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Review

Current Challenges in the Sustainable Valorisation of Agri-Food Wastes: A Review

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Abstract: In the upcoming years, the world will face societal challenges arising, in particular, from the impact of climate change and the inefficient use of natural resources, in addition to an exponential growth of the world population, which according to the United Nations (UN) estimations will be 9.8 billion in 2050. This increasing trend requires optimized management of natural resources with the use of value-added waste and a significant reduction in food loss and food waste. Moreover, the recent pandemic situation, COVID-19, has contributed indisputably. Along with the agri-food supply chain, several amounts of waste or by-products are generated. In most cases, these biomass wastes cause serious environmental concerns and high costs to enterprises. The valorisation of the agri-food loss and food industry wastes emerged as a useful strategy to produce certain value-added compounds with several potential applications, namely in the food, health, pharmaceutical, cosmetic, and environmental fields. Therefore, in this review, some of the crucial sustainable challenges with impacts on the valorisation of agri-food loss/wastes and by-products are discussed and identified, in addition to several opportunities, trends and innovations. Potential applications and usages of the most important compounds found in food loss/waste will be highlighted, with a focus on the food industry, pharmaceutical industry, and the environment.

Keywords: agri-food waste; valorisation; food loss; food waste; bioactive compounds; minerals; potential applications

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1. Introduction

In recent years, the valorisation of agri-food wastes migrated from a trending ecological movement to an urgent need. The destructive effects of unstable and extreme climate variations on agriculture, soil exhaustion, and water scarcity, among other concerns, lead to a decrease in agri-food production. In contrast, the continuous exponential growth of the human population requires more food to feed everyone, and currently, around 700 million people are estimated to be suffering from hunger [1]. Paradoxically, nutrient loss due to agri-food waste is estimated to provide a diet for 2000 million people. On top of this, agri-food waste disposal in landfills is responsible for greenhouse gas (GHG) emissions and air pollution (e.g., dioxins, ash), as well as groundwater contamination. Overall, the impact on the world economy is very high, affecting different features, such as water and land management, energy production, transport, or storage [2] (Figure 1). These enormous societal challenges have been already addressed by the European Commission which included the mitigation of food waste as a priority area of the Action Plan for the European Circular Economy Strategy [3]. To make such a strategy economically viable, the valorisation of agri-food wastes can be achieved by the extraction of valuable compounds for different industrial sectors, like the nutraceutical, cosmetic and pharmaceutical industries [4,5]. A myriad of phytochemicals is available in diverse agri-food wastes

which are mostly from plant origin and less animal-based (Figure 1), such as peels, leaves, seeds, pomace, meat derivatives, egg products, and food industry rejects, constituting promising raw materials for other industries. However, there are other challenges and obstacles to overcome. Overall, most food waste is generated in five different stages in the food value chain (Figure 1). During production, losses of fruits, vegetables and cereals occur mostly during harvesting on the farm. Edible crops, for instance, are rejected due to their non-standardized measures or defects, inadequate harvesting time, or even due to mechanical damage. Another considerable fraction of food loss happens during the transportation, handling, and storage of the products. These losses are often due to the degradation of edible products by fungi, diseases, handling, or even by poor transportation infrastructure. Processing and packing make the lowest contribution to food loss, which can occur through inappropriate packaging or contaminations. During the distribution and market, some products might be lost due to spoilage during transportation or lack of cooling storage, which is a common situation in the distribution of fruits and vegetables. Human consumption is responsible for the highest amount of waste in the food chain, often due to excessive buying, exceeding use dates, and wrong storage [4–6].

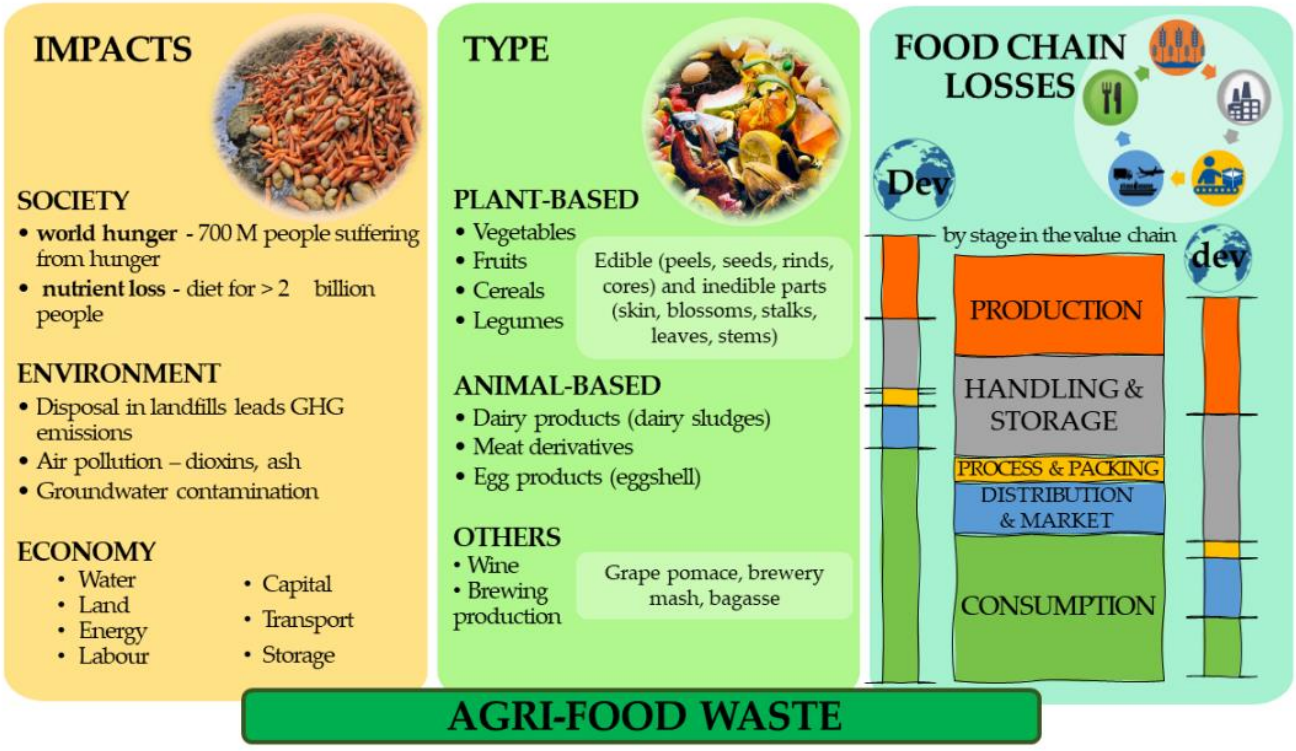

Figure 1. Overview of food waste impact, type, and food chain losses by stage in the value chain in developed (Dev) and developing (dev) countries [2].

In this respect, however, there are significant differences between the performance of developed and developing countries. By far, most of the food waste generated in developed countries occurs during the consumption step. One of the reasons for this paradox is correlated with the fact that proper separation and management of agri-food wastes is still very incipient in many fields, making their valorisation expensive and technologically very demanding for smaller industries [2]. Consequently, it is cheaper to pay to deposit agri-foods in landfills than develop a zero-residue strategy for the value chain of specific food products [2]. In turn, food wastes produced in developing countries are mainly associated with the production, handling and storage stages. This fact is certainly explained by the poor agri-food systems devoted to these stages in developing countries [2]. Irrespective of the stage where agri-food wastes are generated and their respective causes, there is great potential in the extraction of phytochemicals from agri-food wastes, particularly

those obtained from plants, such as fruits and vegetables. These agri-food wastes include edible (peels, seeds, rinds, and cores) and inedible parts (skin, blossoms, stalks, leaves, and stems) which are rich in many bioactive compounds, such as probiotics, dietary fibres, fat-soluble vitamins, essential omega-3 fatty acids, phytoestrogens, and several phytochemicals, namely carotenoids, flavonoids, and phenolic acids, known to exhibit antioxidant, anti-microbial or anti-inflammatory activities [6]. Hence, these compounds can provide the most diverse applications in food, health, pharmaceutical, cosmetic, and environmental fields, as substitutes for synthetic preservatives, pigments, fragrances, and antioxidants in both food and cosmetical products or the addition of health protective effects to our diet [4,5,7,8]. This strategy would allow to obtain better food with less waste, and consequently a better environmental footprint. Therefore, this review comprises some of the crucial sustainable challenges in the valorisation of agri-food wastes and by-products, including green extraction techniques for the recovery of compounds of interest from food waste. The potential applications of the most important compounds found in agricultural by-products, mostly bioactive compounds and valuable chemicals, are highlighted with a focus on the food and pharmaceutical industries.

2. Extraction Techniques for Bioactive Recovery from Agri-Food Wastes

The extraction of bioactive compounds from agri-food wastes using green extraction procedures (e.g., supercritical fluid extraction, pulsed electric fields, ultrasound-assisted extraction, microwave-assisted extraction, enzyme-assisted extraction, pressurized liquid extraction) (Figure 2) has gained special attention due to their exceptional practices focused on economic, environmental, and safety concerns [9]. Moreover, green extraction procedures comprise six principles of green chemistry, namely: (i) the use of renewable and sustainable bio-resources, (ii) use of water or green solvents, (iii) lower energy input, (iv) co-products production from waste, (v) a minimal number of unit operations, and (iv) resulting non-denatured and biodegradable extract [9]. The following sub-sections present the most common green extraction procedures used for the recovery of bioactive compounds from agri-food wastes.

2.1. Supercritical Fluid Extraction

Supercritical fluid extraction (SFE) using carbon dioxide (CO_2) has been proposed as a green extraction procedure, since it requires low volumes of organic solvent to recover the value-added bioactive compounds (e.g., carotenoids, phenolic compounds) from agri-food wastes [10–13]. CO_2 is the most used supercritical fluid due to its mild critical temperature (31.2 °C) and pressure (73.8 bar), which allows for operation at moderate conditions, generally ranging from 40 to 60 °C and 200–400 bar pressure [9]. Additionally, CO_2 is non-carcinogenic, non-toxic, non-mutagenic, non-flammable, thermodynamically stable, and generally identified as safe [14]. The main benefit of this green extraction technique is that the solvent physicochemical properties can be changed by adjusting the pressure and temperature conditions within the system, consequently improving the extraction selectivity and extraction yields due to the fast diffusion of fluid through the solids [12,14]. However, the low polarity of supercritical CO_2 represents the major drawback of this procedure. This problem can be minimized by adding small percentages of co-solvents (e.g., ethanol, methanol, water) or modifiers that change the polarity of the solvent. Consequently, this results in an enhancement of the extraction yield by improving the solubility of the solute or the swelling of the solid matrix that facilitates the solute–solvent contact [15]. This versatility makes SFEs very appealing for several applications in different fields (e.g., industry, pharmaceutical).

