Crucial Challenges in the Development of Green Extraction Technologies to Obtain Antioxidant Bioactive Compounds from Agro-industrial By–products

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Review
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Exploitation of agro-industrial by-products represents an important source of bioactive compounds that can be used both directly as ingredients and for the production of functional ingredients. Among these compounds, polyphenols are capable of strengthening endogenous antioxidant defences in human tissues, preventing cardiovascular and neurodegenerative diseases. The present paper aims to evaluate and review various green extraction technologies for a cheap, fast, eco-friendly procedure to obtain these bioactive molecules. Several physicochemical approaches can be used with the aim of optimizing the use of energy, solvents, and pressure; among them are ultrasound-assisted extraction, subcritical and supercritical fluid extraction, extraction with neoteric solvents (ionic liquids, deep eutectic solvents, and natural deep eutectic solvents), microwave-assisted extraction, pressurized liquid extraction, pulsed electric field, multi-frequency multimode modulated technology, rapid solid liquid dynamic extraction, and enzyme-assisted extraction. The challenges and future work regarding the development of these green products for the commercial markets were comprehensively evaluated.

Keywords:
agro-industrial by-products, green extraction technologies, antioxidant activity, bioactive compounds, polyphenols, food industry

Introduction

The food industry is increasingly globalized, and its constant search for new goods, technologies, services, and cultural practices runs in parallel with several environmental concerns. In fact, it is thought that, due to industrial processing, 30–50 % of agricultural foodstuffs are discarded as agro-industrial by-products, and food by-products are responsible for an estimated 8 % of greenhouse gas emissions.[1,2] Continued growth in the agro-industrial sector has knock effects on the quantity of waste produced in both the retail and consumption of products: this has led international agencies and authorities to search for common strategies to manage the growth of agricultural food processing by-products.[3] On the other hand, agro-industrial by-products are key sources of potentially profitable bioactive compounds (BACs).[4,5] The pharmaceutical, cosmetics, and food industries have shown a great deal of interest in these compounds, especially in those with antioxidant activity.[6] Recovery and recycling are fundamental parts of the process of converting "waste to wealth", a typical approach of the circular economy: the most profitable and sustainable challenge might be the recovery of beneficial compounds and the contemporaneous reduction of the amount of waste generated.

Extraction of natural origin compounds from the agro-industrial by-products is a well-established procedure for several production chains, such as oils, juices, and wine. Techniques are being updated constantly to overcome problems of recovery yield and separation of target compounds from the agro-industrial waste. Over the past ten years, extraction processes have been developing in the search for green solvents and evolving green practices.[7] In fact, the idea of green chemistry has immense importance in manufacturing processes to diminish or remove generation and use of dangerous substances, as well as in helping to develop green approach.[8] The use of GRAS (generally recognized as safe) or Safer Choice Standard solvents should guarantee environmentally friendly processes.[7,9]

Thus, a crucial challenge in agro-industrial by-product exploitation is the development of sustainable and green extraction technologies (GETs). This review focuses on recent approaches utilized in agro-industrial by-product exploitation using

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mainly green techniques. The primary focus of this review is on the most used green extraction procedures and the development of some promising techniques. Furthermore, current research about specific agro-industrial by-products and bioactive compounds recovery with antioxidant activity are discussed, as well as the extraction technologies and conditions used.

General aspects and properties of green extraction techniques

Innovative (non-conventional) solid-liquid GETs have been introduced in the agro-industrial field with the aim of overcoming the limitations of conventional extractions. In fact, counter-current extraction, distillation, extraction through Soxhlet, maceration, percolation, and squeezing can have several negative aspects, such as (1) impact on the thermal decomposition of thermolabile compounds, (2) a large amount of expensive solvents, (3) long extraction times, (4) a low selectivity of extraction, and (5) a high solvent evaporation rate during the process.[10] GETs are defined as methods which decrease energy consumption, and permit the use of renewable natural products and alternative solvents. Moreover, GETs guarantee high quality and safe extract/product.[11] According to Chemat *et al.*,[11] GETs possess 6 main principles: (1) use of renewable plant sources, (2) use of water or agro-solvents as principal alternative solvents, (3) reduced energy consumption due to innovative technologies or energy upturn, (4) replacement of waste with creation co-products including agro- and bio-refining industry, (5) favour controlled, robust, and harmless processes, as well as reduced unit operations, (6) purpose to obtain biodegradable and non-denatured extract free of contaminants.

The most common GETs found in literature are ultrasound-assisted extraction (UAE,[12–33] including pulsed ultrasound-assisted extraction, PUAE),[34–36] supercritical fluid extraction (SFE),[15,23,37–45] subcritical water extraction (SWE, and the more general subcritical fluid extraction, SbFE),[46–55] extraction with deep eutectic solvents (DESs,[56–60] including natural deep eutectic solvents, NaDESs),[61–66] microwave-assisted extraction (MAE, including the solvent-free microwave extraction, SFME)[14,17,20,28,67–72] and pressurized liquid extraction (PLE, including accelerated solvent extraction, ASE, and enhanced solvent extraction, ESE).[20,24,42,73–77] Furthermore, a few other, less common, green extraction methods have been used by scientists in the last decade to extract BACs from agro-industrial by-products; namely, rapid solid liquid dynamic extraction (RSLDE),[78] pulsed electric fields (PEFs) extraction,[79–82] multi-frequency multimode modulated technology (MMMT),[83,84] enzyme-assisted extraction (EAE),[85–88] and ionic liquid extraction (ILE).[89,90] All studies regarding these green processes and their advantages and disadvantages are reported in Table 1. Generally, all these GETs display cheap, fast, and eco-friendly features, and the most common advantages are: reduced energy consumption, safe and renewable products, high quality extract, low environmental impact, selectivity, and better isolation.[91]

With the purpose of diminishing the use of organic, toxic solvents, which have unfavourable effects on food by-products quality and safety, the interest in GETs has been significantly amplified. Modern extraction methods are aimed at optimizing the use of energy, solvents, and other physical properties like pressure, which could also be further improved through enzyme application.[92]

GET focused on use of energy

Regarding the energy used, among these technologies a well-known example would be UAE, also called sonication or ultrasonic extraction. This technology uses ultrasound waves (ca. 20 kHz to 100 MHz) that have an impact on molecules present in the solvent (liquid medium). The UAE can work in an incessant or pulsed (PUAE) way.[93] The UAE process creates a phenomenon of cavitation, in which fast changes of the pressure of the solvent molecules cause growth and collapse of the voids or air bubbles that create pores, thus accelerating the extraction process. Moreover, UAE can be applied in the food extraction industry, on both small and large scale.[14,94] Also, MMMT is a GET that uses UAE approach: in this method many synchronously exciting vibration modes are coupled with sub-harmonics and harmonics in liquid and solids containers. Furthermore, it is characterised by high intensity, uniform, and repeatable multimode vibrations. Hence, the cavitation process is improved, due to the lack of stationary and standing waves, that guarantees full agitation of the whole vibrating system.[84] Taking into account the importance of energy as a part of the extraction method, MAE is another green technology, based on microwave energy. This energy is delivered straight to materials via molecular interactions with the electromagnetic field (300 MHz to 300 GHz) through changes of electromagnetic energy into thermal energy, whilst the solid matrix, penetrated by the solvent via diffusion, dissolves upon reaching its concentration limits.[14,95] Furthermore, PEFs extraction is a method, which increases mass transfer during extraction by destroying structures of the membrane. Not only that, it is a non-thermal technique that reduces the degradation of the thermolabile compounds. The investigated material is placed between two electrodes, while

the pulse amplitude varies from 100–300 V cm^{-1} to 20–80 kV cm^{-1}. The treatment is performed at room temperature or slightly higher.[79,94]

GET focused on extraction solvent and pressure

Uses of non-hazardous and harmless solvents, like water, ethanol, and CO_2 have been applied to the extraction of various agro-industrial by-products, especially combined with pressure control. Two well-known and widely used GETs, which depend entirely on pressure, are SbFE and SFE. The first one, can use liquid water (SWE) at temperatures exceeding its boiling point (100 ºC), but remains under its critical temperature (374 ºC), due to the pressure. The idea of SWE is based on changes in water properties, like pH, density, polarity/dielectric constant, viscosity, and surface tension, caused by the elevated processes of the temperature in the subcritical region.[96] The second uses supercritical fluid (ScF) as the extraction solvent, which is fluid that has characteristics of both gases and liquids above its specific critical pressure and temperature. This extraction method is characterized by changes in pressure and temperature that convert the gas into ScF. The SFE method is composed of the solubilisation process of the chemical compounds occurring in the solid matrix, and separation of these molecules in the ScF. Due to the effects of pressure reduction and an increment of temperature, this solvent converts to a solvent-free extract.[94,97,98] Another GET where pressure plays an important role, is PLE, also called high-pressure solvent extraction, enhanced or accelerated solvent extraction, and accelerated fluid extraction. This process guarantees high penetration of the solvent in the matrix because elevated pressures maintain the solvent in liquid form above its boiling point.[94] Additionally, RSLDE focuses on a negative gradient of pressures between the interior of the material (high pressure) and the exterior of the solid matrix (low pressure). The changes in the pressure have an impact on the solid matrix following an active deed, in which a small quantity of material is extracted at each pressure and depression cycle. Specifically, due to the gradient pressure removal, the solvent runs out of the matrix very fast, transporting with it substances that are not chemically attached to the main structure of the matrix. Furthermore, these compounds are extracted with the force effect, and consequently, are able to be extracted in solvents with different polarity.[10]

Focusing our attention on the solvent used, neoteric solvents (NSs) are unconventional or structurally novel solvents. Generally, they are characterized by adjustable chemical and physical properties used for different applications. NSs such as DESs, ionic liquids (ILs), ILs with co-solvents, and CO_2-expanded ILs, can potentially replace conventional solvents due to their biocompatibility, low toxicity and recyclability.[99] ILE uses ILs (salts with melting point below 100 °C) that are a relatively new class of compounds. They are characterized by simple cationic (organic)-anionic (organic and inorganic) structure, which provides unparalleled and unusual properties.[100] ILs are non-volatile, due to low vapour pressure. Furthermore, they are highly polar, mixable with water and specific organic solvents, as well as being characterized by good solubility of inorganic and organic materials.[101] Many ILs have opposite properties when compared with supercritical CO_2, which is volatile, nonpolar, nonconducting, nonviscous, and unable to dissolve large, unsaturated compounds.[102,103] ILs can be divided into many subclasses, like IL-based surfactants (formed by the long alkyl chains with micellar properties when dissolved in water), room temperature ILs (melting point lower than room temperature), magnetic ILs (possess paramagnetic constituents in the anion or cation moiety), polymeric ILs (synthesized by IL monomers), and task-specific ILs.[102] Among all the NSs, DESs are gaining more attention with regards to the extraction of BACs from food processing by-products due to their high biodegradability when compared to the imidazolium-based ILs. In addition, DESs have great potential in developing extraction procedures that offer clean and highly energy-efficient processes. However, current study on the use of DESs for extraction purposes needs to better understand different aspects, like external factors (time, temperature, and solid:solvent ratio), solvent (pH, polarity, solubility and viscosity), and cytotoxicity to achieve eco-friendly processes.[102] DESs were developed to surmount the environmental problems caused by ILs. Despite similar properties to ILs, they are more stable and cost-competitive, and simpler to synthesize.[99] On the other hand, extraction with NaDESs is carried out by mixing two or more naturally occurring components (Brønsted Lowry acids and bases), which are able to interact between hydrogen bonds. Mixtures are composed of a hydrogen bond acceptor (HBA) with an electric charge and hydrogen bond donator (HBD). Furthermore, DESs can be divided into four main groups: Type I (organic salt and metal), Type II (organic salt and metal salt hydrate), Type III (organic acids and HBD), and Type IV (aluminium/zinc chloride and HBD).[102] In turn, NaDESs are classified into five groups based on their composition (HBA and HBD): amino-acid, ionic liquids, neutral-basic, neutral-acid and neutral.[102] The common components are choline chloride (ChCl), urea, organic acids (citric, lactic, and malic acid), and sugars (fructose, glucose, and sucrose).[57,63] Furthermore, it has been proven that the

