Current Food Science and Technology Reports
https://doi.org/10.1007/s43555-023-00010-8

Current Emerging Green Technologies for the Valorization of Grape and Cherry Wastes

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Accepted: 3 November 2023

Abstract
This review aims to highlight current emerging technologies for the valorization of the bioactive fraction of waste from cherry and grape processing industries through its recovery and conversion into high added-value products. Due to its richness in diverse functional and health-related metabolites, the valorization of cherry residue and grape residue as a source of bioactive compounds for new food, nutraceutical, and pharmaceutical products has great interest and potential. Furthermore, aiming for more sustainable processes, several process intensification technologies (UAE, SWE, MAE, PEF, ASE, and NaDES) have emerged in recent decades to extract bioactive compounds from these byproducts, according to a circular economy approach. These techniques allow a considerable reduction in extraction time, greater mass transfer, lower energy consumption, volume of solvents, and energy. Consequently, these new technologies have potential for application on a pilot scale.

Keywords Agro-industrial residue · Bioactive compounds · Valorization · Grape · Cherry · New technologies

Introduction

A third of global anthropogenic greenhouse gases emissions comes from the agricultural and agro-food sectors, due to inadequate waste management [1]. Indeed, according to the Food and Agriculture Organization (FAO), the disposal of industrial waste generated a total emission of 54 to 62% from 1990 to 2019 [2]. Reducing the food chain carbon footprint is thus mandatory to addressing climate change, and proper waste management is of particular importance [2].

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Most agri-food waste, due to its organic nature, is viewed as a resource in the circular bioeconomy. Rich in carbohydrates, proteins, lipids, wax, and nutraceuticals, food waste can be used to produce energy, biofuels, bioactive compounds, and biodegradable materials, rather than being wasted [3]. In line with this concept, the European Union adopted in 2020 a new action plan for the circular economy, which purposes to increase global competitiveness, promotes sustainable economic growth and generates new jobs [4].

Currently, one of the trending strategies for the valorization of agro-industrial and agricultural waste is the development of novel green processes to recover high value-added fractions according to the needs of local and regional markets [5]. Valorization encompasses redirecting erstwhile food waste toward the transformation of food waste into extracted food and feed components. According to this approach, all plant materials can be used in an integrated manner due to their richness in numerous bioactive compounds that can later be incorporated in novel functional and health-related products with careful consideration of its quality, durability, and composition.

In recent years, the production of cherries and grapes significantly increased, with over 2.7 and 84.8 million tons produced in 2021, respectively. Currently, Turkey is the leading producer of cherry with around 25% of

Published online: 28 November 2023

the global production, followed by the USA, Chile, and Uzbekistan (12.4%, 11.74%, and 7.72%). Concerning grapes, China is the leading producer contributing 26.5% of the global production, followed by Italy with 9.61% [6].

The cherry and grape processing industry generates significant by-products, including skin, stalks, and seeds, posing environmental challenges [6]. Yet, this waste is a valuable, low-cost source for innovative bio-based products. These by-products are rich in compounds like dietary fibers, biopolymers, and phenolics known for their health benefits, especially their antioxidant and anti-inflammatory properties, making their recovery crucial [7–9].

Conventional solid–liquid extraction (SLE) remains the most common method to extract bioactive compounds from grape and cherry waste. However, this process uses harmful organic solvents, leading to long processing times, high temperatures, and excessive energy consumption, impacting both human health and the environment. To combat these challenges, industries explore innovative green extraction methods with eco-friendly solvents and advanced technologies, striving for process sustainability [10]. These promising techniques include microwave-assisted extraction (MAE) [11–13], ultrasound-assisted extraction (UAE) [14], supercritical fluid extraction (SFE) [15], accelerated solvent extraction (ASE) [16], subcritical water extraction (SWE) [17], and pulsed electric fields (PEF) [18], among others [8, 13].

The aim of the present review is to underline the richness in bioactive metabolites of grape and cherry by-products that could be recovered fulfilling the dictates of the circular bioeconomy. A particular focus will be placed on spotlighting the current emerging technologies suitable for sustainable extractions of bio-actives from these agro-wastes.

Bioactive Composition of Grape and Cherry Waste

Grape by-Products

Cultivated globally, grapes yield over 60 thousand tons annually, primarily used for wine, creating significant grape pomace (GP) as a by-product. GP, comprising seedless pomace (pulp, skin, stem) and grapeseed, constitutes 20% of the original grape weight after pressing and fermentation [19]. Its composition varies, with the seed fraction typically comprising 38 to 52% [20]. Rich in bioactive compounds like unsaturated fatty acids, dietary fibers, vitamins, and natural antioxidants (phenolic acids, flavonoids, proanthocyanidins), GP presents valuable potential [20].

GP is a great source of phenolic compounds that are present in the skin (374.6 mg gallic acid equivalent (GAE)/g) and adjacent sections, such as seeds, pulp, stalks, and leaves (2178.8, 23.8, 5.78, and 351.6 mg GAE/g, respectively) [21, 22•] (Fig. 1A). The phenolic composition of grape by-products varies largely in quantity and quality depending on various factors including climatic conditions, ripening stage, geographical origin, variety, and processing techniques [23]. Nonetheless, the most prevalent phenolic compounds found in these by-products include anthocyanins, hydroxybenzoic and