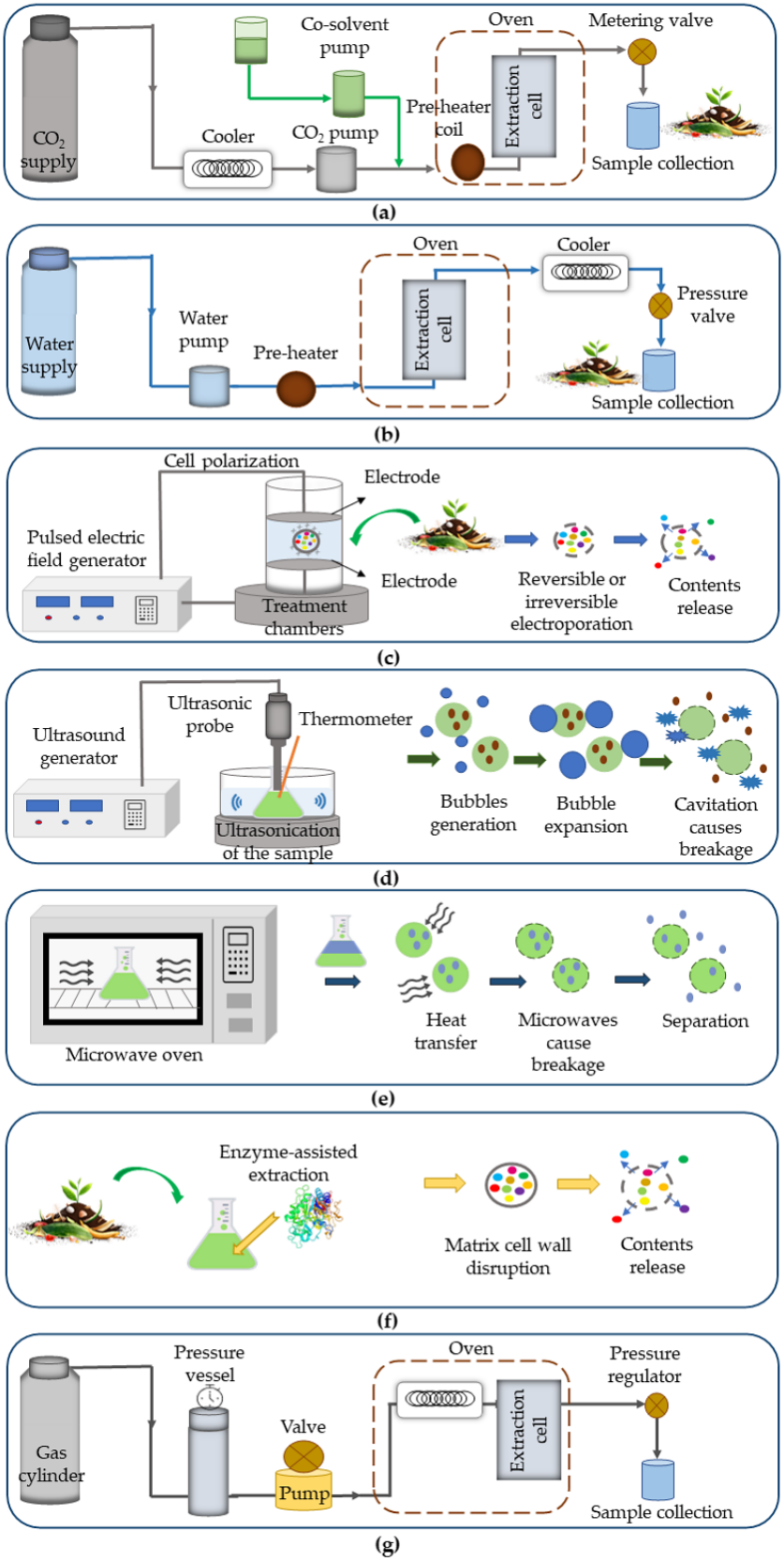

Figure 2. Simplified representation of the most used green extraction procedures, (**a**) supercritical fluid extraction, (**b**) subcritical water extraction, (**c**) pulsed electric fields, (**d**) ultrasound-assisted extraction, (**e**) microwave-assisted extraction, (**f**) enzyme-assisted extraction, and (**g**) pressurized liquid extraction.

Table 1 shows the potential of SFE in the extraction of important value-added compounds from agri-food wastes [10–13]. The bioactive compounds extracted by SFE include a wide diversity, namely, phenolic compounds from onion peels [16], antioxidants and saponins from *Agave salmiana* bagasse [12], and carotenoids from carrot peels [10], among others. The effect of pressure, temperature, and the addition of a co-solvent in the extraction of bioactive compounds by SFE are evaluated in some studies in Table 1. Generally, the extraction was performed by applying pressures, temperatures and co-solvents ranging from 30–400 bar, 33–230 °C and 5–15 % v/v, respectively, while the extraction time and flux ranged from 30–180 min and 1.7–133 g/min, respectively. Some studies compared the extraction efficiency of SFE with other conventional extraction procedures (e.g., Soxhlet extraction). Soldan et al. [13] compared the extraction efficiency of SFE and Soxhlet on the recovery of the bioactive compounds phenolics, flavonoids, fatty acids, and carotenoids, from *Capsicum annuum* waste. The results showed that the total mass yields obtained by SFE ranged from 9.38 to 10.08%, while for Soxhlet the yields ranged from 8.45 to 15.5% (w/w). Despite revealing bioactive compounds, the extracts did not show significant antioxidant activity. Natolino and collaborators [17] also performed a comparison between SFE and Soxhlet on the recovery efficiency of seed oils from pomegranate, which showed no significant difference between these two extraction procedures, as the extraction yield from SFE ($0.18 \pm 0.01\ g/g$) was similar to Soxhlet ($0.19 \pm 0.01\ g/g$). Nevertheless, SFE was faster than Soxhlet (8 h vs. 2 h of SFE) to achieve the asymptotic extraction yield and presented more oxidation stability than Soxhlet. Santos–Zea et al. [12] evaluated the effect of ultrasound on SFE for the recovery of antioxidants and saponins from agave bagasse. The data obtained showed that the use of ultrasound-assisted SFE improved the extraction yield of antioxidants and saponins from agave bagasse when a low mass load (0.043 g/cm^3) was applied. Since the CO_2-SFE demonstrated low extraction efficiency of more polar compounds in some studies (e.g., phenolic compounds), a few authors proposed the use of co-solvents to enhance the extraction yields of polar and medium polar bioactive compounds [13]. Soldan et al. [13] verified that the temperature variation and the addition of a co-solvent (ethanol) were significant in increasing the total extracted mass of oleoresin, although the pressure did not have a significant effect. In sum, this green extraction technique can be easily transferred to an industrial scale to extract large quantities of matrix and obtain a great amount of extract in a single step. However, despite the exceptional extraction properties and outstanding versatility, the high processing costs and the complex industrial equipment are restricting factors [14].

Table 1. Extraction techniques for bioactive recovery from agri-food waste.

Agri-Food Waste (Amount)	Targets	Extraction Conditions	Extraction Efficiency	Ref.
		Supercritical fluid extraction		
Agave salmiana bagasse (10 g)	Antioxidants and saponins	CO_2, 60 °C, 300 bar, 10 % *v*/*v* ethanol, 1.7 g/min, 60 min	Increase in the antioxidant activity in the US-assisted extraction from 11.54 ± 0.06 to 17.61 ± 0.75 µmol of Trolox equivalents/g	[12]
Avocado peel and seeds (-)	Catechin, quercetin	CO_2, 80 °C, 250 bar, ethanol ratio of 1:1.5 S:L, 30 min	Integral biorefineries of avocado seed and peel allow profit margins of 47% and 43%, respectively	[18]
Capsicum annuum waste (20 g)	Phenolics, flavonoids, fatty acids, and carotenoids	CO_2, 40 and 60 °C, 200 and 250 bar, with and without ethanol, 3 g/min, 30 min	Yield 9.38–10.08%, phenols (12.30–23.94 mg/g), flavonoids (0.6–1.52 mg/g), and carotenoids (0.27–2.01 mg/g)	[13]
Carrot peels (50 g)	Carotenoid	CO_2, 59 °C, 349 bar, 15% *v*/*v* ethanol, 15 g/min, 80 min	Carotenoid recovery was (86.1%) with 97 % purity	[10]
Grape seeds (17 g)	Triacylglycerols	CO_2, 40–60 °C, up 400 bar, 1.8–2.8 g/min	Oil yield in the range of 12.0–12.7%, as compared to 12.3% obtained by a conventional *n*-hexane extraction	[19]
Mandarin peel (100 g)	Limonene, hesperidin	CO_2, 130–220 °C, 100–300 bar, 33 g/min, 90 min	Limonene (13.16 and 30.65% at 100 and 300 bar), hesperidin (0.16–15.07 mg/g)	[20]
Mango peel (5 g)	Carotenoids	CO_2, 60 °C, 250 bar, 15% *w*/*w* ethanol, 6.7 g/min, 180 min	Carotenoids (1.9 mg all-trans-β-carotene equivalent/g dried mango peel)	[11]
Melon seeds (5 g)	Phytosterols	CO_2, 33 °C, 200 bar, 11 g/min, 3 h	β-sitosterol (304 mg/kg) and stigmasterol (121 mg/kg)	[21]
Pomegranate seed (100 g)	Seed oil	CO_2, 60 °C, 320 bar 133 g/min, 180 min	Oil (85.4% of punicic acid)	[17]
		Subcritical water extraction		
Citrus peel (3 g)	Hesperidin, narirutin	Water, 110–190 °C, 10 MPa, 3–15 min	Hesperidin (6.96 mg/g peel dw), narirutin (8.76 mg/g peel dw)	[22]
Grape pomace (50–60 g)	Phenolic compounds	Water, 130–190 °C, 100 bar, 10 mL/min	29 g of phenolic compounds (*p*-hydroxyphenyl, guaiacyl, and syringyl)/100 g extract	[23]
Kiwifruit peel (2% S:S ratio)	Phenolic compounds	Water, 160 °C, S:S ratio (2%), pH 2, 20 min	TPC (51.2 mg GAE/g dw), TFC (22.5 mg QE/g dw)	[24]
Onion peel (2 wt % onion skins into 600 mL of H_2O)	Phenolic compounds	Water, 170–230 °C, 30 bar, 400 rpm/30 min	63–75 mg GAE/g, 23–26 QE/g	[16]
Onion skin (4 g)	Phenolic compounds	Water, 105–180 °C, 5 MPa, 2.5 mL/min	Quercetin (15 mg/g onion skin) and quercetin-4-glucoside (8 mg/g onion skin)	[25]
Peach palm (4 g)	Phenolic compounds, sugars	Water, 130 °C, 100 bar, 1 mL/min, 90 min	Soluble sugar (15 g/100 g), TPC (921 mg/100 g)	[26]

Table 1. *Cont.*

Agri-Food Waste (Amount)	Targets	Extraction Conditions	Extraction Efficiency	Ref.
Shellfish waste (1 g)	Protein hydrolysates	Water, 200 °C, heating rate of 6 °C/min	8.5 g protein/100 g dw (improved extraction yield of up to 65%)	[27]
Vine-canes (40 g)	Phenolic compounds	Water, 250 °C, 50 min	181 mg GAE/g dw, 203 mg TE/g dw	[28]
Vine co-products: cane, wood, and root (5 g)	Stilbenes	Water, 160 °C, 100 bar, 5 min	Cane (3.62 g/kg dw), wood (9.32 g/kg dw), and root (12.1 g/kg dw)	[29]
		Pulsed electric fields		
Apple pomace (28.7 g)	Phenolic compounds	E = 2, 3 kV/cm, U = 17, 100 kJ/kg, 40 °C	PEF performed with EtOH:H_2O (70:30, *v*/*v*) showed the highest content of phlorizin (753.84 ± 26.38 µg/g fresh apple pomace)	[30]
Banana peels (-)	Phenolic compounds	E = 1.3–6.45 kV/cm	Increase the TPC and antioxidant activity	[31]
Jackfruit waste (1:20 *w*/*v* solid-to-solvent ratio)	Pectin polysaccharide	E = 5–15 kV/cm	No significant effect on pectin yield	[32]
Lemon peels (30 g)	Phenolic compounds	E = 7 kV/cm, U = 7.6 kJ/kg	Increase the efficiency of phenolic compounds (hesperidin and eriocitrin) extraction by 300%	[33]
Potato peels (5 g)	Phenolic compounds	E = 0.25–3 kV/ cm, U = 1–20 kJ/kg	PEF showed higher TPC yield (10%) and antioxidant activity (9%) compared to conventional solid–liquid extraction with same extraction protocol but without the application of the PEF pre-treatment)	[34]
Pomegranate peels (30 g)	Ellagic acid	E = 10 kV/cm	PEF selectively extracted and enhanced the recovery of ellagic acid (≈740 µg/g dm)	[35]
Pomelo peels (1 g)	Naringenin	E = 2–10 kV/cm	Increase the extraction yield of naringenin	[36]
Olive pomace (850 g)	Phenolic compounds	E = 1–6.5 kV/cm, U = 0.9–51.1 kJ/kg, 50 pulses spaced at 3 s, 20–27.5 °C	PEF allowed a 28.8% increased recovery yield of polyphenols (~3 mg GAE/L) compared to untreated	[37]
Tomato peels (10 g)	Lycopene	E = 5 kV/cm, U = 5 kJ/kg, 20 ± 2 °C	Enhance the extraction rate (27–37%), the lycopene yields (12–18%) and the antioxidant power (18–18.2%)	[38]
		Ultrasound-assisted extraction		
Apple leaves (10 g)	Phloretin	400 W, 20 kHz, 14.4 min, <25 °C	The phloretin concentration ranged from 292 to 726 µg/g	[39]
Apple pomace (1:10 (*w*/*v*) S:L ratio)	Phenolic compounds	45 min, 45 °C	Increase the TPC, antioxidant activity, and recovery of interesting antioxidant compounds (quercetin derivatives, chlorogenic acid, phloridzin)	[40]
Beet leaves (1:20 (*w*/*v*) S:L ratio)	Bioactive compounds	90 W, 16 min	Yields were 14.9 mg/g polyphenols, 949.1 µg/g betaxanthins, and 562.2 µg/g beta-cyanins	[41]