Table 1 – *Advantages and disadvantages of green extraction techniques*

Green extraction techniques	Main process involved*	Advantages	Disadvantages	Ref.
Enzyme assisted extraction (EAE)		– energy efficient – easy to use – high extraction efficiency – safe and friendly working conditions – highly specific enzymes – an alternative method due to mild environmental conditions – small amount of undesired products – high bioactive yielding technology – high catalytic efficiency – efficacy of the natural products – no or small amounts use of harsh substances – no involvement of wasteful protection or deprotection stages – easy isolation and product recovery – process recyclability – removal of the needless components from cell walls	– difficult in terms of cost – difficult processing steps	92
Microwave-assisted extraction (MAE)	E / S	– energy efficient – high reproducibility – low solvent consumption	– not proper for extraction of volatile or non-polar compounds	10, 14, 94, 110
including: Solvent-free microwave extraction (SFME)	E	– compact procedures – enhanced purity of the final product – decreased thermal degradation – selective heating of vegetal material – reduced the usage of organic solvent	– tested samples must be thermostable	
Neoteric solvents (NSs) extraction	S	– negligible volatility – thermal stability – tunability	– high viscosity	57, 61, 63, 99, 100, 102, 114, 115
– Ionic liquid extraction (ILE)		– chemical stability – synthetic versatility – high conductivity – ideal alternative to volatile organic solvents – electrochemical stability – ability to permeate and modify biomass cell walls and tissue following to easy realise of compounds – outstanding solubility for organic, inorganic, and organometallic substances	– potential toxicity – the need to remove them from the environment – multi-stage synthesis – inherent high costs	
– Deep eutectic solvents (DESs) extraction		– low-cost – biodegradability – non-flammability – easier synthesis than in case of ILs – low lattice energy – low melting	– potential toxicity	
– Natural deep eutectic solvents (NaDESs) extraction		– low-cost – biodegradability – easy to use – time-saving – high miscibility – rare solvation ability – solute stabilization – broad range of polarity, – endless opportunities for the design of extraction approaches	– time-consuming solvent transfer operations – slow mass transfer in dissolutions or extractions	
Pressurized liquid extraction (PLE) a.k.a.: Enhanced solvent extraction (ESE) / Accelerated solvent extraction (ASE)	E/P/S	– low-cost – energy efficient – easy to use – time-saving – low solvents consumption – high extraction yield – avoidance of subsequent concentration steps – possibility of adjustment of selectivity parameters, frequent use of environmentally friendly extrahents (e.g. CO_2) – fully automated system – possibility of extraction of 24 samples in one cycle – use of liquids at high temperature under pressure	– high capital cost – large volume of solvent used – labour-consuming – no possibility of automation – usually requires solvents of high dielectric constant, cooling the extraction bomb and filtration of sample after extraction	10, 74, 116, 117

Green extraction techniques	Main process involved*	Advantages	Disadvantages	Ref.
Pulsed electric fields (PEFs) extraction	E	– energy efficient – time-saving – thermal stability – high extraction yield – non destructive – high selectivity – easy to scale up – avoidance of undesirable substances into the extraction liquid – reduced loss of thermosensitive bioactive compounds	– high equipment cost – dependence on medium composition (conductivity)	82, 118, 119
Rapid solid liquid dynamic extraction (RSLDE)	E/S	– low-cost – time-saving – whole process takes place in the order of hours		10
Subcritical fluid extraction (SbFE) including: Subcritical water extraction (SWE)	P/S	– low-cost – time-saving – non-flammable – high diffusion into the plant matrix – elevate mass-transfer properties – low-polar and non-polar compounds – easily available extraction solvent – easy equipment – unique solvation properties	– high temperatures following to undesirable reactions and toxic compounds – possibility of hydrolysis – degradation of labile compounds – possible low selectivity – necessity of use of high quality materials for equipment construction	8, 96
Supercritical fluid extraction (SFE)	P/S	– time-saving – high extraction yield in a single step – small amount of sample – reduced incidence of oxidation reactions – good preservation of labile compounds – selective – not require further cleaning – low viscosity – easily transferred at industrial scale – great versatility	– high-cost – lipophilic nature of CO_2 – low polarity of CO_2 – require support of small % of co-solvent – low effectiveness in extracting more polar compounds – high pressures – difficult design of extraction conditions – insoluble high polar substances	8, 37, 98, 99
Ultrasound-assisted extraction (UAE) including: Pulsed ultrasound assisted extraction (PUAE) and Multi-frequency multimode modulated technology (MMMT)	E	– low-cost – energy efficient – easy to use – time-saving – high extraction yield – small structural and molecular changes in material – low extraction temperatures – low solvent consumption – ability to break the cell walls extracting the intracellular liquids – good for extraction of thermolabile and unstable compounds – applied in the food extraction industry (both small and large scale)	– induce lipid oxidation – increasing temperature by cavitation – formation of free radicals by sonolysis – mechanical forces generated by shockwaves microstreaming – high power consumption – dicult to scale up	14, 36, 84, 94, 120

*Main process involved: E: Energy; P: pressure; S: solvent; TE: thermal energy

DES combined with unconventional and modern technologies (e.g. UAE, PLE, and MAE) has high extraction potential.[104]

GET focused on enzymatic approach

Another GET in use over the last decade is EAE. The practice of enzymes, particularly in biocatalysis of agro-industrial waste, is used as a technique focused on the hydrolytic impact of enzymes on the cell wall and membrane of components, as well as on the macromolecules inside the cell, which enable the release of the natural products.[85–88] Enzymes are very efficient and peculiar, and the enzymes that play the key role in this extraction method are α-amylase, cellulase, pectinase, and tannase.[85,94] The process of enzymatic extraction of polyphenols requires optimal pH, temperature, and time. As an example, application in enzymatic-catalysis of cellulase, pectinase and tannase allowed for extraction of ellagic acid, hesperetin, and naringenin from citrus residues.[85] It was observed that extraction time had very important impact on the extraction yield. The last two compounds reached the highest values after 24 h (decreased slightly at 30 h), while ellagic acid was the highest after 5 h of incubation and decreased up to 90 %. Furthermore, other compounds (naringin and hesperidin) significantly increased. These changes were due to simultaneous extraction and biotransformation during the enzymatic reaction. Possibly, the pectinase and cellulase hydrolysed most of the compounds in the citrus waste wall cell, which led to liberation of glycosylated phenolic compounds to be acted upon by tannase. In this instance, the tannase formed aglycon phenolics (naringenin and hesperetin).

Enzyme manufacture is a significant field in biotechnology, with global sales around 5 billion dollars yearly, and a growth rate of the large number of patents.[85] EAE has attracted many researchers especially due to its advantages: (1) high catalytic efficiency and preservation of original efficacy of the natural products; (2) high bioactive yielding; and (3) improvement in the transparency of the system by elimination of avoidable components from the cell wall.[92] Moreover, EAE can be an alternative method due to mild environmental conditions, without great quantity of unwanted products. Despite its advantages, EAE is not always applicable at an industrial scale, because enzyme behaviour is strictly limited by environmental conditions.[85–88,92]

Advantages and disadvantages of GET

According to the data reported in Table 1, it is not possible to indicate the best GET in terms of cost-effectiveness. First of all, therefore, all necessary information regarding the investigated agro-industrial by-product should be collected, with the aim of finding the most appropriate extraction technique. In this way, it will be possible both to maximize the extraction of the target bioactive molecules, and to recover other components that may have beneficial aspects. For the sake of the environment, emphasis must be placed on development connected to bioeconomy and circular economics. Concerning new designs of processes, researchers propose novel green solvents and the use of less extreme conditions of pressure and temperature. The ecological character and costs of the extraction are determined by the source of BACs, solvent, temperature, processing time, and the occurrence aided extraction modes, like microwave or ultrasound.[99]

Gallego *et al.*[105] observed that SbFE and SFE technologies have a lot of potential for effective extraction of BACs from many different plant matrices. However, some improvements in different aspects (e.g., chemical composition, natural materials), have to be taken into consideration. Water is regarded as the cleanest solvent. Yet, corrosion issues still do not allow for its application at industrial scale.[99] In turn, CO_2 is GRAS, and it is considered as thermodynamically stable, non-flammable, non-mutagenic, non-carcinogenic, and non-toxic.[99] However, its fruitful application in SFE depends on the exhaustive understanding of SFE and experimental strategy methods.[99] Comparing SFE-CO_2 with UAE, the first better isolates essential oils and other lipophilic compounds, but is less effective for extraction of polar compounds (e.g., polyphenols). That is why co-solvents are often used to improve the recovery of polar and medium polar molecules. However, despite many advantages, SFE has some limiting aspects, like complex industrial equipment and high processing expenditures.[99]

Upcoming developments aiming to obtain more efficient extraction, take account of integration of techniques within the same process. An example could be the use of a procedure of enzyme treatments coupled with PLE or SFE process. With this approach, a better recovery of BACs may be obtained; by connecting green technologies, BACs in a diverse, more active, chemical form than that initially present in the natural matrix can be obtained. Another possibility for the future development of GET may be connected to the use of new solvents.[102] Hence, to PLE could be applied ILs or DESs,[106] as well as ScFs. Nevertheless, the toxicity of ILs is controversial and the use of ScFs is expensive. Thus, NaDESs appear to be a more auspicious and greener choice due to their bio-compatibility and low toxicity.[99]

Taking into account that the pharmaceutical, cosmetic, food, and biomaterials industries are interested in exploitation of target high added value

BACs, especially those with antioxidant properties, development of more environmentally sustainable production systems should be sought. This is fundamental in order to increase process efficiency with or without minimal changes in the biological and nutritional properties of natural matrixes.

Green extraction practices for the recovery of antioxidant bioactive compounds from agro-industrial by-products

It is well known that each year, worldwide, 5 billion tons of biomass residues from agrifood by-products cause the emission of 3.3 billion tonnes of CO_2.[99] It is imperative to decrease the amount of this biomass, which can cause serious environmental problems. Nevertheless, these remains can be rich in various BACs in such a way that their utilisation might have a favourable impact on the environment and industry.[104] Over the last 10 years, the number of publications on the application of green extraction practices of agro-industrial by-products has dramatically increased (Fig. 1). Natural BACs from agro-industrial by-product processing are characterized by a wide range of molecules with different functionalities and structures, such as polyphenols, sugars, dietary fibres, amino acids, tocopherols, phytosterols, carotenoids, and vitamins.[99,102,104] Polyphenols are among the most sought after BAC, and their extraction from agro-industrial waste has driven scientists to explore more lucrative, efficient, and sustainable extraction techniques, premised on a green extraction procedure. Polyphenols show interesting properties like cardioprotection, anti-inflammation, anticancer, and antimicrobial activity.[107] Antioxidant activity in particular has been shown to be a very recognisable feature of polyphenols.[108] The antioxidant activity is usually evaluated using different types of assays to carry out a comparative assessment of various methods. A lot of chemical and biological assays are used to investigate the antioxidant activity, but the most commonly applied for agro-industrial by-products are: DPPH$^{\bullet}$ (radical scavenging activity assay), ABTS$^{\bullet+}$ (radical cation scavenging activity assay), ORAC (oxygen radical absorption capacity assay), H-ORAC (hydrophilic oxygen radical absorption capacity assay), CUPRAC (cupric reducing antioxidant capacity assay), FRAP (ferric reducing antioxidant power assay), LPO (lipidic peroxidation inhibition assay), PPO (polyphenol oxidase assay), TBARS (thiobarbituric acid reactive substances assay), β-carotene bleaching method, cellular antioxidant activity, and Rancimat test.[4–6,28,29,33,39,40,42,54,76,85] Also, evaluation of total polyphenolic content (TPC) by Folin-Ciocalteu method can be applied as an antioxidant assay due to its redox chemical mechanism.[4–6,14,67,86] Table 2 reports information regarding the different GET used for an extraction of BACs with antioxidant properties from agro-industrial by-products.

