Review

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Evaluating the Sustainability of Emerging Extraction Technologies for Chemical Analysis of Food Waste: Microwave, Ultrasound, Enzyme-Assisted, and Supercritical Fluid Extraction

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Abstract

The food industry generates substantial waste, raising economic and environmental concerns. Green Chemistry (GC) highlights extraction as key to waste valorization, driving interest in sustainable methods to recover valuable compounds efficiently. This review focuses on evaluating the sustainability of some of the most used emerging technologies, namely Microwave, Ultrasound, Enzyme-assisted, and Supercritical Fluid Extraction, and their compliance with GC principles. The review begins by exploring the principles, key parameters, and main advantages and disadvantages of each technique. Subsequently, the sustainability of these extraction techniques is evaluated in selected studies using the Analytical GREEnness Metric Approach (AGREEprep). By calculating the Greenness Score (GS), this metric assesses the adherence of extraction processes with sustainability standards. The analysis reveals variations within the same extraction technique, driven by factors such as the choice of solvents and operating conditions, as well as differences across the various extraction methods.

Keywords: sustainability; greenness score; valorization; green chemistry; agricultural wastes; microwave; ultrasound; enzymes; supercritical fluids

1. Introduction

Sustainability has aroused a huge concern for the scientific community in the last 4 decades. This term was defined as the development that meets the needs of the present without compromising the ability of future generations to meet their own needs [1], which implies that the industry has to reach a balance of the impact at each stage of the supply chain in terms of economic, social, environmental and technological sustainability [2]. In this sense, the European Community established the European Green Deal as strategy to be transformed into a climate neutral Europe by 2050 [3]. The core of this strategy is to achieve a sustainable food system from a circular bioeconomy point of view [4]. These initiatives agree with the United Nations 2030 Agenda [5] along with its 17 Sustainable Development Goals [6]. Specifically, goals 12.3 and 12.5 are committed to reduce 50 % food loss at production sector and supply chain, employing prevention an 3R strategy for waste management: reduce, reuse, and recycle [5–7].

The food industry, as one of the largest industries around the world, will lead to a sharp increase in food production in the upcoming 50 years due to population increases [2]. In 2022, the European Union (EU) estimated that this production generated 132 kg per inhabitant of which 46 %

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corresponds to the food supply chain. Indeed, from the 59 million tons of fresh mass generated, a 19 % and 8 % accounted for the processing and manufacturing and the primary production sectors, respectively [8,9]. In fact, to be in line with sustainable food production and consumption, the EU target is to reduce food waste to a 10 % for processing and manufacturing sector by 2030 [6]. Thus, one of the major challenges in food industry is to upgrade this biomass loss (co- and by-products) from its production due to the high economic and environmental impact [2,6].

In view of this aim, several strategies have been developed for biomass valorization into animal food, landfilling, biofuel, composting and the recovery and reuse of high-added-value constituents from food residues [2]. From them, this last is in the spotlight due to the application of these compounds as novel food ingredients to increase the product shelf-life, supply sustainable nutrient-dense foods, and reduce food processing contaminants as part of the challenges that the food industry also must face [10–12]. Furthermore, the recovery of high-added-value constituents from biomass comprises five stages according to the Universal Recovery Strategy stated by Galanakis [12,13]. Briefly, from an Analytical Chemistry (AC) point of view, it consisted of sample preparation and isolation of target components, where extraction is the most decisive stage of this process [12,13].

In addition to being the key step from this process, extraction is also the most critical step due to the concern towards environmental issues [14,15]. Bearing the concept of sustainability in mind, concepts like green and white AC have gained attention meaning that AC adopted the 12 principles of the Green Chemistry (GC) to become into Green AC (GAC), or White AC (WAC) if it also comprises analytical efficiency and practical aspects [14–16]. For WAC, the blue applicability grade index (BAGI) has been presented using the Red-Green-Blue model to complement GAC tools [17], whereas for GAC, several metrics such as analytical eco-scale, National Environmental Methods Index (NEMI) [18], Green Analytical Procedure Index (GAPI), Analytical Greenness calculator (AGREE) [19], and the update versions complementary GAPI (ComplexGAPI) [20], complementary modified GAPI (ComplexMoGAPI) [20] and AGREE for sample preparation (AGREEprep) [15] were developed to measure the 12 GC principles.

Although the integration of these principles is hard to accomplish since a “solvent free” extraction and low energy consumption, nowadays, a stunning effort is being done to develop sustainable extraction methods [14,15]. Advanced analytical extraction techniques, namely Microwave-assisted Extraction (MAE), Ultrasound-assisted Extraction (UAE), Enzyme-assisted Extraction (EAE), and Supercritical Fluid Extraction (SFE), among others, and their combinations have been developed as a solution for sustainable extraction [12,21]. These techniques have demonstrated to overcome traditional extraction problems, better recoveries of bioactive compounds, and are considered more sustainable compared to other advanced extraction techniques [22,23]. Furthermore, conventional extractions are also adapting to those principles using non-toxic, reusables and biodegradables reagents such as natural deep eutectic solvents (NADESs) which are considered a sustainable alternative with the benefit of enhancing the antioxidant capacity of the extract [24–26]. Besides, latest articles of advanced analytical extraction techniques also include the use of NADESs as a greener upgrade solution [24]. Thus, the aim of this review is to evaluate and analyze the grade in which the analytical extraction techniques employed for food wastes valorization are aligned with GC principles employing the AGREEprep tool, since it refers to the step of sample preparation focusing on extraction methodology.

2. Methodology

To prepare this review on sustainable extraction techniques, namely MAE, UAE, EAE, and SFE, for managing industrial food waste, a comprehensive search was conducted across the Scopus database, including articles, review papers, and books. Keywords used in the search included “waste”, alongside the full name of the respective technique, “food waste”, “valorization”, “agricultural waste”, “sustainable extraction”, “green chemistry”, “emerging technologies”, “industrial waste”, and “by-products”.

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Given the primary objective of this review, focusing on evaluating the sustainability of common emerging extraction technologies as alternatives to conventional methods, the environment impact of these technologies was assessed. For this purpose, and due to the limited availability of standardized metrics for evaluating extraction processes sustainability, the Greenness Score (GS) was calculated using the AGREEprep tool [15]. This metric reflects compliance with GC criteria, each with assigned weights expressed in brackets, to visually represent adherence to sustainability standards:

1. Favor *in situ* preparation (1)
2. Use safer solvents and reagents (5)
3. Target sustainable, reusable and renewable materials (2)
4. Minimize waste (4)
5. Minimize sample, chemical and material amounts (2)
6. Maximize sample throughput (3)
7. Integrate steps and promote automation (2)
8. Minimize energy consumption (4)
9. Choose the greenest possible post-sample preparation configuration for analysis (2)
10. Ensure safe procedures for the operator (3)

Criterion 2 is assigned the highest weight (5 out of 28) due to the significant impact of solvents and reagents on the green attributes of the entire extraction process. Following this, criteria 4, 8, and 10, which address waste, energy demands, and operator safety, play a critical role in the GS assessment and are assigned the second-highest weight (4 out of 28). This metric uses clear, colorful round pictograms to visually represent the results. The inner circle displays the overall GS, as both a color and a score, resulting from the scores of each criterion and their respective weights. The GS ranges from 0 to 1, where 0 (reddest) indicates the worst performance across all criteria, and 1 (greenest) represents the best, reflecting the greenest possible process. Around the inner circle, the performance of the ten evaluated criteria is depicted as individual segments, with the length of each segment proportional to the criterion's weight in the overall score and the color providing a visual representation of its performance.

To select studies on emerging technologies relevant to this review, specific criteria were applied to ensure a minimum of 20 articles after the literature screening process. The search was limited to English-language articles published within selected dates in the Scopus database. Identified references were screened based on exclusion criteria, and the final selected studies were assessed for their integration into GC principles through GS determination.

For MAE, articles published from 2022 and 2024 were identified using the search terms "microwave" AND "assisted" AND "extraction", in the title, abstract, and keywords, restricted to the "Agricultural and Biological Sciences" subject area. The search included the keywords: "waste", "microwave-assisted extraction", "microwave assisted extraction", "microwave radiation", "microwave-assisted", "microwaves", "microwave irradiation", "microwave", "microwave-assisted technique", "microwave-assisted hydrothermal", and "microwave extraction. This yielded 41 documents, of which 4 were excluded for not using agricultural waste as raw material, 1 for not being in English, 5 for being review articles despite being classified as research papers, 2 for evaluating microwave technology combined with other methods, and 4 for lacking information on operational extraction parameters making GS estimation impossible. After screening, 25 articles were selected for GS determination.

For UAE, articles published between 2023 and 2024 were identified using the terms "ultrasound" AND "assisted" AND "extraction" in the title, abstract, and keywords, within the "Agricultural and Biological Sciences" subject area. The search included keywords "waste", "ultrasound assisted extraction", "ultrasound-assisted extraction", "ultrasonics", and "ultrasound", yielding 48 articles. After screening, 2 articles were excluded for not focusing on agricultural waste, 6 for being review articles mislabeled as research papers, 1 for lacking free access, 4 for insufficient information on operational parameters for GS estimation, and 12 for using combined extraction techniques. This resulted in 23 final articles selected for GS determination.

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For EAE, no date or subject area restrictions were applied. Articles containing "enzyme-assisted" AND "extraction" in the title, abstract, and keywords were searched using "waste" as an additional keyword. This yielded 89 articles, but only 24 articles were selected after applying exclusion criteria. 12 were excluded for focusing on non-agricultural waste, 9 for being review articles instead of research papers, 21 for addressing enzyme-related topics unrelated to agricultural waste valorization, 3 for lacking operational extraction parameters requires for GS estimation, and 20 for employing enzymatic extraction in combination with other technologies.

For SFE, articles published between 2022 and 2024 were identified using the terms "supercritical" AND "fluid" AND "extraction" in the title, abstract, and keywords, within the "Agricultural and Biological Sciences" subject area. The search included keywords "waste", "supercritical fluid extraction" and "supercritical fluid", yielding 50 articles. After screening, 4 articles were excluded for not focusing on agricultural waste, 14 for being review articles mislabeled as research papers, 1 for lacking free access, 6 for insufficient information on operational parameters for GS estimation, and 3 for using combined extraction techniques. This resulted in 22 final articles selected for GS determination.

Figure 1 provides a detailed overview of the literature search process, including the search, screening, and selection after applying the specific exclusion criteria for each technique.