Table 1. *Cont.*

Agri-Food Waste (Amount)	Targets	Extraction Conditions	Extraction Efficiency	Ref.
Brewers' spent grains (1:30 (*w*/*v*) S:L ratio)	Proanthocyanidins	400 w, 75 % acetone, 55 min, 25 °C	High recovery of proanthocyanidins (1023 µg/g dw)	[42]
Citrus peel (1 g)	Citric acid	119–141 W, 5.8–35.5 min, 0–7 % (*v*/*v*) ethanol	Recovery of 6.4 g and 3.4 g of citric acid per 100 g of dry orange and lime peels, respectively	[43]
Grape pomace (280 g)	Phenolic compounds	450 W, 15 min, 20 °C	Increased the TPC (6.68 ± 0.05 mg of gallic acid) and antioxidant activity (ABTS: 23.84 ± 0.57 µmol of Trolox equivalents/g and DPPH: 33.27 ± 2.00 µmol of Trolox equivalents/g)	[44]
Kiwi peel (1.5 g)	Flavonoids	5–500 W, 20 kHz, 1–45 min, 25 °C	46% extract weight and 1.51 mg/g dw of flavonoids	[45]
Orange peels (10 g)	Bioactive compounds	40 kHz,85 min, 55 °C, 61% methanol	The spectra of extracts showed a similar fingerprint of hesperidin	[46]
Tomato peels (72 mL/g, L:S ratio)	Lycopene	20 kHz, 20 min, 65 °C, Microwave-assisted extraction	Lycopene recovery of 1536 µg/g	[47]
Carrot juice waste (flaxseed oil + waste ~ 20 g)	Carotenoids	170 W, 9.46 min, 8:1 g/g oil-to-waste ratio	Carotenoid recovery of 77.48%. The enriched flaxseed oil showed high phenolic content (214.05 ± 1.61 µg GAE/g oil) and antioxidant activity (inhibition % of DPPH = 70.67 ± 0.85)	[48]
Coffee pulp (-)	Phenolic compounds, flavonoids, chlorogenic acid, and caffeine	1000 W, 85 min, 1:100 g/100 mL sample-to-solvent ratio, 42.5 % (*v*/*v*) aqueous ethanol solution	Extraction yields of TPC, flavonoids, chlorogenic acid, and caffeine were 38.68, 27.00, 6.95, and 5.47 (mg/g dw), respectively. The extract showed high antioxidant capacities (ABTS, DPPH, and FRAP assays as 87.95, 9.3, 65.31 (mg TE/g DW), respectively)	[49]
Peach waste (1000 mg)	Phenolic compounds and anthocyanins	500 W, 90 s, 80 % ethanol (*v*/*v*)	TPC of 19.35 mg GAE/g fresh plant matter and total anthocyanin 1.12 mg cyn-3-glu/g fresh plant matter) yields	[50]
Cocoa shell waste (100 g)	β-Sitosterol	500 W, 10 min, 70 °C	The maximum yield obtained was 13% higher than the yield of conventional maceration (3546.1 mg/ 100 g)	[51]
Eggplant peel (-)	Phenolic compounds, flavonoids, anthocyanins	269.82 W, 7.98 min, 5.01 mL/g L:S ratio	The maximum extraction yield (3.27%), TPC (1,049.84 µg GAE/mL), TFC (130.40 µg QE/mL), and total anthocyanin content (6.99 mg/L)	[52]
Lemon peel waste (-)	Essential oil (limonene, β-pinene, and γ-terpinene) and pigment	500 W, 50 min, 80 °C, 80% (*v*/*v*) ethanol, 1:10 L:S ratio	The extraction yields of lemon essential oil and pigment were around 2 wt.% and 6 wt.%, respectively	[53]
Spent sweet potato leaves (0.1 g)	Flavonoids	470 W, 21 min, 54 °C, 70 mg/mL S:L ratio	The yield of TFC was 40.21 ± 0.23 mg rutin equivalents/g	[54]

Table 1. *Cont.*

Agri-Food Waste (Amount)	Targets	Extraction Conditions	Extraction Efficiency	Ref.
Broccoli stems, leaves and florets (2.5 g)	Phenolic compounds (vanillic, sinapic, caffeic, chlorogenic, ferulic, gallic, neochlorogenic, and *p*-coumaric acids)	Stems: 2.45 GHz, 74.54% methanol, 15.9 min, 74.45 °C Leaves: 2.45 GHz, 80% methanol, 10 min, 73.27 °C Florets: 2.45 GHz, 80% methanol, 18.9 min, 75 °C	MAE increased the phenolic yield up to 45.70% (1940.35 ± 0.794 µg GAE/g dw), for broccoli leaves, 133.57% (657.062 ± 0.771 µg GAE/g dw) for broccoli florets, and 65.30% for broccoli stems (225.273 ± 0.897 µg GAE/g dw), in less time compared with maceration extraction	[55]
Spent onion skins (-)	Flavonoids (quercetin, kaempferol, luteolin, and quercetin-3-O-β-D-glucoside)	554 W, 16 min, 76 °C, 14 mg/mL S:L ratio	TFC extraction yields of 47.83 ± 0.21 mg/g	[56]
		Enzyme-assisted extraction		
Unsold tomato (-)	Carotenoids and carotenoid-containing chromoplasts	Enzymatic mix: polygalacturonase, pectin lyase, cellulose, xylanase, 25 U/g for 180 min, 45−55 °C at pH 5–5.5	Recovery yield of 4.30 ± 0.08 mg lycopene/ kg tomato)/U as carotenoid-containing chromoplasts and 5.43 ± 0.04 mg lycopene/ kg tomato)/U as total carotenoids	[57]
Apricot pulp (-)	Polysaccharides (sodium glycocholate and sodium taurocholate)	5 mL/mg liquid-material ratio, 3% enzyme dosage and incubation time 1.5 h, pH 4.5	The yield, sodium glycocholate and sodium taurocholate binding rates were 21.90%, 39.08% and 43.80%, respectively	[58]
Tomato peel and seed (4 g)	Lycopene-rich oleoresins	Enzymatic reaction: 40 °C, 5 h, 0.2 mL/g enzyme:substrate ratio, 5 mL/g solvent:substrate ratio, extraction time 1 h, 1 enzyme:enzyme ratio	Celluclast:Pectinex-ethyl acetate combination yielded the highest content of phenolic compounds (oleoresin with a concentration of 11.5 mg)	[59]
Beetroot cell wall (200 g)	Betalains	Enzymatic mix (cellulase 37%, xylanase 35%, pectinase 28%), 25 U/g total dose of enzymatic mix, 25 °C, 240 min, pH 5.5	Betaxanthins and betacyanins yield 10 and 15 mg/mL U, respectively	[60]
Sweet cherry pomace (15 g)	Non-extractable polyphenols	0.38 g/mL S:L ratio, 70 °C, pH 10, 40 min for Depol (90 µL/g of sample) and Promod (140 µL/g of sample) enzymes and 18.4 min for Pectinase enzyme (2 µL/g of sample)	The extracts obtained by acid hydrolysis (1.87 ± 0.05 mg GAE/g of extraction residue) and Promod enzyme (1.75 ± 0.20 mg GAE/g of extraction residue) followed by alkaline hydrolysis (1.46 ± 0.20 mg GAE/g of extraction residue) and enzymatic hydrolysis with Depol enzyme (1.33 ± 0.13 mg GAE/g of extraction residue) were the richest in terms of phenolic content	[61]
Sugar beet leaves	Protein	54.25 °C, 81.35 min, 27.65 mL/g solvent/solid ratio	EAE increased the protein yield by 43.27% and reached a 79.01% yield	[62]
Raspberry pomace (9 g)	Lipophilic compounds (phytosterols) and polyphenols	1.2 units of thermostable alkaline protease/100 g pomace press-cake, 60 °C, 2 h hydrolysis, pH 9	The recovery of polyphenols and antioxidant activity was, respectively, 48% and 25% higher than the obtained by extraction with methanol/acetone/water mixture	[63]

Table 1. *Cont.*

Agri-Food Waste (Amount)	Targets	Extraction Conditions	Extraction Efficiency	Ref.
		Pressurized liquid extraction		
Pomegranate peel and carpelar (6 g)	Phenolic compounds (α, β punicalagin, and ellagic acid)	60 °C, 80 bar, flow rate of 1 mL/min, 76 min, 10 solvent-to-feed ratio	The highest content of α, β-punicalagins, and ellagic acid obtained was 194.96 mg/100 g and 24.91 mg/100 g, respectively, representing 45% of TPC	[64]
Beetroot leaves and stems (5 g)	Phenolic compounds (ferulic acid, vitexin and sinapaldehyde)	40 °C, 7.5, 10 and 12.5 MPa, flow rate of 3 mL/min	The highest TPC was obtained for beetroot leaves and varied from 7 ± 1 to 252 ± 2 mg GAE/g extract	[65]
Vitis vinifera L. cv. negra criolla pomace (5 g)	Phenolic compounds (flavanols and phenolic acids)	10 atm, 5 min with 250 s of nitrogen purge Flavanols: 20% ethanol, 160 °C Phenolic acids: 60% ethanol, 160 °C	PLE recovered ~2.5 and ~1.5 more polyphenols from skins (6.93 µg/g dw) and seeds (45.34 µg/g dw), respectively, compared to conventional extraction	[66]
Olive pomace (5 g)	Phenolic compounds (phenolic alcohols, secoiridoids, flavonoids, and lignans)	Clean-step with n-hexane as the solvent and 1500 psi at room temperature to remove the lipophilic fraction from the olive pomace. Ethanol (0 to 100%), 40 to 176 °C, 1500 psi, 20 min	PLE showed higher TPC than conventional extraction (1659 mg/kg dw and 281.7 mg/kg dw, respectively)	[67]
Pomegranate peel (3.75 g)	Phenolic compounds (phenolic acids, flavonoids, and hydrolysable tannins)	200 °C, ethanol 77%, 1500 psi, 20 min	TPC of 164.3 ± 10.7 mg GAE/g dw	[68]
Pomegranate seed (1.75 g of waste and 7 g of sand, 1:4 ratio)	Protein and phenolic compounds	Ethanol (0 to 100 %), 28 to 170 °C, 1 to 5 cycles, 3 to 12 min, pH 6.5 to 11, 103 bar	Higher extraction yield by PLE (15.3 ± 0.9 g proteins/100 g pomegranate seed waste) at a cost of a longer extraction time and the co-extraction of phenolic compounds	[69]

Legend: dw—dry weight, L:S—liquid:solid; S:L—solid:liquid; S:S—solid:solvent; GAE—gallic acid equivalent; PEF—Pulsed electric fields; QE—quercetin equivalent; TPC—total phenolic content; TFC—total flavonoid content.

2.2. Subcritical Water Extraction

Subcritical water extraction (SCWE) is an environmentally friendly and techno-economically feasible alternative to conventional extraction procedures, such as solvent extraction. This green extraction technique uses water as an extractant, which is economic, non-flammable, and renewable. Compared to conventional extraction procedures, such as solid–liquid extraction (Soxhlet) using organic solvents, maceration, and hydrodistillation, SCWE shows higher yield and purity while applying lower extraction time [16,24]. This green extraction procedure is also distinguished by the demand for a downstream solid–liquid separation step which increases the energetic requirement of the process [70]. However, and despite large research efforts, corrosion problems have not been completely solved for the application of SCWE at an industrial scale [14].

During SCWE, the feedstock is heated in the aqueous phase at a sub-critical temperature (∼150–320 °C) and pressure (∼20–150 bar). Under these conditions, the dielectric constant (ε), surface tension, and viscosity, among other properties of the water change, enhance mass transfer and the extractability of barely water-soluble bioactive compounds, since subcritical water promotes the hydrolysis of the bonds between phenolic compounds and agri-food waste matrix [25]. Moreover, the mass transfer ratio also rises due to the reduced viscosity and surface tension of the water as well as its diffusivity [24]. A diversity of SCWE applications on the extraction of bioactive compounds from agri-food wastes has been performed, namely, stilbenes from vine co-products (e.g., cane, wood, and root) [29], phenolic compounds from onion peel [16], and protein hydrolysates from shellfish waste [27], among others. Nevertheless, the main drawback of SCWE is the risk of hydrolysis and other degradation reactions during extraction [14]. In this sense, Rodrigues et al. [27] used SCWE to recover antioxidant protein hydrolysates from shellfish waste streams. In this study, the authors assessed the impact of operating temperature (150, 200, and 250 °C), solid/liquid ratio (1:5, 1:10, and 1:15 g/mL), and heating rate (3 and 6 °C/min) on SCWE performance. It was verified that higher temperatures enabled the production of extracts with a higher antioxidant potential, possibly due to an increase of smaller peptides/free amino acids and Maillard reaction products. On the other hand, Hwang and collaborators [22] recovered hesperidin and narirutin from *Citrus unshiu* peel waste using SCWE combined with pulsed electric field (PEF) treatment. The data obtained demonstrated that the concentrations of hesperidin and narirutin increased with PEF treatment time, with increased yields of hesperidin and narirutin by 22.1% and 33.6%, respectively, in PEF pretreatment combined with SCWE.