Among the by-products extracted generally from different plants by various GETs were: peel, stem, pod, seed, leaf, chaff, scape, umbel, skin, te-

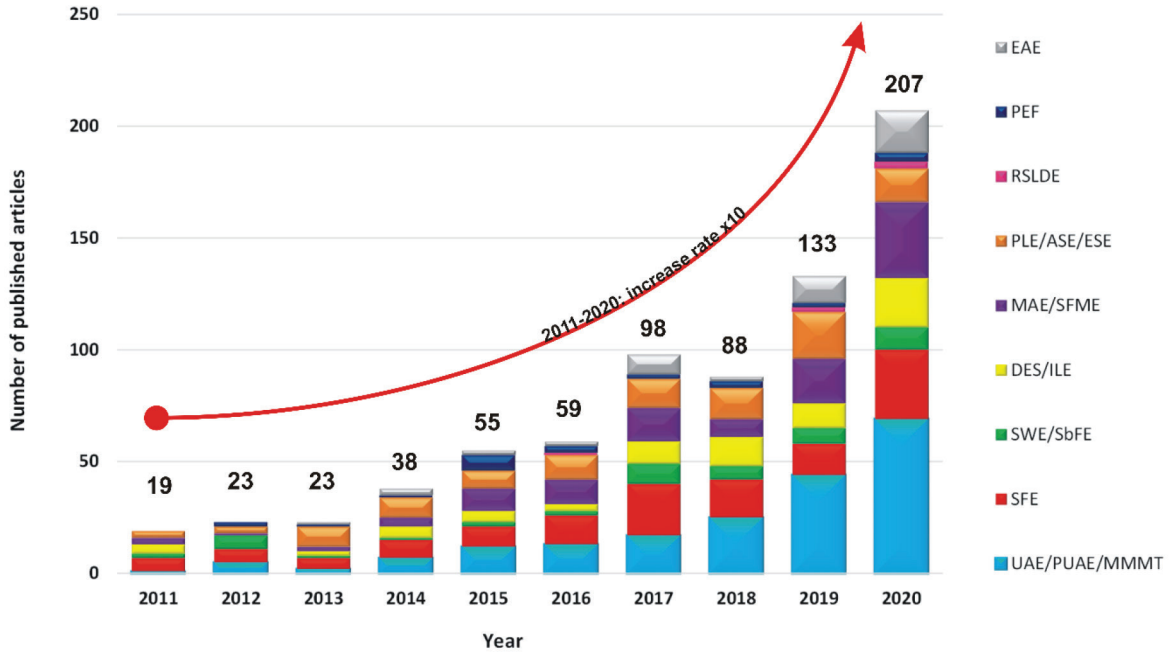

Fig. 1 – *Number of original articles on the application of green extraction technologies on agro-industrial by-products (source Scopus®, 30/01/2021; for the abbreviations, see the list at the end of the manuscript)*

Table 2 – *Green extraction techniques used for extraction of antioxidant bioactive compounds from agro-industrial by-products*

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Enzyme-assisted Extraction (EAE)	tannase, pectinase, cellulase	– temperature (40 °C) – time (30 h) – pH (5.0)	citrus (*Citrus latifolia* and *Citrus sinensis* L.) albedo and flavedo	hesperetin, hesperidin, naringenin, naringin, ellagic acid	ORAC, DPPH$^{\bullet}$	85
	Celluclast® 1.5 L, Pectinex® Ultra, Novoferm®	– temperature (40 °C) – time (48 h) – pH (3.0)	grape (*Vitis vinifera* L.) residues	gallic acid, resorcinol, *o*-coumaric acid	TPC, DPPH$^{\bullet}$	86
	recombinant α-L-rhamnosidase	– temperature (50 °C) – time (1 h)	kinnow mandarin (*Citrus deliciosa* × *Citrus nobilis*) peel waste	naringin	–	87
	water, carbohydrases (cellulase, pectinase, xylanase), proteases (alcalase, neutrase, pepsin, papain)	– incubation temperature (45 °C) – incubation time (1 h) – hydrolysis time (2 h) – pH (0.5 M NaOH or 0.5 M acetic acid) – pH and temperature for carbohydrases (4.5–5 and 45–50 °C)	raspberry (*Rubus idaeus* L.) pomace press-cake	ellagic acid, ellagitannin, ellagic acid pentoside, methyl ellagic acid pentoside, lambertianin C and D, gallic acid, sanguiin H-2, -6, -10, hexahydoxydiphenoyl galloylglucose	TPC, ORAC, DPPH$^{\bullet}$	88
Microwave-assisted Extraction (MAE)	water	– temperature (50 °C) – time (10 min)	pomegranate (*Punica granatum* L. cv. Dente di Cavallo) endocarp and aril residues	polyphenols	TPC, DPPH$^{\bullet}$	14
Including: Solvent-free Microwave Extraction (SFME)	ethanol:water (0–100 %, w/w)	– temperature (25–75 ºC) – time (5–15 min) – solid:solvent ratio (10–30 g mL^{-1})	kiwi (*Actinidia deliciosa*, cv. "Hayward") juice pomace	polyphenols, flavan-3-ols	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	67
	water	– temperature (25–75 ºC) – time (5–65 min) – solid:solvent ratio (1:50–1:150 g mL^{-1})	white-fleshed red (*Hylocereus undatu* (Haworth) Britton & Rose) and yellow (*Hylocereus megalanthus (K. Schumann ex Vaupel) Ralf Bauer*) pitaya peel	phenolic acids, flavonoids, betacyanins	–	68

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	hydroethanolic solvent (60 or 70 %, v/v for the white or reds cultivars, respectively)	– temperature (70 or 60 °C, for whites or reds, respectively) – time (4 min) – pH (1.5) – solvent:solid ratio (6.59 mL g^{-1} skins)	red and white grape (*Vitis vinifera* L.) pomaces	polyphenols	TPC	69
	–	– atmospheric pressure – power density (1 W g^{-1})	sea buckthorn (*Hippophae rhamnoides* L.) pomace	isorhamnetin 3-*O*-rutinoside, isorhamnetin 3-*O*-glucoside, quercetin 3-*O*-glucoside, isorhamnetin	TPC, DPPH$^{\bullet}$	72
	hydroethanolic solvent (50 %, v/v)	– temperature (60–100 °C) – time (6–30 min) – max power (500 W) – frequency (2.45 GHz)	English walnut (*Juglans regia* L.) fresh male flowers and unripe seeds	polyphenols	TPC	70
	hydroethanolic solvent (20 mg/mL)	– time (30–120 s) – power (250–750 W)	avocado (*Persea americana* Mill. var. Hass) peel	polyphenols	TPC, DPPH$^{\bullet}$, FRAP, LPO	28
	hydroetanolic solvent (60 %, v/v)	– time (60–120 s) – solvent:solid ratio (10–50 mL g^{-1}) – power (400–800 W)	supercritical CO_2 pre-extracted mango (*Mangifera indica* L.) peel	gallic acid, mangiferin, quercetin	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	71
	water	– temperature (50–125 °C) – time (20 min) – pressure (2–5 atm)	pine (*Pinus pinaster* var. *Pinus maritime* and *Pinus d'Alpes*) seeds	polyphenols	TPC, ABTS$^{\bullet+}$,	20
	water, methanol (50 %), ethanol (50 %), acetone (50 %)	– temperature (<135 °C) – time (90–240 s) – solvent:solid ratio (15–30 mL g^{-1}) – power (300–600 W)	lemon (*Citrus limon* (L.) Osbeck) peel	polyphenols	TPC, DPPH$^{\bullet}$, FRAP	17

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Neoteric solvents (NSs) extraction						
Ionic Liquid Extraction (ILE)	1-ethyl-3-methylimidazolium acetate	– temperature (25–51.8 ºC) – time (60 min) – solid:solvent ratio (8.2–41.8 g mL^{-1})	red grape (*Vitis* spp.) pomace	anthocyanins	–	90
	tributylmethylphosphonium bis(trifluoromethylsulfonyl)imide, tributylmethylammonium bis(trifluoromethylsulfonyl) imide, trioctylmethylammonium bis-(trifluoromethylsulfonyl)imide	– temperature (30 °C) – time (2 h) – solid:solvent ratio (1:5)	olive (*Olea europaea* L.) mill waste water	tyrosol	–	89
Deep Eutectic Solvents (DESs) Extraction	complexes (ratio 1:1, 5:2, 1:1, or 4:1) of choline chloride with DL-malic acid, citric acid, glycerol, D-(+)-glucose, D-(–)-fructose, D-(+)-galactose, D-(–)-ribose, sucrose, D-(+)-maltose, or maltitol and complexes (ratio 1:1 or 2:1) of citric acid with D-(–)-fructose, D-(+)-maltose, or maltitol	– room temperature – time (45 min) – water:DES ratio (3:7; w/w) – coupled with UAE	grape (*Vitis* spp.) skin	anthocyanins	TPC, DPPH$^{\bullet}$	57
	complexes (ratio 1:2) of choline chloride with urea, citric acid, lactic acid, glucose, sorbitol, xylitol, glycerol, 1,6-hexanediol, triethylene glycol, ethylene glycol, propylene glycol, and complex of betaine with lactic acid, glycerol, ethylene glycol, or triethylene glycol	– temperature (65 ºC) – time (20 min) – heating power (200 W) – frequency (37 kHz)	spent coffee (*Coffea* spp.) ground	chlorogenic acids (3-*O*-caffeoylquinic acid, caffeoyl-epi-quinic acid, 5-*O*-caffeoylquinic acid, 4-*O*-caffeoylquinic acid, 5-*p*-coumaroylquinic acid, quinolactone, 4-feruloylquinic acid, 3-feruloylquinic acid, 3,4-dicaffeoylquinic acid, 3,5-dicaffeoylquinic acid, 4,5-dicaffeoylquinic acid, caffeoylferuloylquinic acid)	–	56
	complexes (ratio 1:1, 1:2, or 1:3) of choline chloride with acetamide, butane-1,4-diol, citric acid, ethylene glycol, glycerol, lactic acid, levulinic acid, malonic acid, malic acid, N-methyl urea, oxalic acid, sorbitol, thiourea, urea, or xylitol	– temperature (30–70 ºC) – time (30–90 min) – amount of water (10–30 %)	mandarin (*Citrus reticulata*) peels	hesperidin	–	58

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	complexes (ratio 1:2, or 1:1) of choline chloride with acetamide, butan 1,4-diole, ethylene glycol, fructose, glycerol, glucose, malic acid, xylitole, levulinic acid, citric acid, malonic acid, lactic acid, oxalic acid, sorbitol, urea, or tartaric acid	– temperature (30–90 °C) – time (5–15 min) – amount of water (10–50 %) – coupled with MAE	cocoa (*Theobroma cacao* L.) bean shell	gallic acid, theobromine, caffeine, catechin, caffeic acid, epicatechin	$DPPH^{\bullet}$	59
	complexes (ratio 2:1, 1:1, or 5:2) of choline chloride with D-(+)-galactose, L-proline, DL-malic acid, xylitol, D-(−)-fructose, sucrose, citric acid, D-(+)-glucose, and complex of glycerol, with xylitol, and D-(−)-fructose in different molar ratios	– temperature (60 °C) – time (20 min) – power (250 or 700 W for UAE or MAE, respectively)	fig (*Ficus carica* L.) leaves	polyphenols (caffeoylmalic acid, rutin), and furanocoumarins (psoralic acid-glucoside, psoralen, bergapten)	–	60
Natural Deep Eutectic Solvents (NaDESs) Extraction	complexes (ratio 1:1) of citric acid with glucose or fructose with citric acid, or complex (ratio 1:5) lactic acid with glucose	– temperature (40 ºC) – time (15–60 min) – solid:solvent ratio (15–75 mg mL^{-1}) – amount of water (0, 40 and 75 %) – power output (200 W) – frequency (20 kHz) – coupled with UAE	olive (*Olea europaea* L.) cake	rutin hydrate, gallic acid	–	63
			onion (*Allium cepa* L.) seed (dry scapes and umbels)	rutin hydrate, caffeic acid, tyrosol, quercetin dihydrate	–	
			tomato (*Solanum lycopersicum* L.) peels, seeds and cull fruits	tyrosol, 3-hydroxytyrosol, trans-ferulic acid, caffeic acid, rutin hydrate, apigenin, luteolin	–	
			pear (*Pyrus* spp.) peels, seeds and cull fruits	caffeic acid, naringenin, catechin hydrate, quercetin dehydrate, rutin hydrate	–	
	complexes (ratio 1:1, or 1:2) of choline chloride with propylene glycol, citric acid, or malic acid, complexes (ratio 1:1:3, 1:1, or 3:1) of citric acid with glucose, and water or with propylene glycol, or with betaine	– temperature (353 K) – time (60 min) – solid:solvent ratio (1:30, g mL^{-1}) – with use of acidified ethanol solution (50 % (v/v) with 0.1 M citric acid) – NaDESs diluted in water (1:1 w/w)	jabuticaba (*Myrciaria cauliflora*) pomace (peel and pulp)	anthocyanins	–	61

