect was selected [30]. In this study, besides water and ethanol, a less common solvent was used in the PLE process, i.e., glycerol. Although water provided the highest recovery of total phenolic compounds (TPC), the water-glycerol mixture (15% glycerol) at 150 and 120 °C provided the higher extraction of procyanidin monomers and dimers, while 15% ethanol in water extracted higher quantities of trimers and tetramers at 90 °C. This extract showed a significant inhibition of the type 2 diabetes mellitus enzymes, higher than that produced by the drug control. In the same way, Perra et al. [42] performed an individual statistical analysis for each of the phenolic groups present in wine waste including tannins, flavonoids, stilbenes, anthocyanins, proanthocyanins, and phenolic acids. Each group presented a different behavior for the tested PLE conditions. For instance, proanthocyanins were highly affected by the temperature and extraction time, while for phenolic acids the most relevant parameters were the temperature and the ethanol content in the extraction solvent, showing the high impact of the

Table 1
Some relevant applications of the use of PLE for the extraction of bioactive compounds from plants published during the period 2019–23.

Matrix	Compounds of interest	Extraction method	Extraction solvent	Temperature (°C)	Pressure (MPa)	Extraction time (min)	Ref.
Food by-products							
Grape pomace	Procyanidins	SWE	Water Water ethanol (15%) Water glycerol (15%)	90-150			[30]
Sweet cherry pomace	Proanthocyanidins	PLE-EAE	Buffer phosphate (100 mM) pH 10	60	9	31	[31]
Feijoa leaves	Phenolic compounds	PLE	Ethanol/water (50:50, *v/v*)	80	10	50	[32]
Beet leaves	Phenolic compounds	PLE	Ethanol/water (70:30, *v/v*)	25	10	87	[33]
Beetroot	Phenolic compounds	PLE	Ethanol	40	10	90	[34]
Beet leaves and stalks	Phenolic compounds	PLE	Ethanol	40	10	90	[35]
Olive leaves	Phenolic compounds	PLE	Ethanol/water (60:40, *v/v*)	190	11.7	5	[36]
Olive leaves	Phenolic compounds	PLE	Ethanol/water (41:59, *v/v*)	119		15	[37]
Olive pomace	Phenolic compounds	PLE	Ethanol/water (52.3:47.7, *v/v*)	136.5	10.3	20	[38]
Maqui leaves	Phenolic compounds	PLE	Ethanol/water (23:77, *v/v*)	200	10.3	3 min × 3 cycles	[39]
Lemon peel	Phenolic compounds	PLE-SPE	Water-ethyl lactate pH 6 Water-ethanol pH 6 (gradient extraction)	80 (hesperidin and no polar compounds) 50 (narirutin)	10	125	[40]
Lime pomace	Phenolic compounds	PLE-UAE	Ethanol/water (3:1, *v/v*)	110	10	50	[41]
Wine-making product	Phenolic compounds	PLE	Ethanol/water (55:45, *v/v*)	130	11	22	[42]
Passion fruit rinds	Phenolic compounds	PLE-UAE	Ethanol/water (70:30, *v/v*)	60	10	68.54	[43]
Passion fruit bagasse	Phenolic compounds	PLE-UAE	Ethanol/water (75:25, *v/v*)	65	10	300	[44]
Brewers spent grain	Phenolic compounds	SWE	Water	120	10	15 min static + 60 min dynamic	[45]
Pomegranate peel	Phenolic compounds	PLE	Ethanol/water (70:30, *v/v*)	200	10	20	[46]
Pomegranate peel and carpelar membrane	Phenolic compounds	PLE	Ethanol	60	4	10 min static + 76 min dynamic	[47]
Strawberry pomace	Phenolic compounds	PLE	Ethanol Water	90 110	10.3	15 min × 3 cycles	[48]
Acai seeds and pulp pomace	Phenolic compounds	PLE	Ethanol/water (75/25, *v/v*)	115	10	60	[49]
Mung bean seed coats	Phenolic compounds	PLE	Ethanol/water (50:50, *v/v*)	160	9	20	[50]
Bean hulls	Anthocyanins	PLE	Ethanol/citric acid 0.1 M (30:70, *v/v*)	60	10	26	[51]
Berry (*Plinia cauliflora*) wastes	Anthocyanins	PLE-SPE	ChCl:LA (1:2) 35% water	90.2	10.3	15	[52]
Grape marc	Anthocyanins	PLE-membrane separation	Ethanol/water (50:50, *v/v*) pH2	40	10	54	[53]
Saffron processing waste	Anthocyanins and phenolic compounds	PLE-stirring-UAE	Citric acid 1% pH 2.9	120		10	[54]
Black rice bran	Anthocyanins	PLE	Ethanol/citric acid 0.1 M (50:50, *v/v*)	55	10	8	[55]
Chokeberry pomace	Anthocyanins	PLE-UAE	Citric acid 1.5%	70	18	45	[56]
Blueberry pomace	Anthocyanins	PLE	Water/propylene glycol (80/20, *v/v*)	40	10	30 min static + 180 min dynamic	[57]
Orange juice waste	Terpenes	PLE	Ethyl acetate	100	10	30	[58]
Pomegranate seeds waste	Peptides and proteins	PLE	20 mM bicarbonate buffer pH 11	170	10.3	12 min × 3 cycles	[59, 60]
Chia seeds (underutilized)	n3-rich oils	PLS	Ethanol	60	10.3	10	[61]
Algae							
Isochrysis galbana	PUFAs	PLE	Hexane/isopropanol (2:1, *v/v*)	100	11	10	[62]
Isochrysis galbana	FFA, TAG, DAG, MAG, PL	PLE	Ethanol/water (90:10, *v/v*)	80	10.3	5 min × 3 cycles	[63]
Nanochloropsis gaditana	PUFAs	PLE	2-MeTHF/ethanol (1:3, *v/v*)	120	11	15	[64]

(*continued on next page*)

Table 1 (*continued*)