2.3. Pulsed Electric Fields

Pulsed electric field (PEF) is a nonthermal agri-food processing method that applies high-intensity electric field pulses to agri-food passing through electrodes. This extraction process causes the electroporation of membranes (permeabilization) that enables the release of intracellular bioactive compounds from the matrix investigated [22,71]. The extraction efficiency of PEF treatment depends on numerous factors, involving electric field strength, total specific energy input, treatment time, and temperature. Previous studies have demonstrated that the PEF pre-treatment of moderate electric field intensity (0.5–10 kV/cm) and relatively low energy input (1–10 kJ/kg) has advantageous effects on the permeabilization of membranes of plant cells, enabling high recovery yields of intracellular compounds of interest from a wide range of food processing wastes and by-products [38]. Furthermore, PFE treatments have shown several advantages, including low solvent consumption, shorter treatment time, energy efficiency, continuous operability, ease of scale-up, non-destructive nature, and high selectivity. However, its dependence on medium composition (conductivity) and the high cost of the equipment represents the main disadvantages of PFE treatments [35,72].

Table 1 presents a diversity of PFE applications for the extraction of bioactive compounds from agri-food wastes, such as lycopene from tomato peels [38], ellagic acid from pomegranate peels [35], and phenolic compounds from lemon peels [33], among

others. Pollini et al. [30] compared different extraction techniques, such as ultrasound-assisted extraction (UAE), ultraturrax extraction (UTE), accelerated solvent extraction (ASE), and PEF extraction pre-treatment to identify the most efficient method to recover phenolic compounds from apple pomace. The extraction efficiency of phloridzin, the main phenolic compound in apples, increased by applying PFE at a low intensity and for a long duration (2 kV/cm and 100 kJ/kg), using EtOH:H_2O (70:30, *v*/*v*). In another study, Lal et al. [32] combined PFE with microwave-assisted extraction to recover pectin polysaccharide from jackfruit waste, but the pectin yield obtained was not significant when compared to conventional processes. Radjha et al. [35] compared the aqueous extraction efficiency and biological activities of phenolic compounds from pomegranate peels assisted by infrared (IR), ultrasound (US), PFE, and high-voltage electrical discharges (HVED). The data showed that the PFE selectively extracted and enhanced the recovery of ellagic acid (≈740 µg/g dm), whereas HVED (≈345 µg/g dm) intensified the gallic acid extraction compared to US, IR, PFE and WB. Peiró and collaborators [33] evaluated the influence of PEF of different intensities (3–9 kV/cm and 0–300 µs) on the extraction of phenolic compounds from lemon peel residues, which increased by around 300%, giving maximum values of 84 mg of hesperidin in 100 g FW and 176 mg of eriocitrin in 100 g FW.

2.4. Ultrasound-Assisted Extraction

Ultrasound-assisted extraction (UAE) is a green extraction procedure and a techno-economically feasible alternative to conventional extraction procedures. This technique has gained attention in recent years, due to its excellent advantages compared to traditional extraction procedures, such as reduced solvent volumes, shorter extraction time, and use of common laboratory equipment (e.g., ultrasonic bath), making it an environmentally sustainable and economical extraction procedure [73,74]. Yet, the solid–liquid separation and drying are certainly the main disadvantages of the UAE process. This extraction procedure is based on the cavitation process induced by compression and expansion cycles associated with the passage of ultrasounds (20 kHz–100 MHz frequency) through the sample. The acoustic waves promote the distance between molecules and consequently generate spaces among them, forming bubbles. The implosion of the cavitation bubbles causes inter-particle collisions resulting in particle disruption and enhanced diffusion of extractable bioactive compounds into the solvent [70,75]. A large amount of energy is released by bubble implosions, causing significant changes in the local temperature and pressure, liquid circulation, and turbulence, consequently increasing the mass transfer rate [47]. Moreover, the extraction efficiency of UAE can be significantly influenced by the sample properties (e.g., consistency, rheology, particle mobility) which affect ultrasound energy dispersion [75].

The UAE has been extensively applied at the lab scale in diverse food fields [70]. Ben-Othman and collaborators [39] used the response surface method (RSM) with a Box–Behnken design to select the best extraction efficiency of UAE for the recovery of phloretin and other phenolic compounds from apple tree leaves (*Malus domestica* Borkh.) from different cultivars from Estonia. The optimal extraction conditions were 14.4 min of extraction time, 10% sonication amplitude, and 10 g of sample per 100 mL solvent (70% ethanol, *w*/*w*). By applying the ideal conditions, the phloretin concentration ranged from 292 to 726 µg/g and the antioxidant activity from 6.06 to 11.42 mg GAE/g in the local winter cultivars "Paide taliõun" and "Tellissaare", respectively. Martín-García et al. [42] used RSM to evaluate the effect of solvent composition, extraction time, and ultrasound power on the recovery of proanthocyanidins from brewers' spent grains. The highest content of proanthocyanidins was obtained using 80/20 acetone/water (*v*/*v*), 55 min, and 400 W, which resulted in 1.01 mg/g dw of proanthocyanidins from brewers' spent grains. In another study, da Rocha et al. [44] compared the extraction efficiency of microwave-assisted extraction and UAE of bioactive compounds from grape pomace. The results showed that both extraction procedures allowed the recovery of 45% of the anthocyanins when compared to the exhaustive extraction with methanol acidified solution.

2.5. Microwave-Assisted Extraction

Microwave-assisted extraction (MAE) is a green and cost-effective extraction technique that has gained a lot of attention recently, due to its enhanced productivity, reduced extraction time, less solvent requirement, simplicity, and low set-up costs [4]. MAE involves electromagnetic radiations, transmitted as waves in the frequency range from 300 MHz to 300 GHz [4]. This technique is based on the principle that the energy absorbed during the passage of microwaves through the medium is converted into thermal energy, which facilitates the processing, due to higher extraction temperature and resultant faster mass transfer rate [4,76]. The heating effect of microwaves depends on the dielectric properties of the mixture of the solvent. When a solvent placed in contact with the sample is heated, MAE leads to the disruption of the hydrogen bonds, which results in the dipole rotation of the molecules and migration of the ions. Consequently, this process allows for the diffusion of the solvent, and thus the dissolution of the components [4]. MAE can be influenced by a wide range of parameters, namely microwave power, frequency, irradiation time, the particle size of the sample matrix, the composition of the solvent, extraction temperature, pressure, and the number of cycles. The choice of a suitable solvent for extraction is important and depends on the solubility, dielectric constant, and dissipation factors. Solvents with both high dielectric constant and dissipation factor can lead to a better extraction, which can be accomplished by mixtures of water with other solvents, such as ethanol or methanol [76].

MAE has been frequently used in the extraction of bioactive compounds, especially for plant materials [76]. Tran, Akanbi, Kirkman, Nguyen and Vuong [49] provided a method for the recovery of total phenolics, flavonoids, chlorogenic acid, and caffeine from coffee pulp using an MAE system. The results of this study showed that the sample-to-solvent ratio and ethanol concentration significantly affected the recovery yields of the bioactive compounds and the antioxidant capacity. Under the optimal conditions (Table 1), the extraction yields of total phenolic compounds, flavonoids, chlorogenic acid, and caffeine were 38.68, 27.00, 6.95, and 5.47 (mg/g dw), respectively. The extracts showed high antioxidant capacities, with values measured by ABTS, DPPH, and FRAP assays as 87.95, 9.3, and 65.31 (mg Trolox equivalents/g dw), respectively. In another study, Kurtulbaş, Sevgen, Samli and Şahin [50] extracted phenolic compounds and anthocyanins from peach peels, with the highest total phenolic content (TPC) being 19.35 mg of gallic acid equivalents/g of fresh plant matter and a total anthocyanin of 1.12 mg of cyn-3-glu/g of fresh plant matter, under the optimal MAE conditions (Table 1). After the extract was obtained, the samples were exposed to several storage media, such as −20 °C, 4 °C, and 25 °C in dark and 25 °C in light and the storage stability was monitored in terms of 4 bioactive properties (TPC and total anthocyanin contents, *p*-hydroxybenzoic acid and *p*-coumaric acid). In a general way, the degradation rate rose with storage temperature. The longest shelf life in terms of total phenols, anthocyanins, and major phenolic compounds (*p*-hydroxybenzoic acid and *p*-coumaric acid) was calculated as 111, 107, 88, and 83 days under deep freezer conditions at −20 °C. Zhang and collaborators [54] extracted flavonoid compounds from spent sweet potato leaves with natural deep eutectic solvents (NADESs) coupled with MAE. The highest extraction yield (40.21 ± 0.23 mg of rutin equivalents/g of sweet potato leaves) was obtained with NADES-2 synthesized by choline chloride and malic acid (molar ratio 1:2). The extracts were recovered by macroporous resin for the biological activity detection of flavonoid compounds, in which the AB-8 macroporous resin provided a recovery yield of 85.46% ± 2.33%. Additionally, the in vitro bioactivity experiments confirmed that the flavonoid compounds had good DPPH and O_2^- radical-scavenging activity, as well as inhibitory effects on *E. coli, S. aureus, E. carotovora*, and *B. subtilis*. Rodríguez García and Raghavan [55] evaluated the potential of MAE as a green technique to obtain phenolics. The authors extracted phenolic compounds (vanillic, sinapic, caffeic, chlorogenic, ferulic, gallic, neochlorogenic, and *p*-coumaric acids, identified by HPLC) from broccoli by-products (stems, leaves, and florets). MAE was found to increase the phenolic yield up to 45.70% for broccoli leaves, 133.57% for broccoli florets, and 65.30% for broccoli stems, in less

time compared with maceration extraction. Despite the advantages of MAE over conventional extraction methods, the high dependency on the solvent nature and the extraction temperature limits the application of MAE [4,76].

2.6. Enzyme-Assisted Extraction

Enzyme-assisted extraction (EAE) is an eco-friendly, efficient, and mild extraction technology applied in the extraction of valuable compounds from plant resources [76]. This technique uses water as a solvent rather than organic chemicals and is generally employed when the phytochemicals are distributed in the cell cytoplasm or the presence of hydrogen or hydrophobic bonds in the polysaccharide-lignin network [4]. EAE is used to catalyze the hydrolysis of components resistant to mass transfer like cell wall or by binding to the target components in the matrix based on the specificity and regioselectivity of enzymes. The addition of specific enzymes like pectinases, cellulases and hemicellulases can enhance the recovery of the target compounds through degradation and disruption of the structural integrity of the cell walls and membranes [76,77]. EAE is influenced by several parameters, including enzyme composition, concentration, water-to-solid ratio, enzyme/substrate ratio, pH, extraction temperature, and hydrolysis time. Due to the high specificity of enzymatic reactions, careful selection should be given to the parameters mentioned to guarantee the extraction of the compounds of interest under optimal conditions. Besides these parameters, particle size and the solvent-to-matrix ratio are also significant influencing factors [4,76]. Nevertheless, EAE shows some limitations. It is not possible to completely hydrolyze the matrix cell walls with currently available enzyme preparations, which can limit the extraction yields of phytochemicals and bioactive compounds. In large- or industrial-scale productions, enzymes can be very expensive and it can be challenging to apply EAE to scale up in the industry because enzymes behave differently due to the possible variations of environmental conditions inside an industrial-scale extractor [76].

Xu et al. [58] extracted apricot hypolipidemic polysaccharides using EAE. The methodology allowed the recovery of sodium glycocholate and sodium taurocholate binding rates at 21.90%, 39.08% and 43.80%, respectively. Compared with polysaccharides extracted by the water method, it was concluded that the enzymatic extraction of apricot polysaccharides had a better hypolipidemic effect, where the reduced viscosity of polysaccharides improved the intermolecular fluidity. Likewise, the reduction of the galacturonic acid content and molecular weight of polysaccharides was beneficial to expose the polysaccharide active groups and combine them with cholate. Catalkaya and Kahveci [59] improved lycopene recovery from tomato waste by applying enzymatic pretreatment. The authors found that the recovery of lycopene from tomato waste was extensively affected by the type of extraction solvent. Lycopene-rich oleoresins (11.5 mg lycopene/g) obtained through pretreatment of waste by a combination of cellulolytic and pectinolytic enzymes followed by ethyl acetate extraction had the highest phenolic compound concentration and improved antioxidant properties, as well as the highest lycopene recovery and one of the highest red colour intensity. Lombardelli and collaborators [60] extracted betalains from unsold red beets to be used as an alternative to synthetic food colourants. The recovery of betalains was carried out by an enzymatic mix considering the polysaccharide composition of the beetroot cell wall (37% cellulase, 35% xylanase, and 28% pectinase), which improved the recovery of these pigments (10 mg/mL U of betaxanthins and 15 mg/mL U of betacyanins). Dominguez-Rodriguez, Marina and Plaza [61] aimed to recover the remains of polyphenols in the extraction residue of sweet cherries, the non-extractable polyphenols. The authors evaluated the EAE efficiency of extraction with three different enzymes, Depol, Promod and Pectinase. In general, EAE with Promod enzyme (1.75 $\pm$ 0.20 mg GAE/g of extraction residue) followed by EAE with Depol enzyme (1.33 $\pm$ 0.13 mg GAE/g of extraction residue) were more efficient than the EAE with Pectinase enzyme (1.11 $\pm$ 0.13 mg GAE/g of extraction residue), which reached extracts with higher TPC, total proanthocyanidin content, antioxidant, and antihypertensive capacities. This study showed that the optimal EAE methods were more suitable to obtain non-extractable polyphenols from the extraction

residue than the extraction by alkaline and acid hydrolysis. Several studies have mentioned that the combination of novel extraction techniques is beneficial and consists of a promising strategy for fast and efficient extraction (as reviewed by [76]). Combinations of microwave, ultrasound and enzyme-aided extraction are used as powerful tools to provide higher extraction yields and better products [76]. For instance, Yin et al. [78] extracted polysaccharides from *Lentinus edodes* through enzyme-microwave-ultrasound-assisted extraction (EMUE). The optimum extraction conditions were assessed by RSM, which included an enzymatic temperature of 48 °C, pH 5.0, microwave power of 440 W, and microwave time of 10 min. Under these conditions, the highest yield of polysaccharides was 9.38%. The yields from EMUE were compared with other extraction methods including hot-water extraction, EAE, MAE, and UAE. When compared to hot-water extraction, EAE, UAE, and MAE, the yield of EMUE polysaccharides improved by 50.32%, 26.59%, 16.38%, and 8.56%, respectively.