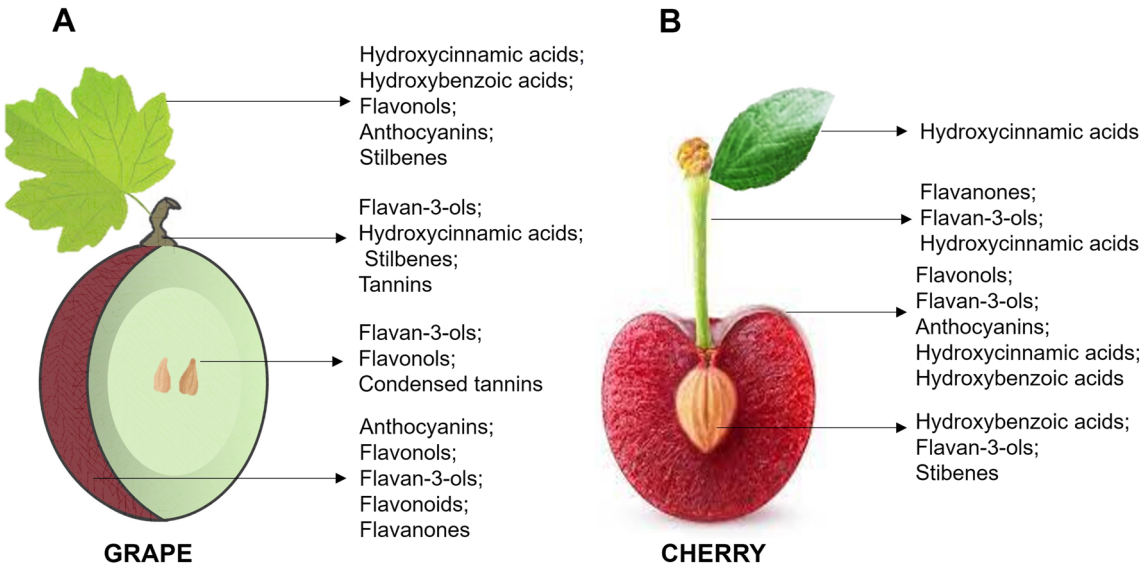

Fig. 1 Main components of cherry and grape

hydroxycinnamic acids, flavan-3-ols, flavonols, and stilbenes [23].

Grape leaves are mainly composed of cellulose and hemicelluloses but have a good concentration of phenolics, tannins, lipids, vitamins, flavanols, and organic acids [24]. In a relevant study conducted by Amarowicz and colleagues [25], *Vitis vinifera* leaves were found to be rich in phenolic acids such as gallic acid that was pointed out to be the major compound, followed by caffeic, and *p*-coumaric acid, caffeoyltartaric acid, and 3-(6-*p*-coumaroyl) glucosides, respectively [25, 26]. Regarding their flavonoid contents, a high amount of flavonols was detected (90%), mainly quercetin together with its glycosides as well as quercetin methyl glucoside, in addition to other flavonols glycosides, such as kaempherol-3-*O*-glucoside, kaempherol-3-*O*-galactoside, and kaempherol-3-*O*-glucuronide, along with some aglycones such as apigenin, kaempferol, and myricetin [27, 28]. Conversely, tannins ((+)-catechin and (-)-epicatechin) and their condensed forms were found with a lesser amount in leaves than in skins [28]. On the other hand, grape leaves contain considerable amounts of anthocyanins (cyanidin-3-*O*-glucoside, delphinidin 3-*O*-glucoside, malvidin 3-*O*-glucoside, and petunidin 3-*O*-glucoside peonidin-3-*O*-glucoside) [29]. Non-flavonoid phenolic stilbenes, namely *cis*- and *trans*-resveratrol and astringin were also detected in grape leaves [28].

Focusing on grape stems, they consist mainly of cellulose, hemicelluloses, and lignin. Additionally, significant amounts of non-flavonoid phenolic compounds like phenolic proanthocyanidins, such as flavan-3-ols, hydroxycinnamic acids, stilbenes, and monomeric and oligomeric flavonols, have been detected [30–32] together with condensed tannins (procyanidin and prodelphinidin) [31]. When analyzing the grape stem extract using gas chromatography, it was found to predominantly contain fatty acids and a higher concentration of sterols, with sitosterol being the most abundant, followed by stigmasterol, ergosterol, and stigmast-4-en-3-one [31]. Lastly, it should be considered that grape stems are also rich in polysaccharides, mainly glucans, xylans, galactans, arabinans, and mannans [24].

As far as grape skin is concerned, white and purple/red varieties present different concentrations of phenolic compounds with anthocyanins identified as the main ones in purple and red grape varieties. Anthocyanins were found to be the major phenolic compounds in purple and red grape skins [33]. Vodnar et al. [34] found in red grape skin different bioactive compounds, namely anthocyanidins forms (malvidin, peonidin); glycosides form (delphinidin-3-*O*-glucoside, petunidin-3-*O*-glucoside, peonidin-3-*O*-glucoside, malvidin-3-*O*-glucoside, and cyanidin 3-*O*-arabinoside); their acylated (malvidin-3-acetyl glucoside); caffeoylated; malvidin-3-caffeoyl glucoside and coumaroyl forms (petunidin 3-*O*-(6″-*p*-coumaroyl)-glucoside), malvidin 3-*O*-(6″-*p*-coumaroyl)-glucoside) [34, 35].

Red grape skin is richer in phenolics due to high contents of anthocyanins, while there is a higher amount of flavonols, catechins, and proanthocyanidins in white skin, a fact which explains the higher antioxidant potential of peels from red grapes than those from white ones [36, 37]. But both grape peels and pomace are rich in flavonoids (quercetin kaempferol and myricetin and their glycosides; quercetin-3- glucoside, quercetin-3-glucoronide, rutin, and myricetin-3-glucoside), in addition to laricitrin-3-glucoside, isorhamnetin-3-glucoside, syringetin-3-glucoside, flavanones; hesperidin and eriodyctiol, flavones; apigenin, vitexin, and luteolin, isoflavone; and genistein [38, 39]. Other phenolic compounds were also found in the skin such as gallic acid (13.7 mg/g), caftaric acid (40.4 mg/g), protocatechuic acid (11.0 mg/g), vanillic acid (9.2 mg/g), syringic acid (4.3 mg/g), (+)-catechin (16.5 mg/g), (-)-epicatechin (23.7 mg/g), rutin (143.1 mg/g), isoquercitrin (212.1 mg/g), kaempferol (362.7 mg/g), and resveratrol (149.2 mg/g) [21].