Matrix	Compounds of interest	Extraction method	Extraction solvent	Temperature (°C)	Pressure (MPa)	Extraction time (min)	Ref.
Spirulina, *Chlorella* and *Phaeodactylum*	Phenolic compounds, chlorophylls, carotenoids, minerals	PLE	Water/DMSO (50:50, *v/v*)	40	10.3	15	[65]
Euglena cantabrica	Carotenoids	PLE	Ethanol	40	10	20	[66]
Spirulina	Phenolic compounds, chlorophylls, carotenoids, carbohydrates, proteins	SWE	Water pH 4	40	10.3	10	[67]
Dunaliella salina	Carotenoids	PLE	Ethyl acetate	120	11	15	[68]
Spirulina	Phenolic compounds, chlorophylls, proteins	PEF-PLE	DMSO	40	10.3	15	[69]
Spirulina, *Chlorella* and *Phaeodactylum*	Phenolic compounds, chlorophylls, proteins	SWE	Water	40	10.3	10	[70]
Laminaria ochroleuca	Fatty acids	PLE	Ethanol Ethyl acetate	120	10	10	[71]
Fucus vesiculosus	Phenolic compounds, alginates, fucoidans	SWE	Water	140–190	1	5	[72]
Fucus virsoides and *Cystoseira barbata*	Fucoidans	PLE	0.1 M H_2SO_4	140	10.3	15	[73]
Fucus vesiculosus	Phenolic compounds	PLE	Ethanol/water (58.65/41.35, *v/v*)	137.18	1	4.68	[74]
Cystoseira barbata, Codium bursa, Cystoseira compressa, and *Fucus virsoides*	Fatty acids, pigments, sterols	PLE	Ethyl acetate/ethanol (1:1, *v/v*)	100	10.3	15 min × 3 cycles	[75]
Durvillaea antarctica	Antioxidant and neuroprotective extracs	PLE	Ethanol/water (29/71, *v/v*)	180	10.3	30	[76]
Kappaphycus alvarezii	Phenolic compounds and carrageenan	PLE	Ethanol	60	10	40	[77]
Coffee and tea							
Green coffee beans	Roasted and chlorogenic acid extraction	SWE	Water	167	10.3	14	[78]
Black tea	Gallic acid, caffeine, flavonoids	PLE-SPE	Water and ethanol	80		208	[79]
Mentha pulegium, *Equisetum giganteum*	Flavonoids	PLE	Ethanol/water (50:50, *v/v*) (*M. pulegium*) Ethanol (*E. giganteum*)	40	10	210	[80]
Fruits							
Campomanesia phaea	Phenolic compounds	PLE	Ethanol	74		6	[81]
Strawberry tree fruit	Phenolic compounds	PLE	Ethanol/water (50:50, *v/v*)	120	10	10 min × 2 cycles	[82]
Açai	Anthocyanins	PLE	Methanol/water pH7 (43:57, *v/v*)	81	20.3	2	[83]
Thinned pears	Isoferulic acid, 4-methyldaphnetin, coniferyl aldehyde and 3,4-dihydroxyacetophenone	PLE	Ethanol/water (50:50, *v/v*)	180	10	20	[84]
Opuntia stricta	Betalains and phenolic compounds	PLE	Ethanol/water (50:50, *v/v*)	25	10.34	10	[85]
Fungi							
Tuber aestivum and *Terfezia claveryi*	Carbohydrates, β-glucans, chitin, proteins, phenolic compounds, and sterols	PLE	Water (carbohydrates and phenolic compounds) Ethanol (sterols)	180	16.7	30	[86]
Lentinula edodes	Eritadenine	SWE	Water	142	10.3	5	[87]
Cannabis sativa	Decarboxylation of cannabinoid acids	SWE	Water	140 (CBC, CBD, CBDV, CBG) 120 (THC)	11	6	[88]
Herbs, species, vegetables and seeds							
Cannabis sativa	Phenolic compounds	PLE	Ethanol	100	6	60	[89]
Stevia	Phenolic compounds	PLE	Ethanol/water (70:30, *v/v*)	125	5	30	[90]
Radish seed	Sterols	PLE	Ethanol	120	10	10	[91]
Chia seeds	Tocochromanols	PLE	Hexane	60	10.3	10 min × 2 cycles	[92]
Laurel	Phenolic compounds	PLE	Ethanol/water (50:50, *v/v*)	150	10.3	5	[93]
Garlic	Phenolic compounds, allicin	PLE	Ethanol/water (53.4:46.6, *v/v*)	140	10	3	[94]
Spinach	Chlorophylls	PLE	Ethanol/water (80:20, *v/v*)	75	8		[95]
Rosemary	Carnosic acid, rosmarinic acid, and carnosol	PLE	Ethanol/water (80:20, *v/v*)	183	13	3	[96]

2-MeTHF, 2-Methyltetrahydrofuran; ChCl:LA, choline chloride:lactic acid; DMSO, Dimethyl sulfoxide; EAE, enzyme-assisted extraction; PLE, pressurized liquid extraction; SPE, solid phase extraction; SWE, subcritical fluid extraction; UAE, ultrasounds-assisted extraction.

solvent in the composition of the final PLE fraction.

Anthocyanins are a group of phenolic compounds that have been related to both bioactivities and organoleptic properties of the food, mainly related to the red, purple, and blue colors of berries, currants, and grapes [97]. Several authors have developed PLE methods for the recovery of these interesting compounds from food by-products. A PLE extract rich in anthocyanins with α-amylase and β-glucosidase inhibition activity was obtained from broken bean hulls and was applied as an ingredient for gelatin formulation [51]. The optimal PLE conditions were achieved at 60 °C, using a mixture of ethanol/citric acid 0.1 M (30:70, *v/v*) as extraction solvent. The temperature was the factor that had the highest impact on the anthocyanin extraction and antioxidant activity. Leonarski et al. [55] reported that the use of high temperatures in the PLE process of anthocyanidin extraction produced the degradation of these compounds by their polymerization or hydrolysis. Polymerization may be increased as well by the use of high ethanol concentration. However, the use of 50% 0.1 M citric acid at 55 °C may help to avoid the polymerization of anthocyanins and therefore, improve their recovery [55].

New processes and couplings have been developed for the fractionation of phenolic compounds present in complex by-product matrices. One of these processes consists of the coupling of PLE and solid phase extraction (SPE) using C18 adsorbent for the fractionation of phenolic compounds from lemon peel [40]. The extraction took place using a gradient of solvents, starting with water to extract the most polar phenolic compounds from the PLE which kept retained in the SPE, then ethyl lactate and ethanol were tested for the extraction and elution of medium and no polar compounds. Ethyl acetate enhanced the extraction and desorption of more polar compounds, while ethanol achieved 1.53 times higher extraction of less polar compounds.

Different strategies have been employed for the purification of anthocyanins using online PLE processes. The integration of PLE with a separation membrane was developed to concentrate the anthocyanins and phenolic compounds from grape marc [53]. The membrane technology consisted of a first microfiltration step for the elimination of large particles and a final nanofiltration for the concentration of the compounds of interest. PLE-SPE was also used for the fractionation of anthocyanins from Brazilian berry waste achieving a high extraction yield and purity level after removing other less interesting compounds [52]. In this work, DES were used as extraction solvents and stabilizing agents. A prediction of the best DES for anthocyanin extraction was performed using the COSMO-RS software. Betaine, choline chloride, lactic acid, citric acid, malic acid, 1,4-butanediol, ethylene glycol, and xylitol were screened, being the combination of choline chloride:lactic acid (1:2) with 35% water the best predicted DES with better ecoscale score.

In another study, the PLE extraction of proanthocyanidins from the non-extractable polyphenol (NEP) fraction from sweet cherry pomace was improved by the combination of PLE and enzyme-assisted extraction (EAE) using Promod enzyme, which consists of a mixture of protease and polygalacturonase activities [31]. The PLE-EAE method was more suitable and selective for proanthocyanidin extraction than the PLE alone, obtaining a higher polymerization degree (>8000 Da). Besides EAE, the combination of PLE with other emergent extraction techniques has been reported, for instance, PLE assisted by ultrasound-assisted extraction (UAE) to enhance the extraction of phenolic compounds from defatted passion fruit bagasse [44], lime pomace [41], or passion fruit rinds [43] have been developed. In these works, the temperature was the key factor to increase the phenolic extraction and UAE highly improved the recovery of the interesting compounds in comparison with the use of PLE alone. Besides, PLE coupled with stirring and ultrasound techniques was applied for the efficient recovery of bioactive phenolic compounds from saffron processing waste [54].

