

Review

Green Extraction Techniques of Bioactive Compounds: A State-of-the-Art Review

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Abstract: Green extraction techniques are more and more relevant due to major sustainable goals set by the United Nations. Greener extraction processes are being designed through the use of unconventional extraction techniques and green solvents, resulting in less hazardous processes which, consequently, reduces environmental impacts. This is also in line with the main principles of green chemistry. Additionally, greener extraction techniques intend to solve different drawbacks that are often related to conventional extraction techniques such as the high environmental impact. Biorefineries are a major player in developing greener extraction processes. These facilities take full advantage of several biomass sources, such as food waste, microalgae, and lignocellulosic biomass, in order to create high-value products, energy, alternative fuels, and bioactive compounds. Herein, a state-of-the-art review is presented, focused on presenting the greenest and least hazardous extraction processes that have been reported on the main biomass sources of a biorefinery—food waste, microalgae, and lignocellulosic biomass. Bioactive compounds such as phenolic compounds, bioactive pigments, and fatty acids are important in several sectors, mainly, the health, pharmaceutical, and agro-food sectors. Moreover, the bioactive compounds obtained through the aforementioned biomass sources and the different extraction procedures used will be presented and the authors will attempt to discuss, compare, and provide information about the most effective extraction techniques for each compound. Therewith, this review article should serve as a guide for industries, academics, and biorefineries in the future development of optimized and greener extraction procedures. Such analysis is lacking and could be very helpful for future research biorefinery projects since it tackles all of the major biomass sources of a biorefinery in a review article. To the best of our knowledge, this brings a novelty to the scientific community.

Keywords: antioxidant; bioactive pigments; bioeconomy; green chemistry; phenolic compounds



Citation: Martins, R.; Barbosa, A.; Advinha, B.; Sales, H.; Pontes, R.; Nunes, J. Green Extraction Techniques of Bioactive Compounds: A State-of-the-Art Review. *Processes* **2023**, *11*, 2255. <https://doi.org/10.3390/pr11082255>

Academic Editor: Maria Angela A. Meireles

Received: 29 June 2023

Revised: 19 July 2023

Accepted: 24 July 2023

Published: 26 July 2023



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1. Introduction

The 12 principles of green chemistry [1], as well as the rising academic and public interest in non-hazardous compounds [2], have been the main drivers of innovation in extraction techniques. A green extraction method or technique is based on the design of an extraction process that decreases energy consumption, permits the use of new-generation solvents, reduces waste by converting it into co-products, and assures a safe and high-quality final product [3,4].

The significance of greener extraction methods has become increasingly apparent due to the United Nations (UN) prioritizing a more sustainable future. As part of these efforts, studies focused on implementing environmentally friendly extraction processes that efficiently convert diverse biomass sources into valuable products such as bioactive compounds and biobased materials have been conducted in recent years. By doing so, waste generation is minimized, environmental impact is reduced, and the economic value of

various biomass sources is enhanced. This holds particular importance for underdeveloped nations [5].

The aforementioned extraction methods produce vital bioactive compounds from different biomass sources (i.e., microalgae species, food waste, and lignocellulosic sources) that reportedly have multiple health-promoting properties [6,7]. Bioactive compounds like polyphenols, vitamins, and fatty acids have attracted great attention due to their role in the prevention of several chronic diseases [8]. Furthermore, bioactive pigments or phytochemicals such as chlorophylls, betalains, carotenoids, phycocyanin, and anthocyanins also have great antimicrobial, antioxidant, and immunologic properties that are of great interest in the pharmaceutical, food, and materials consumer sectors [9,10].

To provide some context, several reviews [11–14] have already covered different innovative extraction methods from different biomass sources, including mainly food waste and microalgae. The number of reviews focused on extraction techniques from food waste, microalgae, and lignocellulosic biomass is not surprising given the great potential of these sources. For instance, microalgae are autotrophic microorganisms that produce high-value compounds such as polysaccharides, polyunsaturated fatty acids (PUFAs), and bioactive pigments like carotenoids (mainly lutein, zeaxanthin, and astaxanthin) [15,16]. On the other hand, food waste has also gained interest in society due to the higher global population and the consequent rise in food waste generation [17]. Food wastage such as peels, seeds, rind, and pulp are generated and often discharged into landfills, thus causing a significant economic and ecological burden due to greenhouse gas emissions (GHGs) that can contribute to climate change [12,18,19]. To solve such issues, researchers have tried to develop innovative outlets for these wastes, mainly related to energy and the extraction of bioactive compounds [11,12].

Lignocellulosic biomass is another great biomass source since it is considered one of the most abundant sources of bioenergy and biobased products. Lignocellulosic biomass includes various agricultural residues such as bark, branches, logs, and leaves [20] from deciduous and coniferous trees and waste from the pulp and paper industry [21,22]. Lignin—a main component of lignocellulosic biomass—may undergo a reversed process of depolymerization, with the release of some important polyphenolic components classified as aromatic aldehydes (vanillin, syringaldehyde), hydroxybenzoic (vanillic, syringic) acids, and hydroxycinnamic (p-coumaric, ferulic) acids [7].

Up to this moment, the reported extraction studies have mainly focused on the use of non-conventional extraction techniques such as microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), pressurized liquid extraction (PLE), pulsed electric fields (PEF), and supercritical fluid extraction (SC) in the extraction of rich bioactive compounds from different biomass sources (i.e., food waste, microalgae, and lignocellulosic sources) [14]. These techniques have been distinguished from conventional techniques such as maceration and Soxhlet due to the use of more sophisticated equipment and higher efficiency in the extraction of several compounds of interest in less time [23]. Nonetheless, in this review, all of these techniques will be addressed for their potential in the extraction of bioactive molecules, taking into account the solvents used.

Solvents are usually employed in order to create a mass transfer of the targeted bioactive molecules [24]. These solvents may bring some issues due to their toxicity and flammability; therefore, it is important to choose the right solvent to be used in the extraction process. Factors such as cost, biocompatibility, and efficiency in extraction must be considered when designing an extraction process [25].

To aid the development of greener extraction methods, academia and industries have spent time and effort in developing tools such as life cycle assessment (LCA) [26], EcoScale analysis [27], quantitative structure-activity relationship (QSAR) [28], National Environmental Methods Index (NEMI), Green Analytical Procedure Index (GAPI), and Analytical GREENness metric (AGREE) [29]. For instance, QSAR could be used to predict solvent ecotoxicity [30]. This poses a great advantage since solvent ecotoxicity could reflect

negatively on a process's overall environmental impact [31]. LCA emphasizes delivering a quantitative analysis of the environmental impacts of material conversion processes.

Therefore, in this state-of-the-art review, we propose an update for existing green extraction technologies, giving a more focused outlook on the use of green solvents coupled with green and innovative extraction techniques. Moreover, we cover three of the major biomass sources of a biorefinery—food waste, microalgae, and lignocellulosic biomass—which, to the best of our knowledge, have still not been addressed by the scientific community.

2. Green Solvents

In recent years, environmental directives and legislation have sought to reduce solvent emissions or regulate the usage of potentially harmful or environmentally damaging chemical substances [32]. However, many existing chemical processes still depend heavily on harmful and toxic solvents. This paradigm is worse in developing countries because of the attractive prices of toxic solvents and their availability. This is a clear case of neglect whereby economic factors are favored above sustainability.

Nonetheless, in recent years, there have been more and more studies focused on the use of green solvents in chemical processes, with publications rising beyond the 10,000 mark from 2010 onwards in ScienceDirect when the term “green solvents” was searched.

Green solvents (Figure 1) can be defined as chemicals that minimize the environmental impact resulting from their use in chemical processes and production [33]. This concept is part of green chemistry, which can be defined as the design of chemical processes that can reduce to eliminate the use and generation of hazardous substances [3]. To provide the readers with a better understanding, a green solvent can be defined as a solvent that possesses one or more of the following properties: low or non-volatility, nonflammable, no inhalation hazards (non-toxic, non-carcinogenic), able to be recycled, and biodegradability [34]. Furthermore, they possess an array of different physical and chemical characteristics that make them suitable for different types of extraction techniques.

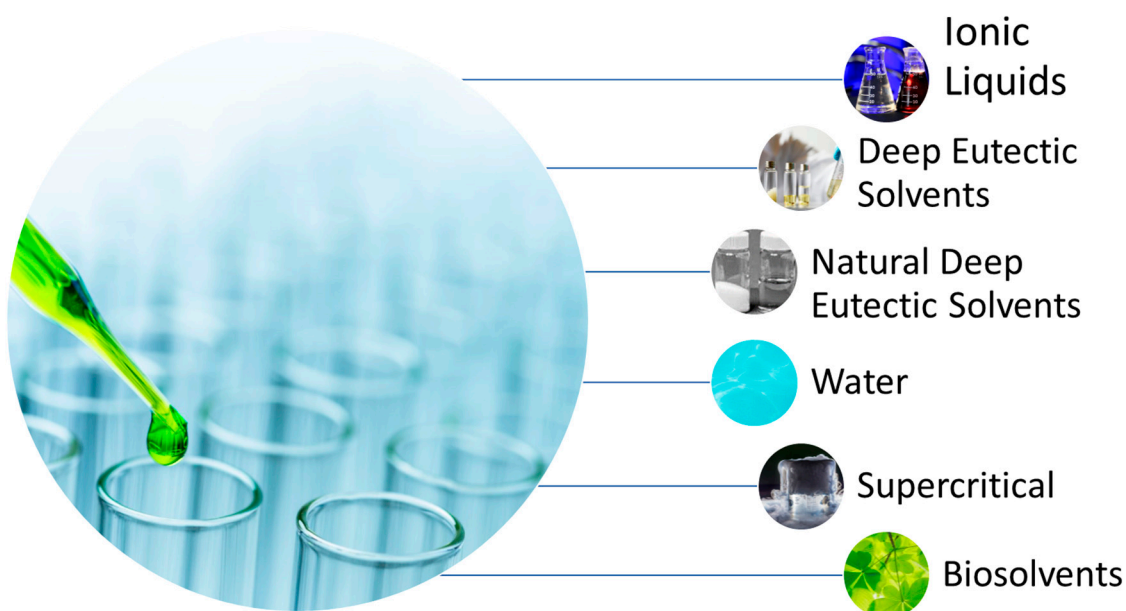


Figure 1. Types of green solvents.

For instance, supercritical solvents are substances that are readily accessible at their critical points. Supercritical carbon dioxide is one of the most used supercritical solvents due to its safety and renewability [32]. Typically, supercritical solvents exhibit high diffusivities similar to the gas state. However, co-solvents such as ethanol are usually required to increase the solubility of solid reagents and products as the polarity of pure supercritical carbon dioxide is very low [32].

Ionic liquids (ILs) are pure compounds composed of ions [35], which present desirable thermodynamic properties such as thermal stability, adjustable viscosity, miscibility, solubility, and extraction capacity for an array of different compounds with distinct polarities [4].

As an alternative to ILs, deep eutectic solvents (DESs) have emerged since they present similar thermodynamic properties to ILs but are more easily synthesized, less detrimental to the environment, and present lower toxicity. DESs are formed by a hydrogen bond acceptor (HBA), such as quaternary ammonium, with a hydrogen bond donor (HBD), such as urea, carboxylic acids, or ammine [14]. When natural components are used for DES synthesis, usually for plant primary metabolites (e.g., sugars), they are called natural deep eutectic solvents (NADESs). Since these solvents are synthesized from natural components, which are inexpensive, abundant, and recyclable materials, they are seen as non-toxic solvents, making them highly compatible with food, pharmaceutical, and cosmetic formulations and use.

Water is considered the most natural of solvents and many researchers have considered water as the greenest solvent in chemistry both from an experimental and an industrial perspective. In recent decades, a new concept has also surfaced, which is switchable water. Switchable water is obtained by adding a base soluble (e.g., N,N,N',N'-tetramethylbutane-1,4-diamine) to water [36]. This enables “the switch”, which consists of the addition or depletion of CO₂ to monitor the ionic strength of an aqueous solution. At the end of the extraction process, it is possible to remove the base from the water solution to make it clean and safe once again [36], making it a great solvent option for the extraction of some polar compounds.

