Industrial Crops & Products 209 (2024) 117906

Contents lists available at ScienceDirect

Industrial Crops & Products

journal homepage: www.elsevier.com/locate/indcrop

New trends in extraction-process intensification: Hybrid and sequential green technologies

Giorgio Capaldi, Arianna Binello, Clelia Aimone, Stefano Mantegna, Giorgio Grillo **, Giancarlo Cravotto *

Dipartimento di Scienza e Tecnologia del Farmaco, University of Turin, Via P. Giuria 9, Turin 10125, Italy

ARTICLE INFO

Keywords:
Green extraction
Coupled synergic technologies
Hybrid technologies
Biomass valorization
Process intensification
Extraction scaling-up

ABSTRACT

The multidisciplinary "Green Extraction" approach has recently been expanded by merging the concepts of Green Chemistry and the Circular Economy. This approach considers waste from different production chains to be a valuable resource for other sectors, and can significantly improve the economic and environmental sustainability of current processes on a global scale. The goal is to produce high-value products from residual biomass in an inherently safe and environmentally friendly way, which is referred to as being "benign-by-design". In this framework, the application of enabling technologies can ideally maximize heat and mass transfer, leading to significant improvements in yield/conversion rates, energy savings, and lower production costs. However, this trend is becoming mandatory and requires even more effort. New solutions are needed to meet increasing demand, and two or more enabling technologies can be combined in sequential or simultaneous systems to dramatically increase process intensification and applicability. These combinations can significantly improve mixing and heating, as well as biomass pretreatment and metabolite dissolution. This literature survey focuses on both hybrid and coupled green-extraction technologies over a five-year period and highlights the differences between the two strategies, their achievements and technology readiness levels (TRLs). The study underlines the potential offered by synergistic combinations of green technologies for the extraction of bioactive compounds from biomass. The information gathered provides an outlook as to the state-of-the-art, and highlights the challenges that must be overcome and the opportunities that exist for future extraction research.

1. Introduction

In recent decades, the new concept of "Green Chemistry" has emerged, in response to growing interest in environmental, economic and safety issues, and has driven the current development of modern chemistry and pioneering innovative, cutting-edge solutions (Hashemi et al., 2022). The principles of Green Chemistry form the basic pillars of sustainable transformations, especially with regards to the exploitation of raw materials and renewable energy sources, along with energy and material savings (Chemat et al., 2021). What constitutes waste for certain production chains can be reused in many other fields. In fact, with appropriate transformations, residues can play the role of secondary raw materials for various applications (e.g., chemistry, energy, biorefineries, nutraceuticals, animal feed), thus operating in perfect

Abbreviations: ABTS, 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid); ATPS, Aqueous Two-Phase System; DIC, Controlled Instantaneous Pressure Drop; DPPH, 2,2-diphenyl-1-picrylhydrazyl; EAE, Enzyme-Assisted Extraction; EASOE, Enzyme-Assisted Salting-Out Extraction; EMAE, Enzyme and MW Assisted Extraction; EMASOE, Enzyme and MW Co-Assisted Salting-Out Extraction; Ext, Extrusion; FRAP, Ferric Reducing Antioxidant Power; FSPs, Fucose-Sulphated Polysaccharides; HD, Hydrodistillation; HIU, High-Intensity US; HPH, High-Pressure Homogenization; HSH, High-Shear Homogenization; HWE, Hot Water Extraction; IPL, Intense Pulsed Light; LIU, Low-Intensity US; MAE, MW-Assisted Extraction; MAHD, MW-Assisted Hydrodistillation; MASOE, MW-Assisted Salting-Out Extraction; MNPs, Magnetic Nanoparticles; MW, Microwaves; NADES, Natural Deep Eutectic Solvents; OH, Ohmic Heating; PEF, Pulsed Electric Field; PLE, Pressurized Liquid Extraction; $ScCO_2$, Supercritical CO_2; SCHE, Spiral-Coil Heat Exchanger; SEM, Scanning Electron Microscope; SFE, Supercritical Fluid Extraction; SWE, Subcritical Water Extraction; TFC, Total Flavonoids Content; TPC, Total Polyphenols Content; UAE, Ultrasound-Assisted Extraction; US, Ultrasound; USAEE, US Assisted Enzyme Extraction; USMAE, US-MW Assisted Extraction; WE, Water Extraction.