The valorization of by-products from food processes implies the extraction of huge amounts of raw material. For this reason, the scale-up of the PLE process is a needed step. In this sense, Arias et al. applied the life cycle assessment (LCA) for the comparison of the greenness of the scale-up of several extraction techniques including PLE, SFE, UAE, maceration, and Soxhlet [35]. For the simulation of the modeling, the authors used the extraction of polyphenols from 100 kg of leaves and stalk beet wastes. The data required for the LCA analysis were obtained from a simulation process with the SuperPro Designer software. The main hotspot that affected the greenness of the processes was the energy consumption, followed by the steam requirement and the use of extraction solvents. As a result, UAE was found to be the less green process, while the techniques with a better environmentally friendly profile were PLE and SFE. PLE was the extraction technique that provided higher antioxidant compounds recovery with a production capacity of 1000 kg antioxidants per year. Additionally, an experimental scale up PLE process was carried out for the phenolics extraction from mung bean seed coats [50]. In this case, a pilot scale PLE extraction of 10 L using 2 kg of mung bean waste material was performed setting 160 °C, 9 MPa, and water-ethanol (1:1, *v/v*) extraction solvent as optimal conditions. In this work, the results of total flavonoids and antioxidant activity obtained under analytical and pilot scales were statistically similar, and the pilot scale process improved the TPC extraction.

Other bioactive compounds different from phenolic compounds have been also extracted using PLE from food waste products. A comprehensive study for the recovery of terpenes from orange juice waste by PLE was developed for the evaluation of the neuroprotective activity of the final extract [58]. The terpene-rich extract with higher neuroprotective activity was obtained by applying 100 °C and 10 MPa for 30 min, using ethyl acetate as extraction solvent. The main compounds related to the observed bioactivity were identified as the monoterpene limonene and some sesquiterpenes such as valencene. Peptides with interesting bioactivities like antioxidant, antihypertensive, and hypocholesterolemic activities from pomegranate seeds were extracted using PLE at 170 °C using three cycles of 12 min and an extraction solvent composed of 20 mM bicarbonate buffer (pH 11) [59,60]. Moreover, n-3 fatty acid-rich oils were also obtained from the PLE extraction of underutilized chia seeds [61]. The oil recovery obtained in this process, using pressurized ethanol at 60 °C, was 100% with a high content of alfa-linolenic acid.

The second most frequent PLE food application reported in the last years is the extraction of valuable compounds from macro- and microalgae. The chemical composition of algae is very complex, presenting a large variety of compounds of interest for health promotion evaluation like n-3 fatty acids, proteins, carbohydrates, carotenoids, chlorophylls, or phenolic compounds. The extraction of these compounds by PLE presents great advantages; in Table 1, the most recent applications for PLE algae extraction with their corresponding PLE settings are summarized.

Lipids from microalgae are one of the most valuable ingredients due to their high content of polyunsaturated n-3 fatty acids [62]. Different solvents have been tested for the PLE extraction of these lipids. A PLE extraction with hexane/isopropanol (2:1, *v/v*) provided an extract rich in docosahexaenoic acid (DHA) from *Isochrysis galbana*. In another study that applied PLE for the extraction of lipids from the same microalgae, ethanol at 90% resulted in an oil extract with a wide composition of lipid families including free fatty acids (FFA), mono-, di, and triacylglycerides (MAG, DAG, and TAG) and phospholipids (PL) [63]. A list of solvents, including 2-methyltetrahydrofuran (2-MeTHF), ethanol, isopropanol, hexane, and isobutane, were tested for the extraction of n-3 PUFAS from the microalgae *Nanochloropsis gaditana*, where the mixture of the bio-based solvents 2-MeTHF and ethanol in a 1:3 (*v/v*) proportion was the most selective and efficient solvent [64]. Hexane, ethyl acetate, and ethanol have been also studied for the extraction and the evaluation of the nutritional value of each lipid extract of the macroalgae *Laminaria ochroleuca* [71]. In this work, ethanol and ethyl acetate produced extracts with a high-quality composition of lipids in terms of n-6/n-3 ratio.

The use of dimethylsufoxide (DMSO) and water has been evaluated

for the recovery of different compounds with high antioxidant activity from the microalgae *Spirulina*, *Chlorella*, and *Phaeodactylum* [65]. The extraction carried out with 100% water was selective for the extraction of proteins while using 100% DMSO, an extract richer in phenolic compounds, chlorophylls, and carotenoids (mainly fucoxanthin, β-carotene, zeaxanthin, and luteolin) was obtained. The recovery of carotenoids from *Euglena cantabrica* was also maximized using 100% organic solvents, in this case, using 100% ethanol [66] while for *Dunaliella salina,* 100% ethyl acetate was optimal [68]. A new extraction process for the recovery of proteins, phenolic compounds, and chlorophylls with antioxidant activity from *Spirulina* consisting of a combined pulsed electric field (PEF) with PLE was developed by Zhou et al. [69]. Compared to the use of PEF and PLE alone, the combination of these two techniques enhanced the extraction yield of all the evaluated compounds thanks to the ability of PEF to break the cell walls, increasing the permeability of the cell membrane of the microalgae. This behavior facilitated the action of the high temperature and pressure used in PLE for the diffusion of the compounds towards the extraction solvent, as can be observed in Fig. 1.

The modification of the polarity and extraction capacity of water under SWE conditions according to the temperature was evaluated for the extraction of different compounds present in the brown macroalga *Fucus vesiculosus* [72]. It could be observed that in a temperature range of 120–200 °C with an increment of 10 °C, the extraction yield, TPC and antioxidant capacity increased with temperature. The maximum recovery of fucoidans was obtained at 160 °C and alginates at 140 °C showing the polarity change of water and its affinity for different compounds according to the temperature increase. According to these findings, another study focused on the optimization of the extraction of fucoidans [73]; the best PLE conditions for the extraction of these sulfated polysaccharides were achieved at similar temperatures (140 °C) with the addition of acid in the extraction solvent (0.1 M H_2SO_4). The polysaccharides extracted under these conditions were characterized to have higher polydispersity index, fucose, and sulfate groups due to the addition of acid that promoted the break between the sulfate esters and allowed the release of more sulfate groups.

Besides by-products and algae, PLE has been used in the last few years to develop novel and interesting applications for different food products. For instance, PLE has been used for the simultaneous roasting and extraction of green coffee beans [78]. The roasting process was studied at different temperatures. The color of the green coffee beans changed when the temperature was increased from 100, 150, and 200 °C from green up to a dark brown color, similar to the color of traditional roasted coffee (Fig. 2). As a quality parameter, the extraction of chlorogenic acid, caffeoylquinic acid, and feruloylquinic acid (some of the most relevant compounds of coffee) was monitored. At 200 °C, similar profiles and concentrations of these compounds of coffee obtained under the traditional roasting process were achieved in PLE as can be observed in Fig. 2 [78]. In another study, a PLE-SPE on-line coupling for the fractionation of gallic acid, caffeine, and flavonoids from black tea has also been developed [79]. In this application, water was firstly used for the extraction and fractionation of gallic acid and caffeine, then, a second step using ethanol was performed for the recovery of the retained flavonoids in the SPE cartridge. The flavonoid fraction obtained in this PLE application was used as an antioxidant ingredient in bakery products.

