Recovery of Bioactive from Food Waste

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Biowastes, especially food wastes, contain bioactive compounds that are suitable for producing functional foods, supplements, and nutricosmetics. Vegetables and fruits have primary metabolites (e.g., amino acids, lipids, dietary fibers, cellulose, hemicellulose, lignin, and fatty acids), and secondary metabolites (e.g., flavonoids, phenols, alkaloids, glucosinolates, carotenoids, and terpenes). The extraction of bioactive compounds from biowastes depends on the source, functionality, chemical properties, and end-use. Various temperatures, pH values, electromagnetic waves, and extraction techniques are used (e.g., supercritical fluid, subcritical water, ultrasonic wave, microwave, and pulsed electric field).

Keywords: food waste ; recycling ; Food chemistry ; nutracosmetic ; nutraceutic

1. Primary Metabolites Recovered from Agri-Food

1.1. Proteins

Proteins are macronutrients with a central role in human nutrition. They are formed by amino acid units. Proteins from food waste sources are used as value-added ingredients and/or products, including human foods and animal feed. The high-value products are used as foaming, thickeners, and gel stabilizers [1], and the low-value products are used as fish and animal feed [2]. Food waste protein sources can be classified into animal and plant sources. Plant byproducts used as protein sources include oat, rice, wheat bran protein [3][4][5], and defatted meals from the oil industry. Wheat bran contains between 13% to 18% of proteins [3], the defatted meals obtained from the oil industry (e.g., canola, sunflower, palm, rapeseed, and peanuts) have between 15% to 50%, and soybean curd residue contain 27% protein [6]. Sugar beet and mushroom flakes are used as a feed ingredient source since they contain 40% essential amino acids [7]. Finally, food waste proteins obtained by animals (e.g., meat, fishmeal, bone meal, yogurt, and cheese) are considered good-quality protein sources that are of high biological value [8]. Some extraction methods were used to isolate protein, including enzyme-assisted, cavitation-assisted, ultrasound-assisted, hydrodynamic cavitation, microwave-assisted, supercritical, liquid biphasic flotation, and hybrid extractions [9]. In enzyme-assisted extraction, the protein recovery depends on the enzyme ratio, substrate characteristics, extraction time, and pH [10]. Protein isolates were generally obtained by defatted pressed legume cakes and animal sources via precipitation at the isoelectric point [11]. Hydrolysate from protein isolates is also used [12][13][14] since it produces higher solubility products and smaller peptides [14][15]. Cavitation-assisted extraction is used in large-scale protein extraction. Low frequency (20 to 100 kHz), temperature, sonication power, and treatment time affect the protein yield [16]. Ultrasound-assisted extraction is coupled with enzyme-assisted or microwave-assisted extraction technologies to improve protein extraction efficiency [12]. Microwave-assisted extraction of proteins can depend on nonuniform temperature distribution and closed- or open-type vessel systems [17][18]. It enhances the proteins' functional properties (e.g., water absorption, emulsifying, foam activity, and foam stability indexes) [12]. Supercritical extraction of proteins depends on temperature [19] and solvent concentration [20]. Chemical dehydration and/or evaporation are required to remove moisture. These procedures can affect protein purity [12]. Liquid biphasic flotation has high separation efficiency and determines the minimal protein loss [21][22]. Cell receptors, drug residues in food, and wastewater treatments were extracted using this technology [23].

Possible Uses of the Recovered Proteins

The food waste proteins can be utilized in feed supplements to enhance the food products' functional properties [24]. Milk protein and whey protein are used to enrich ice cream [25], improve the mixture's viscosity, and decelerate the melting time [26]. The animal proteins can be used as a foaming agent with recycled PET aggregates to produce cementitious concrete composites [27]. Whey protein can be employed to produce plastic films for food packaging materials [28].