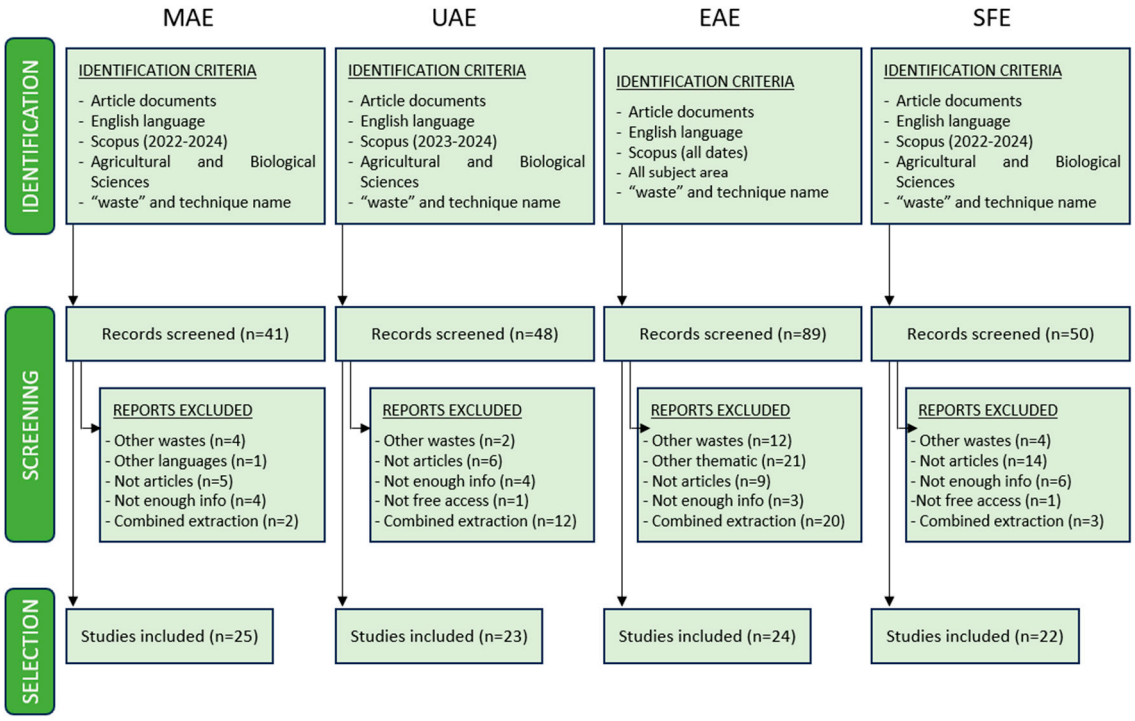

Figure 1. Flow diagram illustrating literature search and screening process, including specific exclusion criteria for each technique, to select relevant studies for GS calculation and evaluation within Green Chemistry principle.

3. Microwave-Assisted Extraction (MAE)

3.1. Principle

Microwave-assisted Extraction (MAE) is an innovative and efficient extraction technology that uses non-ionizing electromagnetic waves within a frequency range of 300 MHz to 300 GHz [27]. These electromagnetic waves are composed of electric and magnetic fields that oscillate perpendicularly to one another and can heat materials by converting absorbed energy into thermal energy [28]. The extraction process using microwaves relies on the capacity of particles within a matrix to absorb this radiation [29]. During MAE, energy transfer occurs through two mechanisms: ionic conduction and

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dipole rotation. When electromagnetic waves are applied, ions migrate through the solution, and the solution resistance to this ions movement generates friction, leading to uniform heat throughout the system. Dipole rotation, on the other hand, involves molecules reorientation in response to the applied electric fields, causing thermal agitation as they return to a disordered state when the waves are removed [27,30]. This microwave-induced heating of solvents and matrices accelerates the extraction process [7].

The MAE process follows these key steps: first, microwave irradiation transfers heat from the solvent to the sample matrix, causing rapid and uniform heating. The rise in temperature causes moisture within the matrix to evaporate, dehydrating the cells and increasing the internal pressure. This increase in pressure leads to cell swelling and rupture, which increases porosity and releases the intracellular components [7,28,30,31]. This enhanced mass transfer, with heat and mass moving in the same direction, shortens extraction time, reduces use of solvents, and improves extraction yield [30]. Efficiency of dielectric heating, which depends on the power and frequency of the microwaves, allows MAE to selectively extract different target compounds [31]. The effectiveness of MAE depends on the dielectric properties of both the solvent and the matrix, making it particularly suitable for extracting compounds of medium to high polarity. Since polar molecules absorb microwave radiation, solvents typically used in MAE are polar, such as methanol, ethanol, and water, as well as mixtures of solvents with varying dielectric constants to prevent overheating [31]. As a result, MAE is usually considered as a combination of traditional solvent extraction methods combined with the use of microwave energy [30,32].

3.2. Influencing Parameters

The key parameters influencing MAE that can be adjusted to optimize extraction efficiency include the solvent selection, SSR, temperature, processing time, irradiation power, and additional factors such as stirring rate and the characteristics of the sample matrix [32–34]. Although MAE can be performed without a solvent, known as microwave-assisted solvent-free extraction, the use of a solvent generally enhances extraction efficiency. As in conventional extraction, solvent selection is crucial, and it must efficiently absorb microwave energy to facilitate rapid heating. A solvent with a higher dielectric constant and dissipation factor will distribute heat more uniformly throughout the sample matrix, resulting in increased extraction yields [30,35]. Other solvent-related factors to consider include its selectivity for the target compound, solubility, penetration capacity, and interactions with the extraction matrix [30]. Regarding the SSR, its effect depends on the nature of the sample matrix. At very low ratios, the reduced solvent surface area hinders microwave penetration into the material, lowering extraction efficiency. Conversely, a high SSR requires more time to reach the needed temperature, reducing the extraction efficiency [35]. The solvent volume should be sufficient to fully immerse the sample matrix during microwave treatment, but not excessively high to avoid increased time and energy consumption [30]. Temperature and irradiation power are closely linked, as microwave power supplies the energy needed to increase the matrix temperature. Higher microwave power leads to increased temperature, which enhances solvent penetration into the matrix and increases extraction yields while reducing extraction time. However, temperature control is essential to avoid unwanted reactions such as isomerization, transesterification, or thermal degradation [35]. While longer processing times generally result in higher extraction yields, prolonged exposure to heat can degrade thermosensitive compounds. Therefore, MAE requires balancing extraction yield and the stability of thermosensitive compounds by carefully adjusting operational parameters [30]. The sample matrix characteristics, including its dielectric properties, pretreatment, particle size, and moisture content, also affect MAE efficiency. For instance, reducing particle size through milling increases the surface area in contact with the solvent, enhancing mass transfer and extraction yield. However, very fine particles may complicate the separation of the extract from the residue, necessitating an additional separation step to obtain a pure extract [31]. Additionally, moisture content in the matrix aids extraction by acting as a solvent that, when heated and evaporated, increases pressure and enhances the extraction [30].

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3.3. Advantages and Limitations

Compared to traditional extraction methods, MAE is a more efficient and cost-effective technology [34,36]. It utilizes a noncontact heating source that generates uniform heat, leading to increased efficiency and selectivity in the extraction process [27]. Additionally, MAE is time-saving, as it requires less time than conventional methods that rely on convention or conduction [29]. MAE consistently demonstrates higher efficiency than conventional extraction techniques, offering greater yields in shorter operational times [27,30,36,37]. During MAE, achieving maximum extraction efficiency depends heavily on selecting the optimal process parameters [38]. Furthermore, it is easy to operate [30] and provides reproducible results [35], with low equipment and energy costs [39] along with reduced solvent consumption, which minimizes environmental contamination [34].

A key limitation of MAE is temperature control. While higher temperatures typically improve extraction yields, excessive heat can damage or degrade thermolabile compounds [27]. Similarly, increasing power levels, which intensify microwave heating, can reduce yields and introduce undesired compounds into the extract. Therefore, finding optimal extraction conditions is essential [7]. The lack of selectivity in MAE, along with the presence of residual materials and extracted compounds, reduces the purity and quality of the extracts, often requiring a post-extraction separation step [7,27]. Additionally, the composition and characteristics of the matrix affect the MAE process, as complex matrices can impede the uniform penetration of microwave energy, resulting in incomplete extraction or compound degradation [7].

3.4. Integration into Green Chemistry

In recent years, numerous studies have evaluated the use of MAE for valorizing food waste, primarily focusing on the recovery of phenolic compounds from various sources such as fruit by-products, dried fruit shells, and grain residues [40–45], as well as the extraction of pectin from fruit peels like pomelo, tangerine, pineapple, orange, and banana [46–50]. The literature review and screening of MAE-related studies identified 41 relevant articles, of which 25 were selected for assessment against GC principles after applying specific exclusion criteria (Figure 1). Table 1 summarizes the raw materials used, operational conditions, target compounds and optimal yields, as well as estimated GS values according to the detailed methodology.

The average GS value across the evaluated studies was 0.42±0.09, with 72 % of the studies scoring from 0.35 to 0.55, and 12 % of the studies scoring above (Table 1). Only four out of the 25 studies scored below 0.35. Although high scores might be expected due to the inherently sustainable nature of microwave technology, several criteria negatively impacted the overall GS values.

The lowest scores were observed for criterion 2, related to the "use of safer solvents and reagents". C2 scored poorly due to the use of hazardous substances such as acidic and basic solutions during extraction, pressurizing agents, and hazardous/toxic reagents in the precipitation or washing steps (e.g., acids, methanol and other alcohols), as the use of more than 10 mL of hazardous solvents leads to a score of 0 for C2, significantly lowering the GS. In contrast, Montemurro et al. [51] valorized spent coffee ground using MAE with water as the solvent, achieving a high C2 score and the highest GS (0.58).

Other criteria with consistently low scores included criterion 6, due to limited sample throughput per hour, and criterion 8, due to high energy consumption from prolonged microwave exposure. The worst scores for C6 were found in studies on pomegranate [52], corn cobs [53], and hazelnut by-products [54] to obtain phenolic-rich extracts, with extraction times ranging from 30 to 80 min. Likewise, low C8 scores were observed in studies using microwave power levels of 500 to 1,500 W for up to 80 min [42,47,52–54], resulting in high energy usage. Significant improvements in these criteria were achieved by Arora et al. [46], who conducted the fastest extraction (1.8 min), enabling 33 samples per hour and reducing energy consumption to just 18 Wh/sample, resulting in high scores for C6 and C8 and an overall GS of 0.49. Similarly, Barrios et al. [55] obtained GS values between 0.45-0.47 by using a multiwave reactor capable of processing 16 samples simultaneously, which reduced both time and energy use (< 35 Wh/sample) and minimized operator exposure risks.

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The lowest GS (0.18) was obtained in a study focusing on tangerine peel valorization using acidified water under 5 bar of pressure [47]. The use of nitrogen as a pressurizing agent reduced scores for criteria 4 and 10 due to waste generation and safety risks. Additional steps in the pressurization process affected C7, while the use of citric acid and HCl (pH 1-2) increased hazard levels and waste generation, lowering C2, C10, and C4. High energy consumption (300 Wh/sample) also led to a poor C8 score, cumulatively resulting in the lowest overall GS.

Conversely, the highest (greenest) GS values were primarily associated with criteria 5 and 9, which generally scored yellow to green. These reflect the small sample quantities used for extraction (typically up to 5 g) and the adoption of spectroscopic methods such as UV-vis spectrophotometry for post-analysis. Among the six studies with GS above 0.5, several achieved high scores in C4 and C10 by using safer solvents like water, ethanol, and weak acid solutions (e.g., citric acid), generating less than 1 g of waste (C4) and fewer than 2 hazards (C10), yielding overall GS values between 0.51 and 0.58 [43,48,51,56–58].

In conclusion, the analyzed MAE studies generally aligned with Green Chemistry principles, but their GS values were not as high as might be expected from a green extraction technology. This is largely due to the use of non-green solvents in extraction and pre/post-processing stages. To further enhance the sustainability of these processes, the use of greener solvents should be prioritized. Additionally, implementing parallel extraction systems could improve throughput and reduce energy consumption, thereby boosting overall GS performance.

4. Ultrasound-Assisted Extraction (UAE)

4.1. Principle

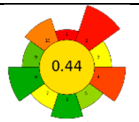
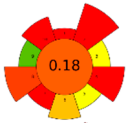
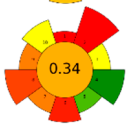
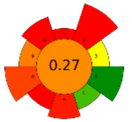
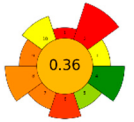
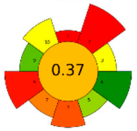
Non-conventional methods are increasingly being applied to overcome the limitations of conventional methods for efficient and sustainable extractions [59]. In this sense, the ultrasound technology has garnered the community interest and has been widely used for the pretreatment or treatment of solid–liquid extraction of a bast number of bioactive compounds from a wide range of sources turning it into a perfect tool for agro-food biomass valorization [60–63].