Fungi are foods that are gaining economic and gastronomic relevance due to their organoleptic characteristics and their biological properties. The extraction of bioactive compounds including carbohydrates, β-glucans, chitin, proteins, phenolic compounds, and sterols from truffles has been performed by PLE [86]. This work reported that the extraction conditions for each compound were different. While water was the best solvent for the extraction of carbohydrates and phenolic compounds, ethanol was more appropriate for the recovery of sterols in the two evaluated species of truffles. The obtained fractions presented good protection against free radical exposure in Caco-2 cells and reduced pro-inflammatory reactions. Eritadine is a cholesterol-lowering component present in the mushroom *Lentinula edodes*. A selective PLE method, consisting of the use of SWE at 142 °C for 5 min, was developed for the extraction of this compound [87].

PLE can be also used for carrying out reactions of compounds to improve their biological activity. This is the case of cannabinoids, which are present as carboxylates in the plant, but they show beneficial effects on human health only after decarboxylation. In this sense, PLE has been proposed as a mild alternative for the common thermal decarboxylation, an aggressive process that produces the degradation of cannabinoids [88].

As a measure of the greenness of the PLE process, an ecofootprint study was conducted for the comparison of PLE and Soxhlet techniques for the extraction of carnosic acid, rosmarinic acid, and carnosol from rosemary [96]. The global processing was evaluated in terms of six principles of green extraction: raw material, solvent, energy, by-products, process, and product recovery. As it can be seen in Fig. 3, values of these principles close to the center of the graph indicate better greenness of the process. PLE could reduce 8-times the extraction time in comparison to Soxhlet as well as PLE required less solvent (50 mL vs. 300 mL, respectively), which highly reduced the production of solvent waste, a big environmental problem. Moreover, in PLE, less amount of raw material was needed for the extraction, and energy consumption was reduced thanks to the high temperature and pressure applied in PLE, that allowed greatly reducing the extraction process time. In total the eco-friendly profile of PLE was 33 times better than the Soxhlet profile.

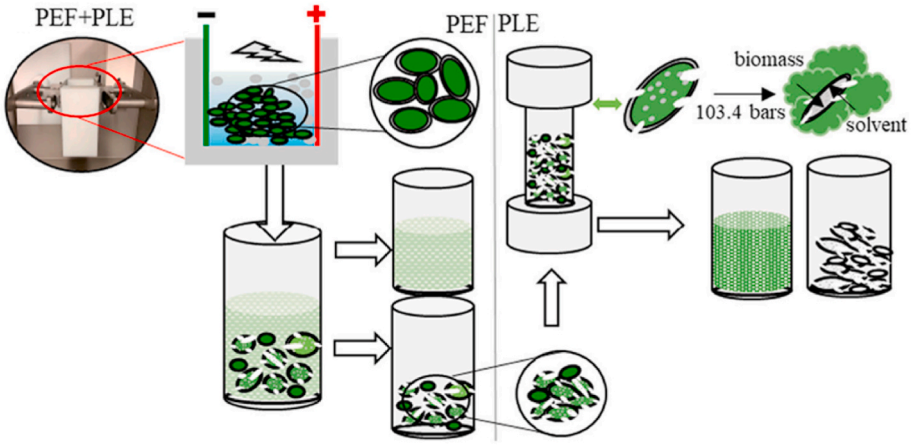

Fig. 1. Schematic diagram of the effect of PEF + PLE on Spirulina and the effect of PEF over the microalga cell membrane that enhances the PLE extraction. Reproduced with permission from Ref. [69].

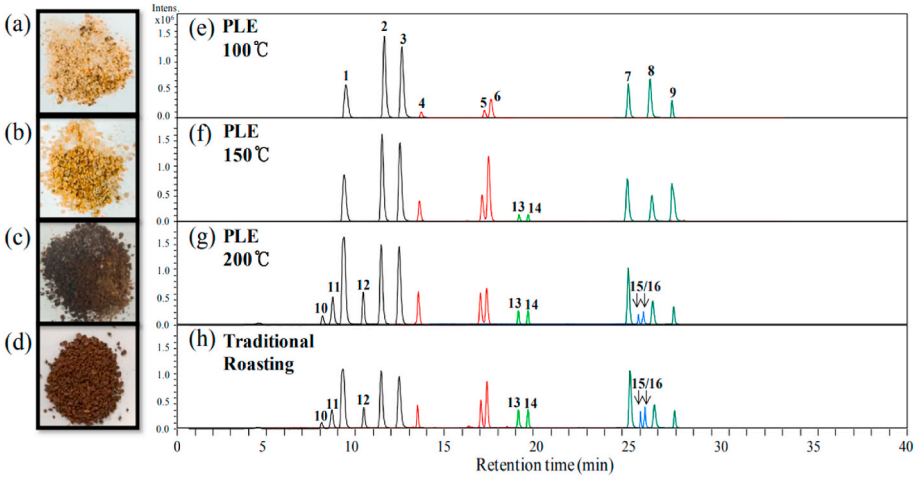

Fig. 2. Coffee bean powder after the different traditional and PLE roasting and extraction conditions and their corresponding chromatograms for 16 chlorogenic acids. (a) and (e): PLE at 100 °C; (b) and (f): PLE at 150 °C; (c) and (g): PLE at 200 °C, (d) and (h): medium-roast coffee extracted by stirring with hot water for 10 min. Reproduced with permission from Ref. [78].

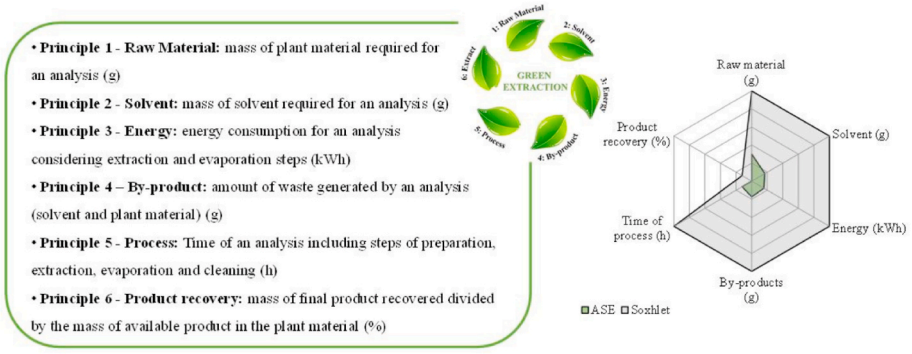

Fig. 3. Principles of green extraction and eco-footprint comparison of PLE vs. Soxhlet for rosemary extraction. Reproduced with permission from Ref. [96]. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3. Supercritical fluid extraction (SFE)

3.1. General aspects

Supercritical fluid extraction (SFE) is one of the techniques most widely regarded as green extraction tools. It is based on the use of solvents at high temperature and pressure conditions over their critical points. Among the advantages that this technique presents, the possibility of obtaining solvent-free extracts is one of the most attractive for the food industry, thanks to the evaporation of the solvent upon depressurization at the end of the extraction [98]. SFE is also capable of increasing the purity and quality of the extracts in comparison with conventional techniques, reducing undesired consequences such as thermal degradation or solvent residues which could be a risk in food industry applications.