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	complexes (ratio 5:1, 7:1, 9:1, 11:1, 13:1) of L-lactic acid with glycine	– temperature (50 °C) – time (150 min) – solvent:solid ratio (35 mL g^{-1}) – amount of water (70 %)	saffron (*Crocus sativus* L.) tepals	flavonols (kaempferol-3-*O*-sophoroside 7-*O*-glucoside, quercetin 3-*O*-sophoroside, kaempferol 3-*O*-sophoroside, kaempferol 3-*O*-glucoside), anthocyanins (delphinidin 3,5-di-*O*-glucoside, petunidin 3,5-di-*O*-glucoside, delphinidin 3-*O*-glucoside)	TPC, DPPH$^{\bullet}$, FRAP	64
	complexes (ratio 1:1, 1:2, and 2:1) of choline chloride with citric acid, glycerol, or glucose and complexes (ratio 1:1 and 1:2) of betaine with citric acid, glycerol, or glucose	– temperature (60 °C) – time (50 min) – power (150 W)	cocoa (*Theobroma cacao* L.) beans	catechin, epicatechin, protocatechuic acid, procyanidin B1 and B2	TPC, ORAC	65
	complex (ratio 2:1) of choline chloride with citric acid	– time 10 min – solid:solvent ratio (0.5 g/10 mL) – microwave power (300 W) – ultrasound power (50 W) – amount of water (30 %)	red grape (*Vitis vinifera* L. cv. Plavac mali) pomace	gallic acid, quercetin-3-*O*-glucoside, delphinidin-3-*O*-monoglucoside, petunidin-3-*O*-monoglucoside, peonidin-3-*O*-monoglucoside, malvidin-3-*O*-monoglucoside, peonidin-3-acetylmonoglucosides, malvidin-3-acetylmonoglucosides, cyanidin-3-(6-*O*-*p*-coumaroyl) monoglucosides, peonidin-3-(6-*O*-*p*-coumaroyl)monoglucosides, malvidin-3-(6-*O*-*p*-coumaroyl) monoglucosides	ORAC	66
			olive (*Olea europaea* L.) pomace	gallic acid, hydroxytyrosol, tyrosol, vanillic acid, vanillin, pinoresinol, catechin	ORAC	
	complexes (ratio 1:2) of choline chloride with citric acid, lactic acid, maltose, or glycerol	– temperature (40 or 60 °C) – time (30 min) – amount of water (20 %) – coupled with MAE, UAE or other	virgin olive (*Olea europaea* L.) pomace	oleuropein, hydroxytyrosol, caffeic acid, vanillin, rutin, luteolin	TPC, DPPH$^{\bullet}$	62

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Pressurized Liquid Extraction (PLE)	0, 50 or 100 % of ethanol in mixture of EtOH/EtOAc (v/v)	– temperature (50–150 °C)	mango (*Mangifera indica* L.) seed kernel	polyphenols	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	73
Including: Enhanced Solvent Extraction (ESE)	water, ethanol:water (50:50), ethanol (PLE)	– temperature (60–100 °C) – time (3 h) – pressure (4–20 and 12–20 MPa, for PLE and ESE, respectively) – flow rate (10 g min^{-1})	mango (*Mangifera indica* L.) leaves	phenolic acids, flavonoids, xanthones, gallotannins, benzophenones	TPC, DPPH$^{\bullet}$	74
	CO_2/ethanol/water (50:25:25) (ESE)					
and Accelerated Solvent Extraction (ASE)	anhydrous ethanol	– temperature (40–80 °C) – time (2–10 min) – flush volume (60–140 %) – number of cycles (1–5)	cherry (*Eugenia uniflora* L.) seeds	ellagic acid, ellagic acid pentoside, ellagic acid deoxyhexose, quercitrin, kaempferol pentoside, quercetin hexoside	TPC, DPPH$^{\bullet}$	75
	ethanol (99.8 %)	– temperature (70 °C) – time (20 min) – pressure (10 MPa)	cocoa (*Theobroma cacao* L.) bean hulls	polyphenols	TPC, DPPH$^{\bullet}$, β-carotene bleaching method	42
	water, ethanol (10 %, v/v)	– temperature (93 °C) – time (5 min) – pressure (1500 psi) – flush volume (150 %) – purge (N_2 100 s) – number of cycles (2)	artichoke (*Cynara cardunculus* L. var. scolymus) bract and leaf	flavone glycosides (luteolin-rutinoside, luteolin-glucoside, apigeninglucuronide), caffeoylquinic acids (caffeoylquinic acids, dicaffeoylquinic acids)	cellular antioxidant activity	76
	water	– temperature (120 °C) – time (two cycles of 10 min) – pressure (1500 psi)	grape (*Vitis vinifera* L.) seeds, pomace and stems	hydroxycinnamic acid derivatives, flavonols, tannins, catechins, anthocyanins	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	24
	water	– temperature (50–125 °C) – time (20–22 min) – pressure (40 atm)	pine (*Pinus pinaster* var. *Pinus maritime* and *Pinus d'Alpes*) seeds	polyphenols	TPC, ABTS$^{\bullet+}$,	20
	water	– temperature (25–200 °C) – time (3–17 min) – pressure (1500 psi)	apple (*Malus domestica* Borkh.) press cake (seeds, cores, stems, skin, and parenchyma)	5-caffeoylquinic acid, hyperoside, isoquercitrin, reinutrin, phloridzin, avicularin, quercitrin, quercetin	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	77

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Pulsed Electric Fields (PEFs) Extraction	water	– initial temperature (20 °C) – final temperature (< 35 °C) – solid:solvent ratio (1:10) – initial voltage peak amplitude (40 kV) – successive pulses (1–2000) – series of 400 pulses	papaya (*Carica papaya* L.) peel	polyphenols	TPC, $ABTS^{\bullet+}$	79
	distilled water	– time (3 μs) – power (1–7 kV cm^{-1}) – energy (0.06–3.77 kJ kg^{-1}) – frequency (1 Hz) – pulses (5–50)	orange (*Citrus sinensis* L.) peel (flavedo and albedo)	naringin, hesperin	TPC, $DPPH^{\bullet}$	80
	rehydrated mixture of water and ethanol, ethanol (0–50 %), supplemented with 0.05–0.3 mol L^{-1} sodium hydroxide for alkaline extraction or with 0.05–0.3 mol L^{-1} citric acid	– temperature (20 °C) – energy (10–20 kV cm^{-1}) – frequency (0.33 Hz) – pulses (40 kV–10 kA)	flaxseed (*Linum usitatissimum* L., cv. Baladin) hulls	polyphenols	TPC	81
	hydromethanolic solvent (0–80 %)	– temperature (15–35 °C) – time (3 μs) – electric field strength (0–5 kV cm^{-1}) – gap (3 cm) – intensity (68–133 A) – pulses (10–50) – energy (0.61–9.98 kJ kg^{-1})	thinned peach (*Prunus persica* L. Batsch var. 'Royal Glory') fruits	catechin, coumaric acid, chlorogenic acid, neochlorogenic acid, quercetin	TPC, $DPPH^{\bullet}$	82
Rapid Solid Liquid Dynamic Extraction (RSLDE)	deionized water	– room temperature – pressure (8–10 bars)	red grape (*Vitis vinifera* L. var Aglianico) peels and seeds	polyphenols, anthocyanins	TPC	78

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Subcritical Fluid Extraction (SbFE) Including: Subcritical Water Extraction (SWE)	distilled water, absolute ethanol	– flow rate (10 g min^{-1}) – pressure (7 MPa)	peanut (*Arachis hypogaea* L.) skin	flavan-3-ol monomers and dimers, flavanols (catechin, epicatechin), flavones (chrysin, luteolin), flavonol (quercetin), *o*-methylated isoflavone (biochanin A), quinic acid, caffeic acid	TPC	48
			sesame (*Sesamum indicum* L.) seeds cake	lignans (sesaminol, sesamolinol, sesamin, sesamolin, episesamin, diasesamin), hydroxycinnamic acid (syringic and ferulic acids), flavonoids (apigenin-7-methylether, epicatechin-3-*O*-galato, 4′-hydroxyflavanone, genistein)	TPC	
			pistachio (*Pistacia vera* L.) nuts cake	flavonoids (daidzein, genistein, naringenin, quercetin, kaempferol), gallic acid and its derivatives, procyanidin B-type dimers, caffeoylquinic acid, anacardic acid, quinic acid	TPC	
	water	– temperature (140–260 °C) – time (2–25 min) – pressure (40–120 bar) – pH (3–9)	potato (*Solanum tuberosum* L.) peel	polyphenols	TPC, FRAP	47
	water	– temperature (100–190 ºC) – time (5–30 min) – pressure (100 bar)	red grapevine (*Vitis vinifera* L. 'Merlot') cane, wood and root	stilbens (piceid, piceatannol, resveratrol, ampelopsin A and F, pallidol, parthenocissin A, ε-viniferin, ω-viniferin, miyabenol C, viniferol E, hopeaphenol, isohopeaphenol, ampelopsin H and E, vitisin A, B, and F)	–	50
	water	– temperature (120–160 ºC) – time (0–30 min) – solid:solvent ratio (2–6 %) – pressure (30 bar) – pH (2–5.5)	green kiwi (*Actinidia deliciosa* (A. Chev) C.F. Liang & A.R. Ferguson) fruit peel	polyphenols	TPC, $DPPH^{\bullet}$, $ABTS^{\bullet+}$, FRAP	51

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	water	– temperature (100–160 ºC) – time (120 min) – pressure (100–200 bar)	asparagus (*Asparagus officinalis* L.) fibrous stem	polyphenols	TPC, DPPH$^{\bullet}$, FRAP	52
	water	– temperature (120–220 ºC) – time (15–75 min) – solvent:solid ratio (10–30 mL g^{-1})	cocoa (*Theobroma cacao* L.) shells	epicatechin, catechin, chlorogenic acid, gallic acid	TPC, DPPH$^{\bullet}$	53
	water	– temperature (120–160 °C) – time (20–60 min) – water:solid ratio (20–40 mL g^{-1})	saffron (*Crocus sativus* L.) petals	polyphenols	TPC, DPPH$^{\bullet}$, FRAP	46
	distilled water	– temperature (180–270 ºC) – time (10 min/each temperature) – pressure (1.0–5.3 MPa)	coffee (*Coffeea arabica* L., and *Coffeea canephora* L.) silverskin	polyphenols, chlorogenic acid hemihydrate,	TPC, DPPH$^{\bullet}$, H-ORAC	54
	water	– temperature (100–200 ºC) – pressure (25·10^5 Pa) – constant flow rate (6 mL min^{-1})	red and white grape (*Vitis vinifera* L.) pomace	anthocyanins, gallic acid, flavan-3-ols (catechin, epicatechin, procyanidin dimers and trimers)	TPC, ABTS$^{\bullet+}$, FRAP, CUPRAC, ORAC	55
	water	– temperature (125–200 ºC) – pressure (20 bar)	rice (*Oryza sativa* L. mix) bran	phenolic acids (gallic acid, caffeic acid, *p*-coumaric acid, ferulic acid)	TPC	49
Supercritical Fluid Extraction (SFE)	CO_2 with ethanol as a co-solvent	– temperature (60–70 ºC) – time (15 min) – pressure (250 bar) – flow rates (2 and 0.4 mL min^{-1} for CO_2 and ethanol, respectively)	red grape (*Vitis vinifera* L. 'Merlot') pomace, skins and seeds	*t*-resveratrol	TPC, DPPH$^{\bullet}$	37
	CO_2 with ethanol/water (85 %) as a co-solvent	– temperature (40 °C) – time (120 min) – pressure (100 bar) – flow rate (10 and 0.5 mL min^{-1} for CO_2 and ethanol/water, respectively)	onion (*Allium cepa* L.) brown dry skin	quercetin dimers and trimers, protocatechuic acid, 2-(3,4-dihydroxybenzoyl)-2,4,6-trihydroxy-3(2h)-benzofuranone, quercetin-7,4′-diglycoside, quercetin 3,4′-diglycoside, isorhamnetin-3,4′diglycoside, quercetin-3-glycoside, quercetin-4′-glycoside, isorhamnetin-4′-glycoside, quercetin, protocatecoyl quercetin, kaempferol, isorhamnetin	DPPH$^{\bullet}$, ABTS$^{\bullet+}$	15