* Corresponding author.
** Co-Corresponding author.
E-mail addresses: giorgio.grillo@unito.it (G. Grillo), giancarlo.cravotto@unito.it (G. Cravotto).

https://doi.org/10.1016/j.indcrop.2023.117906

Available online 23 December 2023

harmony with the Circular-Economy concept (Esposito et al., 2020).

By merging the concepts of Green Chemistry and the Circular Economy, the multidisciplinary approach of "Green Extraction" has recently been established to drive the development of new and sustainable extraction procedures that aim to: save time and energy, enhance extraction yields, reduce organic-solvent consumption and valorize natural resources (Chemat et al., 2012). In particular, the goal is to achieve high-added-value products from renewable biomass, in an intrinsically safer and eco-friendly way, thus introducing the concept of "benign-by-design" (Anastas, 1995). Fig. 1 depicts the trend in interest in green extraction, in terms of publication numbers, from 2000 to the present, with 19,263 results coming from the Web of Science Core Collection, with Food Science leading the publication outcomes, followed by multidisciplinary chemistry and chemical engineering (Web of Science, 2023a). The exponential increase in numbers from the 2010s is worth noticing, as it was driven by the concepts and principles of green natural-product extraction that were exhaustively discussed by Chemat et al. (2012). The field has also experienced a dramatic increase over the last 5 years, with 10,755 articles being observed in the Web of Science Core Collection, with Multidisciplinary Chemistry overtaking Food Science and with the rise of Environmental Science (See Fig. 1) (Web of Science, 2023b).

The most attractive naturally occurring bioactive molecules to be recovered from vegetal biomasses are proteins, cellulose, hemicellulose and lignin, in terms of primary metabolites, and polyphenols, carotenoids, vitamins and essential oils, in terms of secondary metabolites. All of these compounds have interesting prospects and are in demand by industries in the pharmaceutical, cosmetic and nutraceutical sectors because of either their biological activity or their exploitability as building blocks for bioethanol, biogas and solvent conversion. The main enabling technologies involved in green-extraction procedures include ultrasound-assisted extraction (UAE) (Khadhraoui et al., 2021; Grillo et al., 2020), microwave-assisted extraction (MAE) (Angiolillo et al., 2015), pulsed electric field (PEF) (Naliyadhara et al., 2022; Grillo et al., 2022), ohmic heating (Kaur, Singh, 2016) hydrodynamic cavitation (HC) (Verdini et al., 2021), supercritical fluid extraction (SFE) (Sahena et al., 2009) subcritical water extraction (SWE) (Cravotto et al., 2022) and mechano-chemistry-assisted extraction (MCAE) (Liu et al., 2022; Gaudino et al., 2022). In addition to the listed processing methods, sustainable protocols also involve biological strategies, in particular, the use of enzymes (Nadar et al., 2018). Nevertheless, each of these novel strategies have advantages and limitations, and their effectiveness is strongly influenced by the type of matrix and targeted product (Soquetta et al., 2018). The ground-breaking introduction of these procedures, however, may not be enough due to the compelling need to push forward the enhancement of process sustainability and efficiency on an industrial scale. The challenge here then is how to combine technologies to create integrated systems that can overcome individual drawbacks and achieve superior results by exploiting synergistic effects and merging diverse mechanisms (More et al., 2022).

This can be done, for example, via the sequential application of two techniques, in two physically or temporally separate steps. This method is commonly referred to as a pretreatment coupled with a subsequent extraction. The natural evolution of this concept is the simultaneous application of different technologies, carried out *in situ* or in flow, known as "hybrid technologies," which can be conceived either in the fusion of two physical methods, such as PEF and MAE, or US and MAE, or in the combination of physical and biological treatments.

Sequential and hybrid systems are generating intensifying attention within the scientific community. More precisely, where interest in pretreatments appears to be based on well-established values, the trend towards hybrid approaches demonstrates significant growth potential with considerable free space for research to overcome challenges in these developing areas.