This process started to be used in the 1970s in the food industry, taking importance in the caffeine separation, oil and other plant components refining. Its use has been increased in the last decades because SFE provides a green solution for the extraction of natural compounds [99].

Solubility is one of the most important properties to be considered in SFE to enhance the extraction selectivity of target compounds. Depending on the particular interactions between solute and solvent and their corresponding solubility, the temperature and/or pressure must be tuned to increase the extract purity. From a physicochemical point of view, the behavior of supercritical fluids can be defined as intermediate between gases and liquids because the density of supercritical fluids is similar to a liquid, but they present viscosities similar to gases. The most commonly used solvent in SFE is supercritical CO_2 (sc-CO_2) due to the advantageous properties of this compound. Among these properties, its easily attainable supercritical conditions (critical point: 30.9 °C and 7.38 MPa), its higher diffusion rate, and its reduced viscosity allow sc-CO_2 to penetrate in solid matter can be pointed out. Moreover, the high density of sc-CO_2 provides a higher solvation power [100]. Considering this last property, it is well-known that sc-CO_2 density depends on the pressure and temperature. While the density increases with pressure, temperature has an opposite effect, causing the reduction of the density with the increase of this variable [101].

Due to the non-polar nature of sc-CO_2, only non-polar compounds can be solubilized in sc-CO_2. To partially solve this issue, the use of co-solvents or modifiers is necessary to improve the range of compounds with different polarities that can be extracted using sc-CO_2. These are used in low relative proportions, typically below 10%. Among others, ethanol stands out among the used co-solvents because its polar nature makes the mix of solvent and modifier a better extractant of natural bioactive compounds [102], increasing not only the amount of non-polar compounds but also improving the extraction of polar compounds. Therefore, the use of this co-solvent makes possible to increase the extraction of more compounds present in the matrix, resulting in a complex and complete mixture of bioactive compound extract with different properties. Besides, it is widely considered non-toxic at low amounts, may be generated from renewable biomasses, and presents a

good miscibility with sc-CO_2 [103].

3.2. Applications (plants, seaweeds, microalgae and food by-products)

Table 2 summarizes the most relevant works that have recently used SFE for the extraction of plant-based materials. One of the main advantages is the fact that sc-CO_2 could recover non-polar extracts without solvent residues. For example, terpenoids-rich essential oils recovered from orange peel [104], or capsaicin from paprika and pigment-rich oils [105].

In nature, there are a lot of bioactive compounds that can be selectively extracted using sc-CO_2, such as terpenoids or fatty acids. However, there is also an increasing interest on the production of more complex extracts, rich in a variety of potentially bioactive compounds, in a search for multi-activity bioactive extracts. A strategy followed in this sense, is to increase the percentage of co-solvent in the solvent mixture in an effort to improve the extraction of relatively more polar compounds, such as polyphenols. For example, the percentage of co-solvent has been modulated to produce the concentration of different interesting compounds in different polarity steps, from neat CO2 to 30% cosolvent [135]; under these latter conditions, an extract rich in chlorophylls from rosemary was obtained. In other studies, other co-solvents different than ethanol have been applied to extract some specific bioactive compounds, for example, for the extraction of colchicine from autumn crocus. Methanol at a flow rate up to 0.6 mL min^{-1} was used to produce an extract enriched in this compound [106]. Recent studies of some specific plants such as *Lupinus mutabilis* [107] or *Sophora moorcroftiana* [108] have implemented this green technique to obtain extracts rich in alkaloids. *Lupinus mutabilis* bioactives were extracted in only 30 min of extraction, only using 10% ethanol as co-solvent. This reduction of the extraction time, without changing the rest of the extraction conditions (50 °C and 27 MPa of extraction temperature and pressure, respectively), shows the importance of using the correct kind of co-solvent, which could increase the efficiency of the extraction.

Co-solvents are not only used to extract a particular group of compounds, but are also used to increase the amount of compounds that could be extracted in SFE, for example triterpenoids present in trees of Acacia family [109]. The efficiency of extraction obtained from this plant could be improved by replacing ethanol by ethyl acetate as co-solvent, which also enhanced the bioactivity of this extract.

Pressure is another important parameter that can enhance the extraction. Recent studies are focused on obtaining better extracts using high pressure (normally between 10 and 40 MPa) because of the increasing concentration of bioactive compounds observed at these pressures and their greater bioactivity. In this sense, technological developments are capable to provide with higher maximum pressures attainable during SFE. The use of pressures above 40 MPa is sometimes referred as high-pressure SFE or even ultra-high pressure SFE. The use of these higher pressures has been demonstrated to be useful in several applications. For instance, interesting results were obtained for the extraction of *Idesia polycarpa* [110] using 65 MPa as extraction pressure. This high pressure provided an oil more concentrated in γ-tocopherol, α-tocopherol, β-sitosterol and squalene, while possessing less acid value and better antioxidant activity than using lower SFE pressures. This high-pressure SFE extraction is showing an increase at present, providing promising results.

Some applications have explored the potential to extract specific compounds present in some plants like asiaticoside, a triterpenoid that has a very relevant antioxidant bioactivity from *Centella asiatica* [111], or curcuminoids from *Curcuma longa* L [112]. Additionally, *Melissa officinalis* has been studied for years because of its antiviral compounds, which could be extracted only using sc-CO_2 [113]. In the case of *Quercus cerris*, the triterpenoid friedelin needed 5% of ethanol as co-solvent to be extracted [114]. Similarly, 5% of methanol was needed for the extraction of two of furanochromones (khellin and visnagin) present in the plant *Ammi visnaga* [115].

Cannabis is one of the plants where SFE has shown greater advantages to generate a high-value product. For the SFE extraction of cannabinoids, just sc-CO_2 without co-solvents is needed to extract a high amount of tetrahydrocannabinol and other cannabinoids due to the high solubility of cannabinoids in sc-CO_2 [116].

SFE can be used in combination with other techniques. For example, SFE coupled to an ultrasound-assisted extraction step (SFE-UAE), stands out for its improved extraction capacities. In this way, some studies have shown that implementing UAE after the SFE could increase the efficiency of the extraction of polyphenols present *Santolina chamaecypatissus* [119] using 70% ethanol as solvent in the latter case. Even more interesting is the use of ultrasounds simultaneously during SFE extractions. For instance, this type of coupled system was used for the extraction of saponins from agave bagasse (*Agave salmiana*), possessing different interesting bioactivities, using SFE + UAE with 2.3% of ethanol as modifier, producing a 3-fold increase in the recovery of saponins compared to regular SFE [120].