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	CO_2, CO_2 with ethanol as a co-solvent	– temperature (40–60 °C) – pressure (10–30 MPa)	papaya (*Carica papaya* L.) seeds	phenolic acids (chlorogenic acid, caffeic acid, ferulic acid, *p*-hydroxybenzoic acid, *p*-coumaric acid), flavonols (myricetin, quercetin, kaempferol, kaempferol-3-*O*-glycoside, quercetin-3-*O*-glycoside)	TPC, DPPH$^{\bullet}$, evaluation of lipid peroxidation measured by conju–gated dienes method and TBARS assay	39
	CO_2	– temperature (40 °C) – time (60 min) – pressure (350 bar) – extraction rate (2 g min^{-1})	olive (*Olea europaea* L.) oil mill wastes	polyphenols	TPC, DPPH$^{\bullet}$, peroxide value determination, Rancimat® test	40
	CO_2	– temperature (40 °C) – pressure (300 bar) – flow rate (0.194 kg h^{-1})	raspberry (*Rubus idaeus* L.) seeds	ellagic acid	–	41
	CO_2	– temperature (40–60 °C) – time (2 h) – pressure (20–30 MPa) – flow rate (11 g CO_2 min^{-1})	cocoa (*Theobroma cacao* L.) bean hulls	polyphenols	TPC, DPPH$^{\bullet}$, *β*-carotene bleaching method	42
	CO_2	– temperature (40–50 ºC) – pressure (150–300 bar) – flow rate (0.5 kg CO_2 h^{-1})	passion fruit (*Passiflora edulis* sp.) seeds and seed cake	polyphenols	TPC, DPPH$^{\bullet}$ ABTS$^{\bullet+}$, *β*-carotene bleaching method	23
	CO_2	– temperature (40–60 °C) – pressure (10–30 MPa) – flow rate (1–3 kg h^{-1})	sage (*Salvia officinalis* L.) leaves	carnosoic acid, carnosol	TPC, DPPH$^{\bullet}$	43
	CO_2	– temperature (40–60 °C) – pressure (200–300 bar) – flow rate (1.5 L min^{-1})	red grape (*Vitis vinifera* L.) seeds	phenolic acids (gallic acid, caffeic acid, *p*-coumaric acid, ferulic acid, ellagic acid	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	44
	CO_2 with ethanol as a co-solvent	– temperature (50 °C) – time (2.5 h) – pressure (200 bar) – flow rate (6 mL min^{-1}) – amount of co-solvent (10 %)	cacao (*Theobroma cacao* L.) pod husk	polyphenols	TPC, ABTS$^{\bullet+}$	45
	CO_2 with ethanol as a co-solvent	– temperature (40–80 °C) – time (40–80 min) – pressure (150–450 bar) – flow rate (28–32 g min^{-1})	broccoli (*Brassica oleracea* L. var. italica) leaves and stems	polyphenols	TPC, ABTS$^{\bullet+}$	38

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
Ultrasound-assisted Extraction (UAE)	water	– temperature (50 °C) – time (10 min)	pomegranate (*Punica granatum* L. cv. Dente di Cavallo) endocarp and aril residues	polyphenols	TPC, DPPH$^{\bullet}$	14
Including:	65 % (v/v) ethanol (methanol and acetone)	– temperature (50 °C) – time (30 min)	plum (*Prunus armeniaca, Prunus persica and Prunus domestica)* seed	polyphenols	TPC, ABTS$^{\bullet+}$	12
	ethanol (85 %, v/v)	– temperature (25 °C) – time (15 min)	onion (*Allium cepa* L.) brown dry skin	quercetin dimers and trimers, protocatechuic acid, 2-(3,4-dihydroxybenzoyl)-2,4,6-trihydroxy-3(2h)-benzofuranone, quercetin-7,4′-diglycoside, quercetin 3,4′-diglycoside, isorhamnetin-3,4′diglycoside, quercetin-3-glycoside, quercetin-4′-glycoside, isorhamnetin-4′-glycoside, quercetin, protocatecoyl quercetin, kaempferol, isorhamnetin	DPPH$^{\bullet}$, ABTS$^{\bullet+}$	15
	water, ethanol, methanol, butanol	– temperature (25–60 ºC) – time (0–60 min) – ultrasound frequency (15–45 kHz)	flax (*Linum usitatissimum* L.) seeds	lignin (secoisolariciresinol diglucoside), flavonol (herbacetin diglucoside), hydroxycinnamic acids (*p*-coumamic, caffeic, ferulic acid glucosides)	–	16
	dichloromethane, dry extract solubilised in methanol	– solvent evaporation (30 ºC) – time (15 s) – amplitude (50 %) – double extraction	mango (*Mangifera indica* L.) peels and rye (*Secale cereale* L.) grains	alk(en)ylresorcinols	–	18
	water	– temperature (34–76 ºC) – time (4–46 min)	chestnut (*Castanea sativa* Mill.) shells	catechin/epicatechin, ellagic acid, tetrameric PAC, epigallocatechin, apigenin-7-*O*-rutinoside, trimeric PAC, luteolin-7-*O*-rutinoside, caffeic acid derivative, procyanidin polymers	TPC, FRAP, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	19

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	water	– temperature (20–65 °C) – time (30 min) – power (35–100 W)	red beet (*Beta vulgaris* L. var. conditiva) leaves	polyphenols, betalains (betaxanthins, betacyanins)	TPC	22
	hexane, ethyl acetate, ethanol, ethanol:water (ratio 1:1)	– room temperature – time (45 min) – potency (220 V) – frequency (55 kHz)	passion fruit (*Passiflora edulis* sp.) seeds, and seed cake	polyphenols	TPC, DPPH$^{\bullet}$ ABTS$^{\bullet+}$, β-carotene bleaching method	23
	hydroalcoholic solvent (44 % ethanol)	– temperature (<50 °C) – time (3 min) – power (500 W) – frequency (20 KHz) – cycles (15 s turn on and 5 s off)	grape (*Vitis vinifera* L.) seeds, pomace and stems	hydroxycinnamic acid derivatives, flavonols, tannins, catechins, anthocyanins	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	24
	water	– temperature (25 ºC) – time (30 min) – power (7.8 or 49.5 W)	coffee (*Coffea* spp.) silver skin	5-feruloylquinic acid, 3-caffeoylquinic acid, 5-caffeoylquinic acid, 4-caffeoylquinic acid, 3-coumaroylquinic acid, 5-coumaroylquinic acid	TPC	30
			brewer's spent grain	*p*-coumaric acid, caffeic acid, vanillic acid, (+)-catechin, protocatechuic acid	TPC	
			potato (*Solanum tuberosum* L.) peel	*p*-coumaric acid, caffeic acid, chlorogenic acid, protocatechuic acid	TPC	

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	water	– time (15–60 min) – power (1200 W) – frequency (25 kHz)	artichoke (*Cynara scolymus* L.) scraps	caffeoylquinic acids (5-*O*-caffeoylquinic acid, 1,5-di-*o*-caffeoylquinic acid), flavonoids (apigenin 7-*O*-glucoside, luteolin), other polyphenols	TPC, DPPH$^{\bullet}$, ABTS$^{\bullet+}$	25
	pure water, ethanol 60 %	– temperature (20 or 60 ºC) – solid:solvent ratio (1:40) – power (100 W) – frequency (30.8 kHz) – agitation speed (160 rpm)	chicory (*Cichorium intybus* L.) grounds	polyphenols	DPPH$^{\bullet}$	31
	water, ethanol, methanol, acetone	– temperature (45 ºC) – time (30 min) – solid:solvent ratio (1:20–1:50)	walnut (*Juglans regia* L.) green husk	polyphenols	TPC, FRAP, DPPH$^{\bullet}$	26
	hydroethanolic solvent (20 mg mL^{-1})	– temperature (40–60 ºC) – time (15–60 min)	avocado (*Persea americana* Mill.) peel	polyphenols	TPC, DPPH$^{\bullet}$, FRAP, LPO	28
	ethanol (0–100 %,v/v)	– temperature (45 ºC) – time (20–40 min) – solvent:solid ratio (10:1) – frequency (0–45 kHz)	almond (*Prunus dulcis* Mill.) cold-pressed oil residues	phenolic acids (*p*-coumaric acid, protocatechuic acid, chlorogenic acid, *p*-hydroxybenzoic acid)	TPC, CUPRAC, TBARS, DPPH$^{\bullet}$ ABTS$^{\bullet+}$	29
	water	– temperature (20–40 °C) – time (60 min)	bilberry (*Vaccinium myrtillus* L.) juice by-products (cake)	anthocyanins, flavonols	TPC, DPPH$^{\bullet}$	32
	water, 80 % ethanol: 20 % water, 80 % methanol: 20 % water, 80 % acetone: 20 % water	– temperature (20 °C) – time (15 min) – solid:solvent ratio (1:10 w/v) – power (160 W) – frequency (40 kHz)	strawberry (*Fragaria x ananassa* Duch.) by-products	polyphenols and agrimoniin	TPC, FRAP DPPH$^{\bullet}$, PPO	33

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	ethanol (20–80 %, v/v)	– temperature (20–40 ºC) – time (10–20 min) – solvent:solid ratio (25–35 mL g^{-1}) – frequency (20 kHz)	jujube (*Zizyphus lotus* L.) seeds	polyphenols	TPC, FRAP, DPPH$^{\bullet}$	13
	water	– temperature (10–75 ºC) – time (20 min) – pressure (1 atm)	pine (*Pinus pinaster* var. *Pinus maritime* and *Pinus d'Alpes*) seeds	polyphenols	TPC, ABTS$^{\bullet+}$	20
	hydroethanolic solvent	– temperature (10–40 ºC) – time (30 min) – solid:solvent ratio (0.25 g mL^{-1}) – power (50–150 W) – frequency (20–80 kHz)	orange (*Citrus sinensis* L.) peel	flavanones (naringin, hesperidin)	TPC, ORAC, DPPH$^{\bullet}$	21
	ethanol (30–100 %)	– time (5–20 min) – solvent:solid ratio (20–50 mL g^{-1}) – amplitude radiation (20–100 %)	lemon (*Citrus limon* L. Osbeck) peel	polyphenols	TPC, DPPH$^{\bullet}$, FRAP	17
	aqueous glycerol	– temperature (45 °C) – time (60 min) – power (140 W) – frequency (37 kHz)	red grape (*Vitis vinifera* spp.) pomace	polyphenols, anthocyanins	TPC, reducing power	27
*Pulsed Ultrasound-assisted Extraction (PUAE)	water	– temperature (< 40 ºC) – time (10 min) – solvent:solid ratio (40:1) – power (200 W) – frequency (26 kHz)	pomegranate (*Punica granatum* L.) peels	polyphenols, anthocyanins	TPC, DPPH$^{\bullet}$	36

Green extraction technique	Carrier (solvent or gas) or other	Other extraction parameters	Biomass/by-product	Bioactive compounds extracted	Antioxidant activity assay	Ref.
	water/glycerol/ethanol (50/30/20 v/v/v)	– temperature (< 70 °C) – time (5–15 min) – solid:solvent ratio (1:20) – power (200 W) – frequency (26 kHz)	sweet chestnut (*Castanea sativa* Mill.) bud	cinnamic acids, flavonols benzoic acids, catechins, tannins	–	35
and	glycerol and ethanol:water (95:5 v/v)	– temperature (< 70 °C) – time (5–15 min) – solid:solvent ratio (two different levels (first step: 1:40, 1:50, 1:60; second step: 1:20, 1:15 and 1:10) – power (200 W) – frequency (26 kHz)	chestnut (*Castanea* spp.) bud-derivatives	cinnamic acids (ferulic acid, coumaric acid, chlorogenic acid, caffeic acid), flavonols (rutin, quercitrin, quercetin, isoquercitrin, hyperoside), catechins (epicatechin, catechin), and tannins (vescalagin, castalagin)	–	34
Multi-frequency Multimode Modulated Technology (MMMT)	distilled water	– time (300 s) – power (160 W) – frequency (19.8 kHz)	hardy kiwi (*Actinidia arguta* L.) leaves	chlorogenic acid derivatives (quinic acid, 5 CQA, 3 CQA, 4 CQA, caffeic acid-*O*-hexoside, *p*-coumaroylquinic acid), flavonoid derivatives (catechin, quercetin-*O*-rutinoside, quercetin-3-*O*-rutinoside-7-*O*-rhamnoside, quercetin-3-*O*-hexoside, kaempferol-3-*O*-rutinoside, quercetin-3-*O*-(acetyl-rhamnoside)-hexoside, kaempferol-3-*O*-hexoside, quercetin-3-*O*-(acetyl)-hexoside, kaempferol-3-*O*-(acetyl-rhamnoside)-hexoside, kaempferol-3-*O*-(acetyl)-hexoside)	TPC, DPPH$^{\bullet}$, FRAP, scavenging capacity against reactive species ($O_2^{\bullet-}$, H_2O_2, $NO^{\bullet}$, $ROO^{\bullet}$, HOCl, $ONOO^{-}$)	83
	hydroethanolic solvent (1:1)	– temperature (40 ºC) – time (60–600 s) – frequency (19.8 kHz) – magnetic stirring (600 rpm) – input electric power (250–500 W)	coffee (*Coffea* spp.) chaff	3-caffeoylquinic acid, 5-caffeoylquinic acid, 4-caffeoylquinic acid, 5-feruloylquinic acid, 3-feruloylquinic acid, caffeoylferuloylquinic acid, caffeoyltryptopha, dicaffeoylquinic acids	TPC, FRAP	84

