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Review

A Systematic Review on Waste as Sustainable Feedstock for Bioactive Molecules—Extraction as Isolation Technology

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Abstract: In today's linear economy, waste streams, environmental pollution, and social–economic differences are increasing with population growth. The need to develop towards a circular economy is obvious, especially since waste streams are composed of valuable compounds. Waste is a heterogeneous and complex matrix, the selective isolation of, for example, polyphenolic compounds, is challenging due to its energy efficiency and at least partially its selectivity. Extraction is handled as an emerging technology in biorefinery approaches. Conventional solid liquid extraction with organic solvents is hazardous and environmentally unfriendly. New extraction methods and green solvents open a wider scope of applications. This research focuses on the question of whether these methods and solvents are suitable to replace their organic counterparts and on the definition of parameters to optimize the processes. This review deals with the process development of agro-food industrial waste streams for biorefineries. It gives a short overview of the classification of waste streams and focuses on the extraction methods and important process parameters for the isolation of secondary metabolites.

Keywords: waste; residue; bioactive molecules; extraction; secondary metabolite; biomass; circular economy; biorefinery

Citation: Drescher, A.; Kienberger, M. A Systematic Review on Waste as Sustainable Feedstock for Bioactive Molecules—Extraction as Isolation Technology. *Processes* **2022**, *10*, 1668. https://doi.org/10.3390/pr10081668

Academic Editors: Ofelia Anjos, Eugenia Gallardo and Raquel P. F. Guiné

Received: 29 July 2022
Accepted: 15 August 2022
Published: 22 August 2022

1. Introduction

A growing world population and technological innovations have led to a growing economy with higher consumption of energy and materials, leading to an increased utilization of fossil raw materials. According to the United Nations, 10 billion people will needed to be supported by earth by 2050 [1]. The consequences of this population growth are spreading social economic wealth and increased resource and agricultural demands, finally resulting in higher waste streams, increasing air and sea pollution, the acceleration of greenhouse gas emissions, facilitated climate change, and worldwide social–economic conflicts [2,3]. The higher demand of energy and resources will lead to a municipal waste production of up to 3.4 billion tons, with a composition of up to 58% of food and green waste and 28% paper and cardboard [3].

To overcome the growing gap between environmental sustainability and economic growth and to achieve the transition from the current linear economy to a circular economy, the development of a circular bio-based industry, where biomass and its byproducts can be processed into viable bioenergy, biochemicals, biomaterials, feed, and food, is mandatory. This includes the development of specially designed products and complete recyclability. In the best case, these products are already manufactured from recycled sustainable resources [2,4]. At present, an average of about 8.6% of the global material input, which is about 8.4 Gt, is recycled [5]. In 2019 and 2020, about 9.7% and 9.1%, respectively, were kept in the cycle. To keep global warming under 2 °C and to reach the goal of sustainability in 2050, the circularity gap has to be closed by 2030 by an additional 8.6% recycling rate [6]. Besides the development of new technologies and pathways, the redesign of traditional linear economies for better recyclability and further substitution of fossil-based block chemicals with sustainable counterparts is an approach to start with. Every step further will affect the material balance, leading to the desired recycle ratio of 1. Figure 1 shows a schematic representation of a complete transformation to a circular economy.

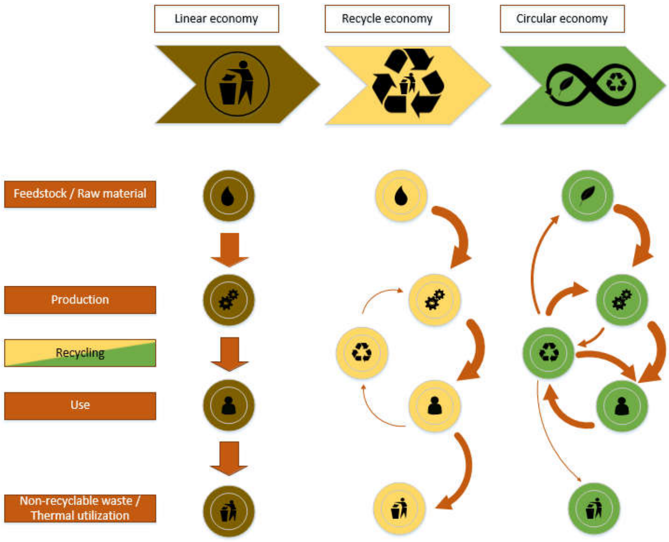

Figure 1. Linear, recycle and circular economy concept.

One tool within this transformation from a linear to a circular, and from a fossil based into a bio-based economy, is biorefineries. Here, renewable feedstock is processed to produce energy and marketable products [7]. In general, all streams generated during downstream processing are converted to marketable intermediates or products. Further, green engineering and green chemistry principles need to be integrated into the overall concept [8]. The most promising feedstocks for biorefineries are waste streams and residues from agricultural production. Approximately 30–40% of the primary agricultural biomass is residues after/during production. Due to their high uniformity, wastes from the bio-based industries are of special interest. An important factor for a positive evaluation is that these feedstocks do not accelerate deforestation or lead to a conflict in land use with food production [9], but the competition with relatively cheap fossil raw materials and the unique and complex chemical structure of biomass requires more effort to receive certain products [10]. In the first approach, biorefineries use agricultural products, mainly edible biomass resources, for the conversion into energy and materials. The competition with food supply, rising basic food prices, as well as deforestation of woodland for agricultural supply and its devastation of formerly fertile land with mono-cultural plantations, paid its tribute in social acceptance of the products all over the world [7,11,12]. Biorefineries based on second and third biomass generations, which do not compete with land use or food production, are now state of the art. The raw materials consist of residues from different agricultural, municipal, and industrial sources as well as algae [12,13]. To help stakeholders and industries classify biorefineries and to develop business opportunities, the IEA Biorefinery Task 42 developed a classification guideline that summarizes a wide range of feedstocks, processes, intermediates, and potential products for biorefinery valorization pathways. The utilization of renewable resources depends on highly interconnected pillars, namely environmental, social, and economic factors. For a long-term business opportunity, we must look at all three pillars. Every product must be environmentally friendly, socially accepted, and economically reasonable to be successful in the long term. Consequently, there needs to be a classification of how the feedstock is cultivated, harvested, and produced to be able to classify it as green or not.

The review is divided into two parts, first, the feedstock classification is discussed, and second, the unit operations for the isolation of bioactive molecules from residual feedstocks, especially extraction methods, are summarized. The majority of the studies for extraction are in a time frame of about 5 years (2016–2021). As feedstocks, agro-industrial food residues were selected according to their homogeneity compared to post-consumer residues.

2. Feedstock Classification

The present review focuses on the utilization of agro-industrial wastes that contain bioactive molecules. Figure 2 gives an overview of the two major categories, namely lignocellulosic residues (LR) and organic residues (OR).

Lignocellulosic residues are mostly leftovers of agricultural use, especially grains and cereal plants, such as grain, corn, or rice, but also leaves, stalks, shells, and different grasses. Residues from logging are counted in this class as well. The main characteristics of these plants are high cellulose, hemicellulose, and lignin content [14–18]. The origins of LR are:

- Agricultural cultivation;
- Residues from nature and landscape management;
- Forest-based management;
- Industrial/municipal residues.

Other organic residues are mostly high in protein and could be a good source of feed and food additives [14–18]. These organic wastes are sourced from:

- Animal waste and manure;
- Processed food waste;
- Industrial/municipal residues;
- Aquatic residues.

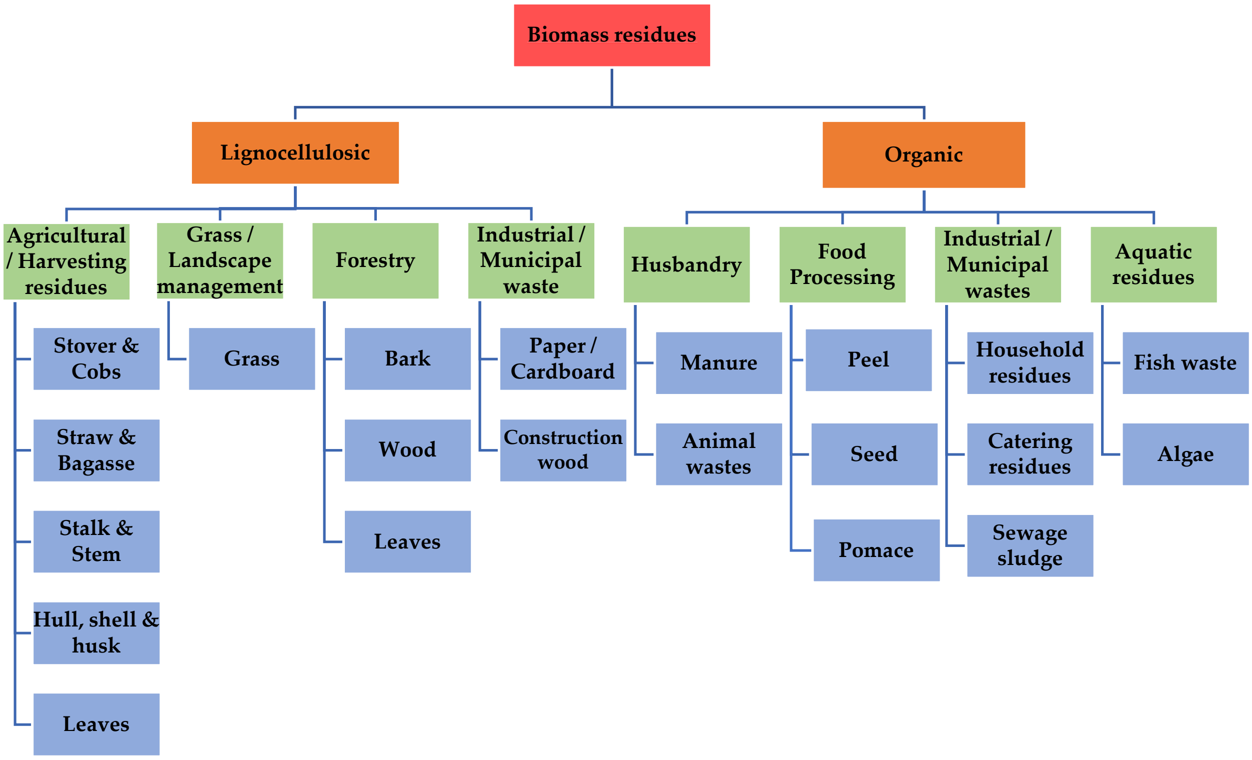

Figure 2. Residue origin and classification.