Besides plants, microalgae are probably the second most used type of biomass in SFE in recent years. This fact is due to the high number of bioactive compounds that those organisms may accumulate thanks to specific changes on the growing conditions that can foster the production of specific compounds by microalgae. An important example of this adaptability is the extremophile species *Cocoomyxa onubensis*, a green microalga with high concentration of valuable bioactive compounds that possesses high contents of lutein and polyphenols as well as an important antioxidant capacity. This microalga could change the concentration of these compounds increasing their antioxidant capacity only with little changes in the ambient conditions. In recent studies, *Cocoomyxa onubensis* was studied to obtain an extract using SFE with different gradients of ethanol as co-solvent obtaining high values of antioxidant capacity in its optimum at 70 °C and 40 MPa [121].

N-3 polyunsaturated fatty acid, specifically eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) are other bioactive compounds of great interest and their extraction is favored by SFE. In this sense, the content of n-3 fatty acids from some diatoms such as *Opephora* sp. (a bacillariophyceae microalga) and *Pseudostautosira trainorii*, stands out. Recently these diatoms were studied to obtain a DHA and EPA enriched extract using SFE using ethanol as co-solvent [122]. This extraction, provided a lower fraction of these fatty acids compared to conventional extraction methods, such as Soxhlet using methanol/chloroform and reflux extraction with acetone, but with lower toxicity. Moreover, the extract was free of solvent, thanks to a sequential depressurization into different fractions, allowing its direct use. Diatoms are not the only target to obtain those kinds of fatty acids-enriched extracts, but also *Nannochloropsis* sp. has been studied in the same way [123]; in this case, 10% of pure ethanol was used as co-solvent, although the extraction time required was up to eight times higher compared to the previously commented application [122].

There are also other compounds such as flavonoids or carotenoids present in microalgae whose extraction can benefit from the use of SFE. For example, the use of SFE for the extraction of carotenoids (astaxanthin, lutein, and β-carotene), chlorophylls and phenolic compounds from *Chlorella vulgaris* using sc-CO2 and 10% ethanol as co-solvent, improved the antioxidant capacity of the extract and allowed a 7-fold lower extraction time in comparison to conventional solid-liquid extraction [125]. *Dunaliella salina* stands out because it is able to accumulate large amounts of β-carotene, but not only in the form of all-*trans* isomer but also as diverse *cis*-isomers. Recent studies have shown that SFE extracts of this microalga could have important bioactivity when obtained using 55 °C and 30 MPa, having the concentrated extract in 180 min [126].

Sc-CO_2 has also been reported to extract bioactive compounds present in macroalgae as, for example, *Azolla pinnata*. This seaweed has a large number of antibacterial compounds that could be useful as nutraceuticals. Normally, the extraction technique traditionally used to obtain this kind of compounds is Soxhlet extraction but, new studies

Table 2
Relevant applications of the use of SFE for the extraction of bioactive compounds from plants published during the period 2019–23.

Matrix	Compounds of interest	Extraction method	Extraction solvent (co-solvent, *w/w*)	Temp. (°C)/ Pressure (MPa)	Extraction time (min)	Flow rate	Ref.
Plants							
Autumn crocus (*Colchicum speciosum)*	Colchicine	SFE	CO_2 (4-40% methanol)	35/24.7	60-350	1.5 mL min^{-1}	[106]
Lupinus mutabilis	Alkaloids	SFE	CO_2 (10% ethanol)	50/27	30	13.5 mL min^{-1}	[107]
Sophora moorcroftiana	Alkaloids	SFE	CO_2	50-70/17-31	60-180		[108]
Acacia dealbata	Triterpenoids	SFE	CO_2 (5% ethanol) CO_2 (5% ethyl acetate)	40-80/20–30 80/30 80/33	360	14 g min^{-1}	[109]
Acacia longifolia	Triterpenoids	SFE	CO_2 (5% ethanol) CO_2 (5% ethyl acetate)	40-80/20–30 80/30 80/31	360	12 g min^{-1}	[109]
Acacia melanoxylon	Triterpenoids	SFE	CO_2 (5% ethanol) CO_2 (5% ethyl acetate)	40-80/20–30 80/30 80/32	360	13 g min^{-1}	[109]
Idesia polycarpa	Essential oil	SFE	CO_2	40/35,50,65			[110]
Gotu kola *(Centella asiatica)*	Asiaticoside	SFE	CO_2 (10% methanol)	80/10.5	90		[111]
Curcuma longa	Curcuminoid	SFE	CO_2	65/35	20		[112]
Lemon balm	Antiviral compounds	SFE-SSI	CO_2	40/10	180	0.18 g min^{-1}	[113]
Quercus cerris	Friedelin	SFE	CO_2 (5% ethanol)	60/30	480	11 g min^{-1}	[114]
Khella *(Ammi visnaga)*	Furanochromones (Khellin and visnagin)	SFE	CO_2 (5% methanol)	45/20	240		[115]
Cannabis	Tetrahydrocannabinol and cannabinoids	SFE	CO_2	60/15-32	240-600	40–150 g min^{-1}	[116]
Silverweed	Fatty acids	SFE	CO_2	47/36	126	11.1 mL min^{-1}	[117]
Sinami fruit *(Oenocarpus mapora)*	Polyphenols	SFE	CO_2	60/35	60		[118]
Cotton lavender *(Santolina chamaecypatissus)*	Polyphenols	SFE + UAE	CO_2	40/30	240		[119]
Agave salmiana	Antioxidants	SFE-UAE	CO_2 (2.3% ethanol)	70/70	60	0.5 mL min^{-1}	[120]
Algae							
Coccomyxa onubensis	Antioxidant oil	SFE	CO_2 (0, 25 and 50% ethanol)	30-70/25-55	60	3.62 g min^{-1}	[121]
Opephora sp.	EPA and DHA oil	SFE	CO_2 (10% ethanol)	60/30	40	10 mL min^{-1}	[122]
Pseudostautosira trainorii	EPA and DHA oil	SFE	CO_2 (10% ethanol)	60/30	40	10 mL min^{-1}	[122]
Nannochloropsis sp.	EPA and DHA oil	SFE	CO_2 (10% ethanol)	50/35	480	8 g min^{-1}	[123]
Spirulina (*Arthrospira platensis)*	Non-polar compounds	SFE	CO_2	50/35	300	3000 mL min^{-1}	[124]
Chlorella vulgaris	Chlorophyll, carotenoids, and phenolic compounds	SFE	CO_2 (10% ethanol)	60/25		10 g min^{-1}	[125]
Dunaliella salina	β-carotene	SFE	CO_2	55/30	180		[126]
Azolla pinnata	Antibiotics	SFE	CO_2 (20% ethanol)	60/24-38	3		[127]
By-products							
Ripe bitter melon pericarp	β-carotene	SFE	CO_2	70/39	190	35 mL min^{-1}	[128]
Pomegranate seed	Polyphenols	SFE	CO_2 (15% ethanol, methanol and iso-propanol)	40-60/20-30	20	2 mL min^{-1}	[129]
Mango seed	Polyphenols	SFE	CO_2 (5-15% ethanol)	40-60/11-21	180	10 g min^{-1}	[130]
Tomato seeds	Fatty acids	SFE	CO_2	40-80/10-40	10-30	25–75 g min^{-1}	[131]
Wine industry by-products	Polyphenols	SFE	CO_2 (10% ethanol:water 57:43, *v/v*)	40/8	240	1.67 g min^{-1}	[132]
Broccoli by-product	β-carotene	SFE	CO_2 (7-32% ethanol)	40-80/15-45	80	28-32 g min^{-1}	[133]
Virgin coconut oil	Lauric acid	SFE	CO_2	30-60/20-30	60-90	10 g min^{-1}	[134]
Orange peel	Essential oil	SFE	CO_2 (ethanol, % n.i.)	55/35	30		[104]
Vegetables and herbs							
Paprika	Paprika oil, capsaicin, and pigments	SFE	CO_2	57.04/40	100.89	3000 mL min^{-1}	[105]
Rosemary	Carnosic acid	SFE-SFC	CO_2 (3% ethanol/water 50:50, *v/v*)	25/10	40	3 mL min^{-1}	[135]
Rosemary	Carotenoids	SFE-SFC	CO_2	25/20	20	3 mL min^{-1}	[135]
Rosemary	Chlorophylls	SFE-SFC	CO_2 (30% ethanol)	25/10	30	3 mL min^{-1}	[135]
Rosemary	Rosmarinic acid	SFE-SFC	CO_2 (10% ethanol/water 50:50, v/v)	25/10	50	3 mL min^{-1}	[135]