pal, bean shell and hull, seed kennel, husk, cake, dust, midrib, grounds, bran, endocarp, aril residues, parenchyma, albedo, flavedo, cores, oil residues, pomace, bract and cull fruit (Fig. 2). The most common plants investigated for their by-products were fruits (kiwi, pomegranate, orange, jujube, strawberry, bilberry, avocado, cherry, grape, passion fruit, mango, raspberry, papaya, peach, pear, apple, fig, mandarin, plum, jabuticaba fruit, sea buckthorn, and pitaya),[12–14,18,23,24,28,32,33,58,60,61,63,67,68,72,75,77,79,80,82,88] vegetables (tomato, chicory, potato, red beet, broccoli, asparagus, artichoke, olive, and onion),[22,25,31,38,47,52,63,89] nuts (chestnut, almond, walnut, pistachio, and peanut),[19,26,29,48] grains (coffee, cacao, rye, sesame, and flax),[18,48,59,81,84] and other plant materials (rice, sage, pine, and saffron).[20,43,49,64] It should be noted that exploited agro-industrial by-products differ greatly in their texture and BACs content, and this affects the choice of the proper GET (Fig. 3).

Agro-industrial by-products extraction with GET based on the energy used

UAE has been widely used for extraction of polyphenols from a variety of fruits, like grape, pomegranate, mango, bilberry, strawberry, jujube, armenian plum, orange, passion fruit, avocado, as well as vegetables, like onion, potato, artichoke, chicory, and red beet. Moreover, UAE has been used for the extraction of polyphenolic compounds from nuts (chestnut, walnut, and almond), as well as grains (flax, rye, and coffee), and other plant sources, like pine. Specifically, Bibi *et al.*[12] extracted new low-cost and eco-friendly phenolic compounds with the use of UAE from seeds of *Prunus armeniaca, Prunus persica* and *Prunus domestica*, while Geerkens *et al.*[18] performed the quantitative isolation of alk(en)ylresorcinols from mango peels and rye grains with the same GET. Regarding different berry by-products, Varo *et al.*[32] used UAE for extraction of anthocyanins and flavonols from bilberry juice by-products, while Villamil-Galindo *et al.*[33] isolated these compounds from strawberry by-products. In the latter's findings, extracts with acidified methanol in two steps yielded the highest polyphenol concentration (15.01 g kg^{-1}), and the highest antioxidant capacity. Moreover, agrimoniin was the main polyphenol detected, and the extraction with acetone in two steps produced the highest yield (2.45 g kg^{-1}). In terms of extraction, another important crop investigated with ultrasound techniques were grape by-products. Trasanidou *et al.*[27] using UAE and water/glycerol mixtures as the solvent, re-

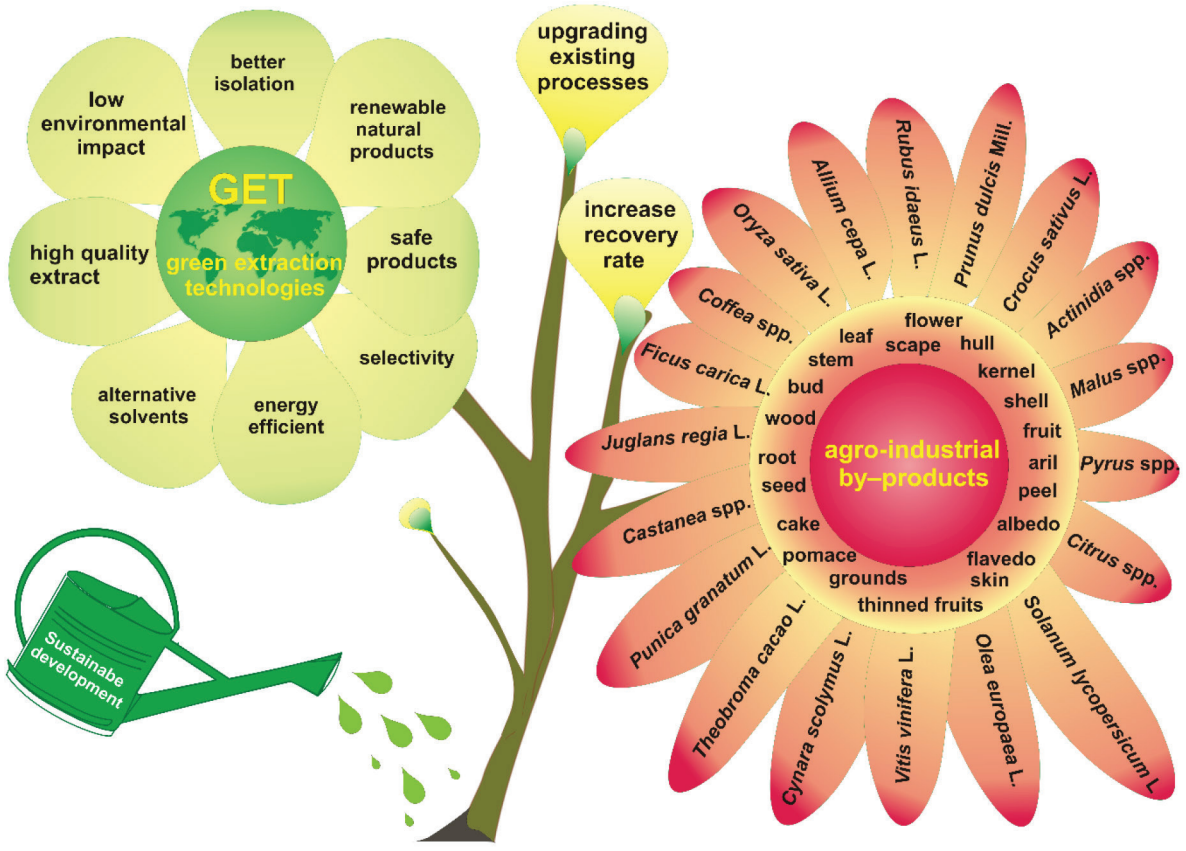

Fig. 2 – *Blooming of the GETs and agro-industrial by-products' exploitation in the framework of sustainable development (for the abbreviations, see the list at the end of the manuscript)*

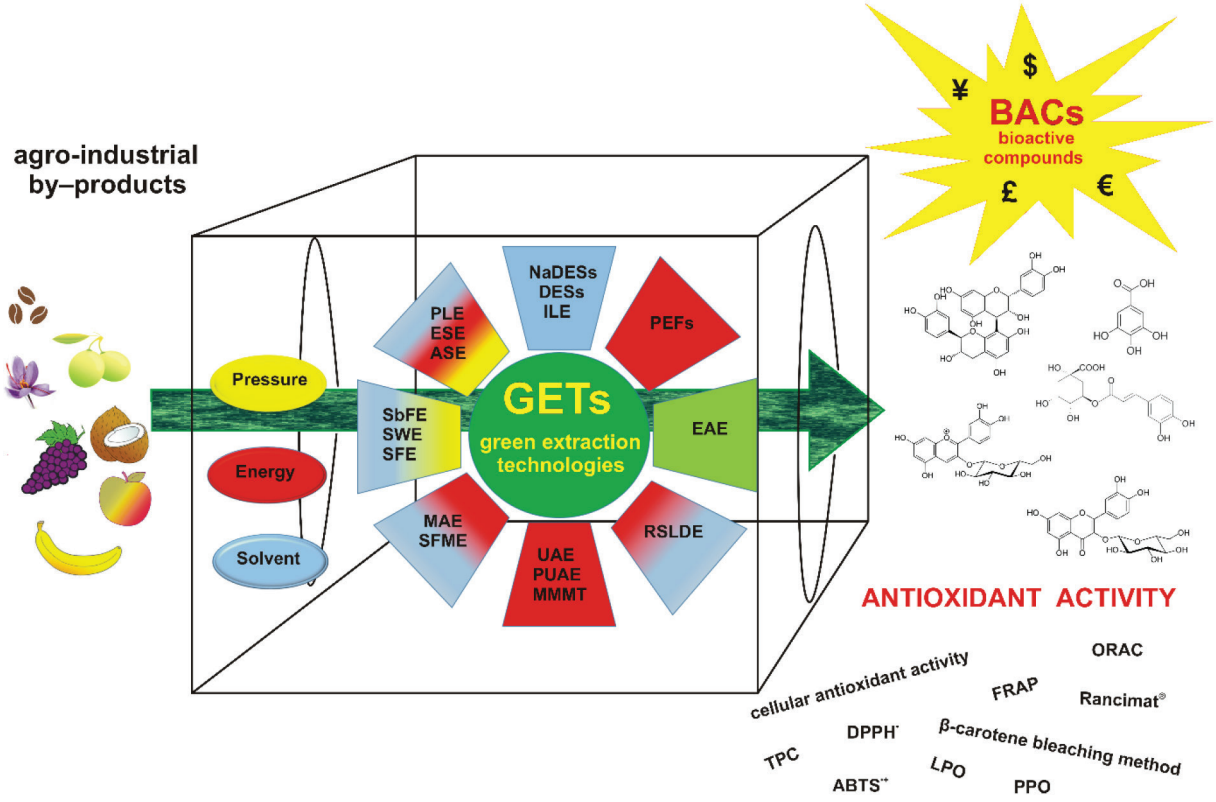

Fig. 3 – *From agro-industrial by-products to bioactive compounds with antioxidant activity (for the abbreviations, see the list at the end of the manuscript)*

covered polyphenols and anthocyanins from red grape pomace (wine industry solid waste). Furthermore, Poveda *et al.*[24] extracted different winemaking wastes (grape seeds, pomace and stems) from *Vitis vinifera* L. cv. Tempranillo, and seeds from *V. vinifera* L. cv. Cabernet Sauvignon, not only using UAE but also applying ASE. As a result, they obtained extracts rich in hydroxycinnamic acid derivatives, flavonols, tannins, catechins, and anthocyanins. Poveda *et al.*[24] also observed strong correlations among antioxidant activity of extracts and phenolic composition (Pearson correlation: 0.879 and 0.778 for $ABTS^{\bullet+}$ and $DPPH^{\bullet}$, respectively). Finally, regarding fruits by-products, Berkani *et al.*[13] applied UAE to extract polyphenols from jujube seeds. A maximum TPC (2383.10 mg GAE/100 g) was obtained under sonication temperature and time (29.01 ºC and 15.94 min, respectively), ethanol concentration (50.16 %), and solvent:solid ratio (34.10:1 mL g^{-1}). Regarding vegetable by-products as a source of polyphenols, several authors used UAE to extract these bioactive molecules. The findings of Nutter *et al.*[22] were focused on obtaining BACs from beet leaves. Moreover, surface methodology was employed to optimize UAE conditions (16 min, 90 W, and solid:solvent ratio, 1:20), under which the yields were 14.9 mg g^{-1}, 949.1 µg g^{-1}, and 562.2 µg g^{-1} for polyphenols, betaxanthins, and betacyanins, respectively. Vauchel *et al.*[31] used UAE under different operating conditions for the extraction of polyphenols from chicory grounds, while Punzi *et al.*[25] applied water UAE to obtain polyphenols from different artichoke parts (TPC: 1446, 1343, 907, and 774 mg kg^{-1} fresh weight, for hearts, leaves, outer bracts, and stems, respectively). Finally, the goal of Trujillo-Mayol *et al.*[28] was to study the combination of UAE and MAE principal factors such as sonication time, temperature, and microwave power in a new extraction procedure (U-MAE) for the maximum recovery of TPC from avocado peel by engaging a response surface methodology. As a result of the combination of optimal parameters (15 min of sonication followed by 95.1 s of microwaving) it was possible to recover 166.3 mg GAE g^{-1} dry matter, which was 1.1, 1.3, and 1.2 times greater than maceration, UAE, and MAE, respectively. Similarly, the U-MAE was higher in TPC yield (281.4 mg GAE g^{-1} dry matter), and antioxidant activity ($DPPH^{\bullet}$, FRAP and LPO: 779.1, and 167.0 µg TEAC, and 70.03 %, respectively). Boggia *et al.*[14] have also evaluated UAE and MAE of the fresh by-products (endocarp and aril residues) obtained after pomegranate juice processing, using water as extraction solvent in both cases. Dahmoune *et al.*[17] used both these methods for the recovery of TPC from lemon peels. The maximum forecasted TPC recoveries under the optimized conditions for UAE and MAE were 15.08 and 15.74 mg GAE g^{-1} dry weight, re-

spectively, which were close to the experimental values, indicating the success of response surface methodology, in optimizing the extraction conditions.[17] Moreover, regarding citrus by-products, Khan *et al.*[21] used ethanol in UAE, in order to obtain polyphenols (275.8 mg of GAE/100 g fresh weight), especially flavanones (70.3 and 205.2 mg of naringin and hesperidin/100 g fresh weight) from orange peel.