1.2. Pectins

Pectins are polysaccharides that are formed by d-galacturonic acid, d-galactose, or l-arabinose units, and are found in the cell walls of plant tissue [29]. The degree of pectin esterification affects the pectins' functional properties as a thickening and gelling agent. Conventional (e.g., extraction with the mineral acids) and innovative techniques (e.g., ultrasound- or enzyme-assisted microwave- extraction) were used to extract them from biowaste. Traditionally, pectin is extracted via continuous stirring with water that is acidified (e.g., in nitric, 0.05–2M sulfuric, phosphoric, hydrochloric, or acetic acid) for 1 h under controlled temperature (80 and 100 °C) [30]. The maximum pectin yield is obtained using hydrochloric acid at pH 2.0 [31]. Innovative extraction methods help in the extraction of pectins, disrupting the cell membrane's structure by electromagnetic or sound waves and facilitating the contact between solvent and bioactive molecules. Among the most innovative approaches, ultrasound-assisted technology improves (+20%) the pectins' molecular weight and extraction yield compared to the traditional method under the same temperature, pH, and time conditions [32]. The microwave-assisted extraction of pectins is affected by the weight of the biomaterial, the power of the wave, the time of extraction, and the pH. For example, the optimum processing conditions to extract pectins from lime bagasse are a sample weight of 6 g, a wave power of 400 W, a time of extraction of 500 s, and a pH of 1 [33]. Finally, enzymes can enhance the extraction process by hydrolyzing the plant cell wall matrix (enzyme-assisted extraction). The enzymes used to extract pectins are protease, cellulase, alcalase, hemicellulase, xylase, α-amylase, polygalacturonase, b-glucosidase, endopolygalacturonase, neutrase, and pectinesterase [34].

Possible Uses of the Recovered Pectins

The food industry employs pectins as emulsifiers, stabilizers, thickeners, and gelling agents.

The pharmaceutical industry uses them as drug-controlled release matrices and prebiotic, hypoglycemic, hypocholesterolemic, and metal-binding agents [35].

Finally, the functionalization of pectins with nanomaterials and phenolics can produce active packaging films with antimicrobial properties [36].

1.3. Omega-3 from Fish Waste

Omega-3 fatty acids (e.g., eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA)) have the first double bond on carbon 3, counting from the terminal carbon. Fish are a good source of omega-3. They accumulate them from plankton, algae, and prey fish [37]. The omega-3 fatty acids regulate cell membranes' architecture and permeability, produce energy and eicosanoids, and modulate the human body's pulmonary, cardiovascular, immune, reproductive, and endocrine systems [37]. Their potential health benefits include the prevention of cancer, cardiovascular disease (CVD), Alzheimer's disease, depression, rheumatoid arthritis, attention deficit hyperactivity disorder (ADHD), dry eyes, and macular degeneration [38]. Numerous apparatuses and techniques were proposed to extract omega-3 fatty acids from fishes. Traditional extraction techniques use organic solvents (e.g., hexane, methanol, petroleum ether, and chloroform), which cannot be employed on an industry scale [39][40]. Soxhlet extractor, ultrasounds, or microwave-assisted extractions decrease the time and use of solvents [41]. On an industrial scale, fish oil extraction is achieved through a wet-reduction or wet-rendering process [42]. Supercritical fluid extraction (SFC) [43] solves the problem of n-hexane use for extraction in traditional extraction methods, uses low temperature to reduce the oxidation of polyunsaturated fatty acids, decreases residual solvent contaminants (polychlorinated biphenyls and heavy metals), does not modify the biomass, and allows other bioactive molecules to recover. The ethanol used as a co-solvent is much more food-compatible than hexane [44]. The disadvantage of this method is the extract's smell due to volatile compounds. Encapsulation decreases this effect and improves the extracts' palatability [45].

Possible Uses of the Recovered Omega-3

Omega-3 fatty acids are employed in supplements and fortified food and feed [46]. Moreover, they can be used to ameliorate cutaneous abnormalities and maintain skin homeostasis in cosmetics [47].

2. Secondary Metabolites Recovered from Agri-Food

2.1. Phenolic Compounds from Biowaste

Phenolic compounds (PC) are secondary metabolites characterized by an aromatic ring with more hydroxyl substituents. Plants produce them in response to environmental stimuli. They vary greatly by type and level based on target species and biotic and abiotic factors. Biocontrol agents, such as *Trichoderma* strains, can interfere with phenolic production in

plants [48][49][50][51][52]. Phenolic compounds have antioxidant and antimicrobial properties [53] and beneficial potential for human health, such as antitumor, anti-obesity, anti-inflammatory, and ultraviolet (UV) radiation protective activities. Isolated and dosed phenolic compounds [54] were used to prepare food supplements [55][56], nutricosmetic formulations [57][58][59], and nutraceutical food [60][61]. The process used to recover PC from biowaste consists of a pre-treatment (thermal treatment of the sample followed by electro-osmotic dewatering, foam mat, pulverization, or micro-filtration) [62], extraction (obtained by maceration, or assisted by microwave, ultrasound, supercritical fluid, pressurized liquid, pulsed electric field, high voltage electrical discharges, or multi-technique) [63], concentration (obtained by distillation, or steam distillation), and purification (obtained by microfiltrations, ultrafiltration, resins, or chromatography) [63].