In ultrasound-assisted enzymatic extraction (UAEE), enzymes promote the recovery of compounds by degrading and disrupting the cell walls and membranes. However, enzymes cannot completely hydrolyze the matrix and as such, UAE has a complementary effect for EAE. The cavitation caused by power ultrasound in UAE can physically disrupt and break down the matrix to facility the enzymatic reaction and thus the release of target compounds. Moreover, the enhancement of enzymatic reaction rates caused by ultrasonication in EAE is thought to increase the collisions between enzyme and substrate, which implies a higher releasing rate [76]. Encalada et al. [79] develop an environmentally friendly extraction method, consisting of an ultrasound-pretreatment (12.27 W/cm^2: 20 kHz, 80% amplitude, 20 min) followed by EAE (5 h, 40 °C, without or with hemicellulase or cellulase) of pectin-enriched fractions from the cell wall of discarded carrots due size and shape out of standards. The methodology based on UAEE aimed to achieve higher yields, through the co-extraction of carotenoids and other natural antioxidants (α- and β-carotenes, lutein, α-tocopherol) from the pectin extracts. The ultrasound-pretreatment increased the extraction yield of all pectin-enriched fractions. The existence of an additional positive effect due to the subsequent enzymatic step depended on the enzyme used. Ultrasound-hemicellulase led to the highest pectin yield (27.1%), extracting the whole pectins of carrot powder. Luo and collaborators [80] extracted tannins from acorns using UAEE, which was shown to be a highly efficient and time-saving extraction method. The optimal extraction conditions were assessed by RSM, which included ultrasonic time (2.51 h), ultrasonic power (97.92 W), cellulose concentration (3.44 g/L), and extraction temperature (38 °C). Under these conditions, the extraction yield was 63.16 ± 0.12%. Furthermore, tannins were found to possess a corrosion inhibition efficiency of 86%.

Due to their higher extraction efficiency, easy handling, low solvent, and energy consumption, EAE and MAE have been recognized as promising techniques in the extraction of phytochemicals. Microwave-assisted enzymatic extraction (MAEE) results from the combination of these two techniques, which can disrupt cell wall structure and increase the permeability of cell membranes or walls so that the target compounds within the matrix cell can be transferred more easily into the solvent [76]. Görgüç and collaborators [81] compared the effects of MAE and MAEE (by alcalase) techniques for the recovery of plant-based protein and antioxidant compounds (polyphenols) from sesame bran wastes using RSM. The process conditions, enzyme concentration (0.12–2.40 AU/100 g), temperature (25–55 °C), and time (10–120 min), were found to have significant effects on protein yield, total phenolic content, and antioxidant capacity values. The optimal conditions for MAE were 51 °C and 29 min, while for MAEE were 1.94 AU/100 g enzyme concentration, 49 °C, and 98 min. The highest protein yield and TPC were obtained with MAEE (94.1%, 8.20 mg GAE/g), followed by MAE (61.6%, 4.90 mg GAE/g), and alkaline extraction, the control method in protein extraction studies (24.5%, 3.45 mg GAE/g).

Ultrasonic microwave-assisted extraction (UMAE) results of the combination of UAE and MAE. UMAE benefits from high efficiency, short extraction time, and high extraction yield. Ultrasound can enhance the penetration of the solvent into the sample matrix,

promotes the solvation of soluble compounds, and increases the contact surface area by disrupting cell walls and enhancing mass transfer. The microwave heats the sample quickly, increasing the mass transfer rate and solubility of solute, accelerating the desorption of the interesting compounds from the sample matrix, and resulting in the enhancement of extraction efficiency [76]. Sun et al. [82] used an UMAE procedure for the extraction of polysaccharides from the fruit of *Camptotheca acuminata*. Under the optimal conditions obtained by RSM (liquid: raw material ratio of 30 mL/g, microwave irradiation time of 20 min, microwave irradiation power of 570 W, and fixed ultrasonic power of 50 W), yields of 6.81 ± 0.04% were achieved. Furthermore, UMAE produced higher yields in a shorter time (20 min) than conventional hot water extraction (120 min). Yang et al. [83] extracted pectin from potato pulp by ultrasound-microwave-assisted HCl extraction. The optimal extraction conditions were evaluated by RSM and comprised 93 °C, pH 2.0, and 50 min. Under these conditions, the yield obtained was 22.86 ± 1.29%, which showed that UMAE was an effective way to increase the extraction efficiency of potato pectin extraction.

2.7. Pressurized Liquid Extraction

Pressurized liquid extraction (PLE) is an automated, economic, and time-efficient green extraction technique. During PLE, the use of elevated pressures allows the liquid solvents to be used at temperatures above their atmospheric boiling point, resulting in enhanced solubility and diffusion rate of the targeted compounds. During this process, the surface tension and solvent viscosity decrease, resulting in a drained matrix after the extraction. The extraction performance can be improved by choosing the adequate solvent, temperature, pressure, and time of extraction. PLE is often combined with chromatographic and spectral techniques such as ultra-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) to identify, characterize, and quantify the compounds of interest as well as to evaluate the antioxidant activity of the extracts [84].

PLE has been used to study the potential of agri-food wastes in potential future industrial applications (Table 1). Toledo-Merma et al. [64] recovered phenolic compounds such as punicalagin and ellagic acid from pomegranate by-products, peel and carpellary membranes. The authors found that PLE at low pressures and temperatures was able to recover phenolic compounds from pomegranate residues. The best phenolic compounds extraction conditions were obtained using pressurized ethanol at 60 °C and 40 bar (37% on d.b.) and 60 °C and 80 bar (45% on d.b.). PLE at 40 bar presented a significantly higher content of α, β punicalagin, and ellagic acid (48 ± 2, 146 ± 11, and 25.6 ± 0.3 mg/100 g, respectively) than at 80 bar (40 ± 2, 126 ± 4, and 22.7 ± 0.3 mg/100 g). Lasta and collaborators [65] evaluated the potential of PLE to enhance the process yield, TPC and antioxidant activity (DPPH, ABTS and FRAP methods) of the recovered extracts from beetroot leaves and stems. In general, PLE was more efficient in comparison to Soxhlet, UAE and SFE methods for the recovery of valuable compounds, as the PLE provided higher global extraction yield, phenolic content and antioxidant activity, especially for beetroot leaves. The chemical composition was analyzed by liquid chromatography, which allowed the identification of 33 and 24 phenolic compounds from beetroot leaves and stems, respectively. Furthermore, ferulic acid, vitexin and sinapaldehyde, were the most abundant phenolic compounds obtained from the extracts. Allcca-Alca et al. [66] studied the recovery of polyphenols from the skin and seeds of a Peruvian pisco-industry grape-pomace waste by hot PLE with water-ethanol solvent mixtures. Compared to the conventional extraction (1 atm, acetone 60%, 30 °C), hot PLE at ethanol 60% and 160 °C recovered ~2.5 and ~1.5 more polyphenols from skins and seeds, respectively. These hot PLE extracts also showed higher antioxidant activity than conventional extracts, with 45% and 58% higher DPPH for skin and seeds, respectively. Additionally, at ethanol 60% and 160 °C, the seed fraction extracts contained ~6 times more total polyphenol and presented ~5 times more antioxidant activity than the skin extract. The highest recovery of flavanols (163.61 μg/g dw from seeds and 10.37 μg/g dw from skins) was seen at ethanol 20% and 160 °C. The recovery of phenolic acids was maximized at ethanol 60% and 160 °C with 45.34 μg/g dw from seeds and

6.93 µg/g dw from skins. Flavonols were only recovered from the skin, maximized (17.53 µg/g dw) at ethanol 20% and 160 °C.

3. Valorisation of Agri-Food Waste

Agri-food wastes comprise peels, seeds, shells, pomace, and leaves, valuable due to their content of bioactive compounds (phenols, peptides, carotenoids, anthocyanins), secondary metabolites (polyphenols, alkaloids, terpenes), polysaccharides, enzymes, lipids, minerals, vitamins, and amino acids, among others (Figure 3) [85,86]. Their composition offers a wide spectrum of applications to provide green and more healthy alternatives as renewable natural resources and substrates in biomedicine, cosmetic or food industries [87,88].

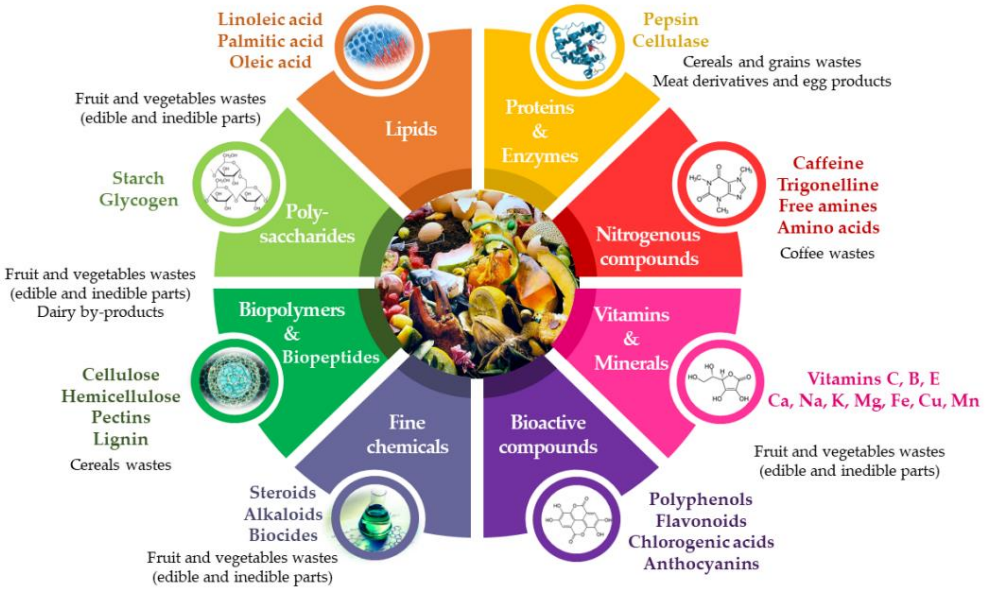

Figure 3. Overview of the different types of bioactive compounds that can be obtained from food wastes.

3.1. As a Source of Biomass for Bioethanol and Biodiesel Production

Fossil fuels are extensively used as major sources of energy. However, fossil fuels are finite reserves which hinder economic development and most of these reserves are geographically located in politically unstable countries. Moreover, its application is considered unsustainable since petroleum-based fuels are non-biodegradable and the emissions of obnoxious gases during their burning result in pollution problems. As an alternative to fossil fuels, the demand for biofuels has been rapidly growing worldwide. Biofuels have been proven to be more beneficial for biotransformation applications as they minimize the emission of harmful gases given their environmentally friendly and cost-effective nature [85,89,90]. Agri-food wastes are a recognized nonedible source of lipids, carbohydrates, amino acids, and phosphates, which can be used as resources to produce biofuels like biodiesel and bioethanol since they contain significant amounts of lipids and carbohydrates [85,86].

Biodiesel is a fatty acid methyl ester produced from the direct or indirect transesterification of fats, greases and plant oils, including soybean, rapeseed, and canola [91]. Biodiesel production consists of a base-catalysed transesterification process operated under low temperatures and pressures, in which a triglyceride (fat or oil) reacts with an alcohol in the presence of a catalyst, usually a strong alkaline like sodium hydroxide, to produce biodiesel and crude glycerol [92]. Biodiesel was designed to replace fossil diesel. This biofuel can be equally used for diesel engine transportation and agricultural vehicles, including tractors, military vehicles, heavy construction vehicles and mining machinery, as well as for heating homes, offices, and industries in addition to electricity generation. The fuel properties of biodiesel depend on the type of feedstock, alcohol and catalyst employed.