Different nuts were investigated by UAE. Tabaraki and Rastgoo[26] evaluated water, methanol, ethanol, and acetone extracts of green walnut husk. The results showed that TPC varied from 6.28 to 7.23 mg GAE g^{-1} dry sample, while FRAP and DPPH$^{\bullet}$ values varied from 0.33 to 0.46 mmol Fe^{2+} g^{-1} of dry sample, and 33.98 to 56.31 % inhibition, respectively. Tungmunnithum *et al.*[29] applied UAE with water and ethanol on Moroccan almond cold-pressed oil residues, and were able to extract protocatechuic acid, *p*-hydroxybenzoic acid, chlorogenic acid, and *p*-coumaric acid from three different native Beldi genotypes. Finally, Lameirão *et al.*[19] used industrial chestnut shells to obtained extracts rich in polyphenols (255.8–408.0 mg GAE g^{-1} dry weight). Furthermore, with the use of UAE under optimal conditions, this research group was able to detect high amounts of ellagic acid, caffeic acid derivative and epigallocatechin (40.4, 15.4, and 15.3 μg mg^{-1} dry weight, respectively).

Regarding grains, Corbin *et al.*,[16] with the use of water, methanol, ethanol, or butanol, applied to UAE technique, extracted flaxseed for phenolic compounds, including a macromolecular complex accumulated in seedcoat composed of flavonol (herbacetin diglucoside), hydroxycinnamic acids (*p*-couramic, caffeic and ferulic acid glucosides), and lignan (secoisolariciresinol diglucoside). Furthermore, Zhang *et al.*[30] used ultrasound water processing in order to recover TPC (2.79, 2.12 and 0.66 mg GAE g^{-1} of dry sample from coffee silver skin, potato peel, and brewer's spent grain, respectively) in 30 min. Liazid *et al.*[20] evaluated various GET, among which were UAE, MAE, and PLE, to obtain polyphenolic extracts from two varieties of pine (*Pinus maritima* and *Pinus d'Alpes*) seeds. Finally yet importantly, Oliveira *et al.*[23] applied a sustainable recovery extraction (UAE with different solvents, and SFE with CO_2) for the valorisation of two juice by-products, the passion fruit seed and the seed cake residue from the seed oil production by cold pressing.

Some authors have investigated agro-industrial by-products using UAE that work via pulses (PUAE). For instance, Donno *et al.*[34] applied this technique to extract different BACs (cinnamic acids, flavonols, benzoic acids, catechins, tannins, organic acids, and vitamin C) from *Castanea* spp. bud-derivatives, and their total content was 160.42 mg/100 g fresh weight. Likewise, Turrini *et al.*[35] used this method to extract the same BACs from the bud waste, remaining after the production of *Castanea sativa* glyceric macerates. The same research group extracted pomegranate juice by-products by PUAE in order to obtain polyphenol fractions.[36] Furthermore, MMMT based on UAE approach was used by some authors to extract BACs from agro-industrial by-products. Marangi *et al.*[83] used this technology to extract polyphenols (including flavonoid, chlorogenic acid and gallic acid derivatives) from hardy kiwi leaves. Moreover, the investigated extract showed a high antioxidant activity (DPPH$^{\bullet}$: IC_{50} = 270.17 μg mL^{-1}, and FRAP: 3219.55 μmol Fe^{2+} g^{-1} freeze-dried plant material). In turn, Puga *et al.*[84] were able to isolate several BACs, like 3-, 4- and 5-caffeoylquinic acid, 3- and 5-feruloylquinic acid, dicaffeoylquinic acids, caffeoyltryptophan, and caffeoylferuloylquinic acid from coffee chaff.

MAE is another GET that bases its extraction process on energy, and that has interested many researchers. Among all studied plant matrixes, it was possible to find kiwi, pitaya, grape, sea buckthorn, mango, and English walnut wastes. Carbone *et al.*[67] were evaluating MAE on kiwi juice pomace. Moreover, they were investigating the influence of the temperature, time, solvent composition and solid:solvent ratio on TPC. Ferreres *et al.*[68] were investigating MAE extracts of white-fleshed red and yellow pitaya peels. Their aim was to find the best conditions for extraction of phytochemicals (phenolic acids, flavonoids, and betacyanins). The study of Kwiatkowski *et al.*[69] was focused on MAE from grape skins of different white and red cultivars at veraison and harvest, while Périno-Issartier *et al.*[72] extracted isorhamnetin-3-*O*-rutinoside, isorhamnetin-3-*O*-glucoside, quercetin-3-*O*-glucoside, isorhamnetin from sea buckthorn pomace. The study of Sanchez-Camargo *et al.*[71] was interesting in terms of different extraction strategies. They performed MAE on supercritical CO_2 pre-extracted mango peel as a valorisation strategy, and with the aim to extract polyphenols. The highest TPC (52.08 mg GAE g^{-1} dry weight) and antioxidant activity (DPPH$^{\bullet}$: 2.75 mmol trolox equivalent g^{-1} dry weight) were obtained in 90 s and at 800 W, and 50 g mL^{-1}. Finally, Rosa *et al.*[70] applied MAE to fresh, male, English walnut flowers and unripe walnut seeds to evaluate the TCP of the extracts, as well as the percentage of water-soluble polyphenols.

Agro-industrial by-products extraction with GET based on the solvent used

Neoteric solvents (ILs, DESs, and NaDESs) have been widely applied to the extraction of polyphenols from cocoa beans, spent coffee grounds,

jabuticaba, grape and olive pomace, grape and mandarin peels, fig leaves, and onion, tomato, and pear by-products. Regarding extraction with ILs, Lima *et al.*[90] performed the extraction of anthocyanins from grape pomace via solid/liquid extraction with aqueous solutions of the ILs (1-ethyl-3-methylimidazolium acetate) and sequential purification in aqueous two-phase systems with additional salts. With the use of response surface methodology, the optimum conditions of extraction were obtained resulting in an anthocyanins yield of up to 3.58 mg g^{-1}. On the other hand, Larriba *et al.*[89] used hydrophobic ILs to replace conventional volatile organic compounds as extraction solvents to recover tyrosol from olive mill wastewater. Furthermore, 15 different DESs extracts of spent coffee grounds were investigated by Fanali *et al.*[56] for green extraction of different chlorogenic acids. Wang *et al.*[60] used ChCl-based DESs and DESs composed of glycerol, xylitol, and D-(–)-fructose in comparison with UAE and MAE based on methanol, for extraction of polyphenols and furanocoumarins present in fig leaves. Under optimal conditions, the extraction yield with DESs was 6.482, 5.207, 16.34, 15.22 and 2.475 mg g^{-1} for caffeoylmalic acid, rutin, psoralic acid-glucoside, psoralen, and bergapten, respectively.[60] In turn, Pavlović *et al.*[59] used 16 different ChCl-based DESs and DESs coupled with MAE to extract gallic acid, theobromine, caffeine, catechin, caffeic acid, and epicatechin from cocoa bean shell. Moreover, Jeong *et al.*[57] used DESs for extraction of anthocyanins from grape peels.

On the other hand, NaDESs have been reported for the extraction of jabuticaba pomace (seeds and peel containing remaining pulp), targeting a selective and sequential extraction of anthocyanin and pectin.[61] Widely produced by-products (pomace) of grape and olive were extracted by Panić *et al.*[66] and Chanioti and Tzia[62] with the use of NaDESs in order to obtain polyphenol (oleuropein, hydroxytyrsol, caffeic acid, vanillin, rutin, and luteolin) fractions. Furthermore, Manuela *et al.*[65] extracted cocoa beans to obtain polyphenol and flavan-3-ol fraction with the use of NaDESs, while Jokić *et al.*[58] performed extractions of mandarin peel, to extract hesperidin as target compound. The results of the last research group showed that the most efficient hesperidin extraction (112.14 mg g^{-1} of plant) was obtained with ChCl:acetamide (1:2), while the lowest hesperidin yield (1.44 mg g^{-1} of plant) was detected with the use of ChCl:citric acid (1:1) solvent. As a further matter, Lakka *et al.*[64] used NaDESs composed of L-lactic acid and glycine in different ratio to extract polyphenols from saffron processing waste. Finally, De los Ángeles Fernández *et al.*[63] investigated NaDESs extracts of onion, tomato, pear and olive by-products for the extraction of 14 different polyphenols, namely, gallic acid, hydroxytyrosol, tyrosol, catechin, caffeic acid, rutin, coumaric acid, *t*-ferulic acid, oleuropein, cinnamic acid, quercetin, luteolin, naringenin, and apigenin.

PEFs, as a non-thermal technique, has shown to be valuable technique for the extraction of BACs by some authors. For instance, Parniakov *et al.*[79] used this method to extract polyphenols, proteins, and carbohydrates from papaya peel, while Redondo *et al.*[82] were able to extract several polyphenols (catechin, coumaric acid, chlorogenic acid, neochlorogenic acid, and quercetin) from thinned peach fruits. Luengo *et al.*[80] applied PEFs treatment to orange peels in order to obtain extract rich in polyphenols (naringin and hesperin), while Boussetta *et al.*,[81] using PEFs, were able to valorise oilseed residues (flaxseed hulls) obtaining extracts with high level of polyphenols.

Agro-industrial by-products extraction with GET based on the solvent and pressure used

GETs that involve pressurized solvents (PLE, ESE and ASE) have been applied by several researchers to obtain BACs fractions from different plant by-products. As an example, Fernández-Ponce *et al.*[74] evaluated PLE and ESE extracts of mango leaves for polyphenols. Interestingly, ethanol improved the selectivity of the PLE process, so extracts present the highest TPC (414.9–854.7 mg g^{-1} dry extract), and the antioxidant activity of PLE and ESE extracts ranged between 3.55–5.64 μg $DPPH^{\bullet}$ μg^{-1}. In turn, Ballesteros-Vivas *et al.*[73] investigated PLE of mango seed kernel for polar fraction (polyphenols), as well as for non-polar fraction (fatty acids and lipids). On the other hand, Pagano *et al.*,[76] using hot water PLE, were able to extract from artichoke bract and leaf, flavone glycosides and caffeoylquinic acids (3–19 and 14–37 mg g^{-1}, respectively). Plaza *et al.*,[77] with the same green approach extracted different polyphenols (5-caffeoylquinic acid, hyperoside, isoquercitrin, reinutrin, phloridzin, avicularin, quercitrin, and quercetin) from apple by-products. Oliveira *et al.*,[75] with the use of PLE, were able to extract a range of polyphenols, like ellagic acid, and its pentoside and deoxyhexose, quercitrin, kaempferol pentoside, quercetin hexoside from Brazilian cherry seeds. Gallo *et al.*[78] used the cyclically pressurized extraction RSLDE to extract polyphenols from the peels and seeds of grapes.