2.1. Lignocellulosic Residues

Lignocellulosic biomass is the most available raw material on earth and can be converted to bioenergy in the form of fuel, heat, and power, and additionally, to various chemicals. The lignocellulosic material consists, beside the main components cellulose, hemicellulose, and lignin, of other organic and inorganic compounds such as proteins, ash, pectin, and secondary metabolites. In total, about 181.5 billion tons of lignocellulosic biomass are produced per year. Besides the approximately 7 billion tons that are used as fodder, about 4.6 billion tons of crop residues are produced, of which only about 25% are intensively used [19].

Based on its source, lignocellulosic residues are divided into four classes:

- Lignocellulosic wastes as agricultural residues. Leftovers of harvesting, as well as secondary residues, which are produced as byproducts from downstream processing of, e.g., maize, sugar cane, wheat, sunflowers, rice, olives, and others such as straw, olive pits, nutshells, leaves, peels, seeds, pomace, or cobs, which also count in this group [14–18].
- Table 1 summarizes the biomass composition of the lignocellulosic biomasses and residues thereof [20–24]. About 44% of the globally produced primary residues can be assigned to wheat and rice residues, which are left on fields. The practice of harvesting primary crop residues could contribute to the reduction of greenhouse gas emissions by hindering the natural degradation of the crops on the field. Nevertheless, these emissions are not counted towards the GHG emissions, because they are kept within the soil–plant–atmosphere continuum. To avoid soil degradation, keeping the nutrient balance as well as the protective behavior of the residues on the soil surface, only a defined percentage of the leftovers can be harvested for further processing [23]. The major problem for producers is the seasonal availability of agricultural residues.

Table 1. Overview of lignocellulosic biomass composition and residues.

Species	Cellulose %	Hemicellulose %	Lignin %	Residue Production [10^6 t/a]	Ref.
Corn stover	37.5	22.4	17.6	1016.7	[20–23]
Corn cobs	45.0	35.0	15.0		
Cotton seed hair	80–95	5–20	0.0	109.5	[21,23]
Wheat straw	38.2, 29–35	21.2	23.4	1069.7	[21,23]
Bagasse	38.2	27.1	20.2	92.0	[20,21]
Sugar cane	25.0	17.0	12.0	563.1	[21–23]
Rice straw	32.0	24.0	13–18	1118.5	[20–23]
Sunflower stalk	31.0	15.6	29.2	44.7	[21,23]
(Cashew) nut shells	25–30	25–30	30–40	4.44	[21,24]

- Residues from nature and landscape management. Worldwide, grasslands account for the largest ecosystems on land, with an area of about 52.5 million km² [25]. Data for the amount of grass from landscape management are hardly available. During the "Forschungsforum" in Austria, innovative technologies for green biorefineries were discussed, and the annual availability of grass from grassland in the very small country of Austria was evaluated to be 750,000 tons of dry matter. This number shows the high potential of grass as feedstock for future biorefineries [26].
- Industrial residues. Sawdust, bark, and spent or black liquor are byproducts of wood processing. Around 53% of the primary biomass from forestry industries consists of residues such as roots, stumps, and bark. Only 47% is stem, which is the main raw material for wood processing. Data from 2020 expel wood residues of about 227 mio m³. According to technical, environmental, and economic restrictions, not all of the

residues can be utilized for further production. In the EU, it is estimated that around 79% of the primary residues, including bark, can be used for the production of bioenergy and biochemicals. This percentage should be transferable to any other wooden-based economy in the world [27]. Sawmill byproducts, bark, and black liquor are mostly burned for power generation. The potential of sawmill byproducts and bark for the isolation of bioactive molecules and the recovery of carbohydrates or organic acid is high [28].

- Lignocellulosic industrial and municipal residues. Mostly paper, cardboard, and wood waste from packaging, construction, and demolition wood is meant here [27]. The paper and cardboard residues are mostly recycled in paper mills, the European Union reports a recycling rate of 73.9% [29]. When the fibers are no longer valid for recycling, the paper is burned to produce energy in the mill. For the valorization of bioactive molecules, this type of lignocellulosic material does not play any role.

2.2. Organic Waste

Organic wastes (OW) contain a wide range of bioactive molecules. In particular, the content of polyphenolic compounds and their utilization in high-value products, such as cosmetics or pharmaceuticals, are very attractive. Around 40% of global waste is classified as organic waste [2]. Organic waste is categorized into animal wastes, like tallow or manure, agro-industrial food wastes, from processing of agricultural feedstocks (industrial wastes) or post-consumer wastes (municipal waste), oil residues, such as used cooking oils, and aquatic biomass, such as algae or seaweed [30]. One of the most commonly used industrial wastes is pomace. Pomace is a residue from paste processing and consists mainly of skin, pulp, and seeds in different content ratios. This feedstock is mostly separated from the juice by evaporation or pressing. Pomace can be produced out of fruits, vegetables, berries, and, depending on the used feedstock, also stems, e.g., grapes, can be part of the pomace. Pomace can be used as fodder, utilized as compost, sent to distilleries to produce alcohol, or can just be discarded [31–34].

Animal wastes, such as tallow, fats, and manure, are inevitably produced. An increase in livestock husbandry production leads to an increase in manure. About 1.4 billion tons of manure are produced in the EU and 1.1 billion in the US every year by cattle, pigs, poultry, and other livestock [35,36]. About 10% of global direct greenhouse gas emissions are linked to the direct application of manure on farmland.

Food waste (FW) from the food processing industry is a highly homogeneous fraction, whereas FW from the consumer side is highly heterogeneous. The composition of FW varies, according to Battista et al., up to 80–90% volatile solids, 10–40% lipids, 5–10% proteins, and starch 10–60% are present [37]. Therefore, food waste from industry is easier to use as fodder and as feedstock in biorefineries [38]. Municipal food waste (MFW) is categorized into four types: [39]

- Fruits and vegetables;
- Starchy foodstuff;
- Meat, fish and byproducts;
- Others, like dairy products, sweets and nonedible products;
- To improve the waste management also the discarding causes of MFWs has to be noted [39];
- Nonedible food: out of date, not consumed full meals, excessive portion sizes, or not processed feedstock;
- Overproduction of food: in restaurants and other services;
- Waste, based on hygienic, quality, and storage standards;
- Aquatic biomass residues consist of seaweed, algae, and fish residues. Fish residues such as skin, heads, frames, and viscera account for 60% of the global fish production, which was 175 million tons in 2017 [40]. Fish waste is a source of many bioactive molecules and peptides, collagen, gelatin, oil, and pigments [41]. The lower amount

of nutrient requirements of algae compared to plants, and the fact that it has not to be cultivated on farmland is beneficial for its footprint. Nevertheless, around 70% of algal biomass remains as waste after the extraction of lipids for food and biodiesel production [42,43].

3. Bioactive Compounds

The utilization of food and agricultural residues (FAR) is a need in a future bioeconomy. At present, high amounts of FAR are discarded unused, with the side effect that dumped and not treated residues cause high uncontrolled greenhouse gas emissions. Technologically, several processes can transform and upcycle FAR into valuable products. FAR consist of complex carbohydrates, cellulose, hemicellulose, lignin, and also ash, as well as proteins and secondary metabolites. To find new or additional valorization paths, the plant constituents, and, especially, the metabolites need to be characterized in more detail. FAR are mostly rich in bioactive molecules, such as polysaccharides, proteins, phenolic compounds, such as flavonoids, carotenoids, and lipids. They can be used as food additives and health-prompting agents according to their biological activity and their ability to modulate metabolic processes. Most bioactive compounds are produced as antioxidizants to promote plant health. The effect of reducing oxidative stress can also be used for humans [44].

The appearance of bioactive molecules in plants varies throughout the day as well as seasonal cycles and the conditions, e.g., temperature, cultivation technique, and soil quality [45,46]. The main challenge is the isolation and separation of the low-concentrated, highly valuable bioactive molecules from the plant matrix.