In a study conducted by Juhaimi et al. [40] on 11 varieties of grape seeds from Italy, France, and Turkey, the authors have proved the presence of high amounts of phenolic compounds (total phenolic content (TPC) fluctuating between 6711 and 8818 mg GAE/g dry weight (dw)) and flavonoids (total flavonoid (TF) ranging from 263.53 to 1706 mg/g catechin equivalent (CE)). Lower amount of anthocyanins (total anthocyanin (TA) varying between 0.31 and 2.55 µmol/g dw) and considerable amounts of proteins (7.51 to 13.2%) were also reported [40].

The flavonoid content was much higher than phenolic acids' content in the seeds. In more details, seeds were found to be rich in flavan-3-ols; (catechin, epicatechin, and epicatechin gallate), as well as condensed tannins, both the dimers (procyanidin B1, procyanidin B2, and procyanidin A2) and catechin oligomers [39, 41]. Other flavonoids were also identified in some cultivars: quercetin, quercetin 3-*O*-glucoside, quercetin 3-*O*-galactoside, and quercetin 3-*O*-glucuronide [42].

The increasing need for natural antioxidants as functional food ingredients stems from their ability to protect cells against oxidative damage and reduce the risk of degenerative diseases linked to oxidative stress [43]. Considering the richness of grape by-products in bioactive compounds, recovering these fractions for natural ingredients presents significant potential for industrial use [7, 20].

Cherry by-Products

Understanding the chemical composition of cherry pomace (CP), which includes stems, seeds, skins, and leaves, will support the development of eco-friendly approaches for the valorization of these wastes. This agro-industrial waste abounds in vitamins and antioxidants, primarily phenolic compounds, renowned for numerous health benefits [44–47].

The CP contains mainly high quantities of hydroxycinnamates, anthocyanins, and flavan-3-ols (the most abundant ones) (Fig. 1B). Anthocyanins are responsible for the attractive color of sweet cherries, with quantities reaching up to 700 mg/100 g in darker fruits [48, 49].

Hydroxycinnamic acids (p-coumaric, ferulic, caffeic, chlorogenic, and neochlorogenic acid), flavanones, and flavan-3-ols are the main phenolic compounds in cherry stem extracts [49, 50]. Other phenolic compounds in cherry stem include sakuranetin, isosakuranetin, p-coumaroylquinic acid, and catechins [50] [51].

Antioxidant capacity in the cherry stem was reported as 433–448 µg ascorbic acid equivalent/mL based on ferric reducing antioxidant power (FRAP) method and 29.7–227.9 mg/mL (IC50) based on 2,2-diphenyl-1-picrylhydrazyl (DPPH) method [52, 53].

The predominant phenolic compound in the leaves and stalks is chlorogenic acid (around 2295 and 473 mg/100 g dw), while the edible part of the fruit exhibited lesser amounts. The seed is also a beneficial source of bioactive compounds and dietary nutrients [54]. The sweet cherry seed oil has more than 80% unsaturated fatty acids (oleic and linoleic acid) [55]. Atik et al. [56] identified β-sitosterol as the major sterol in sweet cherry seed oil (88.9%), followed by campesterol (3.12%), Δ-7-stigmasterol (2.48%), Δ-5-avenasterol (2.12%), and sitostanol (1.42%). In the same study, benzoic acid was demonstrated as the major phenolic compound (58.8 mg/Kg). The sweet cherry seed pomace, after oil extraction, may still have useful compounds to be recovered. For instance, Yüksekkaya et al. [57] reported that cherry seed contains considerable amounts of phenolics, mainly vanillic acid (15.4 mg/Kg), catechin (6.3 mg/Kg), and resveratrol (3.7 mg/Kg).

Polyphenols such as flavonols, flavan-3-ols, anthocyanins, hydroxycinnamic acids, and hydroxybenzoic acids are present in cherry skin [58]. This by-product contains 1.1 mg/g of total phenols with an antioxidant capacity of 5.2 µmol Trolox equivalent/g [59]. The major anthocyanins of sweet cherry skin extract are cyanidin 3-rutinoside (109 mg/100 g), followed by cyanidin 3-glucoside (83.7 mg/100 g), pelargonidin-3-rutinoside (32.9 mg/100 g), peonidin 3-rutinoside (8.41 mg/100 g), and peonidin 3-glucoside (4.97 mg/100 g) [60].

Figure 1. Most abundant phenolic compounds in grape and cherry by-products

Current Emerging Valorization Strategies

Grape by-Products

Currently, the wine-making industry generates significant yearly by-products. Approximately, one ton of grape pomace (GP) contains 225 kg grape seeds, 249 kg stalks, and 425 kg grape skins [21]. These residues, abundant in functional and health-related compounds, are now being recovered using advanced technologies. This emerging strategy addresses the limitations of conventional methods [61].

Traditionally, phenolic compounds are extracted with organic solvents like acetone, methanol, and ethanol [13]. Despite their low cost and high extraction efficiency, these solvents pose challenges due to flammability, toxicity, and improper disposal practices However, ethanol is generally recognized as a safe (GRAS) solvent and still therefore continuously used for the extraction of various metabolites which can further be safely incorporated in food and nutraceutical products [62].

SLE is still the widespread method reported for the extraction of bioactive compounds from grape by-products [63, 64]. The drying or freeze-drying process, grinding, or other pre-treatments of the sample with the respective solvent are usually considered before the extraction step [65].

In recent years, there is a growing demand for plant-based green products prepared using novel sustainable technologies and/or with safe solvents [66]. In recent years, the development of non-conventional techniques, such as UAE, SWE, MAE, PEF, ASE, and extraction with natural deep eutectic solvents (NaDES), for the recovery of the bioactive fraction of agro-industrial by-products has led to significant reductions in the use of organic solvents. These techniques are more efficient, require less extraction time, cost less, and provide a higher purity of the extracted molecules [67], and it have been used to recover valuable biological chemicals from grape by-products as reported in Table 1.