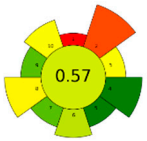
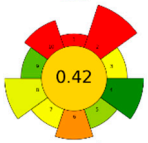
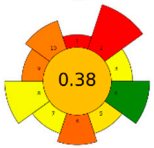
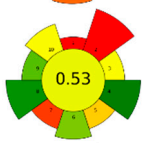
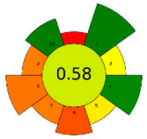
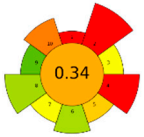
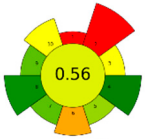
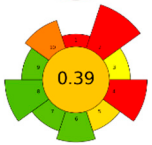
Ultrasound is defined as sound waves with frequencies above the threshold for human hearing (> 16 kHz) [63,64]. These ultrasound waves propagate through the media generating cycles of compression and shearing forces (decompression) [65] due to intense localized changes in pressure and temperature [64,66]. This propagation depends on the excitation characteristics and the properties of the media and generates the rising of cavitation bubbles [64,65,67]. For example, the normal frequency for bubbles generated in water is between 5 and 25 kHz due to their radio (1-100 μm) [67], although in the food industry frequencies from 20 kHz to 10 MHz are employed [64]. The propagation ends with the shock waves and the bubbles collapsing [64,65].

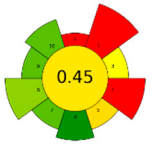
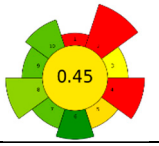
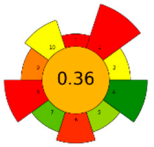
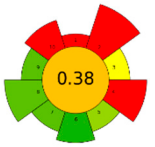
In depth, the process of acoustic cavitation results from several stages (in general, phase changes and nucleation) and two phenomena: sonocapillarity and sonoporation [67]. Thanks to them, plant cell walls are disrupted during this process enhancing the penetration of the solvent into the matrix due to the reduction of particle size and accelerating the mass transfer by the diffusion of the intracellular analytes into the solvent [59,61,63,65–69]. Thus, the use of this principle for the release and solubilization of target compounds is recognized as ultrasound-assisted extraction (UAE), or sonication, [63].

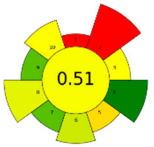
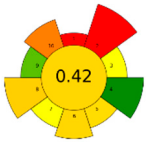
Besides, UAE is also considered as non-thermal technology [62] although the cavitation process induces an increase in temperature in the propagation and sample media. However, the combination of UAE and heat enhances the extraction and changes in functional and structural properties of target compounds [70].

Table 1. Summary of studies on Microwave Assisted Extraction (MAE) from agricultural wastes, detailing the raw materials studied, key extraction parameters, and the greenness score (GS) estimation based on Wojnowski et al. [15].

	Raw material	Solvent	SSR ratio	MP (W)	T (°C)	t (min)	Compound	Optimal yield	GS	Ref.
1	Pomelo peels	Water acidified with HCl (pH 2.0)	1:10-1:20	300-600	n.r.	1.3-1.8	Pectin	3.09-5.57 % EY		[46]
2	Tangerina peels	Aqueous acid solutions (pH 1-2)	1:5-1:50	1500	70-110	4-12	Pectin	30±2 % EY		[47]
3	Corn cobs	30-80 % Ethanol	1:15-1:45	500-800	40-90	5-30	TPC	274,147.2 mAu*s		[53]
		NADESs: Choline chloride/lactic acid (1:2, v/v); Choline chloride/glycerol (1:2, v/v); Choline chloride/1,2-propanediol (1:2, v/v); Choline chloride/urea (1:1, v/v)	1:20-1:40	500-800	50-90	5-30	TPC	86,047.5 mAu*s		
4	Orange waste	50-100 % Ethanol and acetone solutions	1:20	500	45-75	10-20	TPC; Hesperidin; Neohesperidin; Naringenin; Naringin	16.68 %; 2.08 %; 3.82 %; 2.04 %; 6.32 % EY		[40]
5	Pineapple rind	Water, ethanol, acetone	1:3-1:10	100-300	n.r.	5-15	Bromelain	127.8 Units BA/mL; 2.55 mg/mL protein content		[71]
6	Chestnut shell	NaOH (0-0.2 M)	1:25	200-1000	n.r.	3-15	TPC; Melanin	274.09 mgGAE/g; 26.11 % EY		[41]
7	Rice bran	60 % Ethanol	1:10	90-800	n.r.	30	TPC	60.69±0.61 % EY		[42]

8	Pistachio shells	20-90 % Ethanol	1:20-1:35	150-1000	≤ 64	0.83-4.5	TPC	20.57±0.92 mgGAE/g		[43]
9	Pomegranate waste	20-100 % Ethanol	1:10-1:30	150-750	n.r.	2-10	TPC; TEC; TFC	432.05 mgGAE/g; 279.2 mgTAE/g; 25.0 mgQE/g		[45]
10	Black bean waste	Ethanol:water (100:0-0:100 v/v) with 1 % lactic acid	1:20-1:50	100-600	n.r.	2-6	TPC; TFC; TAC	197.23±0.02 mgGAE/g; 87.65±0.06 mgQE/g; 34.14±0.03 mg/g		[72]
11	Banana peel	Water	1:3.4	800	50-170	0-15	Homogalacturonan; Rhamnogalacturonan-I	837.2 mg/g; 111.1 mg/g of alcohol-insoluble solids		[73]
12	Olive pomace	52.7 % Ethanol	1:8.3-1:50	100-800	n.r.	1-3	TPC	15.30 mgGAE/g		[58]
13	Spent coffee ground	Water	1:3	850	55-200	10	Melanoidinds; Sugars; Chlorogenic acid; Caffeic acid	35.55±0.16 mg/g; <10 mg/g; 1.97±0.11 mg/g; 0.05±0.04 mg/g		[51]
14	Onion and garlic waste	0.1 N Citric/acetic acids/HCl/H_2SO_4 solutions	1:30	600	n.r.	4	Galacturonic acid	67.15±0.64 % EY		[74]
15	Jackfruit rags	Citric acid solutions (pH 1-2)	1:20-1:30	50	60-70	5-15	TPC; Pectin; Protein content;	4.64±0.04 mg/g pectin; 29.78 % EY; 2.10±0.01 % EY		[56]
16	Pomegranate by-products	Water	1:10	1,250	< 40	80	TPC; Punicalins; Punicalagins; Ellagic acid	0.296±0.001 gGAE/100g; 0.057±0.002 g/100g;		[52]

								0.195±0.001g/100g; 0.045±0.002 g/100g		
17	Sugarcane waste (bagasse)	60-80 % Ethanol	1:10	100-500	n.r.	1-5	TPC	12.83±0.66 mgGAE/g		[48]
18	Pineapple peels	0.5 N Sulfuric acid (pH 1.5-2.5)	1:10-1:30	400-600	80	2.5	Pectin; AUA	2.44 % EY; 54.40 % EY		[44]
	Brewer's spent grain	NaOH (0-0.64 M)				0-12.56		92.05 % EY; 48.42 mgGAE/g; 8.68 mgCE/g; 13.84 g/L		
19	Spent coffee ground	NaOH (0-1.31 M)	1:10	1,800	56-124	1.59-18.41	Proteins; TPC; TFC; Total sugars	58.99 % EY; 52.08 mgGAE/g; 15.95 mgCE/g; 5.50 g/L		[55]
	Kale stems	Water and NaOH (0.16-1.84 M)				1.59-18.41		95.23 % EY; 34.32 mgGAE/g; 2.46 mgCE/g; 15.20 g/L		
20	Hazelnut by-products	NADESs: Choline chloride/1,2-butandiol (1:4, v/v); Choline chloride/1,2-propylene glycol (1:4, v/v); Choline chloride/glycerol (1:4, v/v); Choline chloride/DL-malic acid:water (1:1:2, v/v); Sucrose/lactic acid:water (1:5:7, v/v); Fructose/lactic acid:water (1:5:5, v/v); Sucrose/choline chloride/water (1:4:4, v/v); Fructose/choline chloride/water (2:5:5 v/v)	1:10-1:20	1,500	50-100	10-40	*D*-(-)-Quinic acid; Gallic acid; Protocatechuic acid; Catechin; Quercetin-3-*O*-rhamnoside	24.38±0.61 mg/kg; 6.80±0.15 mg/kg; 6.95±0.17 mg/kg; 7.32±0.15 mg/kg; 13.99±0.21 mg/kg		[54]
21	Tomato seeds	80 % Methanol, 80 % ethanol, 80 % acetone	1:20-1:50	200-800	n.r.	0.33-2	TPC	265.31±7.87 mgGAE/100g		[75]

22	Seedless sesame capsules	Acidified water with citric acid (pH 1.5-3)	1:20-1:50	300-700	n.r.	1-5	Pectin	138±4 g/kg	 [57]
23	Orange peels	Acidified water with 0.1 N HCl (pH 1.5)	1:25	620	n.r.	3	Pectin	19.3±0.16 % EY	 [49]
24	Tomato seeds	40-80 % Ethanol	1:50-1:80	92.7	40-80	5-15	TPC; Chlorogenic acid; Rutin; Naringenin	1.72±0.04 mgGAE/g; 1.11±0.34 mg/100g; 1.38±0.02 mg/100g; 2.99±0.11 mg/100g	 [76]
25	Banana peels	Citric acid (0.1 M), tartaric acid (0.1 M)	1:20	420-613	n.r.	5-10	Pectin	15.23±0.52 % EY	 [50]

SSR: Solid-to-solvent ratio (w/v); MP: Microwave power; TPC: Total phenolic content; TFC: Total flavonoid content; TAC: Total anthocyanin content; TEC: Total ellagitannin content; AUA: Anhydrouronic acid content; n.r.: not reported; EY: extraction yield; BA: bromelain activity.

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4.2. Influencing Parameters

This technique is considered a green extraction method due to the possibility to select the suitable extraction parameters [77]. In fact, several process parameters affected the extraction efficiency and its suitability as green technique [66,78]. They could be divided into parameters that depends on the ultrasound process such as ultrasound power [59,78], frequency [78], ultrasound amplitude [68] and pulse rate [77], and parameters that depends on the chemical equilibrium like type of solvent [68], solvent composition [68,78], pH [66], raw material to solvent mass ratio [59,66,68,77], temperature [59,66,68,77,78], and extraction time [66,68,77,78]. Among them, it seems that key factor is the solvent (type and concentration) [78].

Moreover, two configurations are available for ultrasound devices that affect the efficiency of the process. The most common is the ultrasonic bath, where the waves pass through the sample container, and the probe, which is directly inserted onto the sample and the waves propagate on it. This last allows working in continuous or pulse mode contributing to the reduction of power consumption [79].

4.3. Advantages and Limitations

In view of the principle and the process parameters, UAE is usually the choice due to its versatility, safety [80], simplicity [64,68], practicality [68], low price [64,68], and because of its environmentally sustainable character [62].