Biosolvents are solvents based on natural ingredients that have been developed to offer an alternative to fossil resources [37]. The major classes of biosolvents are (1) esters of natural organic acids (e.g., ethyl acetate and ethyl lactate), (2) fatty acid esters, (3) bioethanol, (4) terpenes compounds (e.g., eucalyptol, limonene, and others), (5) isosorbide, and (6) glycerol derivatives. These solvents are also considered green solvents since they are environmentally friendly and not harmful to humans [38–40].

3. Extraction Methods

3.1. Conventional Methods

3.1.1. Maceration

Maceration (Figure 2) is a conventional extraction technique that has been extensively used in laboratories and industries to obtain a variety of compounds from different types of matrices [18]. It can be divided into three steps: (1) the sample is ground into fine particles to increase the surface area of the chosen solvent (also called menstruum); (2) the grounded solid material is placed into a closed vessel and completely covered by the solvent during a set period of time where an additional heat source may be used in order to increase the mass transfer and targeted compound diffusion; and (3) the liquid extract is strained off either through sieves or a net and the solid residue is pressed or the extract might also be centrifuged in order to recover the supernatant and remove the pellet [23,41].

The resulting extracts obtained from maceration are usually filtered to remove impurities [42]. The final extract can be concentrated by evaporation. This method is inexpensive and very suitable for thermolabile compounds since the temperature can be adjusted accordingly [41]. On the other hand, it can consume high amounts of solvent depending on the amount of sample used in the extraction and it may need to be performed over long periods of time (from hours to weeks) until high extraction efficiency is obtained [18].

Feed and Food Waste and Non-Compliance

According to the Food and Agricultural Organization (FAO), the world's vegetable and fruit production went from 1194 million tons in 2000 to 2015 million tons in 2020 [43], where four species accounted for 25% of the total production in 2020: tomatoes (9%), onions (dry) (5%), bananas (6%), and watermelons (5%) [43]. Since these cultivars constitute a great percentage of the total world food production, they will be the main focus of this review.

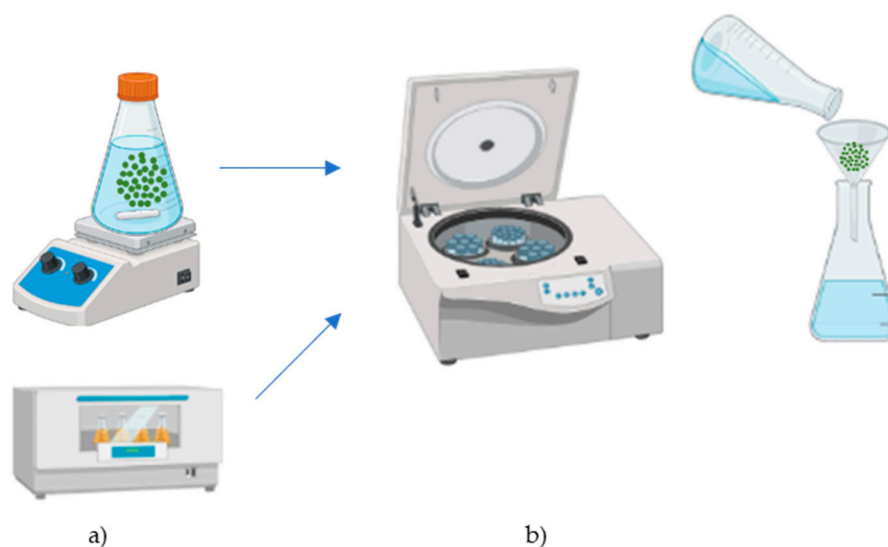


Figure 2. Schematic diagram of maceration extraction: (a) intermittent or continuous agitation; (b) centrifugation and/or filtration.

Table 1 summarizes different studies on the maceration extraction of bioactive compounds using green solvents.

Kehili et al. [44] performed the extraction of lycopene from tomato peels using refined olive oil as a solvent, obtaining a 99.3% overall yield. Lycopene is a red carotenoid abundantly found in tomato peels that presents great antioxidant properties [45]. Like other carotenoids, lycopene is an oil-soluble pigment [40]; thus, the use of vegetable oils as solvents to extract this compound can be an excellent alternative to replace organic solvents because it can produce an extract without contaminants, eliminate the extra cost of evaporation, and retard the oxidation and degradation rates of lycopene [40]. Although interesting, lycopene yield (0.123 mg/g) using refined oil fell short of the results presented by Zuorro et al. [45] (0.856 mg/g), where Ethyl lactate: Ethanol was used as the solvent in the maceration extraction of tomato peels. The use of this solvent showed higher yields of lycopene and it was also possible to achieve such yields during less time and with lower temperatures when compared to the results reported by Kehili et al. [44].

Onion and banana wastes are also rich in phenolics and flavonoid compounds (Table 1). Compared with the onion bulb/flesh, the highest concentrations of phenolics and flavonoids have been observed in onion peel [46]. Stefou et al. [47] developed a sodium propionate-based DES for polyphenol extraction from onion solid wastes, obtaining a total phenolic content (TPC) of 171 mg gallic acid equivalent (GAE)/g, which was higher than the results reported by Milea et al. [48] using water extraction.

Alwazeer et al. [49] applied hydrogen-rich water (HRW) to extract several phenolic compounds from tomato peels (Table 1). The solvent was prepared by bubbling pure hydrogen gas into water for 3 min at 1 L/min. Due to its reducing properties, small size and density, and high diffusion rate in tissues, this type of solvent appears to be beneficial for extracting and preserving several biocompounds from different sources of biomass [49]. The results obtained showed that total phenolic and flavonoid compounds as well as antioxidant capacity are significantly higher in the HRW than in water [50]. However, there are still issues regarding its safeness, especially at an industrial scale, due to its flammability in air (4–75% *v/v*) and detonation limit (18.3–59%). One option is to dilute the H₂ by N₂ to give a safe gaseous mix [49].

Romdhane et al. [51] investigated the optimal conditions for the hot water extraction of polysaccharides from watermelon rind. The optimized parameters resulted in an extract rich in polysaccharides (yield = 34.4%), where galactose was the dominant sugar. The extracted polysaccharides showed fat-binding abilities, foaming properties, and emulsion capacities. Moreover, they also showed important antioxidant activities in vitro and

the potential to inhibit the angiotensin I-converting enzyme. These results showed that the extracts obtained are very promising and could be incorporated into different food formulations to improve their functional and biological activities.

Table 1. Summary of studies reported on maceration extraction of different food waste sources.

Biomass Source	Maceration Conditions	Yield	Ref.
Tomato peels	Ethyl lactate:ethanol (0.667:0.333), 40 °C, 30 min, solvent to biomass ratio (SB): 1:30 (<i>w:v</i>), 350 rpm	Lycopene (0.856 mg/g DW)	[45]
Tomato peels	Refined olive oil, 80 °C, 30 min, solvent to biomass ratio: 2.5% (<i>w:v</i>), 400 rpm	Lycopene (0.123 mg/g DW)	[44]
Tomato peels	Hydrogen-rich water, 25 °C, 24 h, SB: 1:20 (<i>w:v</i>), 120 rpm	Gallic acid (7.89 µg/g extract) Chlorogenic acid (1.11 µg/g extract) Caffeic acid (1.69 µg/g extract) Catechin (41.12 µg/g extract) Rutin (4.49 µg/g extract)	[50]
Onion peels	Hot water, 70 °C, 2 h, SB: 4:100; 500 rpm	Phenolics (21.24 mg GAE/g DW) Flavonoids (20.75 mg QE/g extract)	[48]
Onion waste	Glycerol:sodium propionate, 80 °C, 150 min, 900 rpm, SB: 1:100 (<i>w:v</i>)	Phenolics (137.50 mg GAE/g DW)	[47]
Banana peels	Ethanol 50% (<i>v/v</i>), 40 °C, 20 h, SB: 3:40 (<i>w:v</i>)	Phenolics (28.41 mg GAE/g) Flavonoids (19.07 mg QE/g) Polysaccharides (34.4%)	[52]
Watermelon rind	Water, 60 °C, 80 min, SB: 1:10 (<i>w:v</i>)	(Galactose, arabinose, glucose, galacturonic acid, mannose, rhamnose, xylose, and glucuronic acid)	[51]

Microalgae

Despite being a simple method that is attractive for industrial scale-up, maceration lacks the stimuli to rupture microalgae cells; hence, it can become a time-consuming extraction method when used in microalgae bioactive compound extraction. Thus, it is not used as often as other extraction methods for this biomass source [53]. To further this point, as will be further mentioned in the UAE and MAE extraction sections of this review, several articles have reported these methods' effectiveness in cell-wall disruption, thus enhancing the solvent penetration and overall extraction of the desired molecules.

Table 2 provides an overview of the maceration extraction of different microalgal strains, solvents used for extraction, extraction conditions, extracted products, and yields obtained.

Table 2. Summary of studies reported on maceration extraction of microalgae species.

Biomass Source	Maceration Conditions	Yield	Ref.
<i>Spirulina platensis</i>	Ethanol, 48 h	Flavonoid: 2.68%	[54]
<i>Phaeodactylum tricornutum</i>	Ethanol, 30 min	Fucoxanthin: 15.71 mg/g DW	[55]

Dianursanti et al. [54] aimed to extract flavonoids from *Spirulina platensis* through maceration, using ethanol as a solvent. As a part of the phenolic compound group, flavonoids are natural substances that can be used in several applications such as nutraceutical, pharmaceutical, medicinal, and cosmetic uses due to their antioxidant, anti-inflammatory, and antiallergic properties [54]. The results reported by Dianursanti et al. [54] for the maceration of *Spirulina platensis* for flavonoid extraction (2.68%), although interesting, are far less promising than those reported (5.26%) by the same authors for Soxhlet extraction using the same solvent (ethanol). It is important to note that when extracting flavonoids (phenolic compounds), a polar solvent is required to dissolve and separate them from cells. Among the various solvents used for flavonoid extraction, such as methanol, ethanol, and water, ethanol is preferred as it is safer compared to methanol and more easily separated than water [54].

Fucoxanthin is another compound of great interest that can be extracted from microalgae *P. tricornutum* [55] through maceration with ethanol. This marine carotenoid has been found to have a number of therapeutic activities, including anticancer, antihypertensive, anti-inflammatory, and anti-obesity effects. In the study, acetone, ethanol, water, n-hexane, and ethyl acetate were tested, and ethanol yielded the highest fucoxanthin content (15.71 mg/g DW). When compared to MAE [56], although maceration presents higher yields (1.57% compared to 0.46%), MAE achieves extraction in just 2 min compared to maceration's 30 min of extraction time.

Lignocellulosic Biomass Sources

Many studies (Table 3) have evaluated the use of wood and forest residues as sources of bioactive molecules [57]. Wood extractives are a complex mixture of compounds, among which phenolic compounds, terpenoids, alkaloids, terpenes, and saponins stand out [57].

Pinus pinaster, also called maritime pine, is the conifer occupying the most extended areas in European and Asian forests, and it is the species with the most extended dissemination west of the Iberian Peninsula, where it covers more than 28% of the total forest area [58]. From its wood, bioactive compounds such as catechin can be extracted by maceration. Such was reported by Meullemiestre et al. [59], where the authors used maceration to valorize sawdust from *Pinus pinaster*, obtaining polyphenols such as catechin. Catechins have been studied for their number of promising bactericidal effects on both Gram-positive and Gram-negative bacteria, including multidrug-resistant strains [60]. Additionally, these molecules have shown inhibitory virulence factor activity, particularly toxins, thus reducing the pathogenicity of certain bacteria.

Other interesting studies have also found several important phenolic compounds through the maceration extraction of several lignocellulosic sources. For instance, ethanolic extracts from the maceration of *Robinia pseudoacacia* L. wood showed high levels of two flavonoids (robinetin and dihydrorobinetin) [61]. Moreover, flavonoids, polyphenols, and tannins were reported in ethanolic extracts from *Populus nigra* L. wood after 1 h of maceration extraction [62]. Martínez-Gil et al. [63] showed that the maceration of sawdust, using a hydroalcoholic solution as a solvent, was able to obtain from *Quercus humboldtii* Bonpl. toasted wood the following substances: 5-methylfurfural, guaiacol, trans-isoeugenol, 4-vinylguaiacol, cis-isoeugenol, syringol, furfural, 5-hydroxymethyl-furfural, cis- β -methyl- γ -octalactone, vanillin, eugenol, and trans- β -methyl- γ -octalactone.