While primary metabolites, such as fatty acids and different polysaccharides, are important for plant growth by acting as energy storage or building blocks for hormones, secondary metabolites, which are the main focus, are not mandatory for the plant's survival, but they strengthen the plant against environmental influences. This group of bioactive molecules includes a wide range of polyphenolic compounds, such as terpenoids, phytochemicals, vitamins, and alkaloids, and are produced as byproducts from the syntheses of primary metabolites or for special purposes [47]. Most phytochemicals act as antioxidants and offer potential cancer-inhibitor properties. A good example of polyphenols are carotenoids, which cannot be synthesized by the human body but provide high antioxidant activity and are significantly related to the reduction of incidences of cardiovascular disease [48,49].

The knowledge about waste streams and the composition thereof is just the first part of the story. The second part is to evaluate if the valuable compounds can be isolated from these complex process streams.

4. Isolation

The isolation of bioactive molecules from waste streams is complex. The main topics that need to be considered when working with industrial and agricultural waste streams are:

- Unsteady chemical composition of biomass across the seasons;
- Varying supply masses, and;
- Low concentration of targeted compounds.

To tackle these topics, a combination of different unit operations for pretreatment and isolation is needed. Here, mechanical, mass transfer, chemical, and biochemical unit operations are used. Which unit operation is used for the isolation of a single or a group of bioactive molecules highly depends on the physical and the chemical properties thereof [50].

The present section will review the unit operations used in the last few years to develop new valorization routes to isolate bioactive molecules from various feedstocks.

4.1. Pre–Treatment

To be able to extract (bioactive) molecules from waste streams an effective pretreatment is needed. Methods that are used for this purpose are electric-assisted or biologic-assisted pretreatments like high voltage electric discharge (HVED), hydrolysis, or fermentation.

4.1.1. Electric Discharge Extraction

HVED and other electrical-assisted extraction methods, such as pulsed electric discharge extraction (PEF) or ohmic heating extraction (OHM), damage the cell wall and fragment the cell. It is an effective pretreatment of feedstocks for the valorization of wet plant tissues [51]. Depending on the parameters, energy input, pH-value, gap distance of the electrodes, and electrical field intensity, HVED is also able to degrade the targeted compounds. A range of 2–3 mm and a voltage of 13 kV showed the best results for the performed extraction [52,53]. Pataro et al. investigated the pretreatment of wet tomato tissue with PEF followed by an extraction with acetone and ethyl lactate. Ethyl lactate turned out to be a good solvent after moderate intensity of 5 kV/cm and 5 kJ/kg for the recovery of carotenoids [51]. When Zhang et al. compared HVED, PEF, and UAE, the main outcome was that UAE led to the highest extraction efficiency [54].

The solvent composition is the most important factor for the pretreatment with electric discharges, as the solvent has to be conductive. Therefore, water is the most suitable solvent for PEF pretreatment [55]. For the verification of the PEF pretreatment, experiments with control groups without PEF were conducted, and PEF showed a 55.8% higher extraction efficiency for flavan–3–ols, a 64.34% higher content of flavonoids, a 68.39% higher concentration of phenolic acids, and a 61.20% higher content of total free phenolics. A coupled pretreatment, including MAE or UAE with PEF, is suggested to achieve higher yields [56].

4.1.2. Hydrolysis & Fermentation

The majority of lignocellulosic residues are currently used as an energy source to produce heat and power by burning or are converted into bioethanol or biogas [57].

When dealing with lignocellulosic feedstocks, lignin needs to be removed before the cellulose and hemicellulose can be degraded to sugars that can be further utilized. Lignin removal can either be performed chemically or biochemically. The research focus lies on chemical conversion routes, but biochemical degradation is less harmful to other substances present in lignocellulosic waste streams than chemical treatment [58]. The degradation of lignin, cellulose, and hemicellulose by enzymes and bacteria leads to the formation of low molecular weight components and bioactive molecules either by the degradation process itself, or as secondary metabolites of the enzyme or bacteria activity [59].

The major unit operations for the valorization of protein-rich lignocellulosic materials are hydrolysis, fermentation, and anaerobic digestion. The lignocellulosic materials are hence the basis for biofuel, especially for bioethanol production [60].

For the release of bioactive compounds, which are embedded to cell wall matrices or to cell cytoplasm with polymers, enzymatic hydrolysis can be an effective pretreatment step. Hydrolysis is a part of enzyme-assisted extraction [61]. Enzymatic hydrolysis is an especially important tool for bioactive peptides because the bioactive parts of the protein have to be removed from the parental part without losing its bioactivity [62]. Other polyphenolic compounds, e.g., procyannidin, can be released into aqueous solutions after the depolymerization processes of pectin [61]. The hydrolysis of animal and plant material is therefore crucial for the release of some bioactive compounds which would be inactive and inaccessible in their natural form.

Although fermentation mainly focuses on the degradation of lignocellulosic compounds, there are ambitions to use it as a pretreatment step for the valorization of bioactive molecules. Besides the before mentioned EAE, solid state fermentation (SSF) is the second investigated natural-driven pretreatment step prior to extraction. Results show that the extraction efficiency increase is highly affected by the strain, feedstock, and used extraction technology. While Gassara et al. reported an improvement in aqueous solid–liquid extraction [63], Kitryte et al. [64] and Fernandes et al. [58] observed a decrease in the extraction efficiency of polyphenolic compounds when the feed was hydrolyzed or fermented prior to extraction. Sepulveda et al. showed that the concentration of flavonoids and phenolic compounds increased during the first 12–54 h. Afterwards, the concentrations decreased, a reason therefore could be the demand of the microorganisms. After the available sugars were fermented, the microorganisms started to degrade other available compounds, such as phenolic compounds [65]. To be able to ferment the biomass, it is important to eliminate or significantly reduce inhibiting components. Citrus peels for, example, were first hydro-distillated to ensure that bacteria-inhibiting properties of essential oils were removed. The essential oil was then collected as a valuable byproduct [66]. El Kantar et al. investigated the simultaneous extraction of polyphenolic compounds and fermented sugars by using HVED and EAE. They concluded that the simultaneous use of both technologies hinders the efficiency of the extraction process. A HVED treatment turned out to be beneficial for further EAE extraction processing [52].

4.2. Extraction

Solid/liquid extraction is the unit operation of choice when bioactive molecules are to be isolated from a solid waste stream. Certain pretreatment steps, such as washing, drying, and milling, are needed to ensure a high surface area and good penetration of the liquid phase. As part of the sustainable development goals, the EU green deal and its national counterparts, industry, and academic institutions are encouraged research on and to substitute conventional, toxic environmental hazardous solvents with sustainable, harmless solvents, such as water, ethanol, ethyl lactate, and the like. Solvents such as water, ethanol, ethyl lactate, or supercritical CO_2 can be used for the isolation of bioactive molecules. Due to these solvents, further utilization of the isolated molecules in the food or pharma industry can be done without any limitations. Conventional solvents such as hexane, isopropanol, or methanol lead to limitations in the utilization of the extracted products [67]. Therefore, postprocessing of compounds extracted with conventional solvents is needed. Besides the well-known solid–liquid extraction techniques (SLE) maceration, soxhlet-extraction (SE), hydro-(HD) and steam-distillation (SD), in the last decades' new SLE techniques, such as systems using new (green) solvents, e.g., deep eutectic solvent (NADES) or aqueous two-phase extraction (ATPE), ultrasound-assisted (UAE), microwave-assisted (MAE), agitated bed extraction (ABE), pressurized sub- (PLE), and supercritical extraction (SFE) with water, ethanol, or CO_2, steam explosion extraction (SE), EAE, pulsed electrical discharge extraction (PEF) and HVED, including ohmic heating, have been developed and can be seen in the Appendix A in Tables A1 and A2. All these techniques offer beneficial phase transport and higher contact areas and, therefore, increase the yield, lower the extraction time, and decrease the solvent consumption. The main parameters for the extraction are, besides the solvent itself, the extraction time, the pretreatment, the pH-value, the solid/liquid ratio, and the temperature [68].

SE and SLE are often used as reference processes to evaluate new processes or solvents, such as UAE and MAE, or by using acidified liquids, deep eutectic solvents, and oils. A lot of research is compressed into solid liquid, microwave, and ultrasound-assisted extraction methods. For next generational production plants, it has become important to exchange their fossil-based processes and solvents for renewable ones. MAE, UAE, and electric-assisted extraction ensure an increased permeability of the solvent by disrupting the cells [56,69].

Table A1 summarizes research that was performed for SLE and modified SLE processes, such as UAE and MAE, and more evolved ones, such as SFE or PLE. The process parameters and corresponding results can be seen in Table A2 in Appendix A.

Besides the extraction process itself, solvents are crucial for the selection and the environmental footprint of an extraction process. The development and utilization of green solvents in extraction is under investigation. The water solubility and the recovery or back extraction step are two main points.