In depth, this technique has low energy requirements [59,61,62,79,80], preserves phytochemicals in the extracts by controlling the temperature [63,81], reduces the necessary extraction time compared to conventional methods [61,62,66,79,81], requires a minimum amount of solvent to extract [61,68,79], and is effective for compounds that are difficult to release [60,81], thus improving extraction yields [59,62,66,79] with high compounds purity [66,79].

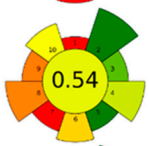
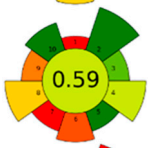
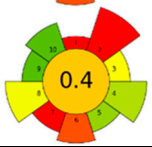
In contrast, the high initial expenses of the equipment are the main UAE limitation, and from the GS point of view, despite being a simple technique, it requires many steps to obtain the final extract, not being possible to fully automatize the technique. However, these drawbacks are compensated by the advantages of the technique [66].

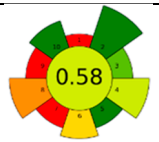
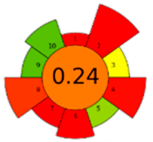
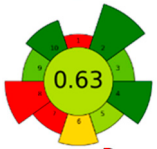
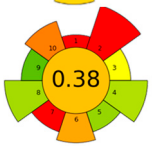
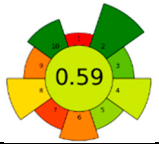
4.4. Integration into Green Chemistry

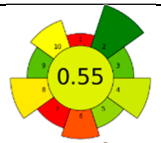
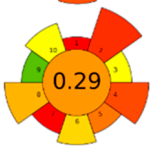
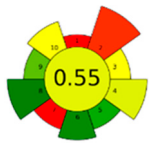
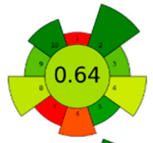
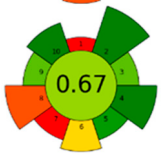
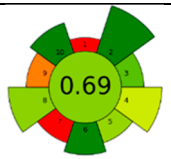
UAE is widely employed as sample treatment for different matrices to recover a bast of compounds of different nature. Besides, the use of low quantities of solvent and short times with good recovery yields make considering it as green technology [82]. For instance, UAE literature review returned 50 potential articles, of which 23 were selected for assessment against GC principles after applying specific exclusion criteria (Figure 1). Table 2 summarizes the raw materials used, operational conditions, target compounds and optimal yields, as well as estimated GS values according to the detailed methodology.

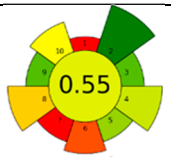
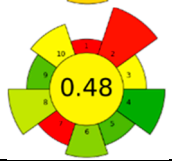
The average GS value for the evaluated studies was 0.51±0.15 with over 35% of the studies scoring above 0.5 (yellow to green), 31% scoring above 0.6 (green) and 27% below 0.5. Four works under a GS of 0.3 notably influence the poor result of this technique. It is noteworthy that the aim of these works is to recover proteins by employing the conventional extraction solvent, alkalinized water [80,83,84]. In fact, a low score is also noticeable for C2, 3, and 10 in other works that employ acidified solvents [84] or solvent such as ethyl acetate [62], methanol [85] or acetone [78] with accounted for several hazards. In contrast, the opposite trend is showed when solvents such as water [65,81,86], ethanol [59,61,68,79,87,88], enzymatic solutions [64], extra virgin olive oil (EVOO) [89], and NADES [60,84,90–92], which are considered safe and GRASS solvents are used. Despite being GRASS, the use of ethanol negatively affects the score due to hazards for operator's safety (C10). Besides, the use of NADES supposes energy consumption due to their viscosity which reduces the capacity of the wave to penetrate implying higher amplitudes and extraction times (C8).

Table 2. Summary of studies on Ultrasound Assisted Extraction (UAE) from agricultural wastes, detailing the raw materials studied, key extraction parameters, and the greenness score (GS) estimation based on Wojnowski et al. [15].

	Raw material	Solvent	SSR	Frecuency (kHz)	UP (W)	Amplitude (%)	t (min)	Compound	Optimal yield	GS	Ref.
1	Grape pomace (GP), jabuticaba peel (JP) and dragon fruit husk (DFH)	Water	1/100	35	160	n.a.	90	TPC; TAC; TBC	GP: 5.01 ± 0.94 mg GAE/g, 0.86 ± 0.05 mg C3G/g, n.d.; JP: 26.82 ± 1.92, 1.03 ± 0.05 mg C3G/g, n.d.; DFH: 3.14 ± 0.08 mg GAE/g, n.d., 78.22 ± 1.35 mg/g		[81]
2	Saffron tepals and stamen	L-Proline:Glycerol (1:2)/water (90:10) (w/w)	1:20	n.r.	180	n.r.	20	Phenolic compounds and flavonoids	Flowers: TPC: 88.96 ± 1.08 mg GAE/g d.w., TFC: 4.36 ± 0.48 mg CE/g d.w. Stigmas: TPC: 95.66 ± 9.34 mg GAE/g d.w.; TFC: 9.56 ± 0.60 mg CE/g d.w.		[90]
3	Grape seeds	Water (pH 11; NaOH 6M)	1:10	40	200	n.r.	180	Proteins	378.31 g/kg	 	[83]
				29	2,000	n.a.					
4	Coffee silverskin	75% aqueous ethanol	0.05:1	37	180	n.a.	60	Phenolic compounds and caffeine	EY: 8.8 % wt; TPC: 36.8 mg GAE/g; 62.7 µmol caffeine/g		[87]
5	Red grapes skins	Nicotinamide-acetic acid (1:1); 40%water.	0.03:1	20	240	n.r.	25	Anthocyanins	21 mg anthocyanins/g biomass		[91]
6	Pineapple pomace	Alkaline water	1:39.88	20	700	20.32	27.23	Dietary fiber (DF)	69.14%		[93]

	Raw material	Solvent	SSR	Frecuency (kHz)	UP (W)	Amplitude (%)	t (min)	Compound	Optimal yield	GS	Ref.
7	Purple guava peels and seeds	choline chloride: Glycerol (1:1), 20%water	0.1:5	37	165	n.a.	60	Phenolic compounds	TPC (LC-ESI-MS/MS) 462.40 ± 16.87 mg/g; TPC (F-C): 1045.15 ± 9.39 mg GAE/g		[60]
8	Pea canning by-product	Alkalized water (pH = 11)	1:20	24	400	80	60	Proteins	66,60% EY		[80]
9	Peach pomace	Pectinase solution (8.5%)	1: 7	37	550	n.a.	50,36	Carotenoids	TPC: 761.10 mg GAE/L		[64]
10	Mandarin peels	80% methanol	1/30	20	500	31%	15	Phenolic compounds	TPC: 3.78 mg GAE/ g d.w.		[85]
11	Almond hulls	80% ethanol	1: 22.28	20	400	50.18	27.26	Phenolic compounds	47.37 ± 0.24 mg GAE/g d.w.		[88]
12	Tomato peels	ethanol: ethyl acetate, 2:3, v/v	1/20	26	200	60%	20	Lycopene	2.92% EY		[62]
13	Tomato peels	EVOO	1: 20	20	400	70%	20	Lycopene	Lycopene content (HPLC-DAD): 0.9 ± 0.2 mg lycopene/g EVOO TPC: 30.95 ± 0.50 mg GAE/g; TFC: 0.07 ± 0.01 mg RE/g		[89]
14	Cocoa pulp mucilage (CPM), cocoa pod husk (CPH), and cocoa bean shell (CBS)	Acidified water with citric acid (pH 2.5)	1/22.5	20	750	n.r.	20	Pectin	EY for CPH, CBS, and CPM (16.2 ± 0.28%, 8.32 ± 0.35%, and 2.98 ± 0.17%), anhydrouronic acid content (68.59 ± 0.2% CPH, 50.7 ± 0.5% CBS, and 43.97 ± 0.17% CPM)		[65]

	Raw material	Solvent	SSR	Frecuency (kHz)	UP (W)	Amplitude (%)	t (min)	Compound	Optimal yield	GS	Ref.
15	Purple waxy corn's cobs	Ethanol 50%	1: 20	20	500	50%	25	Anthocyanin and phenolic compounds	TAC: 305.40 µg C3G/g d.w., TPC: 25.50 mg GAE/g d.w.		[68]
16	Defatted grapeseeds	Alkalinized water (pH = 11; 0.1 M NaOH)	1/16	40	200	n.a.	37	Proteins	EY: 14.3 ± 0.9%; Protein content: 55.1 ± 1.8%		[94]
17	Mexican/Spanish Lime peels	100 mM Tris-HCl buffer [0.25% SDS (w/v) and 0.25% DTT (w/v)/ 0.25% SDS (w/v) and 0% DTT (w/v), pH 7.5]	0.3/5	20	130	30	1	Proteins	Protein content Mexican and Spanish peels: 0.06 ± 0.01 and 0.11 ± 0.01 g protein/100 g d.w.		[84]
		choline chloride ChCl:urea:water (1:1:3)	0.22/ n.r.	20	130	70	30		Protein content Mexican and Spanish peels: 1.00 ± 0.06 and 1.14 ± 0.04 g protein/100 g d.w.		
18	Blueberry leaves	Choline chloride:oxalic acid (1:1)	0.2:1.5	40	350	n.a.	45	Phenolic compounds, anthocyanins	TPC: 195.5 ± 1.1 mg GAE/g d.w.; TAC: 217.9 ± 4.3mg C3GE/100 g d.w.;		[92]
19	Coffee pulp	Water	1:10	37	370	n.a.	5.5	Caffeine and polyphenols	Caffeine: 15.6 ± 0.3 g/kg d.w.; TPC: 12.4 ± 0.2 g GAE/kg,		[86]
20	Ginger herbal dust	50% aqueous ethanol	1/20	24	400	100	2.5	Phenolic compounds; 6-ginerol; 6-shogaol; 8-ginerol	EY: 13.14%; TPC: 112.26 ± 0.06 mg GAE/g d.w.; gingerol (44.57 mg/g dw), 8-gingerol (8.62 mg/g dw), and 6-shogaol (6.92 mg/g dw).		[61]

	Raw material	Solvent	SSR	Frecuency (kHz)	UP (W)	Amplitude (%)	t (min)	Compound	Optimal yield	GS	Ref.
21	Artichoke leaves	50% aqueous ethanol	1: 10	24	400	n.r.	30	Phenolic compounds	TPC: 2.7±0.6 mg GAE/g; TFC: 6.5±0.7 mg CE/g		[79]
				35	240	n.a.			TPC: 2.5±0.6 mg GAE/g; TFC: 5.3±0.2 mg CE/g		
22	Blackberry seeds	56% aqueous ethanol	0.07	n.r.	260	n.a.	60	Phenolic compounds	EY: 0.062 g/ g; TSC: 633.91 mg glucose/g; TPC: 36.21 mg GAE/g; TAC: 3.07 mg C3G/ g		[59]
23	Watermelon rinds and peels	80% aqueous acetone	0.5/7	35	144	n.a.	20	Phenolic compounds	TPC: 3.13 mg GAE/g; TFC 3.76 mg QE/g		[78]

SSR: Solid-to-solvent ratio (w/v); UP: Ultrasounds power; UAE: Ultrasound assisted extraction; EY: extraction yield; TPC: Total phenolic content; TFC: Total flavonoid content; TAC: Total anthocyanin content; TBC: Total betalain content; TSC: Total sugars content; GAE: gallic acid equivalents; CE: catechin equivalents, C3GE: cyanidin-3-glucoside equivalents; d.w.: dry weight; n.a.: not applicable; n.r.: not reported; n.d.: not detected.