Anthocyanins from Agri-Food Waste

Anthocyanins are water-soluble antioxidants pigments that belong to the flavonoid family. They have a C6-C3-C6 skeleton [64]. The flavylium cation gives the red color to the anthocyanins [65]. Temperature, enzymes, pH, light, metallic ions, oxygen, sulfites, and interaction with flavonoids, phenolics, ascorbic acid, and sugars can affect anthocyanins' integrity according to their chemical structure and food concentration. Anthocyanins subjected to high temperatures and prolonged heating oxidize. Moreover, the deglycosylation process, nucleophilic attack of water, cleavage, and polymerization determine the structure breakdown. These structural changes decrease anthocyanin content in the final product [64]. Sulfite adds colorless adducts interrupting the conjugated π-electron system [65]. Anthocyanin association reactions (e.g., self-association between anthocyanins via hydrophobic interactions, co-pigmentation between anthocyanins and other phenols through van der Waals interactions between the planar polarizable nuclei or binding the sugar and the flavylium nucleus covalently, and metal complexing between anthocyanins and with metals, via their o-hydroxy groups) defend the flavylium chromophore from the water's nucleophilic bond, enhancing their stability and color [66]. The anthocyanins can manage and/or prevent chronic degenerative diseases, including cancers, cardiovascular diseases, type 2 diabetes mellitus, dyslipidemias, and neurodegenerative diseases [67]. Several biochemical parameters involved in the inflammatory responses (e.g., interleukins (ILs), tumor necrosis factor-alpha (TNF-α), nuclear factor-kappa B (NF-kB), and cyclooxygenase 2), which are able to improve malonaldehyde and reactive oxygen species, and to reduce the activity/expression of antioxidant enzymes (e.g., catalase and superoxide dismutase), have been related to the prevention or development of these diseases [68][69][70]. The anthocyanins improve the sensitivity, secretion, and lipid profile of insulin by inhibiting the limiting enzyme of cholesterol synthesis (3-hydroxy-3-methylglutaryl-coenzyme A) or adipose triglyceride lipase engaged in triglyceride breakdown in diabetics and prediabetic subjects [71][72]. Moreover, anthocyanins regulate the expression of adipokines, thereby enabling the avoidance of insulin resistance and the progression of type 2 diabetes mellitus [73]. Anthocyanins can reduce triglycerides, cholesterol, LDL-cholesterol, and inflammatory biomarkers regulating the expression/activity of pro-inflammatory cytokines (e.g., IL-6, TNF-α, and IL-1A), pro-inflammatory enzymes (e.g., COX-2), and NF-κB signaling pathways, decreasing the production of pro-inflammatory molecules (e.g., C-reactive protein), and improving SOD and proliferator-activated receptor-γ expression [74][75]. Finally, anthocyanin supplementation can enhance cardiovascular function [76][77], regulate gut microbiota composition [78][79], improve exercise recovery effectiveness [80], decrease ulcerative colitis symptoms [81], reduce ocular fatigue [82], and promote healthy facial skin conditions [80]. Some innovative processes are proposed for the extraction of anthocyanin from biowastes, such as the extractions assisted by a pulsed electric field, microwave, and ultrasound technologies. The enhancement of the extraction of anthocyanin, helped by pulsed electric field technology, is related to permanent (irreversible) or temporary (reversible) pores in the cell membranes, which facilitate the anthocyanins release into the medium [64]. The target compounds' extraction does not constantly improve with the increasing of the electric field strength, specific energy input, treatment time, temperature, and pulse number [83][84]. For example, studies on blueberry extraction showed an improvement (+75%) in the extraction of anthocyanin when the specific energy input was increased [85]. Instead, studies showed that improving electric field intensity and specific energy input does not increase the anthocyanins content extracted from the sweet cherry byproduct [86]. The application of high intensity can lead to anthocyanin degradation. instead, the application of low/moderate-intensity enhanced anthocyanin recovery without anthocyanin's degradation/modification. The pulsed electric field technology allows the selective extraction of the single anthocyanin classes [87]. The pulsed electric field technology improves the monoglucoside anthocyanin extraction compared to acylated glucoside anthocyanins from grape pomace [88] and the extraction of cyanidin, delphinidin, and petunidin glycosides from blueberry byproducts [89]. The anthocyanin recovery from grape pomace enhances when the microwave and irradiation time improve [90], and longer irradiation times enhance anthocyanin recovery from wine lees [90], sour cherry pomace [91], and saffron floral bio-residues [92]. Nevertheless, the excessive intensification of MW extraction process parameters can decrease the extraction of anthocyanin from biowaste due to their degradation (anthocyanins are thermolabile compounds) [93]. Thus, it is recommended to use extraction temperatures below 60 °C to minimize the anthocyanin's losses. The extraction time also impacts MW extraction. The excess time causes the degradation of anthocyanin due to higher exposure to microwave powers and high temperatures [94]. The high microwave power determines internal overheating, leading to carbonization and isomerization, and/or degradation of molecules [95].