The substitution of hexane is of importance for different industries. Ozturk et al., for example, investigated the limonene extraction from orange peel. Besides supercritical CO_2, cyclopentyl–methyl–ether was mentioned as a green alternative [70]. Methanol and ethanol are powerful solvents for the extraction of polar polyphenolic compounds [56,64,71–74]. Zivkovic et al. reported a beneficial extraction behavior with water and an ethanol concentration of up to 40% for polyphenolic compounds. Higher alcohol concentrations led to decreasing extraction efficiencies [75]. The use of sunflower and olive oil was shown to be highly effective in combination with UAE to extract lycopene, also in comparison with conventional organic solvents [31,76–78]. The results showed that, besides the extraction time, the ultrasonic intensity had the highest influence [77,79]. Aqueous two-phase extraction is based two phases that mainly consist of water. The phase composition mainly uses alcohols + salts or ionic liquids + salts [68,80,81]. In pure aqueous systems, ultrasound enhances the extraction for sugars but not for phenolic compounds. In systems with ethanol, the extraction yield was significantly higher for both substance groups, while in both systems with conventional SLE, the extraction yield remained the same. In further investigation and comparison with enhanced ATPE systems, phenolic compounds were enriched in the ethanol-rich top phase, while sugars were enriched in the salt-rich bottom phase. Additionally, lignans were selectively extracted to the top phase, while carbohydrates and proteins remained in the bottom phase [80,81]. The extraction capacity of the ethanol-rich phase can be adjusted by the amount of salt in the second phase. A high amount of salt led to a worse affinity of polyphenols for the ethanol rich phase. Up to 4 wt% ionic liquids were added to the salt-rich phase, which led to an increase of the extraction efficiency from 59.65 to 97.12% [68]. Natural deep eutectic solvents are handled as a green alternative for a lot of compounds. The extraction efficiency, however, depends on the viscosity and, hence, on the water content, which can be adjusted by changing either the molar ration of the NADES or the substances forming the NADES [74,82–86]. It is reported that the water content undergoes a maximum with respect to the extraction efficiency for the extraction of caffeoylmalic acid, psoralic acid–glucoside, rutin, psoralen, and bergapten with glycerol:xylitol:D–(−)–fructose (3:3:3 mol ratio). A maximum of 20% was reported. In conclusion, this maximum is referred to the lower interaction of the NADES with the target compounds [74,83,86]. The water content and the polarity are connected, while cholinium-based NADES offer a low polarity, which is related to an optimal water content of 50%, organic acid-based NADES, which have a higher polarity, show an optimal water content of 25% [83]. NADES showed significantly higher solubility of compounds than water and superior extraction efficiency compared to conventionally used methanol [86]. Although there is still a lot of research needed, the studies show that there is a wide range of green solvents that can be applied in solid/liquid extraction. By the solvent selection, the pH-value becomes important [76,83,87,88]. It is well known that the pH-value is a parameter that highly influences extraction behavior, for example, phenolic compounds are more likely to be extracted at pH-values of around 3–4 [80,81].

Besides the solvent and the extraction method, the solid/liquid ratio is the next influencing factor on the extraction yield [75]. The optimum solvent/solid ration depends on the chosen system. While Ran et al. reported a solid/liquid ratio of 1:20 for ATPS as optimal, Xi et al. demonstrated that this was their lower limit using HVED with water. A general trend cannot be formulated. The systems, solvents, and extracted compounds are too different. It can be concluded that the solid/liquid ratio is one of the key factors for the evaluation of extraction systems [32,53,68,70,81,89]. As an example, ethanol showed solid/liquid ratio independent extraction behavior for the total phenolic content from cranberries, whereas the optimal solid/liquid ratio for the extraction with methanol was between 1:90 and 1:120 [90]. This example shows that the solvent as well as the solvent ratio can make a big difference. High solvent to liquid ratios do not automatically result in good extraction behavior. In addition, the environmental impact has to be considered. In many experiments, the ratio of 1:20 to 1:30 was reported to be beneficial. For UAE and MAE, an increased solvent load led to a decreasing extraction efficiency, which is based on the decreasing energy absorption of the solid material with higher solvent loads [32].

Rahimi et al. and Amyrgialaki et al. stated that the extraction time had a significant influence on the yield of polyphenolic compounds [71,76,77]. During the optimization process, new enhanced extraction processes showed that quite a similar yield could be achieved within a much shorter time, meaning that the phase equilibrium is reached faster than compared to conventional solvents in SE [80]. The extraction times are influenced by the pretreatment of the feedstock. Data show that long pretreatment times with sonication or microwaves lead to a decreased phenolic compound release or/and a degradation processes thereof [81,91,92]. The effective area and the diffusion path length depend on the particle size, hence, the optimal sonication time depends on the particle size [85].

The temperature and, in the case of MAE, the power level of the microwaves [71], are important factors for the extraction [32,69,75]. Higher temperatures show higher extraction yield due to increased diffusivity by loosening the cell wall components [93]. A slightly worse performance of MAE compared to UAE is a result of the higher thermal input of MAE, and that above temperatures of 50 to 65 °C degradation of most bioactive molecules occur [68,79,88,93]. By optimizing the temperature profile with continuous adaption of the MAE power level, better MAE performance can be achieved [84]. Extraction, in general, benefits from higher temperatures due to better solubility of phenolic compounds, higher diffusion rates, improved viscosity of the solvent, and decreased surface tension [84], further, the targeted compounds and solvents used distinguish the extraction efficiency [74].

Pressurized subcritical liquid extraction is an efficient extraction method compared to SLE, UAE, and MAE. As with solvents, water is most often used. Extractions with water can reach up to 9 times higher yields compared to MAE and UAE [94]. An even better performance of PLE, up to 13 times, was achieved by adding 30% NADES to water [95]. As an example, phenolic compounds were extracted from kiwi peel and pomace residues and the results showed that the acidity of the solvent has a big influence on the efficiency. In comparison with conventional SLE extraction using ethanol, methanol, or acetone as solvent [96,97], PLE with supercritical water outperformed acetone as the best solvent with a four-times higher extraction efficiency [96].

The main influencing parameters for supercritical CO_2 extraction are the addition of a cosolvent and the pressure. With elevated pressure and temperature, the selectivity of the solvent increases due to changed polarity [33,98]. The extraction of phenolic compounds from orange and blackberry pomace was investigated, and the main outcomes of the studies were that with increasing temperature, acidity, and the addition of water or ethanol as a cosolvent, the extraction efficiency increases [33,99–101]. Nevertheless, it was discovered that the antioxidative capacity, contrary to the higher phenolic content, decreased with higher pressure. Further, the pretreatment influenced the extraction yield. Compared to dried, the fermented pomace contained simpler molecules with better water solubility and better results for the antioxidative activity. By using more polar cosolvents, a two-times higher yield of total phenolic compounds was achieved [98]. Compared to PLE and SE for black chokeberry, SFE showed a significantly lower efficiency in the extraction of total phenolics. Through the addition of more polar cosolvents, the efficiency of SFE was significantly increased, but was still lower compared to PLE [33,98]. However, SFE is considered the most selective extraction method for monomeric anthocyanins from juçara residues compared to SE, UAE, PLE, and ABE [99]. Further, SFE needed 78% less extraction time and 10 times less EtOH for the extraction of phenolic compounds from orange pomace, which makes it the more sustainable and economical process [98].

Table A2 summarizes feedstocks, applicable processes, and solvents for the isolation of bioactive molecules as well as achieved yields of bioactive molecules, which are polyphenolic compounds, such as carotenoids or flavonoids. The research of the last couple of years has focused on the optimization and evaluation of new solvent systems for solid–liquid extraction as well as the evaluation of pretreatment steps, such as ultrasound, microwaves, or electrical fields. Feedstocks, which were used, were specific or a mixture of different plant parts. A lot of research has focused on the extraction of pectin.

4.3. Pectin Extraction

Pectin is a complex mixture of polysaccharides, mainly galacturonic acid, and is present in the cell walls of plant tissues. Its main function is to keep cells together and to help ripening fruits keep their shape [102]. It is industrially used as a thickener, texturizer, emulsifier, stabilizer, and gelling agent in the food industry as well as a pharmaceutical agent to decrease blood cholesterol, heart disease, and gallstones and to soothe pain [103]. Figure 3 gives an overview of the most important/regularly used processes for the isolation of pectin from, e.g., citrus fruit, mango, or banana peel [104–106].