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Special mention should be made of the work that provided the lowest GS (0.17). In addition to abovementioned, the main drawbacks are that researchers also employed large amounts of sample, solvent and powerful devices at lab and pilot-scales [83]. For both extraction strategies, the same score is obtained due to the limitation of the criteria, thus it is difficult to evaluate properly the differences between scalable processes, something really concerning due to the important role of the industry. In this sense, the use of advanced analytical techniques for the identification and quantification the analytes with higher sensibility are penalized due to the high energy consumption [59,61,86,89]. Nevertheless, these tools are necessary for a precise characterization of the compounds present in the extract to emphasize the biomass valorization.

Regarding the available configurations for this technique several differences are noticed although it is hard to determine which is the best option since similar results were obtained when they were compared at similar and different conditions [79,86,90]. On the one hand, good scores are obtained with an UAE-bath because it allowed the preparation of higher number of samples per hour (C6) even if the extraction time exceed 30 min [60,64,81,87,92], whereas the horn limits the extraction to one sample at a time so it is time-depending [61,65,68,84,88–91]. On the other hand, the GS of UAE, probe are positively influenced in some cases since it facilitates the reduction of energy consumption (C8) thanks to the amplitude modulation [61,62,84,85,89], which is not possible when a bath is employed. In the case of power consumption using pulse cycles, it cannot be compared since it has not been reported in every work, so it has been assumed that they work in continuous mode, as well as for those work that did not include the amplitude.

In conclusion, viewing of the highest GS values [86,90], an effort should be done to align UAE process with Green Chemistry principles controlling extraction time to minimize energy consumption and maximize sample throughput, reducing sample amount and the use of hazard solvents, as well as combining extraction strategies would be promising strategies to further extend the green character of this technique.

5. Enzyme-Assisted Extraction (EAE)

5.1. Principle

Enzyme-assisted Extraction (EAE) is an eco-friendly, non-thermal technology developed over the past decade [95]. This method utilizes enzymes in water under mild conditions and short time frames, leveraging their substrate specificity [31]. Plant cells, however, possess structural barriers that can impede the extraction process. The cell wall, composed of complex structural polysaccharides like cellulose and hemicellulose, provides mechanical strength but also restricts access to bioactive compounds located within the intracellular matrix. Pectin contributes to tissue rigidity, integrity, intracellular adhesion, and water retention, while lignin adds strength and protects against environmental stress, pathogens, and animals. Proteins and other components further enhance the stability and resistance of the cell wall, making the extraction of intracellular components challenging [96]. EAE overcomes these challenges by employing enzymes to hydrolyze cell wall components, disrupting cell integrity, increasing wall permeability, and facilitating the release of target bioactive compounds [7,97]. Enzymes achieve this by undergoing conformational changes that optimize their interaction with substrates, inducing stress and strain that ultimately lead to bond hydrolysis and rupture [97]. During the EAE process, enzymes are added to the sample matrix and incubated under specific temperature, pH, time, and enzyme concentration to maximize enzyme activity and the release of the desired compounds [98]. Hydrolases, which break covalent bonds using water, are the primary enzymes used in EAE. This environmentally friendly approach is gaining popularity in food biotechnology due to its high specificity [99]. Since the plant cell wall comprises diverse components, various enzymes can be employed to degrade it, depending on the target compounds. Cellulases, hemicellulases, and proteases, derived from microorganisms and plants, are the most used enzymes in EAE processes [96]. Overall, EAE represents a promising alternative due to its high substrate

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specificity, efficiency, and minimal environmental impact, making it ideal for applications in sensitive ecosystems [97].

5.2. Influencing Parameters

During EAE, selecting an appropriate extraction plan tailored to the compounds of interest in the raw material is crucial. This requires considering parameters that influence catalytic potential and EAE efficiency [100]. Key factors include enzyme composition, enzyme concentration, temperature, extraction time, pH, substrate particle size, and the enzyme-to-sample ratio [7,36]. To optimize enzyme selection, it is essential to understand the biochemical and morphological characteristics of the biomass undergoing enzymatic treatment. This allows for the choice of specific enzymes or enzyme mixtures that provide complementary activities, facilitating complete cell wall fragmentation [101]. PH and temperature are critical for activating enzymatic catalytic potential. Commercial enzymes generally work across a broad pH and temperature range, but these parameters can vary depending on the substrate [31]. Higher temperatures typically reduce the viscosity of extraction medium, improving mass transfer rates and solubilization, which increases extraction yield. However, excessive heat can denature enzymes and degrade bioactive compounds [101]. In this sense, mild temperatures generally used during EAE, are ideal for recovering thermosensitive components like polyphenols or volatile compounds [100]. Regarding pH, acidic environments destabilize hydrogen bonds, increasing cell wall plasticity [101]. Carbohydrases perform optimally in mildly acidic conditions, while proteases favor slightly alkaline environments [102]. Buffer salts are often used during extraction to maintain a stable pH, preserving enzyme integrity and extract quality. Extraction time is another critical parameter. Although longer durations can increase yields, extended times risk degrading extracted compounds due to heat or oxidation, reducing yield and raising energy costs [100]. Enzyme concentration also affects extraction time, as doubling the enzyme concentration can halve the required time, and vice versa [101]. Lower enzyme concentrations limit contact with the substrate, reducing extraction efficiency [99], while higher concentrations enhance cell wall degradation and improve yields [100]. Conversely, increasing substrate concentration enhances enzymatic efficiency up to a limiting point [97]. Lastly, substrate particle size significantly impacts extraction efficiency. Pretreatment to reduce particle size improves substrate availability for enzyme active sites, resulting in higher yields and cost-effective extraction [100].

5.3. Advantages and Limitations

EAE is a sustainable and environmentally friendly technology that aligns with Green Chemistry principles. Unlike traditional extraction methods that rely on harsh chemicals and organic solvents, EAE uses water or buffer solutions, making it a greener alternative [96]. Key advantages of EAE include the ability to use entire plant materials, fewer processing steps, mild reaction conditions, and substrate specificity. As previously highlighted, controlling temperature and extraction time is crucial in EAE. Operating under mild conditions reduces energy consumption compared to conventional methods, leading to cost savings, minimized equipment corrosion, improved extract quality, and the ability to recover thermosensitive compounds [29,99,100]. The high specificity of enzymes in EAE allows for efficient extraction of targeted biomolecules under optimized conditions, resulting in purer extracts with fewer contaminants. This preserves the biochemical structure and biological activity of the compounds, yielding higher-quality products [100]. Furthermore, EAE does not require expensive equipment, and optimizing extraction parameters can further reduce costs and energy use [96].

However, the primary limitation of EAE is the high cost of enzymes, which poses challenges for scaling up to industrial levels [31]. Strategies such as enzyme immobilization have been developed to address this issue, enabling enzyme reuse while maintaining activity and specificity, thereby reducing process costs [36,100]. Additionally, the production of high-quality compounds through EAE can command premium prices in industries like pharmaceuticals and cosmetics, making the process more economically viable [100].

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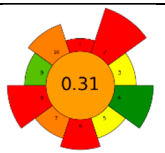
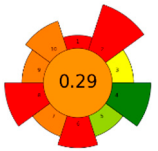
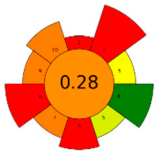
5.4. Integration into Green Chemistry

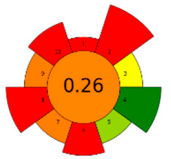
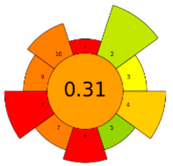
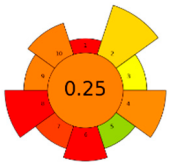
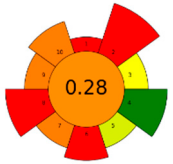
EAE has been widely documented in the literature as a method for recovering multiple bioactive compounds from plant by-products. Most studies are focused on extracting phenolic compounds, including flavonoids, phenolic acids, and anthocyanins, from matrices such as fruit by-products [103–115] and other plant wastes [51,116–120]. Additionally, enzymes have been used to extract the lipid fraction from plant waste [111,113,114], including saturated, monounsaturated, and polyunsaturated fatty acids [105,113,114,117,121], as well as lipidic compounds like tocopherols, phytosterols, and squalene [105,113,114]. Other compounds obtained using EAE include sugars [51,109,111,122], proteins [121,123], pectin [124], and fiber [125]. The GS of the EAE process applied to different plant wastes was estimated and compiled in Table 3 to evaluate environmental impact. From an initial 89 potential articles, only 24 met the inclusion criteria after screening (Figure 1). Many studies treated EAE as a pretreatment step or combined it with other extraction methods, such as conventional solvent extraction, UAE, or MAE. Future research could explore how enzymatic pretreatment could enhance the green character of these techniques. Table 3 shows the GS values for studies using EAE as the primary extraction method, including the key parameters employed during EAE, such as temperature, pH, enzyme type, and concentration.

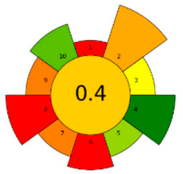
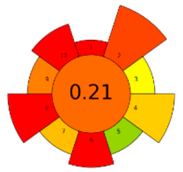
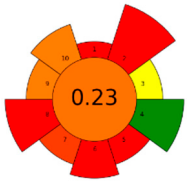
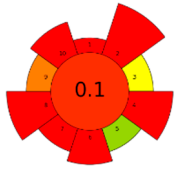
The enzymes used in these studies, including those in commercial preparations, are primarily carbohydrases such as cellulase, hemicellulase, β-glucosidase, xylanase, and proteases and pectinases. These enzymes target the plant cell wall, which is composed of cellulose, hemicellulose, pectin, and proteins, forming a complex structure. As previously stated, cells constitute a limiting factor for reaching the intracellular media, thus the enzymes breaking down these barriers, releasing intracellular content. EAE typically operates under mild conditions, with temperatures around 40°C, and uses green solvents such as water and buffered solutions (Table 1). This makes it a promising alternative to conventional extraction methods. Acidic pH is generally used with carbohydrases, while proteases require more basic conditions. For example, Kaur et al. [125] performed sequential EAE on pearl millet bran using α-amylase, protease, and amyloglucosidase, adjusting the pH to 6.0, 7.5, and 4.5, respectively, for each enzyme optimal activity.

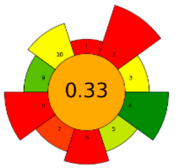
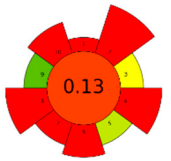
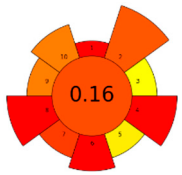
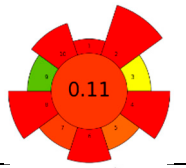
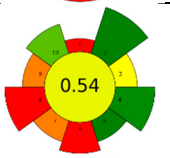
The average GS value for EAE studies was 0.30±0.13, with 52% scoring above, and 24% scoring below 0.20. All the studies scored bad in criteria 1, 6, and 8, reflecting the need for sample transport to labs for analysis, low sample throughput, and high energy consumption. Despite EAE technology simplicity, maintaining a specific temperature for long durations results in significant energy use. For example, 21% of evaluated studies consumed 2,000-5,000 Wh per sample, while 46% exceeded 5,000 Wh. Long extraction times, often over nine hours [51,108,111–113,121,123] and sometimes extending to 24-48 hours [103,104,106,110,115,117,122], further limited process efficiency, negatively affecting criterion 6. One of the lowest GS values (0.1) was collected from a study using enzymes to recover the lipid fraction and total phenolic content (TPC) from raspberry by-products [114]. Although the process used a green extraction approach, conducting it under nitrogen atmosphere introduced a hazardous residue in the process, lowering scores for criteria 2, 4 and 10. Another study, yielding the same poor GS (0.1) extracted phenolic compounds using carbohydrases and their mixtures, scoring poorly in criteria 1, 6, and 8, associated to long extraction times (12h) and high energy consumption (12,000Wh/sample). Moreover, it obtained low scores in criteria 7 because of the high numbers of extraction stages and in criteria 2, 4, and 10 related to the use of hazardous solvents like hexane for the defatting step, and ethanol and formic acid during the extraction and post-extraction stages [108].