These studies are highly relevant since they shed light on the use of a simple extraction technique for obtaining valuable bioactive compounds—mainly phenolic compounds—from several lignocellulosic sources that are available in different parts of the world.

Phenolic compounds (such as those present in the ethanolic extracts of lignocellulosic sources) have the capacity to inhibit reactive oxygen species (ROS), which presents great advantages for the inclusion of such compounds in dermal products [14]. These compounds have reported anti-inflammatory, anti-proliferative, anti-tumor, and cardioprotective properties that should be considered in several health-promoting activities.

Later in this review, the maceration technique will be compared to other unconventional techniques.

Table 3. Summary of studies reported on maceration extraction of lignocellulosic biomass sources.

Biomass Source	Maceration Conditions	Yield	Ref.
<i>Pinus pinaster</i>	Acidified water, SB: 1:17 (<i>w:v</i>), 40 °C	Yield of catechin: 2.34 mg/g of wood	[59]
<i>Robinia pseudoacacia</i> L.	Ethanol (80%), 250 rpm, 4 h	Flavonoids: 3670 mg/L	[61]
<i>Populus nigra</i> L.	Ethanol (70%), SB: 1:12 (<i>w:v</i>), 1 h	Total phenolic content (TPC): 334.87 mg of GAE/g extract	[62]
<i>Quercus humboldtii</i> Bonpl.	Ethanol (70%), SB: 1:5 (<i>w:v</i>) RT, 1 h	Yield: 1.09% TPC: 270.41 mg of GAE/g extract	[63]
<i>Eucalyptus globulus</i> Labill.	Ethanol, SB: 1:10 (<i>w:v</i>), 50 °C, 90 min	TPC: 85.71 g of GAE/100 g extract	[64]

3.1.2. Soxhlet

In 1879, Franz Ritter von Soxhlet invented an extraction technique that is now well-known in academia and industries as the Soxhlet technique [18]. Before the Soxhlet procedure, the sample/biomass to be used in extraction was normally homogenized, ground, pre-dried with an anhydrous substance, and weighted [65]. After sample preparation, the Soxhlet apparatus must be set.

The experimental Soxhlet extraction apparatus (Figure 3) consists of 5 major components: (1) a sample holder (called a thimble); (2) a round-bottomed flask; (3) a siphon; (4) a condenser; and (5) a heat source [65,66]. The sample is packed in filter paper, placed in the thimble, and the round-bottomed flask is filled with a suitable solvent [65]. The solvent is then heated to a set temperature; then, after reaching the boiling state, the solvent reaches the vapor state, passes through the thimble, and reaches the condenser, where it is again liquified [65]. The condensed liquid drips back into the thimble, gradually filling it [65]. As the liquid reaches the overflow level in the thimble, the siphon aspirates the solution and it falls back into the flask, carrying the extracted solutes (desired biomolecules) into the bulk solution [65]. This operation is repeated until complete extraction is achieved [65,66]. The resulting extract may be concentrated through evaporation.

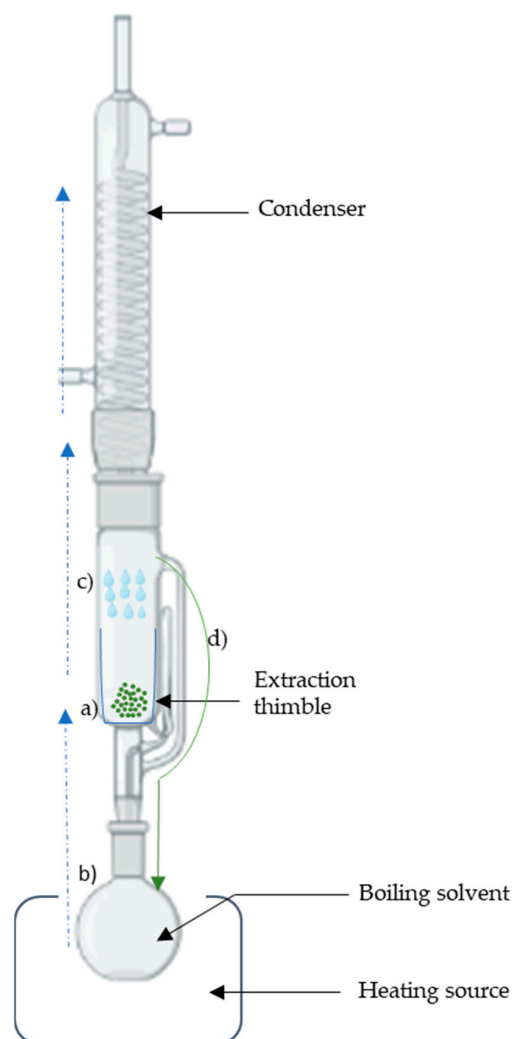


Figure 3. Schematic diagram of Soxhlet extraction: (a) sample is placed in the thimble; (b) solvent vapor passes through the thimble and rises to the condenser; (c) condensed liquid drips back to the thimble, filling it; (c) siphon aspirates the solution (with the solute) back to bulk solution; (d) siphon aspirates the solution (with the solute) back to bulk solution.

In Soxhlet extraction, enhanced mass transfer is usually achieved because of the repeated contact between the sample and fresh portions of solvent. In addition, no filtration or centrifugation is needed after the procedure [18,65,66]. Regarding the main disadvantages, the extraction time is usually long, large amounts of solvent are used, and it is not suitable for highly volatile or thermolabile compounds [23,65,66]. This technique has been applied to extraction processes of several bioactive compounds from different biomass sources and it is used as a standard technique to which the performance of modern extraction techniques is compared [14,65].

Feed and Food Waste and Non-Compliance

To the best of our knowledge, studies concerning the Soxhlet extraction of bioactive compounds using green solvents are scarce. Organic solvents such as n-hexane, methanol, and acetone have been used as the main extractant phases for decades in these types of matrices [63]. Soxhlet can become a greener method for the extraction of phytochemicals by using green solvents; however, it will largely depend on the time and solvent consumption [36]. Table 4 summarizes bioactive compounds extracted from tomato pomace, onion, and banana peels using ethanol and ethyl acetate as solvents. Studies focused on watermelon waste could not be found.

Table 4. Summary of studies reported on Soxhlet extraction of different food waste sources.

Biomass Source	Soxhlet Conditions	Yield	Ref.
Tomato pomace (pulp, seeds, and peels)	Ethyl acetate, 6 h, SB: 2:25 (<i>w:v</i>)	Lycopene (454.64 mg/100 g extract) Beta-carotene (580.96 mg/100 g extract) Phenolics (25.25 mg GAE/g extract)	[38]
Onion peels	Ethanol 70% (<i>v/v</i>) and 90% (<i>v/v</i>), 72 h	Phenolics (301.03 mg GAE/g) Flavonoids (156 QE mg/g) 2-pentenoic acid, 3-ethyl-methyl ester, 4-amino-1-methyl-3-nitropyrazole, Thiophene-2-carboxamide,	[67]
Banana peels	Ethanol 95% (<i>v/v</i>), 6 h	3-ethoxy-N-(4-chlorophenyl) Pentadecane, 1-methoxy-13-methyl-3-Hexadecane, (Z)- 4-Heptafluorobutyryloxyhexadecane 1-Hexadecene, 2-Tetradecene, Pentafluoropropionic acid, 4-hexadecyl ester	[68]

In the study conducted by Popescu et al. [36], bioethanol and ethyl acetate were used to extract lycopene, beta-carotene, and phenolics compounds from tomato pomace. Ethyl acetate extract had higher contents of both lycopene (454.64 mg/100 g extract) and beta-carotene than the bioethanol extracts [36]. This would be expected due to the lipophilic nature of carotenoid compounds. Some studies have demonstrated that ethyl acetate is a better extracting solvent for carotenoids than ethanol and even hexane [36]. Although a promising study, these results fell short of those obtained by Popescu et al. [38] on the SC-CO₂ extraction of lycopene (1016.94 mg/100 g extract) from tomato pomace. Thus, for lycopene extraction from tomato wastes, supercritical CO₂ seems to yield the best results.

Al-Ansari et al. [64] demonstrated that concentrated ethanolic extracts of onion peels are rich in phenolic compounds. These results were also more promising than those reported for the maceration extraction of onion peels using hot water extraction or even DES. In fact, Soxhlet extraction with ethanol yielded higher total phenolics than other extraction techniques mentioned in this review. Thus, although more studies might be missing, mainly, the use of ILs and NADES in the extraction of phenolic compounds from onion peels, Soxhlet extraction seems to be the most efficient, although it also has the highest extraction time, which is very energetically driven and thus environmentally costly.

Several compounds with antioxidant, antifungal, anti-inflammatory, and antibacterial activities were qualitatively determined in ethanolic extracts of banana peels. The highest concentration of these extracts (1000 mg/mL) exhibited 94.13% inhibition of DPPH activity,

very similar to the 96.28% of the AA [65]. These extracts have great potential to be used in food, cosmetics, pharmaceutical, or nutraceutical applications [65].

Microalgae

As a solvent-based lipid extraction method, Soxhlet extraction remains one of the most commonly used techniques for extracting lipids from microalgae [69].

After undergoing extensive development, Soxhlet extraction has established itself as a widely adopted method for assessing lipid extraction efficiency. Over time, researchers have made continuous advancements to address its limitations. However, certain drawbacks persist, such as the prolonged duration of the extraction process and the substantial consumption of solvents which have unfavorable environmental impacts [69].

Table 5 provides an overview of the different microalgal strains, solvents used for extraction, extraction conditions, extracted products, and yields obtained.

Table 5. Summary of studies reported on Soxhlet extraction of different microalgae species.

Biomass Source	Soxhlet Conditions	Yield	Ref.
<i>Spirulina platensis</i>	Ethanol, 60–80 °C, 4 h	Flavonoid: 5.26%	[54]
<i>Chlorella</i> sp.	Ethanol, 78 °C, 3 h	Lipid: 9.40%	[70]
<i>Nannochloropsis oculata</i>	Ethanol, 40 °C, 0.1 MPa, 18h	Lipid: 40.90%	[71]
<i>Synechocystis</i>	Ethanol, 40 °C, 0.1 MPa, 18h	Lipid: 48%	[72]

The studies conducted by Liao et al. [71], Sheng et al. [72], and Ramluckan et al. [70] performed lipid extractions using the Soxhlet method, which resulted in yields of 40.90%, 48%, and 9.40%, respectively. Ethanol was used as the solvent in all cases. The variation in yields can be attributed to the use of different microalgal species, which have distinct lipid compositions [73]. Lipids in microalgae consist of different types, including polar lipids such as phospholipids and glycolipids as well as neutral lipids like triacylglycerol and unsaturated fatty acids (UFAs). The content of lipids in microalgae can also be influenced by factors such as the type of microalgae, light exposure, growth environment, and temperature. However, despite these variations, microalgae are known to be rich sources of UFAs, particularly docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA). Indeed, several reports provide insights into microalgae-derived oils filled with DHA and EPA as easy to use in fish feed formulations [74] and, more importantly, for the promotion of mental health in humans [75]. In summary, microalgae contain a diverse range of lipids, including beneficial UFAs, which have various health-promoting properties [73].

Despite long extraction times, the Soxhlet extraction of microalgae seems to be a valuable technique for lipid extraction, depending on the microalgae species. Nonetheless, faster and more effective techniques for extracting lipids from microalgae will be presented later in this paper as SC extraction, PLE, and PEF extraction showed high yields of lipids in a much shorter time of extraction.