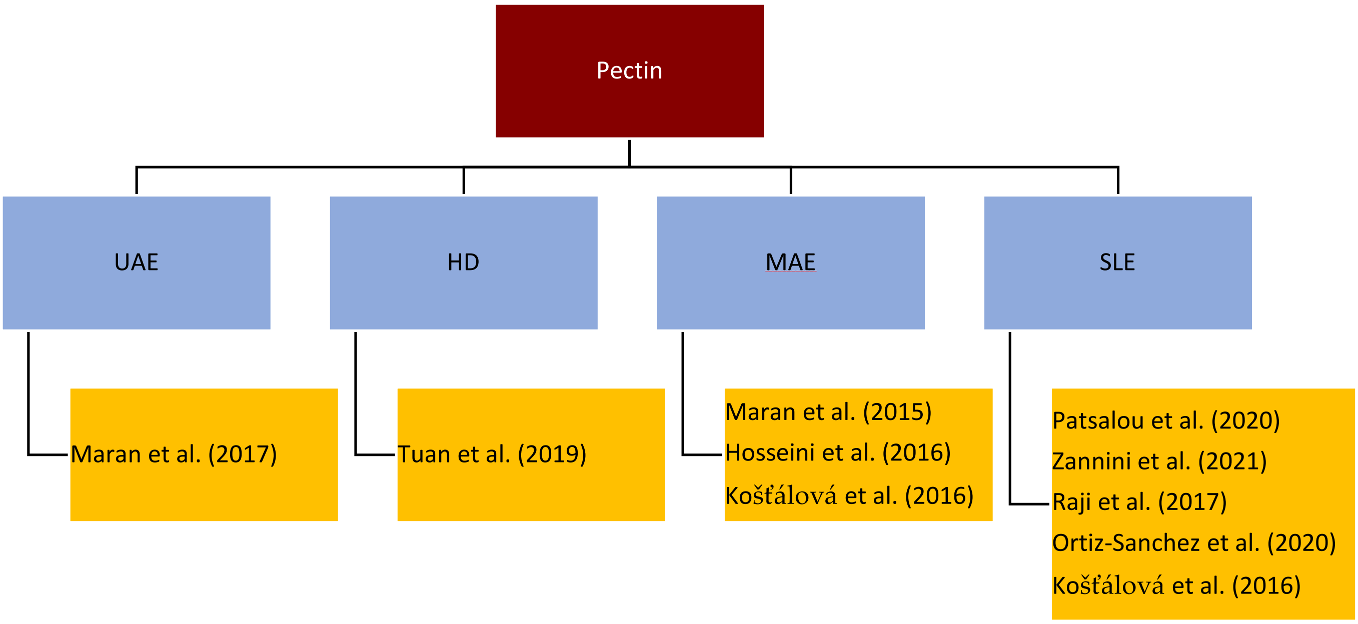

Figure 3. Literature overview pectin extraction [97,98,100–102,104,105].

The main influencing factor of pectin is the degree of esterification (DE), which tackles the functionality, morphology, and bioactivity of pectin. The power duration of the radiation and the pH-value were identified as important factors for the extraction efficiency, the DE, and the yield of pectin. The higher the radiation power and time, the higher the yield but the lower the DE [106]. Further, a decrease of the extraction time by 83% with a constant yield by using MAE compared to conventional hot acid extraction for medium DE was reported [104]. As for other bioactive molecules, the microwave power has to be limited to control the temperature and to avoid fast degradation of the pectin [105]. Raji et al. identified the type of acid as an important factor and confirmed that the pH-value influences the DE and the extraction yield. For the esterification, citric acid, tartaric acid, acetic acid, lactic acid, nitric acid, phosphoric acid, and sulfuric acid were tested. Citric acid was confirmed to work best for the extraction of pectin [107–109]. Many acids lead to pectin depolymerization, like nitric or sulphuric acid. Acetic acid was investigated as an alternative. High acid concentrations lead to high DE. Zannini et al. investigated that a concentration of 3% acetic acid showed a high yield with high DE. Therefore, they concluded that acetic acid is a good alternative to conventionally used organic solvents [108,110]. The yields with citric acid compared to stronger acids, such as HCl, are much lower according to the weaker ability of attacking the cell membrane, but more effective than with nitric acid [109,110]. Weaker organic acids are also more environmentally friendly and cause less corrosion on equipment than their strong inorganic counterparts. Tuan et al. investigated the simultaneous extraction of pectin and essential oils under the influence of citric acid. The citric acid showed no influence on the extracted oil; hence, a simultaneous approach can be implemented [109].

4.4. Pyrolysis

To achieve closed material loops, the solid residues from extraction can still be further utilized. Depending on the used solvents, the residues from extraction can still contain valuable molecules, such as sugars, and not only extracted bioactive molecules. Pyrolysis is used to extract bioactive molecules and to remove agricultural and landscape management wastes, especially waste from greenhouse production, which is mostly not further needed for soil regeneration [111]. In the case of transforming lignocellulosic material to biofuels, pyrolysis is one of the most explored possibilities [112]. The main products from pyrolysis are biochar, condensed biooils, and incondensable gases. Parts of these gases can be further utilized as syngas and industrial energy carriers [111,113]. The focus of today’s biorefineries, and especially the development goals, is on the material utilization of bio-oils. Therefore, single or groups of bioactive compounds have to be isolated or separated, which then can be utilized as pesticides [111,114–118], as antimicrobial agents and preservatives [117–119], for medical treatments [116,120], or as a source for precoursers and chemicals [116,121,122]. A detailed listing of pyrolysis feedstocks, conditions, and major gained chemicals can be found in Table A3.

5. Conclusions

The extraction of bioactive molecules from waste streams is not an easy operational task, but the benefits for the valorization of these streams lie on hand.

Agricultural waste streams mainly consist of lignocellulosic residues, while industrial and municipal vegetative residues are organic materials. Due to the high value and the various applicability for health promoting effects of bioactive molecules, such as polyphenols or pectin, the extraction of these compounds in the waste streams has to be investigated.

Extraction is the most promising unit operation to isolate bioactive molecules from solid feedstocks. Novel enhanced extraction methods and new solvents have been developed and investigated in recent years. The yield and the extraction time of conventional solid liquid extraction can be improved significantly in combination with evolved systems such as microwave-assisted and ultrasound-assisted extraction. The equipment for ultrasound- or microwave-assisted extraction is easy to use and is commercially available. New biological and electric-based unit operations were implemented to disintegrate cells and to enhance the extractability of bioactive molecules. Pressurized extraction methods, such as pressurized liquid extraction or supercritical fluid extraction, were shown to be effective. The pressurized solvents, mainly water and CO_2, are environmentally friendly and can be purchased easily. By adjusting the pressure, the selectivity of single molecules can be improved and fractions can easily be separated. NADES are new and still not very well investigated solvents with a high potential.

The interaction of many process parameters, solvent- or feedstock-based, is a challenge that researchers are focusing on. Due to the sensitivity of the molecules to temperature, light, and oxygen, the extraction parameters are important. While high temperatures foster the extraction of bioactive molecules, the degradation of thermally sensitive compounds starts. This has to be considered during solvent selection and postextraction purifying processes. Besides the temperature, the pH-value of the extraction media and the extraction time play an important role for the evaluation of the extraction processes. Less time with optimized yields leads to more feasible and economical processes. Additionally, pyrolysis can be an effective process for the valorization of the residues to finally obtain biochar and biooils.

Author Contributions: Conceptualization, A.D. and M.K.; writing—original draft preparation, A.D.; writing—review and editing, A.D. and M.K.; visualization, A.D.; supervision, M.K. All authors have read and agreed to the published version of the manuscript.

Funding: Open Access Publishing Fund by the Graz University of Technology.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

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

Appendix A

Table A1 shows a brief process and literature overview. Table A2 gives a broad overview of feedstocks that were used for the extraction of bioactive molecules. Further, the plant part as well as the extraction process used solvents and the used extraction conditions are specified. The table is intended to give an overview of the state of the research for the unit operation extraction when it is used for the purpose of bioactive molecule extraction from waste streams and which targeted single or grouped compounds were isolated or extracted.

Table A1. Literature overview for investigated and developed extraction processes.

Process	**Literature**		
ATPE	Chong et al. [80]	Dordevic et al. [81]	Ran et al. [68]
HVED	Xi et al. [89]	Yan et al. [53]	Zhang et al. [54]
MAE	Cvetanovic et al. [94]	Drevelegka et al. [32]	Klavins et al. [90,124]
	Cvjetko et al. [82]	Hiranvarachat et al. [93]	Routray et al. [71]
	Dahmoune et al. [123]	Ho et al. [69]	Wang et al. [74]
OHM	Loypimai et al. [100]		
SE	Klavins et al. [90]		
SFE	Benito–Roman et al. [101]	Kitryte et al. [64]	
	Garcia–Mendoza et al. [99]	Squillace et al. [125]	
SLE	Allison et al. [54]	Goldsmith et al. [79]	
	Amyrgialaki et al. [76]	Guthrie et al. [97]	Osojnik et al. [72]
	Cvjetko et al. [82]	Kehili et al. [78]	Ozturk et al. [70]
	Dahmoune et al. [124]	Klavins et al. [90]	Routray et al. [71]
	Espinosa–Pardo et al. [98]	Masci et al. [87]	
UAE	Bosiljkov et al. [83]	Fernandez et al. [85]	
	Chanioti et al. [84]	Gassara et al. [63]	Ran et al. [68]
	Cvetanovic et al. [94]	Huang et al. [86]	Routray et al. [71]
	Cvjetko et al. [82]	Klavins et al. [90,123]	Wang et al. [74]
	Dahmoune et al. [124]	Nipornram et al. [92]	Zhang et al. [54]
	Dordevic et al. [81]	Rahimi et al. [77]	Zivkovic et al. [75]
	Drevelegka et al. [32]	Rajha et al. [91]	
PEF	Martin–Garcia et al. [56]	Redondo et al. [55]	
	Pataro et al. [51]	Zhang et al. [54]	
PLE	Cvetanovic et al. [94]	Guthrie et al. [97]	Kitryte et al. [64]
	Garcia–Mendoza et al. [99]	Kheirkhah et al. [96]	Loarce et al. [95]
	Grunovaite et al. [33]	Kim et al. [126]	

Table A2. Extraction process overview.