Compressed fluids-based GET, including sub- (SbFE) and supercritical (SFE) fluid approaches, have been widely applied for the extraction of BACs from plant by-products. Regarding the subcritical approach, Guthrie *et al.*[51] used this technique for the recovery of phenolic antioxidants from green kiwi fruit peel. Under optimum conditions (160 ºC,

20 min, pH 2, and 2 % solid:solvent ratio), TPC and TFC were 51.2 mg GAE g^{-1} dry weight, and 22.5 mg CE g^{-1} dry weight, respectively. Gabaston *et al.*[50] and Yammine *et al.*[55] used SWE for extraction of BACs from grape by-products. The former were able to extract complex stilbenes from grapevine by-products (wood, cane, and root),[50] while the latter recovered different polyphenols (anthocyanins, tannins, monomeric and oligomeric flavan-3-ols) from red and white grape pomace.[55] Vegetable by-products appeared to be interesting for SWE extraction. Alvarez *et al.*[47] extracted phytochemicals, like polyphenols and carbohydrates from potato peel, while Iwassa *et al.*[52] investigated asparagus fibrous stem using this technique. The results of the second research group showed that, at 160 ºC, 60 min and under pressures ranging from 100 to 200, it was possible to quantify TPC (2020.56–2172.35 mg GAE/100 g).

On the other hand, Bodoira *et al.*[48] evaluated the economic importance of oil by-products after the oil extraction processes, by investigating SbFE of peanut skin, sesame seed cake, and pistachio nut cake. They investigated the possibility of using non-polluting extraction technologies for making natural biopesticides from these nut and grain wastes.[48] Narita and Inouye[54] and Jokić *et al.*[53] focused their attention on grain and nut by-products, evaluating SWE extracts of coffee silverskin for polyphenol, 5-caffeoylquinic acid, caffeine, 5-hydroxymethylfurfural, sugar, and protein content. It was observed that the antioxidant activity increased with increments of the temperature. Thus, the highest antioxidant activity was observed with the extracts obtained at 270 ºC (2629 and 379 μmol TE g^{-1} of coffee silverskin extract, for H-ORAC and DPPH•, respectively).[54] Jokić *et al.*[53] extracted cocoa shell, to extract a variety of bioactive molecules, like polyphenols, sugars (mannose, glucose, xylose, arabinose, rhamnose, and fructose), as well as their degradation products such as formic acid, levulinic acid, lactic acid, 5-hydroxymethylfurfural, and furfural. Jokić *et al.*[53] were able to obtain different concentration of epicatechin, catechin, theobromine, caffeine, theophylline, chlorogenic acid and gallic acid, applying different extraction conditions. The TPC and scavenging activity with the use of DPPH• assay was 27.26–130.33 mg GAE g^{-1} extract, and 19.20–91.69 %, respectively. Fabian *et al.*[49] used SWE to extract phenolic acids (gallic acid, caffeic acid, *p*-coumaric acid, and ferulic acid) from rice bran, while Ahmadian-Kouchaksaraie *et al.*[46] investigated saffron petals SWE extracts for polyphenol and flavonoid content. They determined that optimum extraction conditions (159 °C, 54 min and water:solid ratio of 36 mL g^{-1}) resulted in the best TPC (1616 mg/100 g), TFC (239 mg/100 g), FRAP value (5.1 mM), and DPPH• (86.05 %).

In addition, an SFE approach was shown to be a common GET. Castro-Vargas *et al.*[39] investigated recovery of phenolic antioxidants from papaya seeds, and the highest TPC (15.34–34.23 mg GAE g^{-1}) was found in extracts obtained with CO_2-EtOH and ethanol. In contrast, Marić *et al.*[41] applied SFE to separate oil from seeds. Consequently, raspberry seed oils were analysed in terms of fatty acids content, tocopherols, and functional quality indices, while the residues after extractions were investigated in terms of free and total ellagic acid. Cocoa bean by-products were shown to be rich in BACs. Similarly, Valadez-Carmona *et al.*[45] used SFE to extract phenolic compounds from cacao pod husk. The extract gained at the optimum conditions (60 °C, 13.7 % of ethanol, and 299 bar) presented TPC (12.97 mg GAE g^{-1} extract) and antioxidant activity (0.213 mmol TE g^{-1} extract). In turn, Mazzutti *et al.*[42] applied pressurized ethanol extraction as well as SFE on cocoa bean hull, aiming to extract polyphenols and volatile compounds.

Other agro-industrial by-products investigated by SFE are leaves. As an example, Pavić *et al.*[43] were able to recover carnosol (0.46–65.5 μg mg^{-1}) and carnosoic acid (0.29–120.0 μg mg^{-1}) from sage leaves. Borja-Martínez *et al.*[38] extracted polyphenols, chlorophylls, *β*-carotene, *α*-tocopherol, and phytosterols from broccoli leaves and stems, while Lafka *et al.*[40] evaluated polyphenol and antioxidant potential of olive oil mill wastes SFE extracts. Campone *et al.*[15] developed an innovative and green SFE method with ethanol/water as a co-solvent to quantitatively extract flavonoids from onion skin. In the same paper, UAE was used to obtain the chemical profile of secondary metabolites of exhaustive extract. Very important agro-industrial wastes extracted with the use of SFE are grape pomace, skins and seeds. Pérez *et al.*[44] focused their attention on red grape seeds, from which they were able to extract fatty acids, and a range of phenolic acids (gallic acid, caffeic acid, *p*-coumaric acid, ferulic acid, and ellagic acid). In contrast, Aresta *et al.*[37] studied SFE of winemaker by-products (pomace, skins, and seeds) for polyphenol, *t*-resveratrol, *β*-sitosterol, and vitamins (*α*-tocopherol and vitamin C) content. Skin was shown to be the richest in polyphenols (603 μg GAE g^{-1} dry matter), while similar antioxidant activity was observed in skin and pomace (ca. 0.12 mmol trolox/g dry matter).

Agro-industrial by-products extraction with GET based on enzymatic approach

The final GET described in this study is EAE. Gómez-García *et al.*[86] applied EAE on grape residues in order to extract polyphenols (gallic acid,

resorcinol, and *o*-coumaric acid). In their findings, a good correlation between polyphenols and antioxidant activity was obtained. Moreover, the highest antioxidant activities (86.8, 82.9 and 90 %) were registered at 12 h for Celluclast® 1.5 L, Pectinex® Ultra and Novoferm®, respectively. Saad *et al.*[88] applied different combinations of carbohydrases and proteases to raspberry pomace and pomace presscake for the recovery of polyphenols (2.7 and 2.5 g per 100 g of dry sample, respectively), and lipophilic compounds. Madeira and Macedo[85] were to obtain polyphenols from Brazilian citrus (*Citrus latifolia* and *Citrus sinensis* L.) albedo and flavedo, with high bioactivity, using simultaneous extraction (cellulase and pectinase) and biotransformation (tannase) by enzymatic process. The highest hesperetin, naringenin and ellagic acid production in this study (conditions: 40 ºC, 200 rpm, 5.0 U mL^{-1} of cellulose, and 7.0 U mL^{-1} of tannase) were 120, 80, and 11,250 μg g^{-1}, respectively. Likewise, Puri *et al.*[87] investigated EAE of kinnow peel. The aim of this research group was to develop an enzymatic hydrolysis of naringin that would simplify the processing of kinnow peel waste.

Regarding the analysed literature for this review and other experimental findings, it is difficult to choose, among all presented GETs, the most acceptable technique.[109] Each of them has advantages. Furthermore, regarding the BACs, it is not possible to choose just one GET that will allow for the best extraction of active compounds in general. For instance, focusing on polyphenols, the scientific findings show evidently that the extraction efficiency is determined by the type of extracted phenolic compound, its position in plant as well as type of plant material, quality, and selectivity. Furthermore, some GETs are able to extract the intracellular molecules selectively without fragmenting the treated tissue. Therefore, in choosing an extraction process, the selectivity, productivity, source, and total yield have to be taken into consideration. Also from our experience, extracting different plant materials and their by-products with different extraction type and conditions, allow to extract preferentially specific compounds of interest. To sum up, before choosing the right extraction method for a given ingredient or product, the final goal has to be defined, and the characteristics of each available method have to be considered.

In a broader view, the game-changing approach to use agro-industrial by-products in biorefinery allows the production of many biobased products (bioenergy, biofuels and other valuable chemicals). Although biofuels are mainstay of biorefineries, production of improved biomolecules such as biopolymers, biopigments, and biosurfactants has been widely studied in the last years with the use of traditional methods. New approaches including holistic extraction and purification technologies with the use of renewable materials and improved processes should follow to evaluation of the entire production chain.[110] Hence, reduction in the production costs benefits waste recycling, making the environment eco-friendly. Furthermore, agro-industrial by-products favourable application in various industries (e.g., chemical, pharmaceutical, and nutraceutical), drives scientists to develop novel green approaches to make the technology cost-effective, and generating more efficient and healthier products used as active compounds (pharmaceutics), food supplements (nutrition), and ingredients (food, home and personal care).[111,112]

Comprehensive investigation of previous and current literature confirms effectiveness of GETs in biorefinery.[112] Moreover, regarding extraction at industrial scale, conventional technologies, due to their many disadvantages (e.g., time consuming extraction, scarce yield recovery, intensive heating and/or mixing, and toxicity), should be replaced with GET. It is well known that biorefineries play a key role in sustainable bio-based economy. They involve the sustainable processes of converting bio-materials into marketable products. Furthermore, in a biorefinery, the idea of green extraction processes for extraction of natural products is focused on effective energy use, reduction in processing steps and equipment size, as well as on enhanced heat and mass transfer. The scientific findings show that many extraction applications may make a big difference in biorefineries, by significantly decreasing the costs and therefore making the processes competitive. Nevertheless, it is necessary to highpoint the exact separation challenges faced in biorefineries and indicate the effect of separations on the total costs.[113] Numerous separation technology challenges in biorefeneries depend on high temperatures, existence of reactive mixtures, complex organic matrix that can contain inorganic compounds, polarity of components in the mixture, and very diluted water solutions.[113]

Conclusions

Turning unmanaged agro-industrial by-products into valuable source of BACs is a viable solution for both eliminating food wastes and at the same time obtaining high added value products. Thus, nowadays, the agro-industrial sector is pushed to increase its overall efficiency by optimizing the life cycle of their processes and products. This can be reached by upgrading existing processes or discovering new uses for waste. In this context, GETs have proved to be reliable strategies for recovering

different types of BACs, including polyphenols and other antioxidant compounds. Moreover, the reprocessing of agro-industrial by-products may create secondary streams to obtain, for instance, valid models to design semisynthetic derivatives and/or synthetic analogues with enhanced health properties. However, several challenging aspects of GETs to optimize rates of recovery and the degree of BACs purity are yet to be overcome. The possibility of GETs being scaled-up at industrial level will have to take into account the proper choice of separation techniques and conditions (e.g., solvent type, material costs, extraction times, etc.) after economic analyses. However, it is incontrovertible that the development of GET for obtaining BACs from agro-industrial by-products could improve economic viability by creating profits in upcoming sustainable manufacture system.

List of abbreviations

ABTS$^{\bullet+}$ – free radical scavenging ability assay using a stable ABTS radical cation
ASE – accelerated solvent extraction
BACs – bioactive compounds
ChCl – choline chloride
CUPRAC – cupric reducing antioxidant capacity assay
DES/s – deep eutectic solvent/s
DPPH$^{\bullet}$ – free radical scavenging ability assay using DPPH$^{\bullet}$ stable free-radical
EAE – enzyme assisted extraction
ESE – enhanced solvent extraction
FRAP – ferric-reducing ability of plasma assay
GAE – gallic acid equivalent
GET/s – green extraction technique/s
GRAS – generally recognized as safe
HBA – hydrogen bond acceptor
HBD – hydrogen bond donator
H-ORAC – hydrophilic oxygen radical absorption capacity assay
ILE – ionic liquid extraction
ILs – ionic liquids
LPO – lipidic peroxidation inhibition assay
MAE – microwave-assisted extraction
MMMT – multi-frequency multimode modulated technology
NaDES/s – natural deep eutectic solvent/s
NSs – neoteric solvents
ORAC – oxygen radical absorption capacity assay
PEFs – pulsed electric fields extraction
PLE – pressurized liquid extraction
PPO – polyphenol oxidase assay
RSLDE – rapid solid liquid dynamic extraction
PUAE – pulsed ultrasound-assisted extraction
SbFE – subcritical fluid extraction
ScF/s – supercritical fluid/s
SFE – supercritical fluid extraction
SFME – solvent-free microwave extraction
SWE – subcritical water extraction
TBARS – thiobarbituric acid reactive substances assay
TFC – total flavonol content
TPC – total polyphenolic content determined with a Folin-Ciocalteu's method
UAE – ultrasound-assisted extraction

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CONFLICT OF INTEREST

The authors have declared no conflict of interest.

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