According to some authors, anthocyanins' thermal degradation determines the loss of sugar moieties, the formation of a carbinol pseudo base, and chalcone by hydrolysis of the remaining sugar moiety and cut between C2 and C3 [84]. According to others, the degradation of anthocyanin is due to decomposition reactions of water molecules and the production of reactive oxygen species [96]. Studies on anthocyanin recovery from eggplant peel and fig peel showed that the recovery of anthocyanin decreased when the microwave powers and irradiation times improved [97]. Studies on grape pomace [87], blackcurrant bagasse [98], blueberry peel [99], black rice bran [100], and corn husk [101] showed that the extraction of anthocyanin decreased when the irradiation times were long. Finally, anthocyanins' structures impact their recovery from bio matrices. For example, anthocyanin analogs that are unsubstituted at C3 of the C-ring are more stable to MW treatment than other anthocyanins [102], as well as acylated anthocyanins than non-acylated ones [103]. Another technique used to improve the extraction of anthocyanin is ultrasound. Acoustic cavitation can determine the thermal and chemical degradations of the anthocyanins since the acoustic cavitation phenomenon can determine thermal stress and free radical formation [104]. Long processing times can cause severe degradations in anthocyanins [105]. For example, the anthocyanin extracted from black chokeberry wastes degrades when an ultrasound water bath (30.8 kHz) for 60 min at 70 °C, with a nominal ultrasound power of 100 W, and 50% ethanol in water are used [106]. Using enzymes (pectinase compound and pectinase) combined with ultrasound can improve the extraction technique's performance [105].

Possible Uses of the Recovered Phenolics

Phenolics are used as functional food additives. Their antimicrobial and antioxidant activities enhance the shelf-life of foodstuffs [107]. Anthocyanins can be employed as a coloring additive (EFSA code E163).

Phenolic compounds can be employed as supplement ingredients [108], pharmaceutical [107], and cosmeceutical agents [59].

Caffeic and gallic acids can be used in chitosan-based biofilms to inhibit the growth of *Bacillus subtilis* and *Staphylococcus aureus* and enhance the film's oxygen and vapor permeability [109].

Tannins could develop protein-based biofilms since they can interact with proteins through non-covalent bonds and hydrogen bonding [110].

Finally, the phenolics might be helpful to the textile industry as natural dyes with antimicrobial properties [110].

2.2. Carotenoids from Agri-Food Waste

Carotenoids are fat-soluble pigments with excellent antioxidant activity (e.g., singlet oxygen-quenching capacity and free radical activity) [111][112]. The two main classes of carotenoids are xanthophylls (yellow color) that contain oxygen and carotenes (orange color) that consist of linear hydrocarbons, which can cyclize at both ends of the molecule [59]. Carotene supplementation is related to the inhibition of atherosclerosis-related multiple sclerosis [113], cardiovascular diseases [114], macular degeneration [115], and degenerative diseases [116]. Carotenes are most bioavailable in their natural trans-form [117][118]. Light, metals, heat, and pro-oxidants can isomerize the trans-form into cis-form [119]. The extraction of carotenoids is achieved using organic solvents (e.g., hexane, methanol, acetone, and ethanol or solvent combinations). The supercritical fluid extraction with CO_2 improves the extraction's efficiency, as is also the case for the extraction of carotenoids from carrot peels (+86.1% at 349 bar) [120]. In addition, the microwave [121] and ultrasound increase the carotenoids' recovery [122]. Finally, an extraction method of lycopene and pectin from tomato pomace [123] and pink guava decanter [124], which relied on a simple water-induced hydrocolloidal complexation, was tested. The pH, temperature, solid loading, and stirring affected the complexation of carotenoids and pectin [125].

Possible Use of the Recovered Carotenoids

Carotenoids can be used as food and feed additives. Supplementation of the animal diet with carotenoids improves the nutritional quality of animal products [126].

Moreover, they can be employed as a food colorant to improve food desirability and acceptability [127].