Extraction Feedstock	Plant Part	Extraction Process	Extraction Solvent	Extraction Condition	Main Target Compounds	Max. Extraction Yields Content	Possible Utilization	Ref.
Apple	peel	PEF	Water	480–1200 V/cm, 0–2 s, 23–25 °C, 1:1–10 (s/l)	Phenolic compounds	~180 mg/100 g dw		[127,128]
	pomace	SSF–UAE	80% Acetone, *P. chrysoporium*	40 °C, 30 min, 1:2 s/l (*w/v*)		720 mg GAE/l		[63]
		UAE	80% Ethanol			639 mg GAE/l		
Aronia	stem	MAE	Water	580 W, 30 min, 1:25 (*w/v*)	Phenolic compounds flavonoids	16.65 mg CAE/g 12.12 RE/g	Antioxidant	[94]
		PLE		40 bar, 140 °C, 3 Hz stirring, 30 min		48.62 mg CAE/g 39.19 RE/g		
		UAE		1:26 (*w/v*)		5.22 mg CAE/g 3.94 RE/g		
Banana	peel	UAE	Water, citric acid	pH 1–5, 200–500 W, 5–45 min, 1:10–20 g/mL	Pectin	9.02%		[129]
Berry	press pomace	UAE	99.5% ethanol (96%), 0.5% Trifluoroacetic acid	1:30–100 (*w/v*), 100 W, 30 °C; 24 h without ultrasound	Phenolic compounds, anthocyanins	1.68 g/100 g dw 147 mg/100 g dw		[123] [90]
		SLE		1:30–100 (*w/v*), 100 W, 30 °C; 24 h		1.12 g/100 g dw 98 mg/100 g dw		
		MAE		10 min heat up, 600 W, 80 °C, 20 min		1.09 g/100 g dw 54 mg/100 g dw		
		SE		80 °C, 12 h, 25 cycles		1.21 g/100 g dw 65 mg/100 g dw		
Black chokeberry	pomace	SE	Hexane, acetone, ethanol	1:40 (*w:v*)	Phenolic compounds anthocyanins	25.92 g/100 g dw		[33]
		PLE	Hexane, methanol, water, acetone; 80:20 (*v/v*) acetone/water and methanol/water	40 and 130 °C, 10.3 MPa, 45 min		48.13 g/100 g dw		
		SFE	CO2	149 min, 40 MPa, 40 °C, 2 l/min		7.08 g/100 g dw		
Blackberry	pomace	EAE	Citrate buffer, water	Viscozyme L, 50 °C, 250 rpm, 360 min, pH 4.8	Phenolic compounds Lipophilic fraction	2.28 mg GAE/g dw 7.83 g/100 g dw		[64]
			Citrate buffer, water	50 °C, 250 rpm, 360 min, pH 4.8		0.84 mg GAE/g dw 5.78 g/100 g dw		
		PLE	Ethanol	50–90 °C, 10.3 MPa, 3 cycles á 5–15–45 min, after SFE		29.14 mg GAE/g dw 26.34 g/100 g dw		
			Water			7.81 mg GAE/g dw 5.09 g/100 g dw		
		SFE	CO2	25–55 MPa, 50–80 °C, 60–180 min		2.91 mg GAE/g dw 9.93 g/100 g dw		
		SLE	70% Water, 30% ethanol (*v/v*)	800 rpm, 20 °C, 360 min, after SFE		23.34 mg GAE/g dw 19.88 g/100 g dw		
		SE	Hexane	6 h		3.41 mg GAE/g dw 9.53 g/100 g dw		
Blueberry	leaves	MAE	15–30 % Ethanol, 1.5 M citric acid	10–20 % (710.5 W), 4–16 min	Phenolic compounds Anthocyanin Chlorogenic acid	92.719–128.76 mg GAE/g dw 2.419–2.636 mg M 3–G equiv./g dw		[71]

						49.542–53.270 mg/g dw		
		SLE	30% Ethanol, 1.5 M citric acid, 80 mL, 97:3 (*v/v*)	24 h, 1:16 (*w/v*)		89.164 mg GAE/g dw 2.196 mg M 3–G equiv./g dw 47.271 mg/g dw		
		UAE	30% Ethanol, 1.5 M citric acid, 80 mL, 97:3 (*v/v*)	1 h, 1:16 (*w/v*), 40 kHz; 24 h without ultrasound		97.77 mg GAE/g 2.46 mg M 3–G equiv./g dw 48.838 mg/g dw		
Brewers	spend grain	PEF	Ethanol water (4:1 *v/v*)	0.5–2.5 kV/cm, 50–150 Hz, 5–15 s	Tricin	97.936–46.125 μg/g dw		[56]
			Ethanol water (4:1 *v/v*)	0.5–2.5 kV/cm, 50–150 Hz, 5–15 s	Sinapoyl hexose	21.08– 36.08 μg/g dw		
Buckwheat	hull	PLE	Water	80–120 °C, 5 MPa, 1–3 mL/min	Rutin	91%, 24.2 mg/g	Medical use	[126]
		UAE	NADES	20 kHz, 200 W, 40 °C		9.5 mg/g		[86]
Carrot	peel	MAE	50 % Hexane, 25 % ethanol, 25 % acetone (*v/v*)	180 and 300 W	Carotenoids	289.2 mg/100 g (d.b.)		[93]
					β–carotene	132.7 mg/100 g (d.b.)		
Citrus	peel	HD	Water	116 °C, 10 min, 5% (*w/v*) CPW and 0.5% (*v/v*) H2 SO4	Essential oils Pectin	0.43% 30.53%		[66]
		SSF	A. succinogenes Z130		Succinic acid	0.73 g/g		
	pomace	SLE	Acetic acid 3% (*v/v*),	20 g, 200 mL, pH 2.6, 90–100 °C, 6 h	Pectin, flavonoids, polyphenol	23.70%	Bio–composite	[108]
Cupressus lusitanica Mill. Cistus ladanifer L.	leaves	SD	Water	1. 30 °C, 30 min ethanol, s:l 1:20, 320 W, 35 kHz 2. 1. + 30 °C, 30 min acetone	Phenolic compounds	140 mg GAE/g extract 210 mg GAE/g extract		[73]
					Flavonoid	1.3 mg QE/g extract 11.5 mg QE/g extract		
					Tannins	86.8 mg GAE/g extract 133.3 mg GAE/g extract		
		UAE	Ethanol, 70% acetone		Phenolic compounds	251.3 mg GAE/g extract 275.6 mg GAE/gextract		
					Flavonoid	6.3 mg QE/g extract 15.2 mg QE/g extract		
					Tannins	82.2 mg GAE/g extract 116.6 mg GAE/g extract		
Fig	leaves	MAE	NADES	1:20 g/mL, 10 min, 55 °C, 250 W	Phenolic compounds	45.724 mg/g		[74]
		UAE	NADES	1:20 g/mL, 60 min, 60 °C, 700 W	Furanocoumarins	45.724 mg/g		
Grape	pomace	MAE	Water ethanol	100–600 W, 8–24 mL/g (l/s)	Phenolic compounds	30.66 mg GAE/g dry pomace 45.35 GAE/g dry pomace		[32]
		PLE	Water NADES	2 g pomace, 1 g diatomaceous earth, 2*10 min, 10.34 MPa, 40–120 °C,		135.24 mg/g		[95]
		UAE	Water, 0–100 % ethanol (*v/v*)	20–60% (20 kHz), 20–60 °C, 8–24 mL/g (l/s)		48.76 mg GAE/g dry pomace	Food industry	[32]

	seed	UAE–ATPE	Ionic liquids Ethyl acetate Isopropanol Ethanol Water Methanol 70% ethanol 70% methanol	1:5 (s:l), 40 °C, 20 min	Procyanidin B2 catechin epicatechin	0.14 mg/g dry weight 1.21 mg/g dry weight 1.22 mg/g dry weight	Natural anti-oxidants	[68]
	peel	MAE	NADES	50–90 °C, 15–90 min, 100 W	Anthocyanins	26 mg/g dw		[82]
		SLE		12 h, RT, 1:11 (s/l)		18 mg/g dw		
		UAE		50–90 °C, 15–90 min, 35 kHz		30 mg/g dw		
	stalk	SSF	LiP, MnP, MnIP, and Lacc iP MnP MnIP, Lacc, CMCase, xylanase, and avicelase (CMCase xylanase and avicelase)		Phenolic compounds	1.5 mg GAE/l control 0.45 mg GAE/l pre–treated		[58]
Haskap	leaves	ATPE	Salt / ethanol	(NH4)2 SO4, NaH2 PO4 25 °C, 5–120 min	Chlorogenic acid flavonoids Phenolic compounds	89.31%–97.82% 36.43 µg/mg		[80]
		SLE	80% Methanol (*v*/*v*)	75 mg, 150 mL, 24 h		76.28 µg/mg leaves 0.23 mg/mg leaves 0.14 mg/mg leaves		
		ATPE	Sugar / propanol	glucose and maltose, 25 °C, 5–120 min, 0.1–1 wt% sample loading		69.52%–82.13%		
Juçara	residues	PLE	Ethanol, water, acidified water	10 MPa, 40–80 °C, 1.5 mL/min, 90 kg solvent/kg dry residue	Phenolic compounds anthocyanins	51.4 mgGAE/g dw		[99]
						1.76 mg/g dw		
		SFE	CO2, ethanol water 50% (*v*/*v*)	46 min, 60 °C, 20 MPa, 10% (*w*/*w*) co–solvent		30 mg GAE/g dw		
						6.2 mg/g dw		
		SE	Ethanol	180 mL, 3 g, 6 h		21.9 mg GAE/g dw		
						10.5 mg C3 RE/g dw		
		UAE	Ethanol, water 50% (*v*/*v*)	800 W, 19 kHz, 45 min		23.3 mg GAE/g dw		
						8.7 mg C3 RE/g dw		
		ABE		2.5 g, pH 2.0, 45 min, 60 °C		22 mg GAE/g dw		
						7.9 mg C3 RE/g dw		
Kiwifruit	Peel	PLE	Water	500 mL, 30 bar, 30 min, 120–160 °C, pH 2–5.5, 2–6% (s/l)	Phenolic compounds	51.2 mg GAE/g DW	Medical use	[97]
					Flavonoids	22.5 mg CE/g DW		
		SLE	Ethanol 50%	2–24 h, pH 2–7	Phenolic compounds	26.15 mg GAE/g DW		
					Flavonoids	18.93 mg CE/g DW		
	pomace	PLE	Water	50 bar, 170–225 °C, 10–180 min, 1:100 g/mL (M/S)	Phenolic compounds	86.26 mg CaE/g DW		[96]
					Flavonoids	24.18–34.59 mg QE/g DW		
		SLE	Ethanol, methanol (80%), acetone (70%)	2 h, 1 h/70 rpm, 1 h/200 rpm	Phenolic compounds	8.1 CaE/g DW		
					Flavonoids			
Mandarin	peel	SLE	Acetone 80%	1:20 g/mL (s/l), 30–50 °C, 20–40 min	Phenolic compounds	12,519.73 mg GAE/100 g DW		[92]