Lignocellulosic Biomass Sources

Lignocellulosic biomass (Table 6) contains several bioactive molecules of interest due to their antioxidant and antimicrobial activity. For instance, Rodríguez-Cabo et al. [76] studied the ethanolic Soxhlet extraction of *Vitis vinifera* canes and obtained a well-known and valuable polyphenol: catechin. However, the yield was low. Moreover, in a different study, Setiawan et al. [77] used *Caesalpinia sappan* L. in order to extract brazilin—a compound member of homoisoflavonoids, which is a rare subclass of flavonoids—that can be used in foods and pharmaceuticals [78].

In an interesting study conducted by Zhao et al. [79], where leaves from *Eucalyptus loxophleba* ssp. *Lissophloia* were submitted to Soxhlet extraction, it was possible to obtain a moderate yield of oil that could be used as an alternative fuel source.

Although interesting, more studies focusing on different green solvent options such as ILs and NADES might be missing. Additionally, long extraction times can be observed in

these studies, which might be mitigated by other unconventional extraction techniques. Moreover, the Soxhlet technique seems to be the most effective and suitable for lipid extraction, as previously mentioned for microalgae lipid extraction with Soxhlet.

Table 6. Summary of studies reported on Soxhlet extraction of different lignocellulosic sources.

Biomass Source	Soxhlet Conditions	Yield	Ref.
<i>Vitis vinifera</i> canes	Ethanol (50%), SB: 1:20 (<i>w:v</i>), 3 h	Catechin yield: 0.65 mg/g	[76]
<i>Caesalpinia sappan</i> L.	Ethanol (96%), SB: 1:20 (<i>w:v</i>), 3 h	Yield of brazilin: 5.43 mg/g of extract	[77]
<i>Eucalyptus loxophleba</i> ssp. <i>lissophloia</i> leaves	Ethanol, 90 °C, 0.5–8 h	Oil: 36.33%	[79]

3.2. Unconventional Methods

3.2.1. Microwave-Assisted Extraction (MAE)

MAE is an extraction technique (Figure 4) that involves subjecting a sample to microwave energy, which causes alterations in the cells of the material being extracted. These changes facilitate the release and extraction of desired compounds from the sample matrix [80]. In recent decades, MAE has gained significant recognition as a valuable method for extracting bioactive compounds from different sources, particularly in the context of repurposing by-products generated by agro-industrial processes [81].

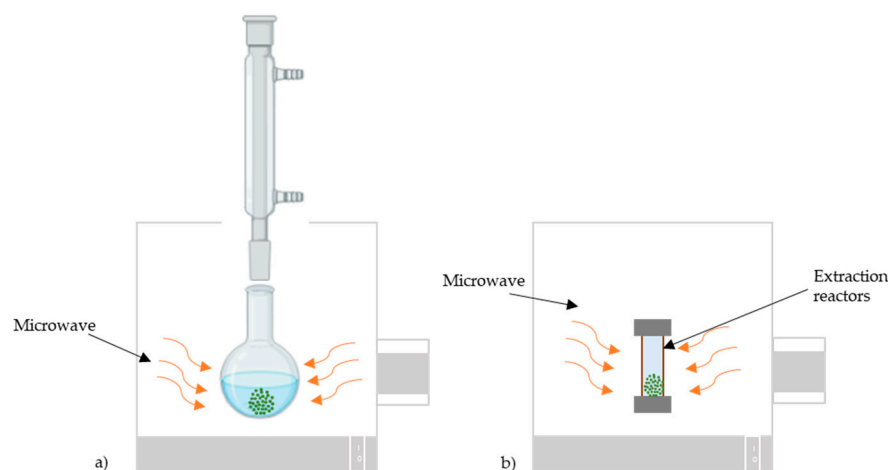


Figure 4. Schematic diagram of microwave-assisted extraction: (a) open vessel (single-mode system) and (b) closed vessel (multimode system).

Microwave devices comprise four major components: (1) the magnetron, (2) the waveguide, (3) the applicator (containing the sample), and (4) the circulator. Based on the microwave energy applied to the sample, MAE devices can be divided into two different categories: multimode systems or single-mode systems [82]. Multimode systems apply microwave radiation, which is dispersed in a space, allowing a uniform treatment of the samples. On the other hand, single-mode systems apply microwaves only to samples for a more efficient extraction [82]. Often, multimode systems are applied to closed vessels, allowing the simultaneous application of high pressure during the process of extraction, while single-mode systems are normally associated with open vessels operating at atmospheric pressure [82].

The moisture content of the sample matrix is the main target of microwave heating during MAE. Moisture evaporation builds up pressure within the plant cell and causes the swelling and subsequent rupturing of the cell, exposing the cell to the surrounding solvent and facilitating solvent penetration [83].

In conventional extraction processes, heat transfer occurs from the heating medium to the inside of the cells, while the mass transfer of solutes follows an opposite direction. In the context of MAE, both the heat and mass gradients act synergistically to enhance the

extraction process by promoting the movement of target compounds from the inside of cells to the solvent. This dynamic facilitates the extraction of high-value compounds more efficiently and reduces the overall extraction time [2,84].

MAE has also been applied to offset drawbacks of pre-existing conventional extraction processes [82], for instance, solvent-free microwave hydrodistillation and microwave hydrodiffusion and gravity [85].

The advantages and disadvantages of MAE are described in Figure 5.

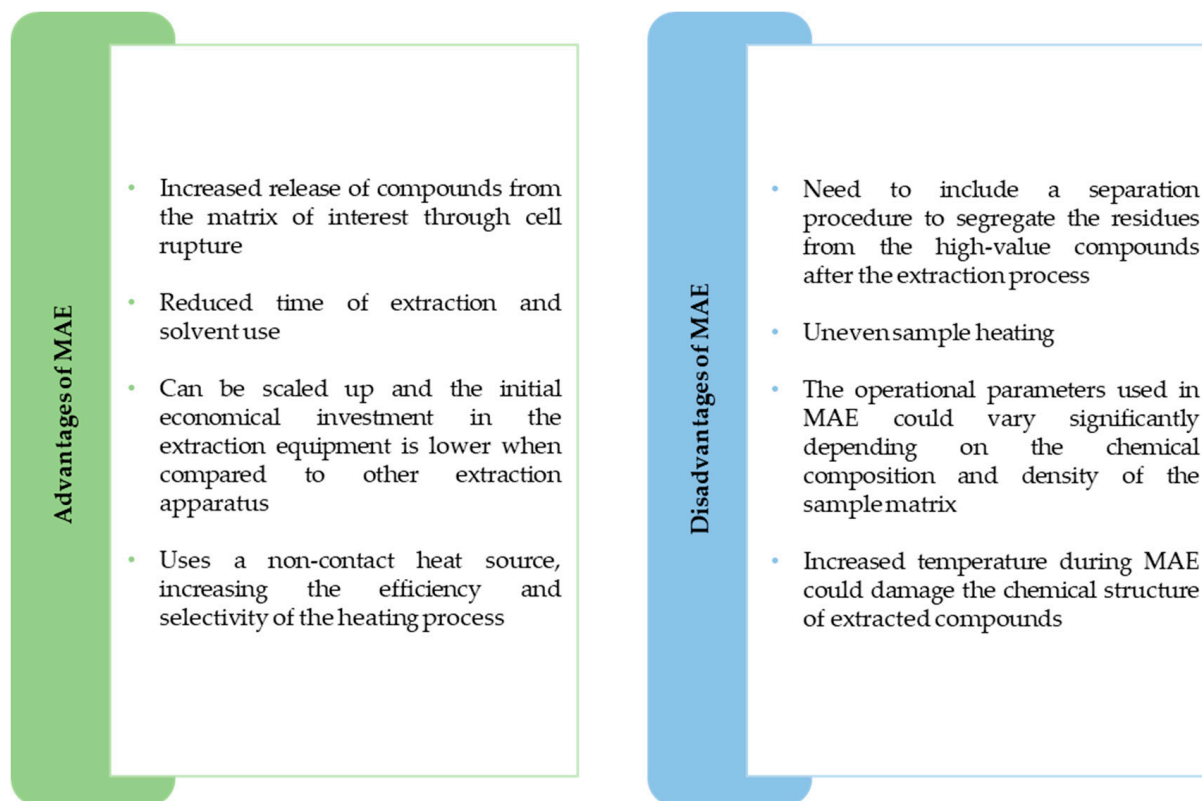


Figure 5. Main advantages and disadvantages of MAE technique.

Feed and Food Waste and Non-Compliance

By employing the MAE technique, a diverse range of phytochemicals, including polyphenolic antioxidants, can be effectively obtained [86,87]. Table 7 summarizes studies focused on the MAE of different food waste sources.

Table 7. Summary of studies reported on MAE of different food waste sources.

Biomass Source	MAE Conditions	Yield	Ref.
Tomato pomace (peels and seeds)	Citric acid solution, 2450 MHz, 600 W, 10 min	Pectin (28.28%), galacturonic acid (22.44 µg/L), lycopene (27.16 µg/g pectin)	[88]
Onion peels	ChCl:Urea:water, 100 W, 15.03 min 1:54.97 (w:v)	Phenolics (80.45 mg GAE/g)	[89]
Banana peels	Water; 2:100 (w:v); 6 min	Phenolics (50.55 mg GAE/g)	[90]
Tomato pomace	Ethanol:ethyl acetate 90:10 (v/v), 3 min, 90 °C	Lycopene (59.66 µg/g) Beta-carotene (39.82%)	[91]

Pectin is used as a functional ingredient in the food industry as a gelling and thickening agent and as a stabilizer [88]. Lasunon and Sengkhamparn [88] applied MAE combined with an acid solution to extract pectin (31.58%) from tomato pomace (Table 8).

Pal and Jadeja [89] used response surface methodology (RSM) to maximize the extraction of phenolic compounds from onion peel by MAE and ChCl (choline chloride):urea:water-based

DES (Table 8). Under optimal conditions, the recovery of phenolics was 80.45 mg GAE/g dry weight (DW) and the maximum reducing power activity was 636.18 $\mu\text{mol AAE g/DW}$. The phenolic content was higher than the maceration (63.28 mg GAE/g extract) (Table 1) and methanolic Soxhlet extract (54.73 mg GAE/g extract) and was achieved with a 12-fold reduction in extraction time [89]. The desorption and release of phenolic compounds from a plant matrix are facilitated by MAE, thus accelerating the extraction process as well as increasing the extraction yield [89].

A study by Vu et al. [90] showed that water could also be used to recover phenolic compounds from banana peels using MAE. Under optimal conditions, 50.55 mg phenolics could be recovered from 1 g dried peel (Table 7).

Microalgae

The MAE technique has been employed to extract various bioactive compounds from microalgae, including polysaccharides and lipids. This method has shown a capability to enhance lipid yield significantly. However, it is important to note that the most effective extraction method may vary depending on the specific microalgae species being targeted [69,92].

Several research studies have utilized MAE as a technique for extracting bioactive compounds from microalgae [56,93–95]. However, it is important to note that MAE operates by applying microwave energy to heat the biomass and induce cell wall breakage. Consequently, one limitation of this method is its unsuitability for extracting heat-sensitive compounds [69].

Table 8 provides an overview of the different microalgal strains, solvents used for extraction, extraction conditions, extracted products, and yields obtained.

In the study conducted on *Spirulina* sp., the biomass was extracted using distilled water, resulting in a yield of phycocyanin at 85.43 ± 0.60 mg/g [96]. *A. platensis* was also subjected to MAE using water under specific conditions, leading to a yield of 127 ± 5 mg of carbohydrate/g of biomass [93]. For *Chlorella* sp., the extraction involved the use of ethanol at 700 W and 78 °C for 6 min, resulting in a lipid yield of 17.11% (dry weight) [94]. *Phaeodactylum tricornutum* was extracted using ethanol at specific MAE conditions, resulting in a yield of 4.51% DW carotenoids and 0.46% DW fucoxanthin [96].

Overall, Table 8 demonstrates the effectiveness of MAE in extracting various bioactive compounds from different microalgae sources. When the temperature during microwave processing is between 80 and 120 °C, raising the temperature results in an increase in the fractal dimension of the cell, indicating greater damage to the cell wall. Conversely, as the extraction time is extended, the pore size on the cell wall gradually enlarges, facilitating the extraction of bioactive compounds [97].

Table 8. Summary of studies reported on MAE of different microalgae species.