Table 1 Application of green technologies for the extraction of phenolic compounds from grape pomace

By-product	Extraction system	Main bioactive compounds	Reference
Skin	• Maceration • UAE	• Total monomeric anthocyanins • Total phenolic content	[68]
Seeds	• UAE	• Fatty acid (palmitic, stearic, oleic, linoleic, linolenic, arachidic, and behenic acid; saturated fatty acid, monosaturated fatty acids, polyunsaturated fatty acids, and unsaturated fatty acids) • α- tocopherol • Total phenolic content	[69]
Syrah grape skin	• UAE	• Anthocyanins (delfinidin-3-O-glucoside, cianidin-3-O-glucoside, petunidin-3-O-glucoside, peonidin-3-O-glucoside malvidin-3-O-glucoside) • Flavan-3-ols (catechin, epicatechin, rutin) • Hydroxynnamic (p-coumaric) • Hydroxybenzoic (gallic acid, syringic acid, vanillic acid, protocatechuic acid) • Flavonols (quercetin, myricetin, kaempferol, isorhamnetin) • Tannins (ellagic acid)	[70]
Sardasht black grape residues	• UAE	• Total anthocyanins • Total phenolic content	[71]
Fresh and freeze-dried *Tannat* grape pomace (skin, seeds, stems)	• UAE	• Total anthocyanins • Total phenolic content	[65]
Peels	• UAE	• Flavan-3-ols (catechin, rutin) • Hydroxynnamic (p-coumaric) • Anthocyanins (cyanidin chlorid) • Flavonols (myricetin) • Hydroxybenzoic (gallic acid) • Total anthocyanins • Total phenolic content • Total flavonoids	[72]
Pomace (skin, seeds)	• UAE	• Total phenolic content • Total flavonols	[73]
Stalks	• UAE combined with NaDES	• Total phenolic content	[22•]
Integral grape	• MAE	• Hydroxybenzoic (vanillic, syringic, gallic acid, protocatechuic Acid) • Hydroxycinnamic (caffeic acid, caftaric acid, ferulic, p-coumaric) • Flavonols (Q-3-glucoside, Q-3-glucuronide, Q-3-rhamnoside, quercetin-3-galactoside, quercetin-3-glucopyranoside, quercetin-3-rhamnoside, quercetin-3-rutinoside, kaempferol, quercetin, myricetin) • Flavan-3-ols (catechin, epicatechin, epicatechin gallate, gallocatechin procyanidin A2, procyanidin B1, procyanidin B2, procyanidin B3, procyanidin C1) • Stilbenes (trans-piceid, trans-resveratrol, trans-e-viniferin)	[74]
Pomace	• UAE • MAE	• Total phenolic content	[11]
Seeds and skin	• MAE	Flavan-3ols (catechin, epicatechin); phenolic acid (caftaric acid); flavonols (quercetin, quercetin-3-glucoside, kaempferol-3-glucoside, dihydrokaempferol-glycoside)	[75]
Stem	• PEF • UAE	• Total phenolic content • Volatile compounds (phenylethyl alcohol; benzene; 1-methoxy-4-methyl; n-hexadecanoic acid; 1,14-tetradecanedoil; 4-dodecanol; hexanedoiic acid, bis(2-ethylhexyl) ester; 14- heptadecenal; 16- heptadecenal)	[76]
Pomace	• PEF	• Total phenolic content (gallic acid, chlorogenic acid, caffeic acid, epicatechin, p-coumaric acid, naringin, rutin and phlorizin) • Total flavonols • Total tannin content • Total anthocyanin content	[77]
Exhausted grape marc	• PEF • SFE	• Quercetin 3-*O*-(6- acetyl-glucoside); kaempferol 3-*O*-galactoside; 6-hydroxyluteolin-7-*O*-rhamnoside; quercetin; petunidin 3-*O*-arabinoside; (+,-) catechin; (+,-)-epicatechin; kaempferol; trans- resveratrol, cis- resveratrol	[78]
Chardonnay grape marc	• MAE	• Total phenolic content	[12]

Abbreviations: *UAE* ultrasound-assisted extraction, *NaDES* natural deep eutectic solvent, *MAE* microwave-assisted extraction, *PEF* pulsed electric field, *SFE* supercritical fluid extraction

The application of ultrasound (US) radiation is a convenient alternative to the conventional dynamic SLE as it increases the extraction efficiency due to the cavitation phenomenon that enhances the mass transfer of the process [13, 79••].

Cavitation involves the formation, growth, and collapse of micro-bubbles, disrupting vegetable tissue and enhancing the release of extractable compounds. This improves mass transfer by allowing better solvent penetration. UAE rapidly extracts bioactive components at low temperatures, conserving the functionality of these compounds and reducing energy and solvent usage [80, 81]. Optimizing parameters such as frequency, power, duty cycle, temperature, time, solvent type, and liquid–solid ratio for each specific by-product is crucial [13].

US power was investigated (50, 70, and 100 W) and different extraction times (15, 40, and 65 min) combined with an alcoholic solution for the extraction of total monomeric anthocyanins and total phenolic content from grape skins of the Tannat variety. The highest total phenolic content was obtained using UAE with 75 W power for 40 min. Extraction with 100 W for 65 min allowed extracts with greater antioxidant capacity by 2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) and oxygen radical absorbance capacity using fluorescein (ORAC-FL). These results seem to indicate that there are antioxidant compounds that are more retained in the cell wall or linked to the fiber. Therefore, a greater energy input seems to be necessary to achieve the release of these compounds with greater antioxidant capacity from the skin [68].

Moreover, employing solvent mixtures containing ethanol and water in UAE enhances extraction efficiency, offering a cost-effective and sustainable approach. Mechanical vibrations from the ultrasound probe increase the solid-solvent contact area, aiding solvent penetration, as demonstrated by Milanovic et al. [69]. This research group achieved higher yields (74 and 85%) recovering oil from various grape seed varieties using UAE compared to cold pressing. The resulting oil exhibited increased α-tocopherol content, improved antioxidant capacity, and enhanced oxidative stability. González-Centeno et al. optimized UAE conditions for grape pomace (GP) by adjusting frequency, time, and power. The best outcomes were achieved with the highest power and longest time at the lowest frequency [73].