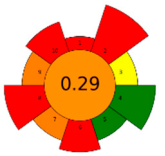
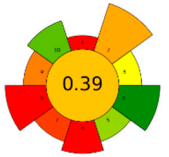
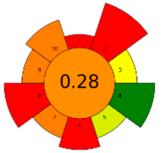
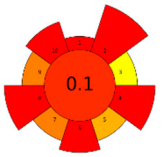
Table 3. Summary of studies on Enzyme-Assisted Extraction (EAE) from agricultural wastes, detailing the raw materials studied, key extraction parameters, and the greenness score (GS) estimation based on Wojnowski et al. [15].

	Raw material	Enzyme	Concentration	Solvent	SSR ratio	T (°C)	t (h)	pH	Compound	Optimal yield	GS	
1	Eggplant peel	Cellulase	5-15 %	Water, ethanol, citric acid (50:48:2, v/v/v)	1:20	35-60	1-4.5	n.r.	TPC; TAC	2,040.87 mgGAE/L; 578.66 mgC3G/L		[116]
2	Citrus by-products	β-glucosidase and tannase	10 U/g	20 mM Acetate buffer	1:25	40	24	5.0	Narirutin; Naringin; Naringenin; Hesperidin; Hesperetin; Diosmetin; Tangeritin	1.11±0.05 µg/mg; 0.33±0.09 µg/mg; 3.86±0.2 µg/mg; 12.05±0.57 µg/mg; 44.08±2.22 µg/mg; 1.22±0.24 µg/mg; 0.36±0.02 µg/mg		[103]
3	Citrus pectin by-product	β-glucosidase, tannase, cellulase, and their mixtures	5 U/g	20 mM Acetate buffer	1:12.5	40	24	5.0	TPC; Gallic acid; Narirutin; Naringenin; Hesperidin; Hesperetin; Tangeretin	>300 mgGAE/100g; 6.75±0.23 mg/100g; 31.93±0.72 mg/100g; 41.48±1.31 mg/100g; 204.53±2.61 mg/100g; 407.90±2.69 mg/100g; 5.67±0.29 mg/100g;		[104]
4	Lemon and orange by-products	Cellulase	150 µL/g	50 mM Phosphate buffer	1:1,000	40	24	5	Fucose; Arabinose; Rhamnose; Galactose; Glucose; Xylose; Mannose; Galacturonic acid; Glucuronic acid	17.3±0.0 µmol/g; 205.4±3.9 µmol/g; 76.2±4.3 µmol/g; 186.1±4.4 µmol/g; 1,205.4±64.0 µmol/g; 173.3±4.6 µmol/g; 242.6±16.0 µmol/g; 449.1±10.1 µmol/g; 2.8±0.2 µmol/g		[122]

5	Onion peel	Zymorouge® EG complex	2 mL	0.2 M Sodium acetate buffer	1:28	40	24	5.0	TPC; TFC; Quercetin; 1,2-Dihydroxybenzene; n-Hexadecanoic acid; 9,12-Octadecadienoic acid	108.36±3.62 mgQE/g; 25.19±3.56 mgGAE/g; 4.92 % TFC r.a.; 21.05 % r.a.; 18.03 % r.a.; 25.81 % r.a.		[117]
6	Pumpkin and exotic fruits by-products	Protease	1:100 (w/w) (enzyme/substrate)	10 mM Phosphate buffer	n.r.	60	16	7.5	Lipids; SFA; MUFA; PFA; Protein	117±25 % EY; 55.3±0.4 % r.a.; 35.6±0.6 % r.a.; 50.72±0.05 % r.a.; 71±2 % EY		[121]
7	Spent coffee ground	Viscozyme®L; Celluclast ®1.5L	0.4-80 µL/g; 0.2-40 µL/g	Acidified water	1:3	25-55	1-14	4.65 - 5.95	Mannose; Glucose; Galactose; Arabinose; Caffeic acid; Chlorogenic acid; Melanoidins	30-40 mg/g; 10-20 mg/g; 10-20 mg/g; 0-10 mg/g; 1.73±0.04 mg/g; 0.15±0.02 mg/g; 32.37±0.08 mg/g		[51]
8	Pomelo seeds	Complex enzyme (alkaline protease, pectinase, cellulase, 1:1:1)	1 % (w/w)	Basified water	1:8	50	2	9	SFA; MUFA; PUFA; Tocopherols; Phytosterol; Squalene; TPC	34.75±0.06 %; 19.60±0.04 %; 45.42±0.04 % of total fatty acids; 95.85±1.41 mg/kg; 2,056.94±14.09 mg/kg; 35.70±0.09 mg/kg; 340.41±1.71 mgGAE/kg		[105]
9	Citrus juice by-products	Tannase, β-glucosidase, cellulase, pectinase, and their mixtures	5-15 U/g	20 mM Sodium acetate buffer	1:12.5	40	6-24	5.0	TPC; Narirutin; Hesperidin; Tangeritin; Naringenin; Hesperetin	aprox. 1000 mgGAE/100g; 50.9±4.5 mg/100g; 255.2±6.9 mg/100g; 1.7±0.2 mg/100g; 24.2±0.9 mg/100g; 148.7±6.8 mg/100g		[106]

10	Guarana seeds	Pectinase, cellulase, and their mixture	1 U/mL	Citrate buffer	1:3	40-50	4	5.70 - 6.10	TPC; Catechin; Epicatechin; Epicatechin gallate; Caffeine; Theobromine; Theophylline	aprox. 520 mgGAE/100g; 17.19±0.07 g/100g; 10.90±0.06 g/100g; 0.08±0.03g/100g; 14.16±0.02 g/100g; 0.12±0 g/100g; 1.30±0.04 g/100g		[107]
11	Hawthorn pomace	Cellulase:pectinase (1:1, w/w)	0.2 mg/mL	Acidified water	1:3	40	3	4.5	TPC; TFC; Quercetin; Epicatechin; Phlorizin; Rutin; Ferulic acid	729.68±5.53 mg/kg; 524.09±3.85 mg/kg; 100.12±13.76 mg/kg; 48.63±5.12 mg/kg; 79.63±0.73 mg/kg; 49.47±2.24 mg/kg; 49.71±3.43 mg/kg		[120]
12	Chicory and fennel by-products	Mix of pectinlyase, polygalacturonase, pectinesterase, arabinase, cellulase, and acid protease / Xylanase	0.03-0.3 mL/0.1 g	Acidified water	1:10-1:15	50	1.5	4-4.5	TPC; Epicatechin; Chlorogenic acid; Rutin; Rosmarinic acid; Kaempferol; Gallic acid; Epigallocatechin; Sinapic acid; Epicatechingallate	6 mg/g; 17.43±0.43 mg/100g; 53.39±0.20 mg/100g; 6.49±0.37 mg/100g; 31.8±0.21 mg/100g; 18.58±0.56 mg/100g; 10.01±0.44 mg/100g; 24.24±0.11 mg/100g; 11.34±0.44 mg/100g; 17.83±0.19 mg/100g		[118]
13	Longan peels	Cellulase, amylase, protease, β-glucosidase, and their mixtures	0.24-210 U	Phosphate buffer and 80 % ethanol with 0.1 % formic acid	1:5	40-50	12	6.5	TPC; Ellagic acid; Gallic acid; Corilagin; o-Coumaric acid; Ferulic acid; Chlorogenic acid; Quercetin; Kaempferol	446.0±22.4 µmolGAE/g; 6,932.50±306.43 µg/g; 120.16±6.10 µg/g; 16.25±1.18 µg/g; 44.71±5.50 µg/g; 26.74±1.21 µg/g; 80.19±3.67 µg/g; 135.28±6.67 µg/g; 15.56±0.65µg/g		[108]

14	Lime pomace	Polygalacturonase	0.115 U/mL	Water	1:31.25	20	0.5-2	3.50	Pectin	15.09±0.44 % EY		[124]
15	Pearl millet bran	α-amylase followed by protease and amyloglucosidase	50 µL, 100 µL, 200 µL	0.08 M Phosphate buffer, 0.275 N NaOH, 0.325 N HCl	1:50	60	1.5	6.0, 7.5, 4.5	Fiber	48 % EY		[125]
16	Bilberry pomace	Viscozyme ® L	2-10 U/g	Citrate buffer	1:10	30-50	1-7	3-5	TPC; Sucrose; Glucose; Fructose; Anthocyanin	13.26 mg/GAE/g; 4.5±0.3 mg/g; 109.5±1.4 mg/g; 121.9±4.7 mg/g; 3,194.0±123.6 µgcyan-glu/g		[109]
17	Rapeseed press cake	Protease	1 %	NaCl (0-1.0 M)	1:9-1:19	20-70	0.75-12	5.8-12	Protein	78.3 % EY		[123]
18	Grape residues	Celluclast ®, Pectinex ® Ultra, Novoferm ®	100 µL	0.2 M Acetate buffer	1:14	40	0-48	3.5	TPC	aprox. 40 mgGAE/100g		[110]
19	Winery solid residue	Ultrazym-Celluclast (3:1, w/w)	2 %	Water	n.r.	35-55	9	n.r.	Oil; Soluble sugars; TPC	aprox. 70 % EY; aprox. 11 mg/g; aprox 39 mg/g		[111]
20	Fruit by-products	Viscozyme ® L	2 %	0.1 M Phosphate buffer	1:20	35-55	0-12	3.0-7.0	TPC; TFC	76.18±2.63 mgGAE/g; 30.57±1.64 mgQE/g		[112]

21	Sweet corn cob	Ferulic acid esterase and endo-1,4-β-xylanase	0.01-18,093.50 U/g	Phosphate citrate buffer	n.r.	45-65	3	4.5-6.5	Ferulic acid	1.45 g/kg		[119]
22	Tomato seeds	Alcalase 2.4L	0.75-3.75 mL	0.6 M Phosphate buffer	n.r.	60	4-12	7.5	Oil; TPC; β-Tocopherol, δ-Tocopherol; Oleic acid; Linoleic acid	9.66 % extraction yield; 3.3±0.00 mgGAE/kg; 128.51±1.14 ppm; 209.88±0.5 ppm; 25.29±0.35 g/100g; 57.77±0.28 g/100g		[113]
23	Citrus by-products	Tannase and β-glucosidase (1:1, w/w)	20 U/g	20 mM Acetate buffer	1:12.5	40	30	5.0	Narirutin; Hesperidin; Naringenin; Hesperetin; Diosmetin; Tangeritin	0.83±0.03 mg/g; 11.11±0.39 mg/g; 3.49±0.10 mg/g; 43.70±0.79 mg/g; 1.03±0.06 mg/g; 0.37±0.02 mg/g		[115]
24	Raspberry pomace	Alcalase 2.4L, neutrase, pepsin, papain, cellulase, pectinase, xylanase	1.2-3.6 U/100g	Water	1:3-1:9	40-60	1-3	7-9	Oil; TPC; PUFA; Total tocols; Total phytosterols	2.64 g/100g; 3.56±0.077 g/100g; 84.3±0.23 % of total fatty acids; 125.9±5.02 mg/100g; 259.7±6.4 mg/100g		[114]

SSR: Solid-to-solvent ratio (w/v); U: Units; TPC: Total phenolic content; TAC: Total anthocyanin content; TFC: Total flavonoid content; SFA: Saturated fatty acids; MUFA: monounsaturated fatty acids; PFA: polyunsaturated fatty acids; EY: extraction yield; r.a.: relative area; n.r.: not reported.