Biomass Source	MAE Conditions	Yield	Ref.
<i>Phaeodactylum tricornutum</i>	Ethanol, 2.45 GHz, 850 W, 30 °C, 2 min	4.51% DW carotenoids and 0.46% DW fucoxanthin (32.26% recovery)	[56]
<i>Spirulina</i> sp.	Distilled water, 120 s, 1400 W, 2.450 MHz	Phycocyanin: 85.43 ± 0.60 mg/g	[96]
<i>A. platensis</i>	Water, 20 min, 434 W, SB: 1:30	127 ± 5 mg of carbohydrate/g of biomass	[93]
<i>Chlorella</i> sp.	Ethanol, 700 W, 78 °C, 6 min	17.11% DW lipid	[94]
<i>Spirulina</i> sp.	Distilled water, 2.74 min, 40 ± 2 °C, 133 W	Phycocyanin: 28.90 mg/g	[95]

Lignocellulosic Biomass Sources

Table 9 summarizes the different studies found focused on the MAE of lignocellulosic biomass.

Fernandez-Agulló et al. [64] showed that MAE, using ethanol as a solvent, 1:10 (*w:v*) solvent to biomass ratio (SB), 50 °C, and microwave power of 150 W, allows an extraction yield of 1.34%, with a TPC of 67.49 g per 100 g of extracts from *Eucalyptus globulus* wood. Nevertheless, these results were inferior compared to those related to maceration extraction,

which yielded 2.87%, with a TPC of 85.71 g per 100 g of extracts. The better performance of the maceration was possibly due to the extraction time because while the MAE was performed for 10 min, the maceration had a time of 90 min. In addition, they concluded that microwaves could have caused the degradation of part of the extracts, which is one of the major drawbacks of the MAE technique.

In two different studies, Meullemiestre et al. [98,99] studied the MAE of *Pinus pinaster*, and the more recent study revealed a yield 0.43% higher than that of the first study. The main differences were in the MAE conditions used: 668 W and 43 min yielded higher yields than 600 W and 60 min. Therefore, higher power in the extraction seems to give higher yields in less time for the same species.

Table 9. Summary of studies reported on MAE of lignocellulosic biomass sources.

Biomass Source	MAE Conditions	Yield	Ref.
<i>Eucalyptus globulus</i> Labill.	Ethanol, SB: 1:10 (w:v), 50 °C, 10 min	TPC: 67.49 g GAE/100 g extract	[64]
<i>Pinus pinaster</i>	Solvent-free, 668 W, 43 min	Yield: 0.43% TPC: 74.62 mg GAE/g extract	[99]
	Solvent-free, 600 W, 60 min	Yield: 0.27% (β -caryophyllene, longifolene, and α -terpineol)	[98]

3.2.2. Ultrasound-Assisted Extraction (UAE)

UAE (Figure 6) uses ultrasound energy and different solvents to efficiently extract target compounds from various plant matrices [100]. Ultrasounds are mechanical waves with a frequency higher than 20 kHz, which is higher than the audible frequency range of human hearing (20 Hz to 20 kHz) [100]. These waves consist of a series of compression and rarefaction cycles that can be propagated through a solid, liquid, or gas medium inducing displacement and dislodgement of the molecules from their original positions. With high-intensity sound waves, the negative pressure during rarefaction exceeds the attractive force joining the molecules together, pulling them apart and creating cavitation bubbles. These bubbles grow through coalescence and later collapse during the compression phase, creating hot spots and extreme local conditions [100]. The collapsing cavitation bubbles generate shockwaves, and accelerated inter-particle collision causes fragmentation in the cellular structure.

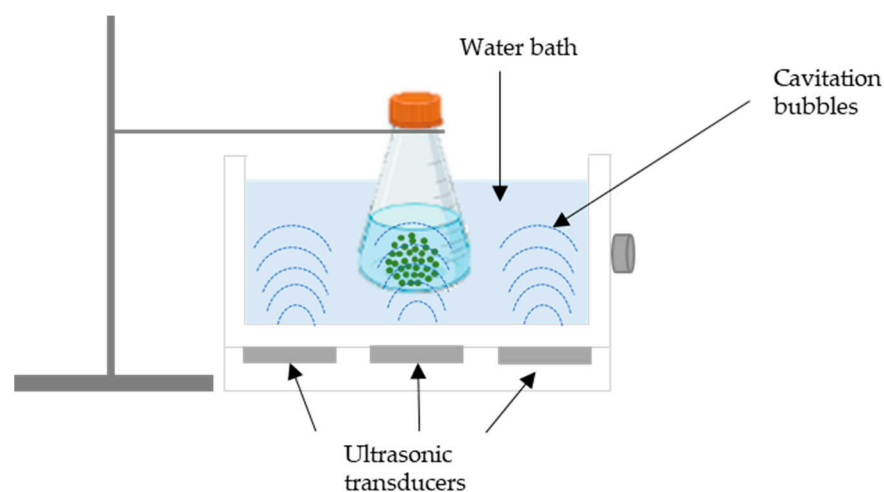


Figure 6. Schematic diagram of ultrasound-assisted extraction.

Feed and Food Waste and Non-Compliance

Several studies (Table 10) were conducted to extract carotenoids from tomato pomace by UAE and green solvents [90,91]. We emphasize the work of Szabo et al. [92]

and Diacon et al. [96], where a mixture of ethyl acetate:ethyl lactate (1:3) and fatty acid ethyl esters were used, respectively. In the latter, the highest lycopene concentration (101.4 mg/100 g) was obtained. As previously reported, the use of oil-derived solvents seems to be a good choice for carotenoid extraction from tomato waste, independent of the extraction technique used.

Table 10. Summary of studies reported on UAE of different food waste sources.

Biomass Source	UAE Conditions	Yield	Ref.
Onion seeds	NADES—Lactic acid, glucose, and 15% water (LGH-15), 40 °C, 30 min, SB: 75 mg/mL	Rutein (67.169 µg/g DW) Tyrosin (139.012 µg/g DW) Caffeic acid (136.314 µg/g DW) Quercetin (2.056 µg/g DW)	[52]
Tomato pomace (peels, seeds, and pulp)	Ethyl acetate:ethyl lactate 1:3 (<i>v/v</i>), SB: 1:20 (<i>w:v</i>), 10 min, 35 °C	Lycopene (254.08 µg/g DW) Beta-carotene (78.74 µg/g DW) Lutein (31.16 µg/g DW)	[101]
Tomato peels	Fatty acid ethyl esters, SB: 2:40 (<i>w:v</i>); 15 min	Lycopene (101.4 mg/100 g)	[102]
Tomato peels and seeds	NADES—Lactic acid, glucose, and 15% water (LGH-15), 40 °C, 30 min, SB: 75 mg/mL	Rutein (325.132 µg/g DW) Caffeic acid (98.087 µg/g DW) Quercetin (62.605 µg/g DW)	[103]
Banana peels	Ethanol (50%), 1:20, 45 °C, SB: 3:60 (<i>w:v</i>), 1 h	Phenolics (31.45 mg GAE/g) Flavonoids (22.11 mg GAE/g)	[104]
Watermelon peels	Ethanol (43.28%), 40 kHz, 44 °C, 110 W, 32 min	Phenolics (8.35 mg GAE/g)	[104]
Watermelon seeds	Ethanol (39.18%), 40 kHz, 50 °C, 38 min	Phenolics (32.2 mg GAE/g) Syringic acid (18.21 µg/mL) Caffeic acid (24.22 µg/mL) Sinapic acid 152.30 µg/mL Ferrulic acid (68.28 µg/mL) Vanillic acid (22.64 µg/mL) Gallic acid (2.56 µg/mL) 4-hydroxy benzoic acid (59.71 µg/mL)	[101]

A combination of lactic acid, glucose, and 15% water at 40 °C, for 30 min was selected as optimal to extract the following phenolic and flavonoid compounds from tomato peels and seeds: rutein (325.12 µg/g DW), caffeic acid (98.09 µg/g DW), and quercetin (62.01 µg/g DW) [93].

Ethanol was the chosen solvent for the extraction of several phenolic and flavonoid compounds from banana and watermelon wastes by UAE. For banana peels, it was observed that sonication leads to better results than maceration [101]. Fadimu et al. [94] studied the use of UAE and ethanol for the extraction of these compounds from watermelon peels and seeds. Contrary to what was expected, the seeds revealed a higher phenolic content than the peels. In fact, in the seed fraction, the following compounds were detected and quantified: syringic acid, caffeic acid, sinapic acid, ferrulic acid, vanillic acid, gallic acid, and 4-hydroxy benzoic acid. However, the DPPH activity was found to be very similar between the two fractions. Further studies must be carried out to evaluate other biological effects that watermelon seeds may have as they seem to be a good functional ingredient.

Microalgae

UAE has proven to be an effective method for extracting a wide range of compounds from microalgae [105]. Table 11 provides an overview of the different microalgal strains, solvents used for extraction, extraction conditions, extracted products, and yields obtained.

Table 11. Summary of studies reported on UAE extraction of microalgae species.

Biomass Source	UAE Conditions	Yield	Ref.
<i>Spirulina platensis</i>	DES/IL, 25 °C, 25 kHz, and 30 min	Allophycocyanin: 6.34 mg g ⁻¹ Phycocyanin: 5.95 mg g ⁻¹ Phycocerythrin: 2.62 mg g ⁻¹	[105]
<i>Spirulina</i> sp.	NADES: glycerol/glucose/water (1:2:4 molar ratio)	Chlorophylls: 0.50 mg/g Carotenoids: 0.22 mg/g Phycocyanin: 3.96 mg/g	[106]
<i>Scenedesmus</i> sp.	NADES: Fen-Thy, 70 min, 60 °C, 40 kHz, 300 W	Carotenoid (lutein): 4.4 mg/g	[107]
<i>Haematococcus pluvialis</i>	Ethanol:ethyl acetate (1:1 (v/v)), 200 W, 16 min	Astaxanthin: 27.58 ± 0.40 (mg/g)	[108]

In the study by Rodrigues et al. [105], *Spirulina platensis* was subjected to UAE using DES/IL at 25 °C, 25 kHz, for 30 min. The extraction resulted in the yields of allophycocyanin (6.34 mg g⁻¹), phycocyanin (5.95 mg g⁻¹), and phycocerythrin (2.62 mg g⁻¹). For *Spirulina* sp., the extraction was carried out using NADES composed of glycerol/glucose/water. The obtained yields were chlorophylls (0.50 mg/g), carotenoids (0.22 mg/g), and phycocyanin (3.96 mg/g) [106]. *Scenedesmus* sp. was subjected to NADES extraction using fenchyl alcohol-thymol (Fen-Thy) for 70 min at 60 °C, 40 kHz, and 300 W. The extraction yielded a concentration of 4.4 mg/g of lutein [107]. In the case of *Haematococcus pluvialis*, extraction was performed using a mixture of ethanol and ethyl acetate (1:1 v/v) under 200 W for 16 min. The extraction resulted in an astaxanthin yield of 27.58 ± 0.40 mg/g [108]. Overall, the studies found showcase the application of UAE for the extraction of various bioactive compounds from different microalgae species, such as pigments and proteins.

Most extraction studies in the field of UAE utilize frequencies of 20–100 kHz and high-power intensities (>1 W/cm²) with the help of an ultrasonic horn. These conditions promote the phenomenon of cavitation, which enhances the extraction efficiency. However, it is important to note that high-power intensities can cause rapid and intense disruption of cell membranes, potentially affecting the purity of the extracts. Therefore, it is crucial to carefully control the parameters of time and power intensity during UAE extraction [109,110].

Lignocellulosic Biomass Sources

Several factors interfere with the efficiency of UAE, particularly the frequency and intensity of the ultrasound, viscosity, temperature and pressure of the medium, moisture content and particle size of the plant sample, solvent used, sonication time, and nature of the plant matrix [57]. In all the studies found (Table 12), different extraction parameters were used. Meullemiestre et al. [98] showed that the UAE of *Pinus pinaster* under temperatures of 40 °C for 43 min of extraction yielded 3.42 mg/g of catechin. This result proved to be 47% higher than the maceration results.