n.i., not indicated; SFC, supercritical fluid chromatography; SFE, supercritical fluid extraction; SSI, supercritical impregnation; UAE, ultrasound-assisted extraction.

have shown that, using SFE, the extraction yield of these compounds grows by 21.20% using methanol as co-solvent. This extraction technique not only increases the extraction yield but also reduces the extraction time to only 3 h. In contrast, soxhlet would need about 12 h to achieve similar results [127].

The need to valorize food by-products in an environmentally friendly way has revealed that SFE is an excellent tool for the extraction of compounds with high added value from these wastes. One of the most employed by-products are fruit peels and pericarp, which are normally discarded but still rich on bioactive compounds. An example of its use is the extraction of orange essential oil from orange peels [104]. SFE has also been used to obtain a β-carotene-rich extract with great antioxidant activity from ripe bitter melon pericarp without the need for co-solvents [128].

Seeds are other type of food by-products that have a lot of compounds with high bioactivities, such as polyphenols or fatty acids. Pomegranate is one matrix that has been studied for years because of its large number of polyphenols and its seeds have a complex polyphenolic profile also. SFE was applied to obtain an extract rich in these bioactives, ready to use, with a particular composition that can be modulated depending on the co-solvent used [129]. Specifically, ethanol, methanol, and 2-propanol were used as co-solvents, obtaining different profiles with different antimicrobial and antioxidant activity. Other seeds rich on bioactives that have been explored include mango seeds [130] as well as tomato seeds [131].

Finally, oil production-related waste is another by-product that could be useful for further valorization. Some oil refineries produce a lot of waste in their process as, for example, coconut oil industry. This coconut oil waste contains a lot of compounds that could be reused, as lauric acid. A recent study has shown that sc-CO_2 with temperatures between 30 and 60 °C and pressures between 20 and 30 MPa provided extracts enriched in lauric acid from the residue of virgin coconut oil [134].

4. Gas-expanded liquids (GXL)

Extractions using gas-expanded liquids (GXL) can be seen as an additional alternative using compressed fluids to the more widely used PLE and SFE. A GXL is a solvent resulting from the mixture of a compressible gas (most commonly, CO_2) being dissolved in an organic solvent [136]. The new solvent system will present different physicochemical properties compared to the two individual phases alone, thus, opening the possibilities for its application in extraction processes. When the compressible gas employed dissolves in the organic liquid under pressure, the liquid expands volumetrically, forming the GXL. As mentioned, CO_2 is usually the compressible gas of choice due to the same characteristics that make this fluid the most-employed for SFE, i.e, safety, availability, price and greenness [137]. When CO_2 is chosen as the compressible gas, the ability of the liquid phase to dissolve it and, therefore, to be expanded, has to be evaluated. Not all the solvents will be able to dissolve the CO_2 in a similar extent. Those that are not able to dissolve it in relatively high amounts, for instance water, will not produce a viable GXL with significantly different solvent properties. On the contrary, most commonly used organic solvents, including ethanol, are able to dissolve great amounts of CO_2 and will expand in great extent with the corresponding significant change in their physicochemical properties [136]. Under these conditions, the organic liquid expanded with CO_2 can be considered as a switchable solvent, since it can be reversibly converted from one form to another differing in more than one physicochemical property depending on the applied pressure to the system. An additional advantage of GXL is that, normally, an improvement in mass transfer can be obtained compared to classical extraction techniques without necessarily using high temperatures [138].

In spite of the potential of the use of this extraction technique, not many applications have been reported for the extraction of valuable bioactive compounds from natural matrices within the period covered by the present review. In an interesting approach, the possibility of modifying the properties of a pressurized liquid by introducing a gas not only to produce its expansion, but also to foster the formation of cavitation bubbles when operated in combination with ultrasound-assisted extraction, for the extraction of polyphenols from pomegranate peels was studied [139]. In this work, the influence of nitrogen at different pressures (0–1.5 MPa) to produce the expansion of pressurized water, together with the use of ultrasounds at different powers (0–600 W) with diverse system pressures (10–30 MPa) was statistically assessed. It was demonstrated that the use of the expansion gas in combination with ultrasounds was able to improve the recovery of the targeted bioactive compounds from pomegranate by-products over a 20%. However, it was also observed that ultrasound power, amount of expansion gas and system pressure should be closely studied and carefully selected, as the application of higher pressures or ultrasound powers did not always benefit the extraction process [139]. Other reports have also explored the use of CO_2-expanded liquids at low temperature (5–25 °C) combined with sonication for the extraction of highly polar natural pigments [140], as well as to extract lipids from berry seeds [141].

A systematic study was presented studying the addition of 0–90% of CO_2 to different widely considered green solvents, i.e., ethanol and ethyl lactate with a percentage of water for the extraction of three very different bioactive components (β-carotene, α-tocopherol and quercetin) from berry juice production by-products [138]. Moderate temperatures, from 40 to 80 °C at pressures from 10 to 30 MPa were tested. Appropriate experimental design was employed to study the influence of the different variables on the recovery of the bioactive compounds. This way, specific optimum extraction conditions were obtained for each bioactive. For instance, the maximum recovery of quercetin was obtained at 20 MPa and 62 °C, using a mixture of CO_2/ethyl lactate/water (33/54/13, *v/v/v*) as solvent. These extraction conditions allowed a 40 % increase in the quercetin recovery compared to a previously published PLE method. The recovery of the other two studied bioactives was also improved with respect to PLE [138], demonstrating the potential interest of this approach for the valorization of agri-food by-products.

The combination of the extraction conditions involved in GXL with the use of novel solvents, such as NADES, has recently been reported for the first time [142]. Specifically, the recovery of phenolic compounds from soy by-products was targeted and the influence of different percentages of CO_2 (25–75%) into choline chloride/citric acid/water (1:1:11 M ratio) was studied at 10 MPa at 60 °C. The procedure was also compared to the use of a PLE process involving the same NADES as extracting solvent at 120 °C. The use of GXL in this application sought the reduction of the total amount of solvent to be employed in a single extraction procedure, at the same time that viscosity and diffusivity of the compounds to the solvent were favored. Although the recovery of polyphenols from the studied by-products was higher using the PLE process, the potential and usefulness of GXL in combination with NADES was also demonstrated [142].