Nevertheless, its energy content is approximately 45 MJ/kg, which is nearly 90% of the heating value of diesel derived from fossil reserves [90,91,93].

Bioethanol production from food wastes can be used as an alternative to gasoline to power automobile engines and mitigate its adverse environmental impacts and negative health effects [90]. This biofuel can be produced by microbial fermentation of carbohydrates of grain starches (wheat, corn, and barley) and sugar-containing or sucrose-containing crops (sweet sorghum, sugarcane, and sugar beet). The leftovers of several agri-food wastes have considerable amounts of carbohydrates (>50%) that can be converted into bioethanol [94]. For instance, cereal crops, such as wheat, barley, rice, and corn can be converted into bioethanol, with corresponding yields of 291, 310, 280, and 290 L/ton. Fruit residues of apple (stem and pomace), orange (bagasse), grape (stem and pomace), and date (cull, pits, and press-cake), provide yields of 241, 171, and 68 L/ton. Vegetables including carrot (pomace), sugar beet (bagasse and molasses), sugarcane (bagasse and molasses), and potato (peel, cull, and green immature potatoes) can provide yields of 228, ~260–375, 260–280, and 211 L/ton, respectively for bioethanol production [85,86,90,94]. Several microorganisms and enzymes are known to hydrolyse agri-food waste into carbohydrates, lipids, amino acids, and phosphates [85,93]. Sugary food wastes can be directly converted into bioethanol using suitable microbial strains such as *Saccharomyces cerevisiae*, but for starchy food waste, a hydrolysis step is required to obtain sugars (glucose) before ethanolic fermentation [90,94]. Starch hydrolysis can be made through thermostable alpha-amylases, which promote a simultaneous gelatinization and liquefaction process [94]. For instance, Arapoglou et al. 2010 [95] converted potato peel waste to bioethanol using environmentally friendly biocatalytic methods. In this study, the potato peel waste was hydrolysed with various enzymes and fermented by *Saccharomyces cerevisiae* var. *bayanus*, in which the enzymatic hydrolysis of a combination of three enzymes released 18.5 g/L reducing sugar and produced 7.6 g/L of ethanol after fermentation [95].

Currently, several technologies are available to produce biodiesel and bioethanol on an industrial scale [93]. However, their industrial production from agri-food waste is largely dependent on the availability of food waste, which is highly fluctuant and depends on the season and geographical locations [90,94]. Other challenges include the efficiency of the hydrolysis process, the content of lipids and carbohydrates obtained from food waste, and the efficiency of fermentation and transesterification methods [85]. Besides, the burning of biofuels results in the emission of increased atmospheric pollutants such as carbon dioxide (CO_2) and other oxides of nitrogen and sulfur, in addition to some harmful oxidative hydrocarbon compounds and volatile organic compounds (VOCs). Therefore, low-cost, greener and advanced technologies are needed to produce fuels from agri-food wastes on an industrial scale [85,90].

3.2. As a Source of Valuable Chemicals

Agri-food wastes are a rich source of organic compounds, namely carbohydrates, biophenols, proteins, lipids, essential oils, and pigments [96]. Given the high content of these wastes in carbohydrates, proteins, fibres, minerals, and vitamins, they can be used as a culture medium for microorganism growth [97]. The production of valuable chemicals such as aroma compounds, pigments, and essential oils from a microbial source using agri-food wastes is a commercially and economically promising alternative to chemical synthesis methods, due to the relative simplicity in controlling growth conditions and the source availability [98]. These valuable chemicals can also be extracted directly from a natural matrix or derived from biotechnological processes using enzymes (reviewed by [99]) [100].

3.2.1. Pigments

Commonly used natural food pigments comprise anthocyanins, carotenoids, betalains, and chlorophylls, among others, which are easily found in agri-food wastes and represent a rich source of bioactive compounds [98]. Besides possessing beneficial health effects,

pigments can also be used as a source of natural colourants [101]. Tiwari et al. [101] extracted carotenoids from carrot pomace using flaxseed oil in combination with ultrasonication, which provided carotenoid and β-carotene yields of 82.66 µg/g and 78.37 µg/g, respectively. Goula et al. [102] used sunflower oil and soy oil as green solvents for the ultrasound-assisted extraction of carotenoids from pomegranate peels, recovering 0.6134 and 0.6715 mg carotenoids/100 g of dry peels in sunflower oil and soy oil, respectively. Ultrasound-assisted extraction was also applied for the extraction of betalains from red beetroot waste, at 44 kHz for 30 min at 30 °C [103]. Supercritical CO_2 has also been applied for the extraction of pigments in agri-food waste. This method was used in the extraction of β-carotene from tomato by-products (28.4 to 58.8%) [104] and pumpkin seeds (18.50 mg/100 g sample) [105]. Ghada et al. [106] extracted anthocyanins from the epicarp of passion fruit, a by-product of the manufacture of juices from exotic fruits. The extraction of these pigments was made through HPLC-DAD-ESI/MS and yielded contents of 3.4 mg/g dried epicarp and 9 mg/g extract. In a different study, Mitrea et al. [107] investigated the physicochemical features of poly(vinyl alcohol) biofilms, enriched with eco-friendly polyols and carotenoid-rich extracts. The authors used tomato peels and seeds as substrates for pigment synthesis of β-carotene by ultrasound extraction. This approach proposed the replacement of synthetic colourants with natural pigments derived from agro-industrial by-products, and the use of a combination of biodegradable polymers and polyols, as a solution for packaging applications in the bioplastic industry. Mourtzinos et al. [108] proposed a green extraction method for the extraction of polyphenols and pigments from onion wastes. Quercetin and quercetin derivatives were the main polyphenols found, while cyanidin 3-O-glucoside was the main colourant. Moreover, cyanidin 3-O-glucoside extracted from the onion leaves proved to be a stable natural colorant in a yoghurt matrix.

Agri-food wastes can be used as a substrate for microbial pigment synthesis, which is beneficial in terms of nontoxic and superior quality, biodegradability, compatibility with the environment, and independence from season variation [97,109]. Moreover, microorganisms, such as bacteria and fungi, produce a variety of different pigments [97]. Kantifedaki et al. [110] used the fungal strains *Monascus purpureus* and *Penicillium purpurogenum* to extract pigments from orange peels through solid-state and submerged fermentation. In this study, yields of 9 AU/g, 0.95 AU/mL, and 0.58 AU/mL were accomplished for yellow, orange, and red pigments, respectively. Sugarcane bagasse is also an interesting source of pigments. Majumdar et al. [111] used it as a resource for the growth of the bacteria *Planococcus* sp. to obtain β-carotene (47.13 ± 1.9 mg/g) through solid-state fermentation. In another study, Terán Hilares et al. [112] used sugarcane bagasse hydrolysate for red pigments (18.71 AU) by the fungi *Monascus ruber*. However, this approach still faces a few challenges concerning the safety of the final products of pigment production, due to mycotoxin co-produced by some microorganisms, as well as the stability of the natural pigments [97].

3.2.2. Essential Oils

Essential oils are obtained by steam distillation of flowers, roots, leaves, and fruit peels of aromatic plants [87]. During cultivation techniques and preparation of plants for the market (processing and packaging phases), the fruits and vegetables that do not meet the required standards are discarded, namely food that has been minimally altered (cut, peeled, shredded), and flowered portions. These processes result in a large quantity of residual aromatic biomass still rich in secondary metabolites. Hence, these wastes can be recovered as by-products, to be used in health-promoting ingredients for additives and nutraceuticals [88]. Da Silva and Jorge [113] extracted oils from seeds of apple, citron, grape, guava, kumquat, mango, melon, orange, papaya, passion fruit, pumpkin, soursop, strawberry, and tomato through cold extraction with chloroform, methanol, and water. Citrus fruit wastes are an important source of essential oils, such as limonene, geranial, linalool, citronellol, neral, humulene, α-phellandrene, *o*-cimene, among others [53,114,115]. Boluda-Aguilar and López-Gómez [116] extracted the essential oil D-limonene from lemon

peel wastes, which was recovered from steam explosion treatment with yields of 6 L D-limonene/1000 kg fresh lemon citrus peel wastes. The citrus leaves also were characterized as another important source of essential oils [117]. Grape seeds are another source of essential oils, namely linoleic, oleic, palmitic, and α-linoleic acids. For instance, Kovalcik et al. [118] extracted essential oils from grape seeds by n-hexane after enzymatic hydrolysis, which yielded 4.2 g of oil per 100 g of grape seeds. While Manna et al. [15] extracted polyphenols from the skin and seed fraction of the grape pomace through supercritical CO_2.

3.3. As a Source of Bioactive Compounds

Agri-food wastes are important sources of bioactive compounds, such as phenolic compounds, bioactive peptides, carotenoids, and dietary fibres [89]. These compounds can be used as food supplements, as well as additives for the functionalization of materials to be used in biomedicine, cosmetic or food industries [75].

3.3.1. Phenolic Compounds

Phenolic compounds are a ubiquitous family of secondary metabolites synthesized through the shikimic acid and phenylpropanoid pathways [119]. These phytochemicals can be found in most plant tissues, including in several agri-food by-products [120]. Additionally, these compounds present interesting bioactivities, such as antioxidant, antimicrobial, anti-inflammatory, and anti-proliferative activities, making them useful ingredients in several products in the industry [121].

Dietary polyphenols have been shown to play important roles in human health, due to lowered risks of many chronic diseases including cancer, cardiovascular disease, chronic inflammation and many degenerative diseases [121]. Many of these diseases seem to be related to oxidative stress from reactive oxygen and nitrogen species. Polyphenols are the main ones responsible for the total antioxidant activities of food, as they can neutralize free radicals by donating an electron or hydrogen atom. Thus, polyphenols can reduce the rate of oxidation by inhibiting the formation or deactivating the active species and precursors of free radicals. These compounds can also act as direct radical scavengers of the lipid peroxidation chain reactions, by donation of an electron to the free radical, neutralizing the radicals that become less reactive and stopping the chain reactions. Polyphenols are also known as metal chelators of transition metals such as Fe^{2+}, being able to reduce the rate of Fenton reaction, and thus preventing oxidation caused by highly reactive hydroxyl radicals [122].

Grape and wine produce approximately nine million tons of waste per year and have been reported to have a diverse phenolic composition, composed mainly of lignin, tannins, phenolic acids (caffeic, gallic, protocatechuic, 4-hydroxybenzoic, and syringic acids), hydroxytyrosol, flavonoids, and anthocyanins [75]. Silva et al. [123] assessed the chemical composition of grape stems, seeds and skins and were able to identify 24 polyphenols in these by-products, using solid–liquid extraction with water/ethanol (50:50) and HPLC-DAD. Citrus peels, seeds and pulp are the main by-products of the orange and lemon juice industry, with a total of about 15 million tons of waste being originated yearly. These are an important source of hydroxycinnamic acids and flavonoids (mainly hesperidin, naringin, naringenin, and luteolin) [124]. A work done by Barrales et al. [125] allowed the identification of nine phenolic compounds in citrus peels, through HPLC analysis. Other fruit by-products generated by the agri-food industry that contain phenolic compounds include apples, pomegranates, olives, bananas, and pineapples, among others [75]. Likewise, various vegetable by-products, such as onion skin, carrot pomace, potato peels, tomato peels and seeds, and broccoli leaves can be a source of anthocyanins, quercetin, kaempferol, rutin, naringenin, and chlorogenic acids [75]. For instance, Kelebek et al. [126] identified 4 flavonols, 1 flavanone and 7 phenolic acids in tomato pastes, using liquid chromatography coupled to diode array detection and electrospray ionization tandem mass spectrometry (LC-DAD-ESI-MS/MS) methodology. Several phenolic compounds have been found in cereal processing by-products, including corn, wheat, rice, and barley. The

phenolic compounds previously identified in these by-products included hydroxybenzoic, ferulic, sinapic, syringic, *p*-coumaric, protocatechuic, vanillic, chlorogenic, and caffeic acids, as well as vanillin and catechin [127]. Other lignocellulosic agri-food by-products are spent coffee grounds, originating mainly from the soluble coffee industry, which can be a source of chlorogenic and protocatechuic acids [128]. Andrade et al. [129] assessed phenolic compounds from spent coffee grounds, via micro solid phase extraction (μ-SPEed) and ultrahigh-pressure liquid chromatography (UHPLC-PDA), which led to the identification of 7 compounds including 5 chlorogenic acids, caffeic acid and caffeine [129].