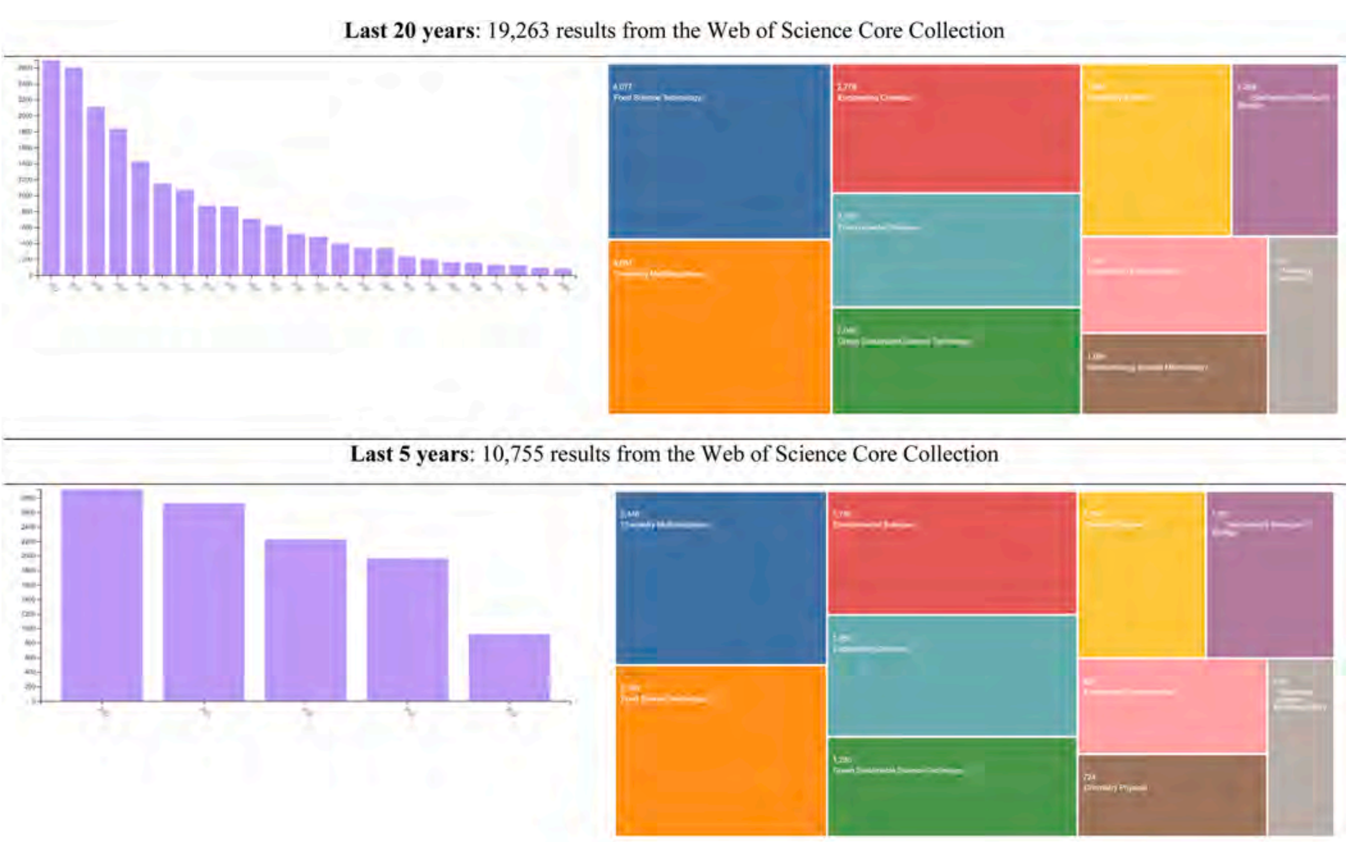
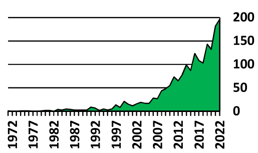
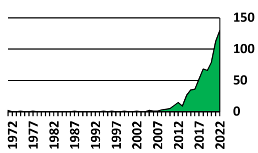
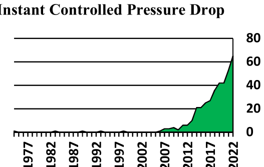
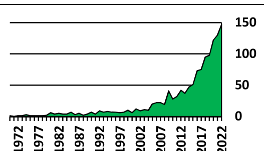
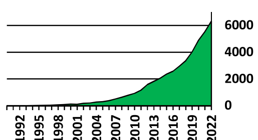
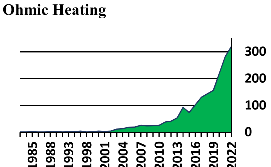