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The highest scores were obtained in criteria 4 and 5, due to reduced waste generation and minimal sample usage, respectively. Gómez-García et al., who used grape residues to obtain a phenolic-enriched extract using different commercial enzymatic formulations, achieved the highest GS value (0.54). Their study performed strongly in criteria 2 and 10, in addition to 4 and 5, thanks to the use of safer solvents and reagents, as well as non-hazardous materials for extraction. The study with the second-highest GS value (0.51) focused on extracting soluble sugars and TPC from winery solid residues using spectrophotometric detection and quantification. It achieved a high score in criterion 9, which, along with strong performance in criteria 2 and 4 due to the use of safe reagents, contributed to its overall high GS. The third-highest GS value (0.5) was reported in a study on the valorization of fruit by-products. The use of safe and less hazardous solvents and reagents, combined with the spectroscopic techniques to assess TPC and TFC recovery, led to good performance in criteria 2, 4, 9, and 10.

Based on the above findings and considering the weighted contribution of each criterion to the overall GS, the difference between high (≥ 0.5) and poor (< 0.2) performance in GS during EAE is primarily determined by criteria 2, 4, and 10. The use of safer, non-hazardous solvents and reagents, along with waste minimization, has been key to achieving high GS values. Conversely, the use of acids, bases, and other hazardous substances significantly lowered GS performance.

6. Supercritical Fluid Extraction (SFE)

6.1. Principle

SFE is an extraction technique based on the use of a solvent in its supercritical conditions which are limited by the critical pressure (Pc) and temperature (Tc). At these conditions, the solvent behaviour is between liquid and gas favouring the extraction thanks to a higher diffusivity and solvating capacity compared to a liquid solvent [126,127], with solubility of target compounds being affected at these conditions [127]. Thus, the main extraction mechanism is based on the diffusion of target compounds from solid matrix to the extraction medium [128]. In fact, three mass-transfer mechanism are clearly differentiated in the extraction curve. Briefly, a convective mass transfer establishes a constant extraction rate period until the thermodynamic equilibrium of the solute followed by slower extraction rate due to the competition of two mechanisms, diffusion and convection. Finally, the curve ends controlled by a diffusion period when target compounds are recovered [128].

Among the solvents used as fluids, carbon dioxide (CO_2) is the most used due to its unique properties [126,128–130]. The principal reason is that Tc and Pc values of this solvent are easily achievable (31.1 °C and 7.3 MPa, respectively) [128]. Moreover, other benefits of using CO_2 include: its non-toxicity, chemical stability, non-flammability, non-explosiveness, as well as the obtention of free-solvent extracts [128,129]. Therefore, in addition to the inert atmosphere, the possibility of working at low temperatures makes this technique and this solvent suitable for heat-sensitive compounds [128,129,131]. Furthermore, due to the nature of CO_2, this technique is efficient for non-polar o low-polar compounds [128,132] although its selectivity could be varied by its combination with an organic solvent to recover a wider range of target compounds [127,129].

6.2. Influencing Parameters

As has been stated in the principle of the technique, the extraction process is dominated by mass-transfer mechanism. Hence, parameters affecting the properties of CO_2 and sample will contribute to accelerating or decreasing the extraction kinetics [128]. Specifically, the optimization of parameters such as pressure, temperature, flow rate, extraction time, and mean particle size improves the extraction yield [128,133]. Besides, the use of co-solvent and the optimization of its percentage, partially enhance the effectiveness of polar bioactive compounds [131,134].

Among them, temperature and pressure are the factors that greatly influence the solvent density of CO_2 and solubility of target compounds. In fact, an increase in the fluid density allows a better

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mass transfer rate and improves the extraction yield. However, the combination of both parameters could affect the diffusivity of the fluid decreasing the extraction yield [126,127,135].

On the one hand, temperature is usually established above the Tc and up to 60 °C to avoid thermal degradation of target compounds [126]. However, several works employed lower values when the aim is to work under CO_2 liquid conditions [135,136], or higher values due to previous experiences or parameters optimization [22,127,137–139]. This parameter has two opposite effects: an increase implies a lower density of the fluid and consequently, its solvation power and the target compounds solubility, lowering the yield, and implies an enhancement of the yield due to the increase of the target compounds vapor pressure and solubility [127,135]. On the other hand, selected pressure is above the Pc considering that values near 30 MPa provided better extraction yields, and below (15-20 MPa) lower yields are obtained, whereas higher values could increase the energy consumption and the cost of the process [126]. Contrary to the effect of the temperature, the density of CO_2 increases when the pressure is increased, although the solubility of target compounds is also increased. In contrast, higher values affect the diffusivity of CO_2 and lead sample compaction [128].

Furthermore, increases in flow rate, extraction time, and the use of co-solvent generally enhance the extraction yield since the mass-transfer of target compounds in the extraction medium is favored, while the opposite trend is shown with particle size due to a higher exchange surface although the diffusion inside the solid is limited [128].

6.3. Advantages and Limitations

Traditionally, extraction of non-polar bioactive compounds has been done by distillation employing organic solvents. This technique requires long extraction time and labor operations to reach low yields due to the degradation of thermolabile compounds and toxic residues [22]. To overcome the main drawbacks of the conventional extraction techniques, SFE-CO_2 has merged as a sustainable alternative for the recovery of non-polar compounds [128].

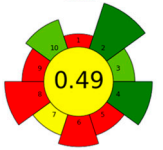
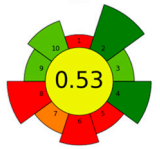
The main advantages that it offers are related to the unique properties of using an environment-friendly solvent which allows the recovery of free-solvent extracts with high purity yield at mild conditions [22,128,132,140]. In addition, this technique exhibits great selectivity for thermolabile non and low-polar compounds, that can be modified by changing the extraction conditions (P y T), and the possibility of enhancing the solubility of polar compounds by the addition of a low percentage of an organic solvent [131,140]. The amount of organic solvents employed for tuning the solubility of target compounds is minimal; thus, the environmental impact is low [131]. Besides, this technique also minimizes the number of extraction steps, increasing the automatization of the process and contributing to reducing the environmental impact [128].

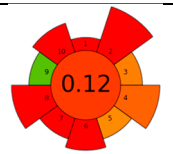
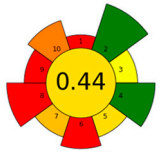
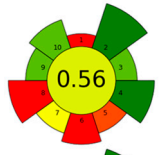
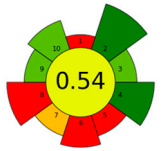
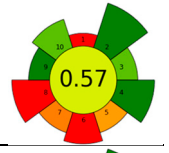
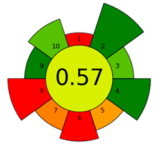
In contrast, the main drawbacks are related to the high energy consumption of the equipment and the long time required to complete the extraction of valuable compounds [140]. In fact, although the technique is scalable as it shows efficiency at industrial scale [132], the operational costs hinder this application [131,140]. In this sense, the economic profitability of the process could also be affected by the conditions employed for the extraction [128].

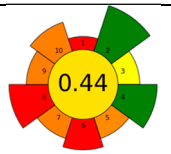
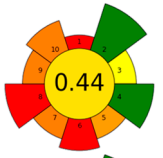
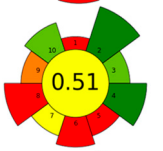
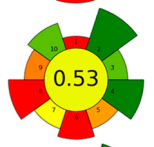
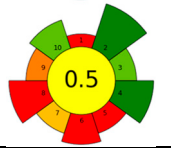
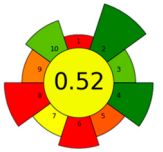
6.4. Integration into Green Chemistry

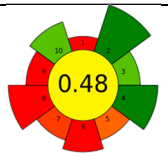
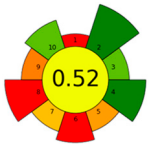
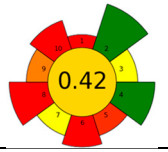
SFE is widely employed as sample treatment for different matrices to recover mostly non-polar compounds, and a low content of polar compounds if extraction conditions are modified employing small amounts of an organic co-solvent. As has been above mentioned, the use of this kind of solvent, even in small quantity, as well as energy consumption, are the major limitations of this extraction technique. For the purpose of the work, SFE literature search returned 50 potential articles, of which 22 were selected for assessment against GC principles after applying specific exclusion criteria (Figure 1). Table 4 summarizes the raw materials used, operational conditions, target compounds, and optimal yields, as well as estimated GS values according to the detailed methodology.

Table 4. Summary of studies on Supercritical Fluid Extraction (SFE) from agricultural wastes, detailing the raw materials studied, key extraction parameters, and the greenness score (GS) estimation based on Wojnowski et al. [15].

	Raw material	Sample (g)	CO_2 (kg)	Flow rate (mL/min)	Co-solvent (%, v/v)	Energy (Wh)	Temperature (°C)	Pressure (MPa)	t (min)	Compound	Optimal yield	GS	Ref.
1	*Picea abies* (cones, branches, needles and bark)	50	4.8	46	-	4,400	50	30	120	Lipophilic extractives	Branches (5.3 %), needles (3.3%), and bark (2.4 %)		[132]
2	Stalks (wine by-product)	40	338.3	2,000	-	7,128	50	30	194.4	Bioactive compounds	1.4 % EY		[126]
3	Sage herbal dust	35	-	n.r.	-	8,800	40	10	240	Essential oil	-		[141]
4	Sage herbal dust	35	-	n.r.	-	8,800	40	30	240	Essential oil	-		
5	Rotten onion waste	30	98.4	2,000	-	2,200	80	40	60	Oleoresin	1 % EY		[22]
6	*Viburnum opulus* (VOP) pomace	131	1,444.8	2,000	-	30,800	60	35	840	Triacylglycerol; tocopherol; phytosterol; fatty acids	26.24% of lipids, β-sitosterol: 514.5 mg/100 g; α-tocopherol 118.6 mg/100 g.		[140]
	Hippophae rhamnoides (SBP) berry pomace		1,612.8				50	50			16.99 % of lipids; β-sitosterol 359.5 mg/100 g and α-tocopherol 65.38 mg/100 g		
7	Apple seeds	80	2.0	16.7	-	2,567	40	24	140	TPC	20.5±1.5 % EY		[142]