3.3.2. Bioactive Peptides

Bioactive peptides are small amino acid chains that can be formed during enzymatic proteolysis of proteins (gastrointestinal digestion, in vitro hydrolysis, using proteolytic enzymes) and during food processing (cooking, fermentation, ripening). These peptides are recognized for having high tissue affinity, specificity, and efficiency in promoting health [130].

Bioactive peptides with 5–16 amino acid residues have been found to possess an antioxidant capacity. Antioxidative peptides from foods are safe and healthy compounds, given their simpler structure compared to enzymatic antioxidants, with low molecular weight, low cost, high activity, easy absorption, and stability. The exact mechanism underlying the antioxidant activity of peptides has not been fully understood yet. Nevertheless, studies suggest that they can act as inhibitors of lipid peroxidation, scavengers of free radicals, and chelators of transition metal ions. The antioxidative properties of bioactive peptides are more related to their composition, structure, peptide sequence, and hydrophobicity, where tyrosine, tryptophane, methionine, lysine, cysteine, and histamine are known to possess antioxidant activity (reviewed by [131]).

Agri-food by-products that are rich in protein can be sources of bioactive peptides. These include pumpkin seeds, rapeseed and soybean meal, sesame and rice bran, coconut pulp, tomato and apple pomace, and potato and orange peels [132]. An investigation conducted by Yu et al. [133] permitted the isolation of 4 peptide fractions from soybean meal hydrolysate, using an ultrafiltration membrane green method, that presented antioxidant activity.

3.3.3. Carotenoids

Carotenoids are a family of pigmented compounds synthesized by plants and microorganisms [134], divided into carotenoids and xanthophylls [135]. Carotenoids are phytochemicals thought to be responsible for the beneficial properties of fruits and vegetables with a well-known role in the prevention of human diseases, including cardiovascular diseases, cancer, and other chronic diseases [134,135]. In addition to their potent antioxidant activity, some carotenoids contribute to dietary vitamin A. Carotenoids such as α- and β-carotene, along with xanthophylls like β-cryptoxanthin are converted to vitamin A [134,135]. Vitamin A is an anti-inflammation vitamin and possesses an important role in the human development and in disease prevention, due to its promoting and regulatory roles in the innate immune system and adaptive immunity. In turn, this vitamin is being able to enhance the organism's immune function and provide an enhanced defense against multiple infectious diseases [134–136]. Vitamin A is crucial for maintaining vision, promoting growth and development, and protecting epithelium and mucus integrity in the body. Moreover, this vitamin has demonstrated a therapeutic effect in the treatment of various infectious diseases (reviewed by [136]). Vitamin A is one of the main dietary deficiencies worldwide in children. Under vitamin A deficiency the epithelial cells shrink. Consequently, the resistance of keratinized epithelial tissues to foreign pathogens decreases, reducing innate immune function, and promoting respiratory tract infections, diarrhoea, and other diseases, particularly in children [134–136]. Hence, the development of functional food using agri-food wastes rich in provitamin A compounds can present an effective strategy to prevent different diseases, particularly in developing countries with malnutrition issues [136].

In the human diet, the major sources of carotenoids are fruit and vegetables. Therefore, they can also be found in several agri-food wastes, making them a potential source of carotenoids. [134]. As a major agri-food residue worldwide, tomato pulp, skins and seeds can be a great source of carotenoids, including high amounts of lycopene and lower amounts of -carotene, α-, β-, γ- and ε-carotenes, phytoene, phytofluene, neurosporene, and lutein [137]. Banana peels, which are a significant by-product of banana fruit processing, have been shown to have carotenoids in their composition, mainly α- and β-carotenes and xanthophylls [138]. Other fruit wastes, such as mango peels, durian, hog plum, peanut butter fruit, and kwai muk by-products have been shown to have high amounts of carotenoids [139]. Additionally, the carrot industry processing wastes (carrot rejects, peels, and pomace) present a significant concentration of carotenoids [140]. The quantification of the total carotenoid content in agri-food by-products can be achieved by spectrophotometric analysis, as described in the work done by Ordóñez-Santos et al. [141], who extracted carotenoids from peach palm fruit through UAE. However, the identification and quantification of specific carotenoids can be attained using chromatographic methods, as demonstrated by Kelebek et al. [126] who used LC-DAD-ESI-MS/MS to identify and quantify eight carotenoids, including four lycopenes, three carotenes, and one luteolin.

3.3.4. Dietary Fibres

Dietary fibres are complex, nonstarch carbohydrates and lignin that cannot be digested by the human organism but contribute to energy production through the metabolites originating from their fermentation by resident bacteria in the colon [142]. Dietary fibres are divided into insoluble and soluble. Water-insoluble fibres include cellulose, hemicellulose, and lignin. Soluble fibres include viscous fibres, such as β-glucans, fructans, pectin, gums, and mucilage [143,144]. Diets rich in fibre, such as cereals, nuts, fruits and vegetables have a positive effect on human health, as their consumption has been related to decreased incidence of several diseases [142,143]. Furthermore, the protective effects of dietary fibre against chronic diseases, including cardiovascular disease, diabetes, metabolic syndrome, obesity, and colorectal cancer have been described [144]. For instance, dietary fibre adds bulk to the diet and adsorbs and sequesters cholesterol. In turn, this helps to decrease hepatic absorption and increases the excretion through bile and faecal lipids and bile acids. Soluble fibres are more easily fermentable by gut bacteria, possessing prebiotic functions. They are also a source of short-chain fatty acids, which are rapidly absorbed from the large intestine and can be oxidized for energy production. Consequently, the absorption of short-chain fatty acids has been shown to decrease cholesterol synthesis in the liver, leading to decreased blood cholesterol and increased sodium and water absorption into the colonic mucosal cells [144,145]. Insoluble fibers decrease transit time and increases fecal bulk, helping to relieve constipation. They also target scarcinogens, mutagens, and toxins for elimination, preventing their absorption and harmful effects to the body [144,146].

Since these carbohydrates are considered an important component in the healthy human diet, they can be used for several applications [138]. Dietary fibres are complex structures present in high concentrations in several fruits and vegetables, as well as in their by-products. Apple pomace is an important source of dietary fibre, with its content being as high as 47.3 g of dietary fibre per 100 g of apple pomace [147]. Moreover, citrus shells and pomace have been shown to present great amounts of dietary fibre [148]. Studies have demonstrated that mango peels have high levels of dietary fibres (literature reports that their contents range from 51.2 to 78.4%) [149]. Pineapple peel and pomace have also been shown to present dietary fibres in their composition (about 45% in pomace and 76 % in peel) [150]. Another fruit by-product that contains high amounts of dietary fibre is banana peels. Approximately 50% of its dry matter is fibre [151]. Some vegetable by-products that are rich in dietary fibres are cabbage leaves, core and outer leaves, artichoke leaves, bracts, stems, and carrot pomace [148]. The extraction of dietary fibres from agri-food by-products can be achieved through several methods (enzymatic, chemical, ultrasound-assisted, wet milling, microbiological, and microwave-assisted methods). However, the combination of

two different extraction methods, such as ultrasound-assisted enzymatic hydrolysis, has been demonstrated to increase the yield of extraction, particularly in the case of soluble dietary fibers [152,153].

4. Potential Applications and Challenges of Agri-Food Wastes Management

Agri-food wastes are highly biodegradable and comprise an important source of bioactive compounds, antibiotics, and pigments, which can be easily managed via their valorisation to value-added products, food additives, therapeutics, or other environmental applications [89,100]. These residues are also important subtracts for phytochemicals including phenolics, carotenoids and tocopherols, which provide antioxidant, therapeutic, and nutritional properties, of great interest to produce functional foods and drugs against acute and chronic diseases [119]. Hence, agri-food wastes represent a promising opportunity for the food, pharmaceutical and cosmetic sectors (Figure 4) [154].

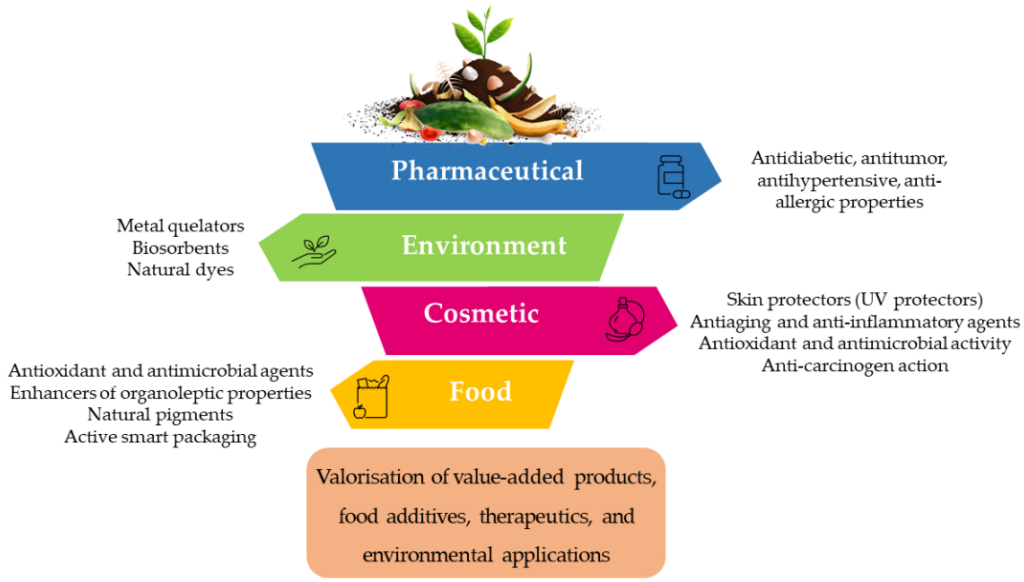

Figure 4. Overview of the main fields of application in which bioactive compounds recovered from food wastes can be applied.

4.1. Food Additives and Nutraceuticals

Aroma compounds can be produced using agri-food wastes as substrates. This approach generates food additives suitable for different industrial applications, which can be labelled as environmentally friendly since it requires fewer solvents for extraction, compared to chemical synthesis methods [100]. In fermentation processes, the catabolism of carbohydrates, proteins, and lipids contributes to the production of the primary metabolites needed to produce aroma compounds [100]. Moreover, the addition of precursors has been shown to generate the production of specific compounds. For instance, the addition of leucine and valine to agri-food waste led to the formation of isoamyl acetate with a strong banana aroma by *Saccharomyces cerevisiae* [155]. In another study, sugarcane bagasse was used to produce the rose-scented volatile compounds 2-phenylethanol and 2-phenethyl acetate upon the addition of L-phenylalanine from the yeasts *K. marxianus* and *Pichia kudriavzevii* [156,157]. After the fermentation processes, bioseparation processes, such as extraction, purification, and recovery of the compound of interest, are employed to obtain the final aroma compound [100]. Furthermore, enzymes, like lipase, protease, glucosidase, and cellulase, are often applied to the substrate during the flavour-production process, where they act as precursors of the aroma compounds [100,158].

Agri-food wastes can be used to extract colourants that can be applied in the food industry as additives and as a source of nutrients [119]. Microbial pigments possess interesting biological properties in addition to food colour, such as antioxidant, antimicrobial,

antimutagenic, anticancer, immunoregulation, anti-inflammatory, antiproliferative, and immunosuppressive activities [97].

4.2. Cosmetic and Pharmaceutical Industries

The interest in natural ingredients, particularly bioactive compounds, has been increasing in the cosmetic and pharmaceutical industries. Agri-food wastes are an important source of nutrients, and bioactive and phytochemical compounds, including organic acids, sugars, and phenolic compounds like flavonoids and anthocyanins. These compounds possess antibacterial, antifungal, anti-inflammatory, and immunomodulatory activities under various conditions and antioxidant potential against free radicals, providing an important source of therapeutic applications [119]. For instance, volatile aroma compounds obtained from microbial processes, such as vanillin, isoamyl acetate, ε-pinene, 6-pentyl-α-pyrone and ethyl acetate, can be applied in the pharmaceutical and cosmetic industries to enhance or modify the original aroma of a product [100]. Moreover, pigments are precursors of vitamins, which help to reduce cancer, chronic diseases, and cataracts and are also used to produce biopharmaceutics and cosmetic products [97,109].

The interest in natural antioxidants has been increasing in the food industry to prevent harmful chemical additives and inhibit oxidation processes in the final product [91]. Cosmetics such as creams, powders, and oils for the skin, are mainly composed of antioxidants due to their properties to reduce cell ageing, by scavenging free radicals and preventing oxidation in body cells. The extraction and application of phenolic compounds from agri-food wastes commonly found in the pulp, seeds, skins, and pomace of vegetables, fruits, and legumes, has shown great interest due to their antioxidant capacity [119]. Phenolic compounds like resveratrol and its derivatives, curcumin, flavan-3-ols and procyanidins, among others, have been incorporated in the formulations of cosmetic products as moisturizers and antioxidants for skincare (as reviewed by [159]), to reduce the use of preservatives, such as parabens and formaldehyde, which seem to harm human metabolism [119,160].