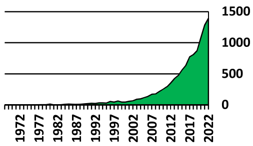
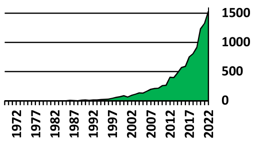
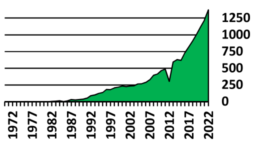
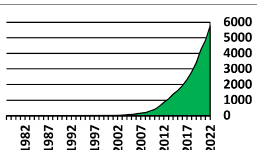
Fig. 1. Web of Science research (Clarivate); Keyword: Green extraction, accessed on 5 May 2023.

Table 1
Summary and description of the main green technologies considered in this review. (Abad et al., 2023; Kamble et al., 2022; Lee et al., 2012; Taha et al., 2022; Teresa-Martínez et al., 2022; Vidovic et al., 2020).

Technology	*Abbr.*	*Method description*	*Modulable parameters*	*Advantages*	*Disadvantages*	*Ref.*
Extrusion 	-	Continuous and low-cost thermo-mechanical process. The apparatus consists of a heated chamber (or orifice) in which the biomass is squeezed by one or more screws. The imprinted shear forces, together with the high temperature, deconstruct the inner structure of the matrix compressing it in a pellet-shaped material. The product is more porous than the starting material and allows better solvents penetration.	▪ Chamber temperature (°C), ▪ Screw rotational speed (rpm), ▪ Number and geometry of screws.	▪ No solvents ▪ Continuous work mode ▪ Low cost ▪ Easy usable and scalable ▪ High flexibility	▪ Thermal and shear damage	(Guiao et al., 2022; Prabha et al., 2021)
High-Shear Homogenization 	HSH	Process driven by the high shear effect caused by the high-speed movement between a rotor and a stator, fluids or raw matrices undergoes to hydraulic and shear stress, by breaking large dimension particles or aggregates of vegetal fibres and tissues, resulting in a more homogeneous sample.	▪ Rotational speed (rpm) ▪ Shear rate (s^{-1}), ▪ Design and size of rotor and stator.	▪ Only water ▪ Crushing and blending vegetal matrix ▪ Highly efficient in cell disruption ▪ Flow-mode working possibility	▪ Feed limitation: clogging of the system ▪ Risk of damaging the most sensitive compounds ▪ Too much finely sized debris led to separation issues ▪ Product overheating	(Lee et al., 2012; Kamble et al., 2022)
Instant Controlled Pressure Drop 	DIC	Process that involves a thermomechanical effect induced by subjecting the material to a fast transition from saturated steam high pressure to vacuum, for a short period. The pressure drop causes the auto vaporization of the water, the swelling of the matrix and a possible breakdown of the cell wall. This treatment induces a change in the structure of the material, making it more porous and permeable to solvent.	▪ Temperature (°C), ▪ Number of DIC cycles ▪ Total heating time (s)	▪ No solvents, only steam ▪ Increasing water diffusivity and surface exchange area (as a pretreatment) ▪ Quick process ▪ Higher extract quality for essential oils extraction	▪ Thermal degradation phenomena	(Allaf et al., 2013; Teresa-Martinez et al., 2022)
Intense Pulsed Light 	IPL	Non-thermal technology, the sample is irradiated with intermittent bursts, usually provided by a xenon lamp (λ=200 - 1100 nm), for few hundreds of μs.	▪ Voltages (V) ▪ Time (μs) ▪ Wavelength (nm)	▪ No solvent or only water ▪ Low loss of nutrients and energy cost ▪ Very short treatment times	▪ Possible degradation of the phenolic metabolites ▪ Biomass disinfection	(Kim et al., 2019)
Microwave-Assisted Extraction 	MAE	The transformation of electromagnetic energy into heat is operated by two mechanism: ionic conduction and dipole rotation. Thus, causing a heating of the solvent/matrix allowing the extraction of several compounds. Typically, it works with a frequency of 900mHz or 2.45 GHz.	▪ Power (W) ▪ Frequency (GHz) ▪ Temperature (°C) ▪ Stirring mode and speed.	▪ Efficient and fast heating ▪ Small size equipment ▪ Low energy cost and easy-usable ▪ Possibility of working under inert atmosphere ▪ Increasing extraction yield and product quality	▪ Thermal and chemical degradation ▪ Extraction solvent must absorb microwave energy ▪ Different outcomes according to the polarity of treated compounds	(Khadhraoui et al., 2021)
Ohmic Heating 	OH	An electric current pass through the sample, which acts as a resistor, dissipating electrical energy into thermal energy by means of Joule effect. Thus, a rapid and volumetric heating is generated directly within the material. Usually works in the kHz field.	▪ Frequency (kHz) ▪ Electrical conductivity (S/cm), peculiar of solvent/biomass system ▪ Electric field intensity (V/cm) ▪ Particle size/concentration/orientation ▪ Ionic concentration ▪ Electrodes materials	▪ Electroosmosis effect ▪ Several possible applications ▪ Easily- scalable ▪ Avoiding thermal damages by means of flash volumetric heating ▪ High energy efficiency	▪ Limited feasibility on low conductivity samples ▪ Possibility of thermal runaway ▪ Electrodes obsolescence, according to the involved material.	(Kaur et al., 2016)
Pulsed Electric Field	PEF	Electroporation phenomena achieved using electrodes able to send high-voltage electrical pulses of μs. Facilitating solvent penetration into the biomass and the subsequent elution of the target compounds	▪ Load voltage (kV) ▪ Pulse width (μs), ▪ Number of pulses ▪ Frequency (Hz), ▪ Total specific energy (kJ/kg) ▪ Field strength (kV/cm).	▪ Only water ▪ Very short time ▪ Non-thermal degradation ▪ Efficient pretreatment for recalcitrant matrixes ▪ High scalability ▪ Flow-mode working possibility	▪ High energy consumption ▪ Existence of bubbles during treatment, with consequent operational problems and non-uniform treatment ▪ Not widely available in many regions worldwide	(Abad et al., 2023; Taha et al., 2022)