Even if an increase in temperature (20 and 50 °C) does not lead to a necessary increase in yield, it must carefully be evaluated during the UAE process, as elevated temperatures can degrade the thermolabile compounds and mitigate cavitacional phenomena. Conversely, during the UAE, an increase in the power is usually linked to higher extraction efficiency [82]. This can be explained by the increase in the effect of violent collapse of cavitation bubbles due to the increase in power leading to a rupture of the cell wall as reported by Kianoush et al. [83]. In the latter study conducted on unripe GP, scanning electron microscopy indicated disintegrated regions attributed to sonication effects during the extraction process.

Mazza et al. [70] observed good recovery of the TPC of the extracts obtained by UAE ranged from 6485 to 11732 mg of GAE/100 g of grape skin. The antioxidant capacity measured by ORAC and ABTS methods ranged from 230 to 516 µmol Trolox/g and from 442 to 939 µmol Trolox/g, respectively. The extraction yield was satisfactory, with a recovery of 59% of the quantified phenolic compounds, with only 3 min of processing. The US was considered a suitable method as compared to conventional extraction (CE), improving the extraction of phenolic acids and facilitating their release.

Another advantage of the US is their less time-consuming character, Matini et al. [71] achieved significantly higher efficiency using UAE in just 15 min, in contrast to the CEM, which required 24 h for Sardasht black grape residue. Under these conditions, the phenolic compounds, anthocyanins, and antioxidant activity obtained by the CE and UAE method were 96.779 mg GAE/100 mL, 118.345 mg/L, 55.49% and 114.115 mg GAE/100 mL, 121.645 mg/L, 64.89%, respectively.

The influence of different US powers was evaluated in the valorization of GP that has undergone different pre-treatment. When evaluating the freeze-dried pomace extract, no significant differences were observed between applying 50 or 100 W of US power. While it is true that increasing the power of US can enhance the extraction of bio-actives by disrupting GP, its effect is less pronounced when dealing with an already disrupted matrix, as is the case with ground freeze-dried pomace [65].

However, when the power was evaluated in fresh pomace, more energy is required to break skin and seed structures to extract the biocomponents [65]. Whereas the linear effect of power on the yield of phenolic, total flavonoid, and total antioxidant has been reported by Gonzalez-Centeno et al. [73].

The solid to liquid ratio is also a key parameter to achieve higher extraction efficiencies [70]. According to the mass transfer principle, the diffusion rate of compounds from the sample to the solvent is directly proportional to the concentration gradient, which increases with the increase in the extractive solution volume [11].

In addition to conventional solvents generally employed for US-assisted grape valorization, a new class of environmentally friendly alternatives has emerged, namely NaDES. For instance, UAE (40 kHz, 200 W) demonstrated to be an excellent technique for grape stem delignification when combined with NaDES (choline chloride (ChCl) and levulinic acid (LeV), 1:2 molar ratio). The developed approach by Salgado-Ramos et al. (2022) enabled the isolation of more than 70% of the lignin fraction [22•].

While UAE is widely adopted in the food sector for its ease of use, rapid extraction capabilities, and ability to enhance extract volume, there are relatively few studies documenting its application in valorizing winemaking by-products at pilot scale [79••].

In this context, noteworthy is the pilot scale US-assisted flow-mode extraction procedure using 2 kg of grape stalks

that was carried out in a 15 L US reactor (45 min). The pivotal components of this process were the downstream operations: a semi-industrial decanter unit followed by a filtration step using a pilot skid membrane. These units facilitated the production of a final polyphenols-enriched stream concentrated up to 355.91%, as evidenced by its antioxidant activity and TPC. Thus, further investigations in this field are of urgent need for exploring the potential use of US technology at industrial scale for the recovery of the bioactive fraction of grape by-products [79••].

MAE is one of the most widely used green extraction techniques for recovering natural compounds from agricultural and agro-food matrices [84]. This technique exploits the interaction of microwave (MW) radiation with the polar and ionic molecules present in the cells, resulting in direct dielectric heating. The increase in temperature and pressure inside the cells promotes the physical rupture of the membrane and cell wall, facilitating the release of metabolites [85]. Therefore, MAE allowed shorter extraction times and solvent consumption, as well as greater extraction yields and selectivity in relation to the target molecules. However, extended exposure to high-potency microwaves might cause the degradation of phenolic compounds, which is regarded as the main disadvantage of MAE [12, 86].

Moreover, recently, this technology has been applied to grape by-product valorization with promising results [11, 87, 88]. For instance, MAE as a GP pretreatment and documented a 57% improvement in polyphenol extraction efficiency from this matrix [89]. This method, when applied to GP, proved to be efficient in terms of recovering more phenolics in less time [90] and saving up to 83% of the diffusion time [91].

The MAE method requires the evaluation of different parameters such as solvent selection, solvent/solid ratio, applied power and temperature, and extraction periods [74]. Individual or combined effects on extract yield and composition are regarded as critical elements in optimizing the MAE process [12, 13].

Azaroual et al. [75] evaluated the main parameters that influence MAE extraction efficiency. They concluded that the best conditions were 65% methanol in water as an optimum extraction solvent, 0.5 g of grape skin or seed in a volume of 25 mL of solvent, a power of 500 W with the maximum stirring speed (100%), and an extraction time of 5 min. The phenolic compounds proved to be stable against degradation in the optimized extraction conditions. The resulting repeatability and the intermediate precision of the optimized method showed a relative standard deviation below 7%. This newly optimized protocol applied on Napoleon grape allowed the determination of a plethora of antioxidant compounds in the resulting extract: catechin (453.2 mg kg^{-1}), epicatechin (306.3 mg kg^{-1}), caftaric acid (22.37 mg caffeic acid equivalents kg^{-1}), dihydrokaempferol-glycoside (11.13 mg kaempferol equivalents kg^{-1}), quercetin (18.28 mg kg^{-1}), quercetin-3-glucoside (20.09 mg quercetin equivalents kg^{-1}), and kaempferol-3-glucoside (11.10 mg kaempferol equivalents kg^{-1}).