UAE was also used to valorize forest industry residues (i.e., *Acer saccharum* wood). A yield of 2.3% of extractives with a TPC of 286 mg of GAE/g extract was obtained in only 30 min. Maceration was also performed to compare the results, and it was found that maceration needs approximately 24 h to obtain similar yield values [111].

Thus, due to the cavitations caused by UAE, which cause ruptures in biomass cell walls, higher yields are obtained through the UAE technique. Moreover, ethanol seems to be a suitable solvent to extract phenolic compounds from lignocellulosic matrices due to its polarity. Other solvents, such as ILs and NADES, should also be studied in order to gage the results against these studies.

Table 12. Summary of studies reported on UAE of lignocellulosic biomass sources.

Biomass Source	UAE Conditions	Yield	Ref.
<i>Pinus pinaster</i>	Acidified water, SB: 1:17 (w:v), 40 °C, 43 min	Yield of catechin: 3.42 mg/g of wood	[98]
<i>Olea europaea</i> L.	Ethanol (70%), SB: 1:5 (w:v), RT, 1 h	Yield: 9.0% TPC: 156.04 mg GAE/g extract	[112]

Table 12. Cont.

Biomass Source	UAE Conditions	Yield	Ref.
<i>Acer saccharum</i> Marsh	Ethanol (95%), SB: 1:10 (<i>w:v</i>), 30 min	Yield: 2.3% TPC: 286 mg GAE/g extract	[111]
<i>Quercus cerris</i> L.	Ethanol (70%), SB: 1:5 (<i>w:v</i>), 1 h	Yield: 1.20% TPC: 350.28 mg GAE/g extract	[113]

3.2.3. Supercritical Extraction (SC)

Supercritical fluid extraction (SC) can be defined as the process that occurs when a substance (e.g., carbon dioxide) reaches its critical point, where the distinction between gas and liquid phases becomes indistinguishable [114]. At this critical point, a supercritical fluid exhibits physical properties that are characteristic of both a gas and a liquid [114]. This unique state enables the supercritical fluid to be utilized effectively in extraction processes due to the solubilization and separation of extractable chemicals [23].

The SC process (Figure 7) involves the use of a solvent that dissolves the desired chemicals present in the sample. The solvent is then circulated through a packed bed, where it interacts with the sample and extracts the target compounds. Subsequently, the solvent exits the extraction vessel. As the solvent flows out, a change in temperature and pressure occurs, leading to an increase in temperature and a drop in pressure. These changes in conditions cause the solvent to transition back to a gaseous state, leaving the extracted compounds behind in a solvent-free form [23].

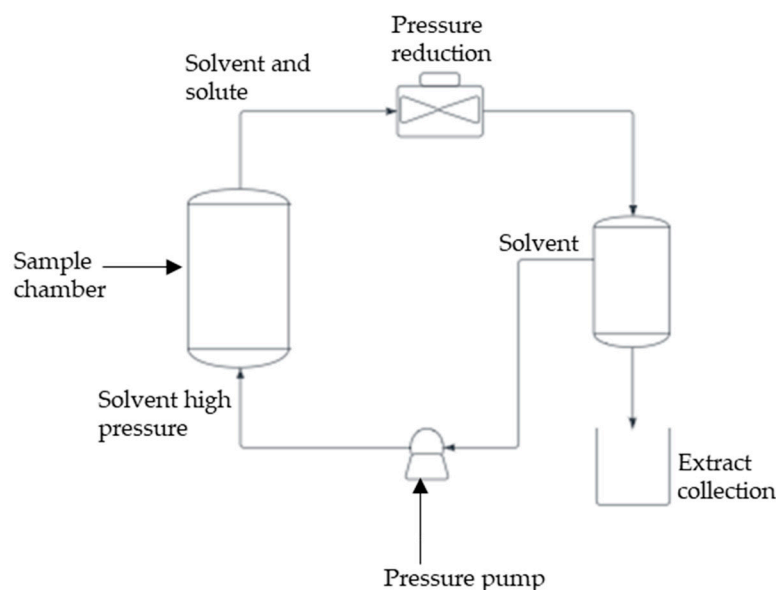


Figure 7. Schematic diagram of supercritical fluid extraction.

One notable example of a supercritical fluid is carbon dioxide, which becomes supercritical at temperatures above 31.1 °C and pressures of 7380 kPa. The utilization of supercritical CO₂ (SC-CO₂) in extraction processes offers several advantages, which are primarily attributed to its robust solvation capacity for nonpolar phytochemicals.

However, polarized phytochemicals often exhibit low solubility in SC-CO₂ extraction. To enhance the solubility of polar phytochemicals in SC-CO₂, co-solvents such as ethyl alcohol, methanol, water, acetone, ethyl acetate, and acetonitrile are added to the extraction process. This adjustment effectively increases the yield of phytochemicals. Due to its versatility and scalability, SC-CO₂ extraction is being applied in several sectors such as food, cosmetics, and pharmaceuticals. It is used for weakly polar compounds of low molecular weight such as carotenoids, triglycerides, fatty acids, aromas, etc. [115–118].

Furthermore, CO₂ has several advantages, including low toxicity, wide availability, and low cost [119–121].

The SC-CO₂ extraction method is widely employed for commercial extractions from natural resources. However, the precise adjustment of temperature and pressure parameters is crucial to achieving optimal yields while preserving the uncompromised biological activities of the extracted compounds [122,123]. While higher temperatures can increase the solubility of solutes in supercritical CO₂, it is important to be cautious when dealing with thermolabile molecules [124]. To extract thermolabile phytochemicals effectively without compromising their quality, it is recommended to maintain low temperatures, increase pressure, and ensure proper sample preparation with no moisture present [125].

Figure 8 summarizes the main advantages and disadvantages of the supercritical extraction process.

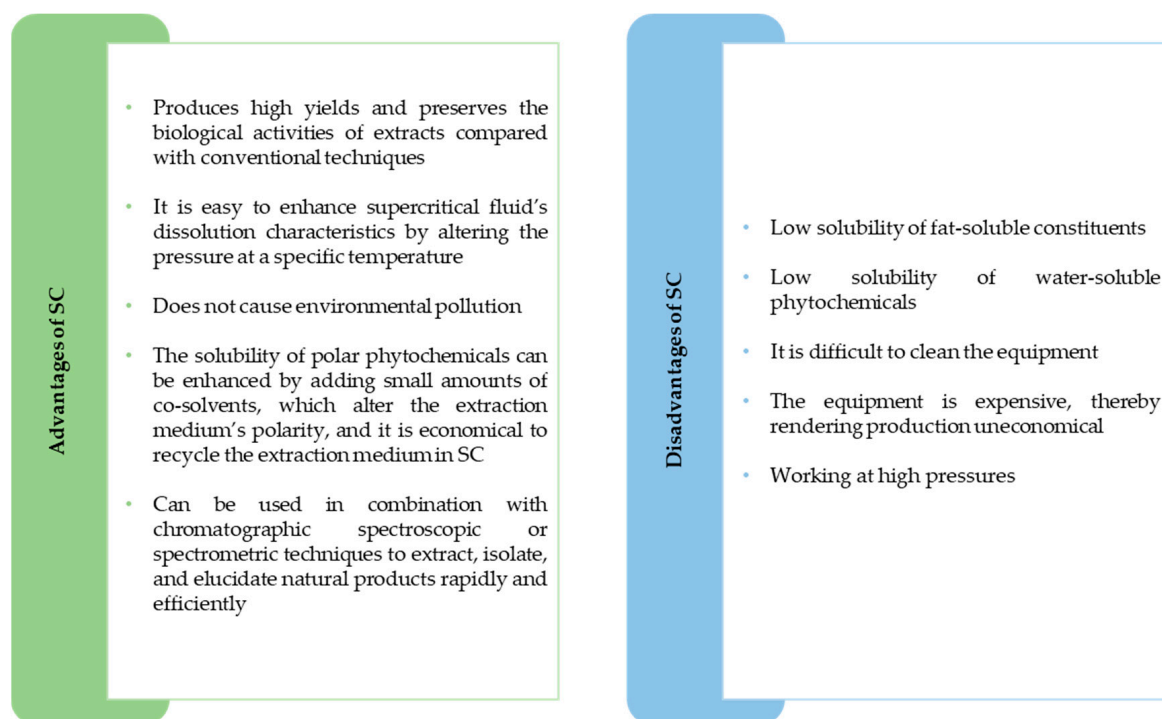


Figure 8. Main advantages and disadvantages of supercritical extraction (SC) process.

The main drawbacks are high initial investment and difficulties in performing continuous extractions [3].

Feed and Food Waste and Non-Compliance

Depending on the conditions applied, Alwazeer et al. [113], Popescu et al. [36], and Hatami et al. [114] demonstrated that SC-CO₂ can be used to extract lycopene and more hydrophilic bioactive compounds from tomato waste.

Alwazeer et al. [47] evaluated the extraction of phytochemicals using hydrogen-rich water and supercritical extraction methods from tomato peels (Table 13). Total phenolic and flavonoid contents, total anthocyanin, and antioxidant activity (DPPH and ABTS) were higher in hydrogen-rich water extracts. On the other hand, the results obtained by Popescu et al. [36] showed that supercritical methods allow for greater lycopene extraction (1016.94 mg/100 g extract) than Soxhlet extraction with ethyl acetate (454.54 mg/100g extract), more than 2-fold. As previously mentioned, flavonoids, quercetin, and kaempferol have several biological effects like antioxidant, anti-inflammatory, immunoprotective, and even anti-carcinogenic properties [23,115].

Table 13. Summary of studies reported on SC extraction of food waste.

Biomass Source	SC Conditions	Yield	Ref.
Tomato peels	CO ₂ , 30 °C, 7 MPa, 2 h	Gallic acid (2.77 µg/g) Chlorogenic acid (2.35 µg/g) P-coumaric acid (1.64 µg/g) Catechin (23.71 µg/g) Rutin (1.69 µg/g)	[49]
Tomato peels and seeds	CO ₂ , 50 MPa, 80 °C, 220 min	Lycopene (55%, 1.32 mg/kg raw material)	[126]
Tomato pomace	CO ₂ , 45 MPa, 70 °C, 11 kg/h	Lycopene (1016.94 mg/100 g extract) Beta-carotene (154.87 mg/100 g extract) Phenolics (35.25 mg GAE/G extract)	[38]
Onion peels	CO ₂ , 40 MPa, 55 °C, ethanol as co-solvent (2.5 to 4 h), SB: 1:8 (<i>w:v</i>)	Phenolics (212.56 mg GAE/g) Flavonoids (211.51 mg QE/g) Quercetin 7,4-diglycoside (6.63%) Quercetin 3,4-diglycoside (45.19%) Quercetin 4-glucoside (0.12%) Quercetin (2.41%) Kaempferol (1.00%)	[127]
	CO ₂ , 40 MPa, 55 °C, ethanol as co-solvent (2.5 to 4 h), SB: 1:8 (<i>w:v</i>)	Phenolics (202.31 mg GAE/g) Flavonoids (282.80 mg QE/g) Quercetin 7,4-diglycoside (1.39%) Quercetin 3,4-diglycoside (3.99%) Quercetin 4-glucoside (5.60%) Quercetin (39.94%) Kaempferol (1.27%)	[127]

Constantin et al. [116] studied the use of SC-CO₂ in combination with ethanol as a co-solvent to extract bioactive compounds from onion waste. The results revealed five main compounds, mainly, quercetin, 7,4-diglycoside, quercetin 3,4-diglycoside, quercetin 4-glucoside, and kaempferol. From the total flavonoid content, the compound that registered the highest content of 45.19% was quercetin 3,4-diglycoside. Quercetin and kaempferol, two of the essential compounds responsible for the antioxidant activity of onion peel extracts, displayed 39.94 and 1.27%, respectively.

Microalgae

Continuous research in the field of extraction has led to significant advancements, particularly in the extraction of biocompounds from microalgae using supercritical fluids [69].

Table 14 provides an overview of the different microalgal strains, solvents used for extraction, extraction conditions, extracted products, and yields obtained.

Table 14. Summary of studies reported on SC extraction of microalgae species.