Synthetic antimicrobial compounds are used against microorganisms that cause respiratory diseases, infections, or conditions in humans. However, these synthetic pharmaceutical products contain contraindications and side effects that can lead to the appearance of new health alterations and bacterial resistance. Due to these concerns, medicinal research has focused on bioactive compounds from plants and fruits by-products, as new alternatives to the preparation of drugs and medicines, to be applied in the treatment of acute and chronic diseases. Moreover, these residues, namely peels, seeds, shells, pomace, and leaves, are also rich in phytochemical compounds, which allow an increase in the bioavailability to produce new drugs [119,161]. For instance, citrus fruit by-products are rich in phenolic compounds such as hesperidin, nobiletin, and tangeretin, known for their antiviral, anti-inflammatory, antibacterial, and antifungal properties [119].

Despite the major applications of bioactive compounds from agri-food wastes in food, cosmetic, and pharmaceutical industries, along with the higher demand and consumer acceptance for natural antioxidants, their reduced bioavailability and bioactivity are a few challenges that prevent them to be widely implemented in the industries. Additionally, most of these compounds present poor solubility which hinders their direct incorporation in certain foods and products, as well as the sensitivity to oxygen, light, heat, enzymes, salts, acid or alkaline media, humidity, and pH variations and instability under normal processing and storage conditions. Consequently, this leads to losses in their beneficial effects and activity, limiting their use [91,162]. In recent years, nanotechnological approaches such as encapsulation have been developed to overcome these limitations and improve the stability of bioactive compounds obtained from agri-food wastes during food processing and gastrointestinal digestion [91]. The next section describes how encapsulation strategies can be used to improve bioactive stability and increase biological availability.

4.3. Encapsulation Strategies

Recently, encapsulation techniques have been applied to increase the bioactivity, bioavailability, stability, and solubility of bioactive compounds protecting them against the environmental adverse conditions of food processing. This capability not only allows for increased shelf-life, but also a controlled release of the substances [91,162]. Therefore, these approaches can lead to innovative and functional products, especially in the food and pharmaceutical industries [91,163].

The appropriate encapsulation techniques and wall materials can improve the stability and solubility of bioactive compounds from food by-products. The chosen methodology depends on the bioactive compound entrapment method and its combination with the wall material, since it can be a solution, an emulsion, or dispersion [162]. Edible particles composed of proteins, carbohydrates, lipids, phospholipids, or surfactants, trap the phytochemicals inside capsules through spray drying, freeze-drying, coacervation, crystallization, molecular encapsulation, extrusion, or electrostatic extrusion to increase the efficiency and management of the compounds of interest [91,162]. Common wall materials include hydrocolloids (gum arabic, alginate, chitosan, pectin), carbohydrates (modified starch, maltodextrin, cyclodextrins), cellulose, proteins (casein, whey protein, gelatin, soy protein), and lipids (hydrogenated vegetable oils, phospholipids, mono- and triglycerides) [162,163].

Encapsulation can be used to isolate compounds of interest, as well as for improving their controlled release in possible applications as functional food ingredients, increasing their stability and improving their bioavailability and water solubility (Table 2) [163]. Encapsulated bioactive compounds can be added to food to improve the stability against oxidation and microbial proliferation and for enrichment, fortification, and colouring purposes, in addition to antioxidant, antimicrobial, anti-inflammatory properties, and enzymatic inhibition [162]. Spray-drying, freeze-drying, and coacervation are the most used encapsulation strategies to produce encapsulated bioactive compounds from agri-food wastes [163]. Spray-drying and freeze-drying are widely used in the controlled release of functional compounds and recovery of by-products from agri-food wastes. The coacervation technique provides high loading capacity, low temperature, improved thermal stability, and offers a controlled release of active materials. Moreover, this procedure is simple, uses non-toxic solvents, has low agitation, and does not require any specific equipment [162,163]. Santos et al. [164] extracted anthocyanins from blackberry pomace and encapsulated them by spray-drying them with maltodextrin. The microcapsule developed was analysed regarding its stability under different pH conditions and their influence on colour variation, anthocyanins, antioxidant activity, and kinetic degradation of anthocyanins in 7 days. The study showed that microencapsulation with maltodextrin was efficient in protecting the colour of the samples and reducing the degradation of anthocyanins against increased pH. Aliakbarian et al. [165] aimed to stabilise the phenolic compounds in olive pomace using a microcapsule with maltodextrin produced by spray-drying. The encapsulation efficiency varied between 85–92%, with yields between 65–82%, while the specific total polyphenol content and specific antioxidant activity ranged between 38–52 mg gallic acid/g dw and 230–487 µg Trolox/g dw, respectively. The microcapsules revealed good stability under storage conditions with significant antioxidant activity. In another study, de Araújo and collaborators [166] evaluated the use of maltodextrin and gelatin as wall materials to encapsulate orange peel essential oil. Both microcapsules were produced by freeze-drying and all achieved high yields, stability and preservation of antioxidant and antimicrobial properties.

Table 2. Encapsulated bioactive compounds from agri-food wastes and possible applications in the food industry.

Agri-Food Waste	Encapsulated Bioactive Compound	Wall Materials/Encapsulation Method	Main Findings	Ref.
Encapsulated bioactive compounds from agri-food wastes				
Blackberry pomace	Anthocyanins (Antioxidant activity)	Maltodextrin/Spray-drying	Microencapsulation with maltodextrin was efficient in reducing the degradation of anthocyanins against increased pH, increased stability, protected the colour of the samples, and longer half-life were achieved (2–7 times greater than extracts)	[164]
Espresso spent coffee	Phenolic compounds, caffeine (Antioxidant activity)	Maltodextrin, whey protein isolate, gum Arabic, and inulin (1:1 proportion)/Spray-drying	The encapsulates obtained carried and protected considerable amounts of antioxidants present in the coffee extracts The whey protein isolate was the most efficient material in the maintenance of antioxidant activity	[167]
Olive pomace	Phenolic compounds (Antioxidant activity)	Maltodextrin/Spray-drying	Maltodextrin-based microcapsules showed good stability under storage conditions, high recovery of polyphenols, and remarkable antioxidant activity	[165]
Orange peel oil	Phenolic compounds (Antioxidant and antibacterial activity)	Maltodextrin, gelatin/Freeze-drying	High encapsulation efficiency (75.75%) and encapsulation yield (90.19%). All encapsulated oil samples showed antioxidant and antibacterial properties. The presence of gelatin in the microencapsulated systems positively influenced their properties, especially the thermo-oxidative stability	[166]
Pomegranate seeds	Polyunsaturated fatty acids	Whey protein isolate, gum Arabic/ Complex coacervation	Microcapsules produced with polymer concentration (5%) and wall material: oil ratio of 2.75 showed the highest punicic acid content and oil retention	[168]
Food applications of encapsulated bioactive compounds				
Carrot waste	β-Carotene (Antioxidant activity)	Sodium alginate/Electrostatic extrusion	Yoghurt fortification with concentrations of 2.5 and 5 g/100 g of carrot wastes beads provided a part of the β-carotene recommended daily intake. The microbiological and physicochemical properties of the fortified yoghurts were well-maintained during the storage period. The encapsulated carotenoids improved the antioxidant activity of the yoghurt (4.21 and 9.36 μmol of Trolox equivalents/ 180 g of yoghurt)	[169]

Table 2. *Cont.*

Agri-Food Waste	Encapsulated Bioactive Compound	Wall Materials/Encapsulation Method	Main Findings	Ref.
Cocoa hulls	Phenolic compounds (Antioxidant activity)	Maltodextrin, gum arabic/Spray-drying	The best-performing extract (water/ethanol 50:50), contained 93.3 mg of total polyphenols/g of dry extract. The microencapsulation using an 80:20 ratio of maltodextrins to the dry extract allowed obtaining the most stable powder, with a total polyphenol content unaffected by the baking process, and an antioxidant activity stable up to 90 days	[170]
Grape seeds	Phenolic compounds, carvacrol (Antioxidant and antimicrobial activity)	Chitosan/Ionic gelation	The microcapsules affected the physicochemical properties of the developed films, increasing their thickness, moisture content, and colour parameters when compared with the unmodified chitosan films. The carvacrol microcapsules showed stable antimicrobial and antioxidant activity, protecting the salmon samples from environmental degradation	[171]
Tomato peel	Lycopene (Antioxidant activity)	Whey protein isolate, acacia gum/Complex coacervation and freeze-drying	The sample powder displayed 27.34 ± 0.18 mg lycopene/g dw and antioxidant activity of 2.15 ± 0.02 mMol Trolox/g dw. Retention of 63% in lycopene was found after storage at 4 °C in the dark for 14 days. An inhibitory effect against α-amylase of 79.89 ± 1.74% was identified. The powder was used for functionalization in dressing samples, showing an increased antioxidant activity	[172]

Legend: dw—dry weight.

The encapsulation of bioactive compounds from agri-food wastes has been recognized as a promising alternative to the production of food with added natural ingredients [163]. Given the superb health benefits of bioactive compounds, their encapsulation allows the production of functional foods, through the controlled delivery of phytochemicals to the target site [91,162]. Šeregelj et al. [169] extracted carotenoids from carrot waste to be encapsulated with sodium alginate by electrostatic extrusion. The authors then added the beads created to the yoghurt for its fortification, which were able to maintain the microbiological and physicochemical properties of the yoghurt while providing a part of the recommended daily intake of β-carotene. The encapsulated carotenoids also improved the antioxidant activity of the yoghurt ranging from 4.21 to 9.36 μmol of Trolox equivalents/180 g of yoghurt. Gheonea et al. [172] developed lycopene microparticles from tomato peels with whey protein isolate and acacia gum by complex coacervation and freeze-drying. The encapsulation efficiency for lycopene was 83.6%, while the powder displayed 27.34 mg lycopene/g dw and antioxidant activity of 2.15 mMol Trolox/g dw. The powder was used for functionalization in dressing samples, where it showed an increased (23–33%) antioxidant activity. In the food industry, the packaging process produces large amounts of waste and residues. Encapsulated bioactive compounds, such as phenolics, anthocyanins, and essential oils have shown promising results in the development of active packaging. These compounds allow adequate protection against oxidative and microbial deterioration when incorporated in biodegradable films of food products [162,163]. Alves and collaborators [171] prepared chitosan films with grape seed extract and carvacrol microcapsules to extend the life of refrigerated salmon. The salmon packed into carvacrol microcapsules provided better protection against microorganisms after 7 days of storage, increasing the shelf-life of refrigerated salmon. Despite the potential of encapsulation strategies, their industrial application still shows some limitations. The extraction yield of bioactive compounds from agri-food wastes is generally low, which hinders the application of encapsulated bioactive compounds. Additionally, various procedures are required to purify the compound of interest and the technology is costly [162].

5. Conclusions

The destructive effects caused by climate variations on agriculture along with the continued exponential growth of the human population have led to a decrease in agri-food production and, consequently, to nutrient loss. Hence, the valorisation of agri-food wastes has become an urgent need. Agri-food wastes mostly from plant origin represent significant societal challenges, from a high impact on the world economy to environmental concerns related to pollution and the emission of GHG. This review focused on wastes obtained from fruits and vegetables, which comprise edible and inedible parts rich in valuable compounds, such as bioactive compounds, secondary metabolites, polysaccharides, enzymes, lipids, minerals, vitamins, and amino acids, among others, known to possess antioxidant, anti-microbial or anti-inflammatory activities. Their composition offers a wide spectrum of applications to produce value-added and marketable products, as renewable natural resources and more healthy alternatives to synthetic and potentially harmful products in the food, health, pharmaceutical, cosmetic, and environmental fields. To make such a strategy economically viable, innovative techniques based on the principles of green analytical chemistry—SFE, SCWE, PEF, UAE, MAE, EAE, and PLE—have proved to be efficient in the extraction and recovery of valuable compounds. Moreover, the combination of these techniques has been demonstrated to be beneficial and a promising strategy for fast and efficient extraction, providing higher extraction yields and better products. Nevertheless, most of these compounds present poor solubility and sensitivity to normal processing and storage conditions, which hinders their direct incorporation into certain foods and products. In recent years, encapsulation strategies have been applied to increase the bioactivity, bioavailability, stability, and solubility of bioactive compounds from agri-food wastes, protecting them against the environmental adverse conditions of food processing and increasing their shelf-life. These approaches can lead to innovative and

functional products, especially in the food and pharmaceutical industries, through the isolation of compounds of interest and the controlled release of substances. However, the industrial application of encapsulation strategies still faces some limitations, since the extraction yield of bioactive compounds from agri-food wastes is generally low, in addition to the technological costs required in the procedures for the purification of the compounds of interest. Hopefully, the continuous research and improvement in innovative extraction techniques will allow for the development of proper strategies for the sustainable development and utilization of valuable compounds from agri-food wastes in real life and their application in various industries.

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