2.3. Essential Oil from Agri-Food Waste

Essential oils are lipophilic substances, including terpenic hydrocarbons (e.g., monoterpenes and sesquiterpenes) and oxygenated derivatives (e.g., aldehydes, phenols, esters, and alcohols) [128]. The traditional methods used to extract the essential oils from plant matrices are liquid–solvent extraction and steam- and hydro-distillation. High temperatures (around 100 °C) can lead to the decomposition of essential oils, and the use of pentane and hexane can determine toxic organic residues. Supercritical fluid extraction (using carbon dioxide) is the most widely applied innovative technological

process. Carbon dioxide is suitable for the extraction of lipophilic compounds since it has a polarity similar to pentane as well as other desirable characteristics such as being non-flammable, non-toxic, available in high purity, and easily removed from the extract, as well as having low temperature (32 °C) and critical pressure (74 bar). Favorable extraction conditions are related to the solubility of essential oil compounds in supercritical CO_2 [113], the plant's pretreatment (used to facilitate the extraction by improving solvent contact and breaking cells), and online fractionation (used to achieve the separation of the essential oil from cuticular waxes) [129]. Essential oils from *Lamiaceae* (e.g., oregano, thyme, rosemary, sage, basil, and marjoram) and citrus (e.g., lemon, orange, etc.) family plants [129] were extracted using this technology. The fractionization, which can be achieved in the mode of multi-step fractionation (successive steps in which CO_2 density improves) or online fractionation (using a cascade decompression system consisting of two or three separators in series), can improve the selectivity of supercritical fluid extraction. This strategy can be employed when molecules with different solubilities in supercritical CO_2 are extracted from the same matrix [129].

Possible Uses of the Recovered Essential Oils

Essential oils can be used as flavors, fragrances [130], and antimicrobial agents [131] in food and cosmetic products.

Moreover, they can be incorporated into food packaging to improve the UV barrier property and surface hydrophobicity to protect foods against oxidation and microbial injuries [132].

Finally, they can be used as green pesticides in agriculture [133].

2.4. Organosulfur Compounds from Agri-Food Waste

Organosulfur compounds are thiols with sulfur in their structure. They include glucosinolates (isothiocyanates), allyl sulfides, indoles, and sulforaphane [134]. Glucosinolates are thioglucosides that are produced in cruciferous vegetables of the Brassica family (e.g., broccoli, radish, cabbage, and cauliflower). They are responsible for the plant's defense against insects and pathogens. Moreover, they have some health-beneficial properties, including antioxidant (e.g., scavenge free radicals) [135], anti-inflammatory (e.g., activating detoxification enzymes, suppression of interferon regulatory factor 3, and macrophage migration inhibitory factor) [136][137], cardioprotective (e.g., they reduce low-density lipoproteins), neuroprotective, and anti-carcinogenic attributes (detoxifying carcinogens and toxicants) [138][139]. The conventional technologies used to extract them employ boiling water or aqueous organic solvent extraction [140] in a single extraction process or repetitive cycles [141]. Among the non-conventional extraction technologies, ultrasound techniques (20 kHz and 400 W) were used to improve the extraction's yield of the sinigrin from Indian mustard (+70.67% recovery than conventional techniques), microwave techniques were sued to decrease the extraction time of sulforaphane from cabbages (30 min for conventional extraction to 1.5–3 min for microwave-assisted technique) [142][143], and supercritical technology was used to extract allyl isothiocyanates from wasabi (SC-CO_2, 35 °C, and 25 Mpa) [144].

Allyl sulfides, produced by alliaceous vegetables (e.g., shallots, chives, leeks, and scallions), originate from S-alk(en)yl-L-cysteine sulfoxides and γ-glutamyl-S-alk(en)yl-L-cysteines. Among these, diallyl sulfides are responsible for the pungent aroma [145][146]. Organosulfur compounds have some health-beneficial properties, including anticancer (e.g., they promote apoptosis, xenobiotic-metabolizing enzyme production, and carcinogen detoxification, in addition to the production of the enzymes that are responsible for DNA repair, are engaged in cell cycle arrest, and decrease the metabolism of nitrosamines and hydrocarbons), antioxidant (e.g., they scavenge free radicals), and antimicrobial properties [147][148]. They are thermally unstable and are lost during sterilization, pasteurization, drying, and cooking [149]. High-temperature processing decreases their bioavailability [150][151], while high-pressure processing reduces their anticancer, antimicrobial, and antioxidative properties, decreases their enzyme alliinase activity [152] and improves their enzyme alliinase activity [153][154]. Moreover, freeze-drying and infrared-drying technologies [155] and microwave-assisted and pressurized liquid extractions have negative thermal effects. Therefore, only supercritical fluid extraction is suitable for efficient extraction among the new technologies used to replace conventional ones [156].

Possible Uses of the Recovered Organosulfur Compounds

Organosulfur compounds can be used as supplements, food additives (because of their aromatic taste and smell) [157], and biopesticides [158].

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