					Hesperidin	6153.22 mg/100 g DW		
		UAE		1:20 g/mL (s/l), 38.5 kHz, 30.34–59.36 W, 30–50 °C, 20–40 min	Phenolic compounds	15,263.32 mg GAE/100 g DW		
					Hesperidin	6435.53 mg/100 g DW		
Mango	peel	MAE	Water	413 W, 2450 MHz, pH 2.7, 134 s, 1:18 g/mL (s/l)	Pectin	28.86%		[105]
Melon	peel	SLE	Water, tartaric acid, citric acid, hydrochloric acid, acetic acid, lactic acid, nitric acid, Phosphoric acid, and sulfuric acid	35–95 °C, 40–200 min, pH (1–3) 10–50:1 v/w (l/s)	Pectin	29.48% with citric acid		[107]
			Ethanol / 0.1 M nitric acid	1:25 *w*/*v* (s/l), 1 h, boiling point		19.3% (fresh) ; 14.2% (dried)		[130]
Microalgae pomace		PEF	Ethanol 95% (*v*/*v*)	24 h, 1:20 *w*/*w* (s/l), 150 rpm	Carbohydrates	37.50%		[54]
					Proteins	10.10%		
					Chlorophyll α	9 mg/g dw		
		HVED			Carbohydrates	19%		
					Proteins	5.5%		
					Chlorophyll α	10.5 mg/g dw		
		UAE			Carbohydrates	31%		
					Proteins	4%		
					Chlorophyll α	25 mg/g dw		
		SLE			Carbohydrates	10%		
					Proteins	3%		
					Chlorophyll α	4.5 mg/g dw		
Olive	cake	UAE	Lactic acid and glucose, 5:1; citric acid and glucose, 1:1; fructose and citric acid, 1:1, water 0–15%	40–80 °C, buffer 0–0.1%, 24 h	Secoiridoids, simple phenol, flavonoids, hydroxycinnamic and hydroxybenzoic acids	669.433 μg/g dry by–product	Pharmaceutical, cosmetic, agricultural and food industries	[85]
	pomace	SLE	Hexane			62.05 μM TYE/g		[79]
		UAE	NADES	2:25 g/mL (s/l), 40–60 °C, 30 min, 60 kHz, 280 W		20.14 GA/g dw		[84]
		PLE	NADES	300–600 MPa, 5–10 min	Phenolic compounds	5.31 mg/g dw		
Onion	pomace	SLE	Water or ethanol 70% (*v*/*v*)	1:10 *w*/*v* (s/l), 90 min, 25 °C, 175 rpm	Quercetin	400 mg/kg dw		[72]
	seed	UAE	Lactic acid and glucose, 5:1; citric acid and glucose, 1:1; fructose and citric acid, 1:1, water 0–15%	15–60 min ultrasound, 15–75:1 mg/mL (s/l), 40 °C, 20 kHz, 200 W	Secoiridoids, simple phenol, flavonoids, hydroxycinnamic and hydroxybenzoic acids	344.551 μg/g dry by–product	Pharmaceutical, cosmetic, agricultural and food industries	[85]
Orange	peel	SSF	Aspergillus fumigatus, water	1:5 (s/l), 60 °C, 30 min	Ellagic acid	18.68 mg/g		[65]
		EAE	Viscozyme L	1:8 (s/l), 50 °C, 180 min, pH 4.5	Sugar	48%		[52]
					Phenolic compounds	0.85%		
		MAE	Water	1:10 g/mL (s/l), 240 min, 100 °C, 300–1000 W	Essential oils	1.80%		[131]
			Water, citric acid	15:1 v/w (l/s), pH 1.5–3, 300–700 W, 1–3 min	Pectin	29.10%		[106]
		SLE	Water, 5.7 mM citric acid, 0.7% sodium hexametaphosphate	348.15 K, pH 2, 90 min, 200 rpm	Pectin	15.85% 0.15±0.77 g/g	D–galacturonic acid as a platform chemical, mucic acid	[110]
			Hexane, CPME, EL, IPA, PEG 300,	1:10 (s/l), 120 min, 30 °C, 900 rpm	Limonene	0.81%	Limonene	[70]

			IAc, DMC, MEK, 2–MeTHF, EAc					
	pomace		Water	1:8 *w/w* (s/l), 50 °C, 160 rpm	Phenolic compounds	0.4–0.6 g/100 g dw		[52]
			Ethanol	1/15 g/mL (*w/v*), 6 h		51–71 mg GAE/g extract		[98]
		HVED	Water, Viscozyme® L	200 W, 1:8 *w/w* (s/l), 50 °C, 40 kV, 10 kA, 0.5 Hz, 160 J/pulse, 180 min, pH 4.5, 44–448 kJ/kg		700 mg/100 g DM		[52]
		EAE–SFE	CO_2, ethanol/water 9:1 (*v/v*), Paecilomyces variotii	124 ± 2 kg solvent/kg pomace, 15–35 MPa, 95 min, 40–60 °C, 2.67 × 10 4 kg/s		18–47 mg GAE/g dry extract		[98]
Peach	pomace	PEF	Water, 0–80% methanol	10–50 pulses, 3 μs, 0–5 kV/cm, 0.61–9.98 kJ/kg, 15–35 °C	Phenolic compounds	6.4–83.3 mg GEA/100 g		[55]
					Flavonoids	0.6–54.3 CE/100 g		
Peanut	shell	HVED	Water, 0–30% ethanol	1000 Hz, 2 μs, 12–16 kV, 10 kA, 20:1–60:1 mL/g (l/s), 20–100 mL/min	Flavonoids	0.117–0.948%		[53]
		SLE	Water, 80% ethanol	30 mL/g (l/s), 60°C, 120 min		0.866%		
Pear	pomace	UAE	NADES	15–60 min ultrasound, 15–75:1 mg/mL (s/l), 40 °C, 20 kHz, 200 W	Secoiridoids, simple phenol, flavonoids, hydroxycinnamic and hydroxybenzoic acids	32.808 μg/g dry by–product		[85]
Pistachio green	hull	SLE	Citric acid	pH 0.5–2.5, 50–90 °C, 30–150 min, 10–50:1 v/w (l/s)	Pectin	22.10%	Health and cosmetic applications	[103]
Pistacia lentiscus	leaves	MAE	20–100% Ethanol (*v/v*)	30–210 s, 300–900 W, 10–40:1 mL/g (l/s)	Phenolic compounds Flavonoid Tannin	185.69 mg GAE/g dw 5.16 mg QE/g dw 40.21 mg/g dw	Antioxidant	[88]
		SLE	60% Ethanol (*v/v*)	60 °C, 2 h, 110 rpm, 50:1 mL/g (l/s)		178.00 mg GAE/g dw 4.79 mg QE/g dw 31.15 mg/g dw		
		UAE	40% Ethanol (*v/v*)	20 kHz, 15 min, 27 °C, 0.01 W/mL, 1:250 g/mL (s/l)		142.76 mg GAE/g dw 4.61 mg QE/g dw 35.94 mg/g dw		
Pomelo	peels	HD	Water, 0–0.55% (*w/w*) citric acid	pH 2.1–7.3, 2.5 h	Pectin	11–24% (0–0.55% citric acid)		[109]
					Essential oils (limonene, β–pinene, α–phellandrene)	4.6% /g dw (89.87%, 2.83%, 1.38%)		
Pomegranate	peel	HVED	Ethanol, methanol, water, acetic acid, ethyl acetate	8–14 mL/min, 18–45 kV/cm, 20–50 mL/g (l/s), 30 min	Phenolic compounds	196.7 mg/g		[89]
		SLE		70 °C, 35 mL/g (l/s), 60 min	Phenolic compounds, Flavonoids, anthocyanins	158.9 mg/g		
			Ethanol, water, 1 g/l citric acid, 1 N NaOH	1:10 *w/v* (s/l), 400 rpm, 22 °C	Phenolic compounds	324.9 mg GAE/g dry weight		[76]
			Ethanol, methanol, Ethanol/water 10% acetic acid (1:1, 3:1), ethyl acetate	1:4 g/mL (s/l), RT, 24 h	Phenolic compounds, Flavonoids, anthocyanins	26.6 mg GAE/g fw 1.9 mg/100 g fw		[87]
	seed peel		Water	50 °C, 10:1 *w/w* (l/s), 200 min, 20 rpm		38 mg GAE/g DM (peel)		[91]