Concerning the exploitation of more sustainable solvents, Neto et al. [92] combined different NaDES with MAE in the extraction of proanthocyanidins from GP. The combination of choline chloride with lactic acid was shown to be the most effective combination for proanthocyanidins extraction yield, and despite the occurrence of some de-polymerization, also leaded to achieve the highest mean degree of polymerization. Additionally, the combination with MAE enabled the process to be completed in 3.56 min, resulting in a considerably reduced extraction time. The efficiency, selectivity, and speed of obtaining organic compounds are desired characteristics found in the MAE, and studies have shown its implementation and operational capacity [11].

Treatment with PEF is considered an emerging non-thermal, ecological, and economical technology to inactivate microorganisms and improve mass transfer for SLE of bioactive compounds (anthocyanins, polyphenols, and pigments) from plant matrices and by-products [18, 93, 94]. PEF can minimize the degradation of heat sensitive compounds, using a moderate electric field (500 and 1000 V/cm; for 10^{-4} to 10^{-2} s) [84]. PEF technology has been used to recover antioxidant compounds in agro-industrial and winemaking by-products [78, 95, 96]. However, the concentration of recovered antioxidant compounds is variable and depends on the matrix and PEF treatment conditions applied (room temperature 23 ± 1 °C, for less than 1 s) [97], with the main advantage of PEF is related to its selectivity and non-thermal mechanism [94].

PEF has been shown to be effective in extracting natural components from GP, seeds, and vine sprouts. The results showed high extraction yields of polyphenols, anthocyanins, and proteins [97]. The PEF application (1 to 7 kV/cm) as a pretreatment before the maceration step, significantly increased the extraction rate of TA and TPC from red cultivars, reducing the maceration time and increasing the color intensity, throughout the winemaking process [98, 99].

PEF was used to value the oily fraction of wine and GP, with this good yield was found (81.8 g/Kg) in addition to having the possibility of more selective extraction (phenolic acids and trans-resveratrol) compared to pressing cold processing of grape grains and this in turn had a yield of 67.1 g/Kg and extracted significantly higher concentrations of tocochromanols, hydrophilic antioxidants, and the main fatty acid, linoleic acid [95].

Valorization of Cherry Bio-Products

Even though cherry is mainly commercialized as fresh fruit, a considerable quantity is industrially processed as frozen, jam, jelly, canned, brined, juice, wine, dried forms, and alcoholic beverages [100, 101]. After its processing, a large amount of solid waste is therefore generated [52].

Although cherry waste contains significant bioactive metabolites, research on this topic has been limited. Solid–liquid extraction (SLE), liquid–liquid extraction (LLE), and Soxhlet extraction (SE) are the most used conventional techniques for isolating bioactive compounds from these by-products [87]. In these processes, solvent selection (ethanol, acetone, methanol, hexane, ethyl acetate, and water) has remarkable importance as it significantly affects the yield and extract composition [102].

In recent years, process intensification techniques such as MAE, UAE, ASE, and SWE, among others, have been used to recover valuable biological chemicals from cherry by-products as reported in Table 2.

ASE is an automated rapid extraction technique that utilizes common solvents at elevated temperature and pressure and thereby increases the efficiency of extraction of organic compounds from solid and semisolid matrices. The higher temperatures increased the mass transfer and the extraction rates, leading to shorter extraction times and lower amount of solvents than conventional methods [16].

Agulló-Chazarra et al. [15] conducted a study that investigated different extraction methods for the recovery of bioactive compounds with antiaging properties. The extractions were performed in the presence of an alcoholic solution consisting of ethanol: water (1:1 v/v), while the extraction times were 60 and 30 min for ASE and SFE, respectively. A total of 57 compounds were identified from different families: organic acids, phenolic acids, and derivatives; flavonoids and derivatives; fatty acid derivatives and terpenes. Moreover, flavonoids were more abundant in the ASE extract (65%), followed by fatty acid derivatives (39%) and terpenes (18%), while nonpolar compounds were better extracted by SFE. ASE thus resulted more efficient than SFE at removing a wide range of compounds with varying polarity, with phenols being the most abundant, while SFE afforded a greater number of fatty acids and derivatives. Chemical analysis of the extracts identified 22 compounds that were discovered for the first time in *P. avium* stems [103].

MAE demonstrated to be effective in the extraction of hydroxycinnamic acids, flavan-3-ols, flavonols, and anthocyanins as it used less time of extraction when compared with conventional methods, affording higher yields (17.03% and 12–13%) [105]. The MAE extracts were analyzed by HPLC–DAD, showing highest amounts of anthocyanins, hydroxycinnamic acid, flavonols, and flavan-3-ols (3.82; 2.85; 0.32; 0.03 mg/g dry extract) when compared with conventional method. In another study, the authors concluded that MAE afforded highest TPC (275.31 GAE/100 g fresh weight (fw)) and highest antioxidant activity (89.9%), while other techniques, such as high hydrostatic pressure (HHP) and UAE provided a TPC of 227.51 and 239.84 mg GAE/100 g fw) [106]. Other studies found that the phenolic content after MAE was significantly influenced by solvent concentration, extraction time, and MW power [109].