5. Biorefinery approaches

Biorefinery is defined as the application of sustainable processing of biomass into a spectrum of marketable products and energy [143]. Within this wide concept, a lot of processing approaches can be considered, including also the application of sustainable extraction techniques based on compressed fluids. Moreover, biorefinery processes target the creation of products involving the food industry, pharma, and chemicals, among many others. Therefore, a biorefinery approach may be generated ad-hoc depending on the biomass available as well as on the target or most valuable components that can be generated from this biomass. Moreover, the idea of zero-wastes is also present, as, ideally, the whole biomass would be transformed into different useable products leaving no side-streams or wastes and generating co-products instead of by-products. The application of biorefinery approaches including the use of compressed fluids-based technologies is starting. Considering the characteristics of those extraction techniques, previously described, the

potential for their coupling in biorefineries is excellent [12]. However, there are not too many published papers under the scope of the present review, which clearly gives an idea of the still immature state of this concept.

An approximation to this approach has been presented for the valorization of microalgal biomass [144]. Microalgae represent an excellent starting biomass for biorefinery purposes, considering their characteristics, chemical composition as well as for the possibility of culturing strains with no other use today [145]. *Porphyridium cruentum* microalgae represents a clear example of a natural biomass with great potential for the development of different products. *P. cruentum* is a red microalga presenting in its chemical composition a wide array of interesting bioactive components of diverse chemical nature, including carotenoids, unsaturated fatty acids, sulfated polysaccharides, and proteins, specifically B-phycoerythrin, which possesses a high value for biomedical applications. For this reason, the development of biorefinery-like procedures able to produce an effective fractionation is interesting. In this context, the use of different compressed fluids has been explored, using ethanol and water as pressurized solvents to obtain different fractions from the biomass enriched on the particular bioactives. A three-step approach was finally optimized, thanks to the use of an experimental design and statistical optimization. The extraction cascade started with a SWE at 25 °C mainly directed to the extraction of phycoerythrin, followed by a second SWE at higher temperature able to recover a higher amount of polysaccharides. Lastly, a third extraction process was applied to the remaining biomass in order to preferentially extract carotenoids, using ethanol at 125 °C [144]. It has to be noted that, depending on the sample and the bioactive compounds targeted, the direction of the cascade extraction might need to be inverted. The final result has to consider the heat sensitivity of the components, since the chance of producing a degradation of the compounds that are not extracted during the first processes is high. For this reason, the combination of techniques, working at different pressure conditions, going from pure liquids (PLE) to supercritical fluids but also taking advantage of the intermediate points (GXL) retains a very good potential.

Other possibilities are related to the use of a single compressed fluid-based extraction procedure within a wider processing scheme using other technologies. For instance, the use of SWE was combined with bleaching and acid hydrolysis to produce hemicelluloses and cellulose nanocrystals from rice husk, an important agricultural by-product [146]. SWE allowed the recovery of arabinoxylans at 160 °C and pH 7. Besides, even using relatively high extraction temperatures, the SWE process was demonstrated to preserve the antioxidant and antibacterial activities of the extracted arabinoxylans that are lost during a conventional alkaline extraction process. This tool has also demonstrated its potential when employed together with hydrothermal carbonization from spent coffee grounds to produce a bioactive extract rich in antioxidants, a protein isolate and a solid fuel [147].

6. Conclusions and future outlook

As described in this review, the use of compressed fluids-based extraction technologies for the recovery of natural bioactive compounds is moving forward from emerging/promising tools to properly settled techniques in the field. In any case, their expansion in the near future is foreseen. There are several important aspects that will surely help to this expected increase in their use, that are listed below:

i) The recovery of potential bioactive compounds is obviously linked to the selected natural source. In this regard, future efforts on identifying new interesting species will bring new advancements in the field, as novel biomasses will be the aim of study. Improvements in bioprospecting of species, both terrestrial and marine, will influence the potential results. In fact, underused biomasses could be the key for future successful implementations. Moreover, new agrifood by-products will be explored as efforts on circular economy and zero-waste approaches will push the research on ways for their valorization. Lastly, the cultivation of new microalgae strains could also provide new and interesting but yet unexplored biomasses.

ii) The use of green solvents, such as NADES, is opening its way in every application. The first applications and studies that have demonstrated their potential are related to conventional extraction processes. For this reason, their use under high pressure conditions will also be explored in the following years. Although some advancements have been already done in this field [142], there is plenty of room for improvements. A lot of research is still needed to perfectly untap the potential that novel solvents, such as biobased solvents and NADES, may have when used under compressed conditions. As a common point to other applications of these solvents, relevant issues related to solvent evaporation and recycling have to be assessed in the future. A further alternative that can be included in this point is the use of switchable solvents. The smart use of these solvent systems may provide new ways for the isolation and recovery of interesting bioactive fractions from natural matrices without compromising the subsequent use of the remaining biomass.

iii) An increased number of couplings between compressed fluids-based techniques and other extraction technologies are also expected in the following years. Some studies have already been presented coupling a compressed fluid with the use of enzymes or ultrasound [138,148], for instance. However, the potential of these approaches has not completely been explored. In fact, intensification of processes may be a good tool for the development of sustainable processing for the attainment of natural bioactive compounds. The combined use of ultrasounds, microwaves, pulsed electric fields, among other widely considered green extraction technologies may offer enhanced results.

iv) As can be observed from this overview, in the recent years the works dealing with scale-up of processes and with the implementation of biorefinery approaches are scarce. As the fundamental research grows constantly, it is expected that this kind of research will also increase. The further development of the biorefinery concept, integrating the use of compressed fluids, may help to solve some of the challenges that our society has ahead for the true implementation of circular economy. The full valorization of selected biomasses into valuable products is a feasible possibility that has to be undoubtedly explored. These studies should also include proper life cycle analyses and techno-economic assessments to fully demonstrate that the proposed processes can be implemented in real-life conditions producing significant improvements from a sustainability point of view.

v) Lastly, the particular development of each compressed fluids-based tool is expected. Improvement in technology can make the instruments more available worldwide, increasing the chances to find proper applications. Other applications of compressed fluids closely related to the extraction of natural bioactive compounds, such as those devoted to encapsulation or particle formation have also the potential to produce excellent results when coupled to the extraction techniques. These latter developments would also help to close the gap between the research and the market, producing ready-to-use ingredients based on natural bioactive compounds that could be employed in a wide range of applications.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgements

V.M.A.L. acknowledge the financial support from the Spanish Ministry of Universities for the predoctoral contract in the "Formación del Profesorado Universitario" grant (FPU21/00054). L.M. acknowledges a "Ramon y Cajal" grant RYC2021-033148-I funded by MCIN/AEI/10.13039/501100011033. This work was supported by grants PID2020-113050RB-I00 and PDC2021-120814-I00 funded by MCIN/AEI/10.13039/501100011033 (Spain) and by the "European Union Next-GenerationEU/PRTR".

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