Biomass Source	SC Conditions	Yield	Ref.
<i>Nannochloropsis oculata</i>	CO ₂ and ethanol, 45 bar, 50 °C, 250 min	Lipid: 83%	[128]
<i>Haematococcus pluvialis</i>	CO ₂ , 55 MPa, 50 °C, 120 min	Astaxanthin: 98.6%	[129]
<i>Chlorella protothecoides</i>	CO ₂ , 30 MPa, 70 °C	Lipid: 21%	[130]
<i>Nannochloropsis oculata</i>	CO ₂ , 80 °C, 20.7 MPa, 240 min	Lipid: 71%	[131]
<i>Scenedesmus obliquus</i>	CO ₂ , 20 °C, 12 MPa, 540 min	Lipid: 59%	[132]

SC has been proven to be one of the most effective methods for extracting lipids [69]. For example, Obeid et al. [128] conducted a study where they utilized SC-CO₂ in combination with ethanol as a co-solvent to extract neutral lipids from freeze-dried *Nannochloropsis oculata* and *Chlorella vulgaris*. By optimizing the extraction conditions, they achieved a remarkable lipid extraction rate of 83% for *Nannochloropsis oculata*. Similarly, Bong et al. [131] also optimized the conditions for the SC-CO₂ extraction of lipids from *Nannochloropsis oculata* and achieved a high yield of lipid extraction (71%). On the other hand,

Viguera et al. [130] optimized the conditions for the SC-CO₂ extraction of lipids from microalgae (*Chlorella protothecoides*), showing the highest lipids extraction rate at 300 bars and 70 °C. Finally, Lorezen et al. [132] optimized the extraction conditions for microalga *Scenedesmus obliquus* and achieved a yield of 59%.

SC extraction can also be used to extract other types of biocompounds, such as bioactive pigments [129]. Sanzo et al. [129] utilized SC extraction to extract astaxanthin from *Haematococcus pluvialis*, achieving a yield of 98.6%. Astaxanthin is a highly valuable carotenoid that holds significant market importance. Due to its reported antioxidant, anti-inflammatory, and immune-enhancing properties [133], this carotenoid finds applications across various industrial sectors such as pharmaceuticals, nutraceuticals, functional foods, natural medicine, and cosmetics [134]. For instance, this bioactive pigment has shown important activities in skin protection and repair [133]. In addition, due to the reported antioxidant activities, the ingestion of this pigment might also provide benefits such as a lower risk of chronic diseases such as cardiovascular diseases, cataract development, macular degeneration, and some types of cancer [135].

Lignocellulosic Biomass Sources

Acacia dealbata Link. is an invasive species that has spread throughout Mediterranean countries, from the Iberian Peninsula to France and Italy [136]. Thus, finding value in such lignocellulosic sources is quite relevant since it can both aid in bioactive compound production and relieve the environmental burden. In a study conducted by Rodrigues et al. [136], supercritical fluid extraction was performed. Lupenone is a triterpenoid and is often consumed in human vegetarian diets. Moreover, pharmacological screening of lupenone revealed various pharmacological activities including anti-inflammatory, anti-virus, anti-diabetes, anti-cancer activities, improving Chagas disease without major toxicity [137].

In two other interesting studies, Bukhanko et al. [138] and Ribas et al. [139] were able to valorize *Picea abies* branches and *Eremanthus erythropappus*. In both cases, SC-CO₂ was performed. SC extraction might be a viable extraction technique for several lignocellulosic biomass sources, although optimization studies must be performed in order to take full advantage of such innovative extraction techniques. In addition, small amounts of co-solvents might aid in the extraction of some valuable compounds.

Table 15 summarizes the different studies focused on the SC extraction of lignocellulosic sources.

Table 15. Summary of studies reported on SC extraction of different lignocellulosic sources.

Biomass Source	SC Conditions	Yield	Ref.
<i>Picea abies</i> branches	CO ₂ , 50 °C, 2 h, 30 MPa	Yield: 5.3%	[138]
<i>Eremanthus erythropappus</i>	CO ₂ , 60 °C, 1 h, 12 MPa, Flow rate: 3 mL/min	Yield: 0.36% α-Bisabolol: 58.02%	[139]
<i>Acacia dealbata</i> Link.	25 MPa, 40 °C, no cosolvent	Lupenone: 0.4748%	[136]

3.2.4. Pressurized Liquid Extraction (PLE)

PLE (Figure 9) is an extraction method that performs under high temperatures and pressure [140]. In PLE, the sample is enclosed in an extraction cell. Then, the cell is filled with the extraction solvent and subjected to high pressure and temperatures. Subsequently, the extract is removed from the cell and the cell is flushed with a fresh solvent. After extraction is completed, the remaining solvent is purged with nitrogen (N₂) into the collection vials [141,142]. The use of a closed system allows for extraction at elevated temperatures since the boiling point of the solvent increases. At higher temperatures, solvation power increases, viscosity decreases, and diffusion rate increases. Thus, the extraction rate is improved and extraction time is reduced [140]. Hence, PLE is regarded as an advanced extraction technique due to the advantages it presents over other traditional extraction mechanisms: faster, fewer volumes of organic solvents, the possibility of automation, and higher extraction yields [143].

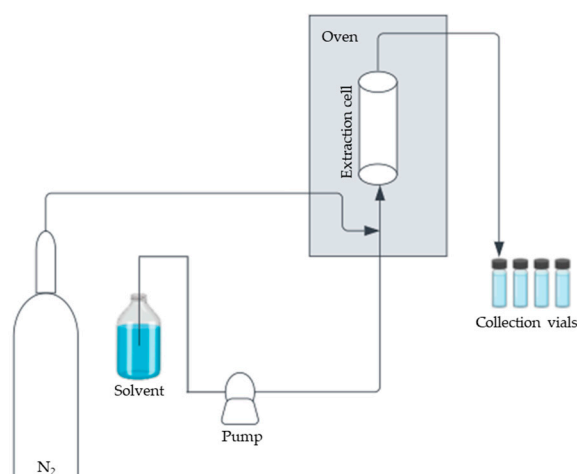


Figure 9. Schematic diagram of pressurized liquid extraction.

Such advantages are mainly explained by the fact that an increase in the extraction temperature positively influences the analyte's solubility, increasing the mass transfer rate. In addition, at these high-temperature conditions, the viscosity and surface tension of the solvents used is decreased, which helps the solvent to interact more easily with the entire matrix, likewise enhancing the extraction rate [144].

The main disadvantage of this extraction technique is the high cost of devices and equipment necessary for the operation of this process, which is primarily a consequence of working with high pressure [145].

Feed and Food Waste and Non-Compliance

To the best of our knowledge, only one study has been published regarding the use of this non-conventional technique for the extraction of bioactive compounds from the food waste previously mentioned (Table 16). Chada et al. [91] optimized the PLE parameters (90 °C, ethyl acetate:ethanol, 50:50, 50 min, 2 mL/min) to obtain tomato pomace extracts with high antioxidant activity (19.10 µmol TE/g) and lycopene contents (20.09 µg/g), which stood out from the Soxhlet extracts with ethyl acetate for 6 h (18.43 µmol TE/g and 10.75 µg lycopene/g). In relation to MAE, the PLE extract exhibited the highest antioxidant activity, whereas the MAE extract showed the highest lycopene content (59.66 µg lycopene/g extract) (Table 8), which represents a 66.93% lycopene recovery compared to a standard technique with acetone. PLE can be a viable alternative to several conventional extraction methods since it can provide higher lycopene recovery in a shorter time.

Table 16. Summary of studies reported on PLE of different food waste sources.

Biomass Source	PLE Conditions	Yield	Ref.
Tomato pomace	Ethanol:ethyl acetate 50:50 (v/v), SB: 1:20 (w:v), 90 °C, 10 MPa, 2 mL/min	Lycopene (20.09 µg/g) Beta-carotene (46.51%)	[91]

Microalgae

PLE offers benefits such as reduced extraction times, oxygen-free conditions, and the ability to selectively extract various lipid classes by modifying the polarity of the extraction solvents. These advantages contribute to the effectiveness and versatility of PLE as a method for lipid extraction from microalgae [146]. Looking at the studies conducted by Pieber et al. [147] and Golmakani et al. [148], presented in Table 17, the efficiency of PLE in lipid extraction can be observed. Furthermore, comparing the three studies, it is evident that the study with the highest yield was the one conducted at a higher temperature. Thus, it can be inferred that temperature influences the yields obtained in lipid extraction from microalgae by PLE [146].

Table 17. Summary of studies reported on PLE of different microalgae species.

Biomass Source	PLE Conditions	Yield	Ref.
<i>Nannochloropsis oculata</i>	Ethanol, 60 °C, 10–12 MPa, 10 min	Lipid: 36%	[147]
<i>Nannochloropsis oculata</i>	Ethanol, 60 °C, 10–12 MPa, 48 min	Lipid: 36.4%	[147]
<i>Arthrospira platensis</i>	Limonene/ethanol, 200 °C, 20.7 MPa, 15 min	Lipid: 70%	[148]
<i>Chlorella ellipsoidea</i>	Ethanol, 115.4 °C, 10.3 MPa, 23.3 min	Zeaxanthin: 4.28 mg g ^{−1}	[149]
<i>Scenedesmus obliquus</i>	Water, 50 °C, 10 MPa, 45 min	Lutein: 7.50 ± 0.8%	[56]

On the other hand, there are many other bioactive compounds that can be extracted from microalgae using PLE, such as carotenoids [56]. These natural pigments have been associated with a wide range of potential benefits. One of the main reasons for these benefits is their ability to act as antioxidants or protectors against free radicals within cells. Due to the numerous health benefits associated with carotenoids, researchers have been actively working on developing environmentally friendly methods to extract carotenoid-rich extracts from microalgae. However, it should be noted that there is no universal extraction method that can be applied to all microalgae species. The composition of carotenoids varies among different microalgae, requiring customized approaches for efficient extraction. [146]. In Table 17, we can observe that Koo et al. [149] and Gilbert-López et al. [56] conducted studies where Zeaxanthin was extracted from *Chlorella ellipsoidea* and Lutein from *Scenedesmus obliquus*, respectively.

Lignocellulosic Biomass Sources

Compared to other techniques reviewed in this article, the use of PLE seems to provide reduced extraction times, low solvent consumption, high selectivity, and highly biologically active extracts [57]. For instance, in both studies developed by D’auria et al. [150] and Todaro et al. [62] (Table 18), just 15 min of extraction time was needed; this is much quicker than that of the previously reviewed studies and extraction techniques. In addition, in the case of *Castanea sativa* Mill., PLE showed higher yields when compared to SC.

Table 18. Summary of studies reported on PLE of lignocellulosic biomass sources.

Biomass Source	PLE Conditions	Yield	Ref.
<i>Castanea sativa</i> Mill.	Ethanol (70%), 110 °C, 3 cycles of 5 min, 1 MPa	Yield: 12.5%	[150]
<i>Quercus cerris</i> L.	Ethanol (70%), SB: 1:5 (<i>w:v</i>), 1 h	Yield: 1.20% TPC: 350.28 mg GAE/g extract	[113]
<i>Populus nigra</i>	Ethanol (70%), 100 °C, 3 cycles of 5 min, 10.34 MPa	-	[62]

Despite working under high pressure and high temperatures, PLE might be the quickest extraction technique for lignocellulosic biomass sources.

3.2.5. Pulsed Electric Fields (PEFs)

Non-thermal processing technologies have been widely studied to extract natural food colorants and pigments. These technologies employ lower temperatures and small amounts of solvent, increasing the chemical and physical stability of the colorants, energy efficiency, and extraction yield. Furthermore, by not using heat as the primary agent for the extraction processes, non-thermal technologies enable better conservation of thermosensitive components such as pigments. The leading innovative non-thermal technology that stands out in the market is pulsed electric fields (PEFs) [151].

PEF technology (Figure 10), in turn, is a promising treatment of short duration, which provides high-intensity pulsed electric fields from a high current flow. These high-intensity pulsed electric fields cause the electroporation of cell membranes. This phenomenon destabilizes the cell’s bilipid layer, making it more permeable and facilitating the extraction of intracellular compounds [151]. The system is composed of a high-voltage pulse generator, treatment chamber, fluid-handling system, and monitoring device. The pulses generated

are applied to two electrodes present in the PEF chamber and the sample is placed between them [152,153].