Another MAE process that showed great potential includes the application of pressure, allowing extraction to be carried out under subcritical conditions. Extraction with pressurized liquid leads to a change in the properties of the solvent by reducing its viscosity, surface tension, and polarity, increasing the solubility of the analyte in the medium [17]. Various solvents can be used in these conditions, but water holds more promising advantages. Recently, Soares et al. [17] investigated and compare the potential of MAE and SWE against the maceration for the evaluation of the phenolic profile and bioactivity of cherry seed and stem. The results of this work show that SWE allowed the highest total amount of recovered polyphenolic compounds (1390 mg/100 g), followed by MAE (1090 mg/100 g) and CEM (184 mg/100 g). White and black cherry skin presented similar phenolic profiles, with the highest total amount for SWE, followed by MAE. Gallic acid was the most abundant antioxidant compound (945–809 mg/100 g dw), while other phenolics identified in these extracts were catechin, vanillic acid, caffeic acid, ferulic acid, and sinapic acid. Epicatechin and naringin were detected only in SWE, while p-coumaric acid was detected in MAE and CEM. Despite the fact that phenolic acids and derivatives are present at higher concentrations in the extract obtained by SWE, the cost of using high-pressure techniques must be considered against the advantages observed to see if such an extraction method is economically viable.

Among process intensification technologies, UAE has also been demonstrated to significantly improve the recovery of phenolic compounds from cherry by-products, compared with CEM. Different extraction time (5, 10, and 15 min) using 24 kHz, 400 W, and 100% of amplitude and 80% (v/v) methanol: water was evaluated to perform the extraction of phenolic compounds from sour cherry pomace. Compared to SLE processes alone, UAE afforded higher total phenolics content after 15 min of ultrasonic cavitation [106].

Although 80% aqueous methanol is the most suitable solvent for extracting phenolics from CP (TPC 239.84 mg GAE/100 g fw), other studies have shown that ethanol/water mixtures can also be very efficient [45]. Milea et al. [100] evaluated the anthocyanin recovery from cherry skin, combining SLE (ethanol 70%) with UAE (40 kHz, 100W) and the extract for TPC; TFC and antioxidant activity were 445.93 mg GAE/g dw, 152.22 mg catechin equivalent (CE)/g dw, and 10.60 mmol Trolox/g dw, respectively.

Phytochemical composition of cherry stem extracts obtained through CEM (70 °C, 20 min) and UAE (40 kHz) was assessed using HPLC–DAD. The presence of neochlorogenic and chlorogenic acids, p-coumaric and

Table 2 Application of green technologies for the extraction of phenolic compounds from cherry pomace

By-product cherry	Extraction system	Main bioactive compounds	Reference
Stem	• Maceration • UAE	• Hydroxycinnamic acid (*p*-coumaric acid, cholorogenic acid, neochlorogenic acid, ferulic acid, hydroxycynnamic acid derivative, *p*-coumaroylquinic acid • Flavan-3-ol, (+)-catechin, (-)-epicatechin • Flavonol (apigenin, kaempferol-3-*O*-rutinoside, quercetin-3-*O*-rutinoside) • Flavanone (sakuranetin, isosakuranetin)	[50]
Stem	• SWE	• 2-furancarboxylic • 2-butanedionic acid • Pyrrole-2-carboxylic • 2-isopropyl-3-hydroxy butenoic acids • Hydroxyquinol	[52]
Stem	• Solvent extraction	• Sinapic acid • Quercetin • Caffeic acid • Catechin	[51]
Stem	• ASE • SFE • SWE	• p-coumaric acid ohexoside (SFE and ASE) • Quercetin-rutinosideglucoside (SWE) • Caffeic acid hexoside (SFE and SWE) • Rutin (SFE and SWE); epicatechin-O-glucuronide (ASE, SFE and SWE)	[15]
Stem	• ASE • SFE	• Proanthocyanidin • *p*-coumaric acid • Catechin • Linolenic acid • Hydroxy-octadecanoic acid	[103]
Stem	• SWE	• Benzoic acid • Cinnamaldehyde, 4-hydroxy-3-methoxy • Cinnamic acid • Catechol	[52]
Seed	• UAE • MAE combined with aqueous enzymatic extraction	• Linoleic acid • Oleic acid • Palmitic • β-sitosterol • Campesterol	[55]
Leaf	• SFE-CO_2 • Solvent extraction	• Hydroxycinnamic acids • Sugars • Alcohols	[104]
Cherry (without leaf and pits)	• MAE • UAE	• Hydroxycinnamic acids (3-Ca_eoylquinic acid; 5-Ca_eoylquinic acid; 4-Ca_eoylquinic acid; 4,5-dica_eoylquinic acid; caffeic acid and p-coumaric acid) • Flavan-3-ols ((-)-epicatechin-3-gallate) • Flavonols (quercetin-3-rutinoside; kaempferol-3-glucoside; quercetin) • Anthocyanins (cyanidin-3-rutinoside; cyanidin-3-glucoside; peonidin-3-rutinoside; pelargonidin-3-rutinoside)	[105]
Sour cherry pomace	• MAE • HHP • UAE	• Neo-chlorogenic acid and cyanidin-3-glucosylrutinoside • Total antioxidant activity	[106]
Sour cherry	• UAE	• Total phenols content • Total flavonoids content • Total monomeric anthocyanins content	[107]
Cherry pomace	• MAE combined with NaDES	• Anthocyanins (cyanidin 3- sophoroside; cyanidin 3- glucosylrutinoside; cyanidin 3- rutinoside) • Flavonoids (quercétines 3- glucoside; quercetin 3- rutinoside; quercetin glycoside isorhamnetin 3- rutinoside) • Phenolic acids (3-*O*-caffeoylquinic acid; 5-*O*-caffeoylquinic acid; *p*-coumaroylquinic acid derivative)	[108•]

Table 2 (continued)

By-product cherry	Extraction system	Main bioactive compounds	Reference
White and black cherries (seeds, stem)	• SWE • MAE • Maceration (20 and 60 °C)	• Hydroxybenzoic acids (gallic acid; protocatechuic acid; vanillic acid; syringic acid; β-resorcylic acid) • Flavan-3-ols ((+)-catechin; (−)-epicatechin • Hydroxycinnamic acids (chlorogenic acid caffeic acid; *p*-coumaric acid; ferulic acid; sinapic acid; cinnamic acid) • Flavanones (naringenin) • Flavonols (quercetin; kaempferol; rutin)	[17]
Skin	• MAE	• Total phenolic contents • Anthocyanins	[109]
Skin	• UAE	• Monomeric anthocyanins content • Total polyphenolic content • Total flavonoids content	[100]