	Raw material	Sample (g)	CO_2 (kg)	Flow rate (mL/min)	Co-solvent (%, v/v)	Energy (Wh)	Temperature (°C)	Pressure (MPa)	t (min)	Compound	Optimal yield	GS	Ref.
8	Cherimoya peel and leaves	15	3.6	85.7	Methanol 15 %	6,600	75	10	180	Alkaloids and phenolic compounds	862.51±18.89 - 3496.49±280.68 - µg BE/g		[127]
9	Dried *Lentinus edodes* (Berk.) sing stipe	150	15.7	500	-	1,467	50	20	40	Flavour compounds	50.47±3.19 µg/mL TPC		[143]
10	Celery (*Apium graveolens* L.) waste	4.8	n.r.	n.r.	Isopropyl Alcohol 15, 25, 100 %	9,900	50	30	270	Bioactive compounds	10.84 ± 1.2 % EY		[131]
11	Wild thyme (*Thymus serpyllum* L.) herbal dust	35	1.2	7.7	-	9,900	50	35	180	Oil recovery	3.36 % EY		[128]
12	Guava (*Psidium guava*) seeds	250	4.5	33.5	-	5,500	52	35.7	150	Phenolic compounds; tocopherols; phytosterols	8.6±1.2 g oil/100 g guava seeds		[144]
13	Brewer spent grains	80	0.6	13.3	-	2,200	55	20	60	Oil recovery and encapsulation	Mx. encapsulation efficiency: 63.8 ± 0.8%		[142]
14	Tomato seeds and peels	12	0.5	10	-	2,200	60	34	60	Oil recovery	12.5 % EY		[136]
			0.3	5	-	1,000	20	15			12.9 % EY		

	Raw material	Sample (g)	CO_2 (kg)	Flow rate (mL/min)	Co-solvent (%, v/v)	Energy (Wh)	Temperature (°C)	Pressure (MPa)	t (min)	Compound	Optimal yield	GS	Ref.
15	Walnut green husk	15	1.7	10	Ethanol 20 %	7,150	50	30	195	Phenolic compounds; juglone; fatty acids; VOCs	Polyphenols (10750 mg GAE/100 g) and juglone (1192 mg/100 g)		[145]
16	Orange (*Citrus sinensis*), tangerine (*Citrus reticulata*) and lemon (*Citrus limon*) peels	12	1.2	10	Ethanol 20 %	11,000	60	30	300	Oil; phenolic compounds; VOCs	17.20, 17.60 and 31.24 % in orange, tangerine and lemon, respect.		[135]
			1.4	10	Ethanol 20 %	5,000	20	20	300		17.49, 17.60 and 28.84 % in orange, tangerine and lemon respect.		
17	Waste salt from the salting process of mullet raw roes	500	n.r.	n.r.	-	17,600	40	30	480	n-3 PUFAs	28.4 %; 122±7 g n-3 PUFA/ kg of oil		[146]
18	Tomato waste	12	178.9	1,000	-	9,533	80	30	240	Lycopene	n.r.		[137]
19	Tomato pomace	1,000	70	305	-	93,333	80	38	280	Lycopene	48.4 % EY		[139]
20	Ginger herbal dust	30	2.0	9.2	-	10,267	40	30	240	Nonpolar and low-polar bioactive compounds	7.60±0.21 % EY		[147]

	Raw material	Sample (g)	CO_2 (kg)	Flow rate (mL/min)	Co-solvent (%, v/v)	Energy (Wh)	Temperature (°C)	Pressure (MPa)	t (min)	Compound	Optimal yield	GS	Ref.
21	Pomegranate peels and seeds	25.2	1,147	10,000	-	4,400	40	40	120	Bioactive compounds	11.5 % EY		[148]
22	Tomato pomace	12	179	1,000	-	8,800	80	30	240	Lycopene and other nonpolar and low-polar bioactive compounds	11.5 % EY; (Z)-lycopene 69 % EY		[138]
23	Red raspberries wasted fruit	50	0.8	24	Ethanol 7 % (with 0.2 % acetic acid)	1,467	40	20	40	Oleoresin; TPC; TFC	TPC: 185 mg GAE/g; TFC: 11.0 mg QE/g		[149]

EY: extraction yield; n.r.: not reported; TPC: Total phenolic content; BE: boldine equivalent; VOCs: volatile organic compounds; GAE: gallic acid equivalents; n-3 PUFAs: n-3 polyunsaturated fatty acids; TFC: Total flavonoid content.

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As can be seen, the average GS value for the evaluated studies was 0.49±0.09 with over 44 % of the studies scoring above 0.5 (yellow to green), 28 % scoring above 0.6 (green) and 28 % below 0.5. Only one work under a GS of 0.12 notably influences the poor result of this technique. In view of the results, as happened with the other techniques, C1 and C9 penalized all the reviewed works, unless they used simple techniques to determine the total content of the analyzed compounds or were only interested in the yield. Furthermore, it is noteworthy in every reviewed work that although the technique reduces the extraction time compared to the traditional method, SFE still requires long extraction times which causes the score to decrease due to high throughput needed to prepare a sample (C5). This fact clearly impacts criterion 8 causing energy consumption to skyrocket. Another important factor which contributes to this technique having such a low result, is the use of large quantities of sample due to the large size of the extractors both on laboratory and industrial scales (C6). Fortunately, this only showed a worst impact on the score if the authors used any toxic solvent (C2), that causes the sample to be considered waste (C4) [127]. In addition to this, the use of co-solvents, a reduction on the sustainability, renewability and reusability of the materials employed (C3), and the increasing number of hazards (C10) is also observed [131,135,145,149] and is especially noticeable in the case of using methanol [127].

In this regard, these differences have been noticed among Romano et al. works from 2020 to 2022 [135,136,145]. The influence of the use of ethanol to recover phenolic compounds in this extraction technology introduces more steps to the process and it also implies more hazards for the operator's safety. Although in their last work [135] cosolvent was eliminated under vacuum instead of using a nitrogen stream [145], the last criterium does not reflect it and in both instances contribute equally. Despite the goal of recovering the greatest amounts of bioactive compounds, the values obtained with this technique demonstrate how unsustainable it is. This confirms the technique's limitations due to the lipophilic nature of the compounds to be obtained. Regarding the yield, the extraction with liquid CO_2 provided the same results, thus it would be an option to reduce energy consumption. However, even though the criterium is still unfavorable, the energy calculated for that process is reduced by half.

To sum up and in view of the highest GS values [136,141], a little effort should be made to align SFE process with GC principles. In this sense, controlling extraction time to minimize energy consumption and maximize sample throughput, and reducing sample amount and organic solvents, would be promising strategies to further extend the green character of this technique. In contrast, the low score obtained in reviewed articles reflects the need to adapt these metrics to semi-industrial and industrial-scale processes, since sustainability is as important as the needs of industry and the population demands.

7. Comparative of Emerging Technologies

When compared to other emerging extraction technologies, MAE, with its moderate equipment costs, proves to be less expensive than SFE [30], though slightly more expensive than UAE. UAE demonstrates superior mass transfer and cell wall disruption, leading to higher extraction efficiency. However, MAE offers faster and simpler extraction processes (Table 5). While MAE operates at lower temperatures than traditional methods, it generally uses higher temperatures than UAE, increasing the risk of degrading thermolabile compounds [30]. Conversely, EAE requires minimal equipment but relies on the use of enzymes, making it particularly expensive for large-scale applications.

Table 5. Comparison of emerging technologies: key parameters, strengths, weaknesses, solvent use, energy consumption, and scalability.

	Microwave-assisted Extraction	Ultrasound-assisted Extraction	Enzyme-assisted Extraction	Supercritical Fluid Extraction
Key parameters	Solvent, SSR, temperature, time, irradiation power, sample particle size.	Solvent, SSR, temperature, time, pH, ultrasound power, frequency, amplitude, pulse rate, sample particle size.	Enzyme composition, enzyme concentration, temperature, time, pH, ESR, SSR, sample particle size.	Temperature, pressure, time, solvent flow rate, SSR, co-solvent, sample particle size.
Strengths	Easy to operate, shorter operational times, low equipment costs, reduced solvent consumption, short extraction times.	Simplicity of the process, compatible with thermosensitive compounds, low cost, short extraction times.	Suitable for whole plant materials, fewer processing steps, simplicity of the process, high specificity, mild reaction conditions, compatible with thermosensitive compounds, high-quality pure extracts, cheap equipment.	Solvent recyclability, tunable solvent power (modifying T and P), selectivity, suitable for thermosensitive compounds, pure and solvent-free extracts, no purification stage needed, short extraction times.
Weaknesses	High initial investment, difficulty maintaining temperature, excessive heat/power can degrade thermolabile compounds, high energy consumption, low selectivity and extract purity (purification steps).	High initial investment, many steps to obtain the final extract, difficult to automatize.	Enzyme cost, long extraction times (high energy consumption).	High initial investment, significant energy consumption, requires co-solvents for polar and intermediate-polar compounds.
Solvent use	Methanol, ethanol, acetone, water, NADESs. Acid/basic solutions. Microwave-assisted solvent-free extraction (without solvent): lower extraction efficiency.	Methanol, ethyl acetate, acetone, water, ethanol, enzyme solutions, NADESs. Acid/basic solutions.	Water and buffer solutions. Acid/basic solutions.	CO_2 - Most used: mild critical conditions (31.1 °C and 73.8 bar), inert, nonflammable, non-corrosive, eco-friendly, non-polar (co-solvents like ethanol or water for polar compounds).

SSR: solvent-to-solid ratio; ESR: enzyme-to-sample ratio; T: temperature; P: pressure; NADESs: natural deep eutectic solvents.

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In terms of environmental sustainability, UAE is associated with long extraction times and high ultrasound power, negatively impacting several criteria in the metrics and resulting in a low GS (Table 6). For MAE, although extraction times are generally shorter, the high-power consumption also leads to a low. On the other hand, while EAE has the lowest equipment requirements, it demands extremely long extraction times, leading to high energy consumption and low sample yield efficiency. Furthermore, achieving a high (green) GS depends significantly on the use of a green solvent. Environmental results indicated that SFE technology exhibits the lowest environmental impact, whereas UAE performs the worst due to its high energy consumption. Electricity represents the primary hotspot with the greatest impact, followed by steam demands and solvent use. Comparing the information for the different techniques, is noticeable that in SFE no post-treatment is needed, contributing to increase the GS due to the absence of solvents prior to the analysis.

Table 6. Comparison of greenness score (GS) performance of studies on the analyzed emerging technologies from agricultural waste collected from **Tables 1-4**.

	Microwave-assisted Extraction	Ultrasound-assisted Extraction	Enzyme-assisted Extraction	Supercritical Fluid Extraction
Average GS	0.42±0.09	0.51±0.15	0.30±0.13	0.49±0.09
Best performed criteria	C5 and C9	C2 and C10	C4 and C5	C2 and C4
Worst performed criteria	C1, C2, C6, and C8	C1 and C7	C1, C6, and C8	C5, C6 and C8
Gaps for GS improving	Prioritize the use of greener solvents and employ parallel extraction (multi-sample system) to enhance sample throughput and reduce energy consumption	Control extraction time to minimize energy consumption and maximize sample throughput, reduce the use of hazardous solvents, promote combining extraction techniques	Use safer, non-hazardous solvents and reagents to minimize waste generation, and perform simultaneous extractions to reduce energy consumption	Discrimination among different scale technologies, advanced analytical techniques, increase flow to reduce time and save energy

C1: Favor *in situ* preparation; C2: Use safer solvents and reagents; C3: Target sustainable, reusable and renewable materials; C4: Minimize waste; C5: Minimize sample, chemical and material amounts; C6: Maximize sample throughput; C7: Integrate steps and promote automation; C8: Minimize energy consumption; C9: Choose the greenest possible post-sample preparation configuration for analysis; C10: Ensure safe procedures for the operator.

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8. Conclusions and Future Challenges

Based on the Greenness Score (GS) results for MAE, UAE, EAE, and SFE, it can be concluded that these technologies generally align with Green Chemistry principles. However, further efforts are needed to enhance their alignment by optimizing extraction times or implementing parallel extraction processes to reduce energy consumption and increase sample throughput. Additionally, a crucial factor in achieving a higher GS and, thus, better adherence to GC principles is the substitution of hazardous solvents with greener alternatives.

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