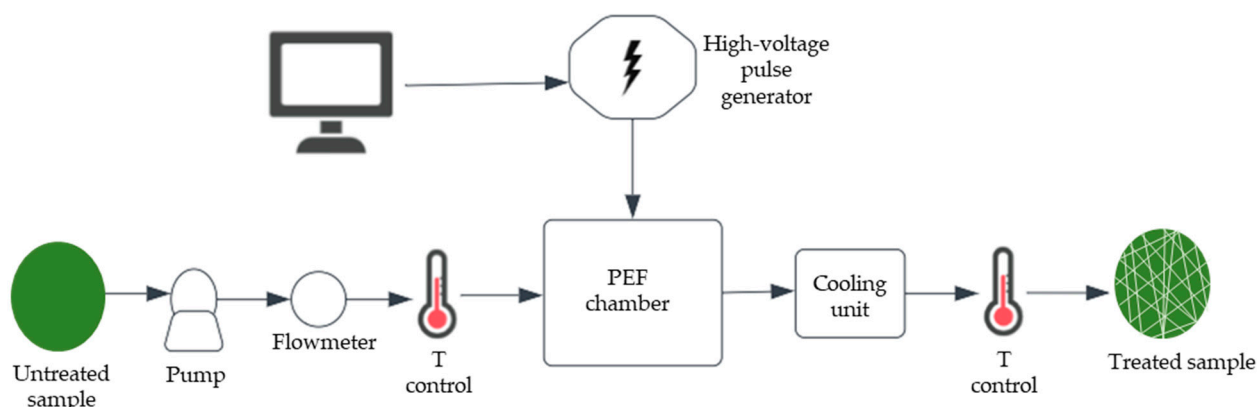


Figure 10. Schematic diagram of pulsed electric fields.

By increasing the permeability of the cells, PEF enables a higher mass transfer of intracellular components. Thus, this emerging technology decreases the need for high temperatures and amounts of solvent, reducing environmental impacts and enhancing the energy efficiency of the processes. Moreover, it promotes minimal changes when it comes to the nutritional and sensory aspects of the product due to the low temperature and holding time of the extraction. However, for the integration of PEF on a large industrial scale, more studies to optimize the process conditions and parameters are still needed [151].

Feed and Food Waste and Non-Compliance

The study of Pataro et al. [154] explored various parameters affecting the extraction process, including electric field strength (1–5 kV/cm), pulse duration, and number of pulses (10–833 pulses). The authors also used set parameters such as total specific energy input (5 and 10 kJ/kg), a pulse frequency of 10 Hz, and a pulse width of 20 μ s. The results indicated that higher electric field strengths and increased pulse durations generally led to higher lycopene extraction yields. However, excessively high electric field strengths or prolonged pulse durations could result in undesirable effects, such as increased energy consumption or degradation of lycopene. Furthermore, the researchers examined the impact of PEF-assisted extraction on lycopene stability. They found that the extracted lycopene exhibited good stability and retained its antioxidant properties, highlighting the potential of PEF as a suitable method for the recovery of high-quality lycopene from tomato processing by-products.

Kim et al. [155] investigated the use of PEF as a pre-treatment method to enhance the extraction of quercetin from onion skin using subcritical water extraction. By subjecting onion skin samples to PEF pre-treatment prior to subcritical water extraction, the study aimed to disrupt the plant cell structure and facilitate the release of quercetin. The results demonstrated that PEF pre-treatment significantly improved the extraction efficiency of quercetin from onion skin compared to conventional subcritical water extraction methods. Optimal PEF parameters were determined (electric field strength: 2.5 kV/cm, pulse frequency: 25 Hz, width: 25 μ s, duration: 15 s), leading to higher quercetin yields and shorter extraction times. Moreover, the study confirmed the preservation of quercetin stability during the PEF pre-treatment and subsequent subcritical water extraction, ensuring the quality of the extracted compound. The summary results of both works are presented in Table 19. The application of PEFs in combination with subcritical water extraction offers a sustainable and efficient approach to the recovery of quercetin from onion skin.

Table 19. Summary of studies reported on PEF extraction of different food waste sources.

Biomass Source	PEF Conditions	Yield	Ref.
Tomato peels	3 kV/cm, 10 kJ/kg, ethyl lactate 1:20 (<i>w:v</i>)	Lycopene (10,140 mg/kg DW)	[154]
Onion peels	2.5 kV/cm, 25 Hz, 25 μ s, 15 s	Total quercetin (19.25 mg/g DW)	[155]

Microalgae

An appropriate cell disintegration process must maximize the yield and value of the compounds extracted [156]. In other words, it disintegrates all the cells precisely without the chemical contamination or degradation of the desired compounds. For large-scale production, it is also important that the disintegration process can be scaled up. All these properties influence the overall efficiency of the disintegration process and, therefore, its overall energy consumption, which is a crucial issue in biofuel production.

PEF treatment may be a promising alternative to conventional cell disintegration methods. The exposure of biological cells to high-intensity electric field pulses can alter the structure of the cell membrane. The external field provokes a charging of the membrane. As a result, the cell membranes lose their barrier function as it becomes more permeable, a phenomenon often referred to as “electroporation” or “electropermeabilization” [156]. This phenomenon aids solvent penetration and overall extraction efficiency [157].

Table 20 presents the results of studies conducted on the application of PEFs for the extraction of bioactive compounds from different microalgae species.

Table 20. Summary of studies reported on PEF extraction of different microalgae species.

Biomass Source	PEF Conditions	Yield	Ref.
<i>Spirulina</i> sp.	Deionized water, 2 h, 41 °C, 20–25 kV cm ^{−1} , 300 Hz, 100 kJ kg ^{−1}	Phycocyanin: 119.48 ± 6.7 mg g ^{−1}	[158]
	Distilled water, 360 min, 40 °C, 25 kV cm ^{−1} , 150 μ s, 110 kJ kg ^{−1}	Phycocyanin: 151.94 ± 14.22 mg g ^{−1}	[159]
<i>Heterochlorella luteoviridis</i>	Ethanol/water, 180 V, 50 min	Carotenoids: 73%	[160]
	Ethanol/water, 180 V, 50 min	Lipid: 83%	

In two studies, *Spirulina* sp. biomass was subjected to PEFs using different conditions. Distilled water was used as the extraction solvent, and various parameters such as temperature, voltage, frequency, and energy input were adjusted and evaluated. The extraction yields of phycocyanin were reported to be 119.48 ± 6.7 mg/g and 151.94 ± 14.22 mg/g, respectively. These results indicate that PEF can effectively extract phycocyanin from *Spirulina* sp. and the extraction yield can be influenced by the specific PEF conditions applied [158,159].

Another study focused on *Heterochlorella luteoviridis*, where PEFs were applied using an ethanol/water solvent mixture. The electric field strength was set at 180 V for 50 min. The study evaluated the extraction of carotenoids and lipids separately, reporting extraction yields of 73% for carotenoids and 83% for lipids. This indicates that PEFs can be utilized to extract both carotenoids and lipids from *Heterochlorella luteoviridis*, with high extraction efficiencies [160].

Overall, the table demonstrates the potential of PEFs as a promising technique for the extraction of bioactive compounds from microalgae, including phycocyanin, carotenoids, and lipids. The specific PEF conditions, such as the choice of solvent, duration, voltage, and energy input, play a crucial role in determining extraction yields and efficiency.

Lignocellulosic Biomass Sources

A key effect of PEF applications in biomass processing today is the enhanced mass transport rate of extraction of different molecules, such as carbohydrates, lipids, pigments, phenols, lipids, and water. In addition, PEFs have been shown to affect biomass struc-

ture by decreasing lignin contents, which can assist in the deconstruction of the complex lignocellulose cell walls [161].

To the best of our knowledge, studies focused on the PEF extraction of lignocellulosic sources are very scarce. Bouras et al. [162] used PEF extraction on *Picea abies* (L.) Karst, where the main parameters for the diffusion kinetics and characterization of extracts were studied (i.e., moisture content, pH, electrical conductivity, polyphenols concentration, and antioxidant activity). The results showed positive effects of PEFs on intracellular compound extraction since the TPC showed an increase of more than eight times with the use of PEF treatment when compared to PEF-untreated biomass also analyzed in the study (Table 21).

Table 21. Summary of studies reported on PEF extraction of different lignocellulosic sources.

Biomass Source	PEF Conditions	Yield	Ref.
<i>Picea abies</i> (L.) Karst.	20 kV/cm, SB: 10 (<i>w/w</i>)	Polyphenols	[162]

4. Conclusions and Future Perspectives

Herein, a state-of-the-art review was performed, shedding light on the existing technologies available for bioactive compound extraction from major biorefinery sources (i.e., food waste, microalgae, and lignocellulosic biomass). From these biomass sources, phenolic compounds such as catechin, flavonoids, bioactive pigments (carotenoids, chlorophylls, anthocyanins, phycocyanin), fatty acids, and others can be obtained. These compounds have several health-promoting capabilities such as antioxidant, anti-carcinogenic, anti-bacterial, and anti-microbial properties that are of great interest to several industries and society at large. From the literature review, it was possible to deduce that maceration studies of bioactive compound extraction from microalgae species are very scarce, probably due to the inability of this technique to rupture microalgae cell walls. In addition, studies focused on the use of the PLE technique in lignocellulosic sources is also very undeveloped and should be further researched. Moreover, studies focused on the use of natural deep eutectic solvents, ionic liquids, and deep eutectic solvents are very scarce and should be further studied in order to better understand the potential of these solvents to extract valuable bioactive molecules from the three biomass sources. Overall, future studies focused on optimizing bioactive compound extraction should be conducted, and life cycle assessment and EcoScale analysis should be conducted in order to fully evaluate the environmental impacts and overall potential of the processes.

Author Contributions: Investigation, writing—original draft preparation, R.M., A.B. and B.A.; Conceptualization, R.M.; writing—review and editing, H.S. and R.P.; supervision, funding acquisition, J.N. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the BioPinus project, CENTRO-01-0247-FEDER-072630—Centro Portugal Regional Operational Program (Centro2020), under the PORTUGAL 2020 Partnership Agreement, through the European Regional Development Fund (ERDF); RHAQ CENTRO, CENTRO-04-3559-FSE-000146—Centro Portugal Regional Operational Program (Centro2020), under the PORTUGAL 2020 Partnership Agreement, through the European Social Fund (ESF); the Centre Bio R&D Unit (UIDB/05083/2020); and the Interface Mission RE-C05-i02 under the Portuguese Recovery and Resilience Plan through the European Union NextGenerationEU Fund.

Data Availability Statement: No new data created.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

AA	acid ascorbic
AAE	ascorbic acid equivalent
ABTS	2,2'-azino-bis-3-ethylbenzthiazoline-6-sulphonic acid
AGREE	analytical greenness metric
ChCl	choline chloride
DES	deep eutectic solvents

DHA	docosahexaenoic acid
DPPH	2,2-diphenyl-1-picrylhydrazyl
DW	dry weight
EPA	eicosapentaenoic acid
FAO	food and agricultural organization
Fen-Thy	fenchyl alcohol-thymol
GAE	gallic acid equivalents
GAPI	green analytical procedure index
GHCs	green house emissions
HRW	hydrogen-rich water
LCA	life cycle assessment
NADES	natural deep eutectic solvents
NEMI	national environmental methods index
MAE	microwave-assisted extraction
MHG	microwave hydrodiffusion and gravity
PLE	pressurized liquid extraction
PEF	pulsed electric fields
PUFAs	poly-unsaturated fatty acids
QE	quercetin equivalents
QSAR	quantitative structure activity relationship
ROS	reactive oxygen species
SB	solvent to biomass ratio
SC	supercritical fluid extraction
SC-CO ₂	supercritical carbon dioxide
SFMH	solvent-free microwave hydrodistillation
TE	trolox equivalent
TPC	total phenolic content
UAE	ultrasound-assisted extraction
UFAs	unsaturated fatty acids
UN	united nations

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