Abbreviations: *UAE* ultrasound-assisted extraction, *SWE* subcritical water extraction, *ASE* accelerated solvent extraction, *SFE* supercritical fluid extraction, *MAE* microwave-assisted extraction, *SFE-CO_2* supercritical fluid extraction with dioxide of carbon, *HHP* high hydrostatic pressure, *NaDES* natural deep eutectic solvent

p-coumaroylquinic acids, ferulic acid, and (+) catechin and (+) epicatechin was detected in US extracts. These resulted richer in phenolic compounds (2377 μg GAE/g dw) than those obtained with CEM (2019.3 μg GAE/g dw). The increment of pressure during sonication causes the decomposition of radicals and the hydroxylation of aromatic rings, increasing their solubility and movement from cells toward the solvents and thus enhancing their recovery [50].

Hu et al. [55] found that the combined ultrasound- and microwave-assisted aqueous enzymatic extraction (UMAAEE) did not induce significant differences in the fatty acid compositions of cheery seed oil compared to SE. However, the bioactive composition after UMAAEE was higher, including α-tocopherol, β-carotene, phospholipids, phytosterols, β-sitosterol, stigmasterol, campesterol, and phytosterol. TPC was also higher than after SE (78.85 mg GAE/Kg oil and 52.76 mg GAE/Kg oil, respectively). The reason for the recovery of more bioactive by UMAAEE than those found in oil after SE might be ascribed to the disruption of cellular structures due to the combined action of different process intensification technologies.

Finally, a process using three different enzymes to recover non-extractable bioactive polyphenols from the solid remaining sweet cherry pomace was tested, and it can be highlighted that this residue may still contain valuable antioxidant compounds, even if the pomace has been subjected to a primary extraction of polyphenol [59].

NaDES are considered excellent greener alternatives to conventional organic solvents, and they were synthesized and tested for CP extractions under non-conventional conditions (MAE and UAE) and with CEM method (40 °C for 30 min). Comparing different extraction techniques, it could be observed that CEM was superior to MAE and UAE when choline chloride: malic acid (ChCl:MalA) and choline chloride: urea (ChCl:U) were used for all polyphenol parameters (total phenol (3238.32 μg/g dw), total anthocyanins (2442.93 μg/g dw), total flavonoids (377.39 μg/g dw), and total phenolic acids (418.00 μg/g dw). However, when NaDES choline chloride: fructose (ChCl:Fru) was used in combination with UAE, the results were better than using MAE and CEM. A possible explanation is that ChCl:Fru solvent system is apparently more viscous than others, and US treatment enables the best mass transfer through a viscous system. However, ChCl:MalA resulted the most efficient solvent for cherry pomace reutilization, while MAE was the fastest solution [108•].

Conclusion and Perspectives

The cherry and grape industries are responsible for the annual production of substantial amounts of by-products, which cause significant environmental concerns. However, due to their richness in various functional and health-related metabolites, the valorization of these residues as a source of bioactive compounds for new food, nutraceutical, and pharmaceutical products has great potential. Aiming for more sustainable processes, several process intensification technologies have emerged in recent decades to extract bioactive compounds from these by-products, according to a circular economy approach. These techniques allow a considerable reduction in extraction time, greater mass transfer, lower energy consumption, volume of solvents, and energy. But, to evaluate the potential application of any extraction technique, pilot plants with larger-scale tests, using larger quantities of biomass and industrial production conditions, are essential to determine the economic viability and sustainability of the process. Optimizing the extraction method is

a critical step in obtaining high-value extracts, considering the complexity and chemical characteristics of agricultural by-products. Special attention must be paid to heat and mass transfer and the corresponding energy consumption.

Abbreviations GP: Grape pomace; CP: Cherry pomace; FAO: Food and Agriculture Organization; UAE: Ultrasound-assisted extraction; SWE: Subcritical water extraction; ASE: Accelerated solvent extraction; SFE: Supercritical fluid extraction; MAE: Microwave-assisted extraction; SFE-CO2: Supercritical fluid extraction with dioxide of carbon; HHP: High hydrostatic pressure; NaDES: Natural deep eutectic solvent; PEF: Pulsed electric fields; SLE: Solid liquid extraction; GAE: Gallic acid equivalent; GC-MS: Gas chromatography coupled with mass spectrometry; DW: Dry weight; FW: Fresh weight; CEM: Conventional extraction method; TPC: Total phenolic compounds; TA: Total anthocyanin; TF: Total flavonoid; ORAC-FL: Oxygen radical absorbance capacity using fluorescein; ABTS: 2,2′-Azinobis-(3-ethylbenzothiazoline-6-sulfonic acid); DPPH: 2,2-Diphenyl-1-picrylhydrazyl; FRAP: Ferric reducing antioxidant power; SE: Soxhlet extraction; LLE: Liquid-liquid extraction; HPLC-DAD: High-performance liquid chromatography with diode-array detection; GRAS: Generally recognized as safe; US: Ultrasound; MW: Microwave; CE: Catechin equivalent; UMAAEE: Ultrasound- and microwave-assisted aqueous enzymatic extraction; ChCl:LevA: Choline chloride (ChCl) and levulinic acid (LevA); ChCl:U: Choline chloride: urea; ChCl:MalA: Choline chloride: malic acid; ChCl:Fru: Choline chloride: fructose

Author Contributions M.V. wrote the main manuscript text, E.C.G., S.T and G.C. reviewed the manuscript. C.F and S.T. supervised the writing work.

Funding Open access funding provided by Università degli Studi di Torino within the CRUI-CARE Agreement.

Declarations

Human and Animal Rights and Informed Consent This article does not contain any studies with human or animal subjects performed by any of the authors.

Competing Interests The authors declare no competing interests.

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Papers of particular interest, published recently, have been highlighted as:

- Of importance

•• Of major importance

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