					Phenolic compounds (ellagic acid, chlorogenic acid, gallic acid)	10.6 mg GAE/g DM (mix)		
		UAE				13.7 mg GAE/g DM (mix)		
						51 mg GAE/g DM (peel)		
			10–90% Ethanol	1:10–1:50 (s/l), 20–80 °C, 5–65 min	Phenolic compounds	118.01–190.94 mg GAE/g dw		[75]
					Ellagic acid	4.05–12.54 mg/g dw		
					Gallic acid	1.13–3.58 mg/g dw		
					Punicalin	28.38–65.67 mg/g dw		
					Punicalagin	7.04–35.05 mg/g dw		
Pumpkin	pomace	SLE	Water, HCl	30:1–50:1 mL/g (l/s), pH 2.5, 85 °C, 1 h, 100 rpm	Pectin	5.7–7.3%		[104]
		MAE	Water	30:1–50:1 mL/g (l/s), pH 2.5, 1200 W, 80–120 °C, 2–10 min		3.1–7.4%		
Rice	bran	OHM	Water: 95% ethanol, 1:1, 0.1 M HCl	30–40% moist, RT, pH 2.5, 100 rpm, 90 min, 1:5 g/mL (s/l)	Tocols and γ–oryzanol	53,077μg/g		[100]
					Anthocyanins	10818.5 μg/g		
		SD	water	RT pH 2.5, 100 rpm, 90 min, 1:5 g/mL (s/l)	Tocols and γ–oryzanol	28.54 μg/g		
					Anthocyanins	7806.9 μg/g		
		SFE	CO_2, ethanol (modifier, 0–10%)	40–60 °C, 30–40 MPa, 0.40 kg/h, 2 h, 100 g	Phenolic compounds	1 mg GAE/g 1.61–3.42 GAE/g (EtOH modifier)		[101]
					Flavonoids	1.05 mg QE/g 4.47 mg QE/g (EtOH modifier)		
					γ–oryzanol	13.19–20.63 mg/g		
		SLE	Hexane	Soxhlet, 5 g, 25 cycles	Phenolic compounds	1.58 mg GAE/g		
					Flavonoids	1.01 mg QE/g		
					γ–oryzanol	12.47 0.25 mg/g		
					Fatty acids	784 mg/g of oil		
					Sesamin	25.7 mg SES/100 g,		
					Sesaminol triglucoside	537.5 mg SE/100 g		
		SSF	Bg352, Bw367 Bg406, H 4 Methanolic extraction solvent		Phenolic compounds	8.81 mg GAE/g FW		[132]
					Flavonoids	14.75 mg RE/g FW		
					Carotenoids	1.65 mg Cy 3 glc/g FW		
Tomato	peel	MAE	Hexane, ethyl acetate (1:0, 1.5:0.5, 1:1, 2:8, 1:9, 0:1 *v/v*)	1:20–4:20 g/mL (s/l), 400–1600 W, 24–48 kJ	Lycopene	13.592 mg/100 g		[69]
		PEF	Acetone, ethyl lactate	10 Hz, 20 μs pulse width, 0.012–0.475 kJ/kg, 0–5 kV/cm, 10–833 pulses, 1:40 g/mL (s/l), 25 °C, 160 rpm, 0–1440 min		17.532 g/kg Dw		[51]
		SLE		1:40 g/mL (s/l), 25 °C, 160 rpm, 0–1440 min		14.823 g/kg Dw		
	pomace	SFE	CO_2	80 °C, 380 bar, 15 kg/h	Lycopene	406 μg g^{-1}		[125]
		SLE	50% Hexane, 25% ethanol, 25% acetone	1:100 g/mL (s/l)	Lycopene	293–476 mg/g dry pomace	Antioxidant, health applications	[31]

			Olive oil	40–80 °C, 200–400 rpm, 2.5–5.5% *w/v* (s/l), 15–150 min	Lycopene	1235.7 mg/kg dw	Lycopene	[78]
			Hexane, hexane: methanol:acetone (2:1:1 *v/v*)	1 g, 1 h, RT	Lycopene	63.66 mg/100 g, 74.89 mg/100 g		[77]
		UAE	NADES	15–60 min ultrasound, 15–75:1 mg/mL (s/l), 40 °C, 20 kHz, 200 W	Secoiridoids, simple phenol, flavonoids, hydroxycinnamic and hydroxybenzoic acids	1,093.475 µg/g dry by–product	Pharmaceutical, cosmetic, agricultural and food industries	[85]
			Sunflower oil	3.18–36.82% *w/v* (s/l), 1.59–18.41 min, 30–70 W/m2	Lycopene	91.49 mg/100 g		[77]
Wheat	chaff	SLE	22.5% *w/w* Ethanol, water	10 min, 25 °C, 3% *w/w* (s/l)	Phenolic compounds	2 mg GAE/g (ethanol), 1.8 mg GAE/g (water)		[81]
		UAE	22.5% *w/w* Ethanol/ammonium–sulfate	10 min, 25 °C, 30 kHz, 500 W, 3% *w/w* (s/l)		2.1 mg GAE/g (water), 2.5 mg GAE/g (ethanol)		
		ATPE	22.5–24.5% *w/w* Ethanol, 20–23.8% *w/w* salt, water	10 min, 25 °C, 3% *w/w* (s/l), ammonium–sulfate		2.1 mg GAE/g		
		ATPE UAE		10 min, 25 °C, 30 kHz, 500 W, 3% *w/w* (s/l), ammonium–sulfate,		2.67 mg GAE/g		
Wine	lees	UAE	Ethanol/water/formic acid, 50:48.5:1.5, *v/v*/v	380 W, 37 kHz, 35°C, 1:10 g/mL (s/l), pH 2.7, 3 h	Anthocyanin	4.35 mg/g dw		[83]
			NADES, 10–50% water	190–380 W, 37 kHz, 35°C, 1:10 g/mL (s/l), pH 2.7, 15–45 min		6.42 mg/g dw		

Table A3. Pyrolysis overview.

Feedstock	Plant Part	Condition	Liquid Yield	Major Chemicals	Possible Utilization of Bioactive Molecules	Reference
Camellia oleifera	shell extraction residue	800 °C		Acetic acid, (E)–Stilbene, 4,4′–(1–methylethylidene)bis–phenol, and 33 other constituents	Bio–oil	[133]
Cocoa	shell	114–514 °C	33.58% at 214 °C	Acetic acid, cyclopropyl carbinol, 1,6–anhydrous–beta and 24 other constituents.	Liquid fuels, source of chemicals	[121]
Coconut		400 °C	51%	Phenol, 2–methoxy phenol, furfural, 29 other constituents.	A traumatic ulcer healing agent	[120]
Corn	cob	300–500 °C	47.78% at 400 °C	Not analyzed	Pesticides, liquid smoke	[115]
Cotton	shell	400–600 °C	51% at 450 °C	Thiofanox sulfoxide, 2–hexane, 3–hexane, Linalool, Conyrine, and 260 other compounds listed	Medical, industrial, and agricultural products	[116]
Durian	peel	340–380 °C		Acetic acid, phenol and small amounts of ketones, aldehydes, and carboxylic acids	Pesticide, natural preservative for mackerel	[118]
Groundnut	press cake	200–500 °C	50% at 450 °C	Oleic acid amide, oleanitrile, p–cresol, palmitamide, n–methyloctadecanamide, indole, and 64 constituents	Bio–fuel	[134]
Hemp	herds	275–350 °C	38%	Methanol, acetic acid, furfural, formic acid, 1–hydroxybutan–2–one		[135]
	fiber	350 °C		Propionic acid, acetic acid, methanol, formic acid, hydroxymethylfurfural, and furfural	Pesticide	[117]
Jatropha	husk shell branch	550 °C	42.7%	Benzene, toluene, xylenes, methyl indene, methylnaphthalene, phenols, alkylphenols, methoxy phenols, and eugenols		[122]

Palm	fruit bunch	425–550 °C	72.4% at 500 °C	not analyzed	Bio–fuel	[136]
Rice	hull	450–500 °C		161 compounds characterized		[137]
Soybean	hulls	300 °C		Levoglucosenone	Antimicrobial activity	[138]
Sunflower	seed hull	450 °C	36%	Acetic acid, methoxy–phenolics, furfural	Insecticide	[114]
Switchgrass		400–600 °C	48% at 400 °C	36% hydrocarbons and alkanes, 20.5% phenolic compounds, 14.1% aromatics and 29.4% furans, acids, ketones, alcohols, esters, and amides	Bio–fuel	[113]
Tomato	leaves stems roots fruits	300–500 °C	37.8% at 500 °C	Neophytadiene, diene, phytol, and 404 minor constituents	Pesticide	[111]
Wood	bark	350 °C		Liquid smoke Propionic acid, acetic acid, methanol, formic acid, hydroxymethylfurfural, and furfural	Wood preservative	[117,119]

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