(*continued on next page*)

Table 1 (*continued*)

						
Subcritical Water Extraction 	SWE	The subcritical state tailor the nature of water, decreasing its dielectric constant and favouring the solute transfer processes such as diffusion, partition equilibrium and convection. Requires pressure to maintain the water in the liquid form.	▪ Temperature (100°C - 374°C) ▪ Pressure (bar and different atmospheres).	▪ Only water ▪ High temperature and pression can be exploited for water removal (flash evaporation) ▪ Short extraction time ▪ High extraction yield	▪ General cost of water evaporation ▪ Thermal degradation phenomena.	(Vidovic et al., 2020; Zhang et al., 2020a)
Supercritical CO_2 	ScCO_2	Most common application of Supercritical Fluid Extraction (SFE). The supercritical state of a fluid is reached above its critical temperature and pressure, typically used for non-polar compounds recovery.	▪ Temperature (°C) ▪ CO_2 flow rate (kg/h) ▪ Pressure (bar) ▪ Additives (*i.e.* EtOH)	▪ Reducing the use of organic solvents ▪ Fast extraction kinetic ▪ Possibility of changing the selectivity by adding co-solvents	▪ Not feasible for heat-sensible compounds ▪ Formation of carbonic acid in presence of residual water: possible degradations.	(Taha et al., 2022)
Ultrasound-Assisted Extraction 	UAE	Low-frequency ultrasound (US), typically 20kHz-100kHz, usually exploited at high intensity (10-1000 W/cm^2), finds several applications in food industry for their cavitation effects, consisting in the formation and subsequential collapse of micro-bubbles inside a liquid medium. The implosion generates forces able to damage materials, (detexturisation of the matrix, increasing contact surface area and solvent penetration capacity), thus enhancing the mass transfer of the target molecules.	▪ Amplitude (μm) ▪ Frequency (Hz) ▪ Acoustic energy intensity (W/cm^2) ▪ Energy density (J/mL) ▪ Power density (W/mL) ▪ Temperature (°C).	▪ Easy usable and scalable ▪ Flow-mode working possibility ▪ High mass-transfer	▪ Possible degradation of targeted compounds by free radicals formation ▪ Biomass comminution could lead to filtration issues ▪ High noise levels ▪ Temperature control required (system overheating) ▪ High S/L ratio not allowed.	(Khadhraoui et al., 2021)

This review presents an overview of sequential (i.e., pretreatment) and hybrid extraction technologies presented in the literature over the last 5 years.

The information has been collected in this paper with the aims of providing scientists, from universities and industry, with an overview of the state-of-the-art of hybrid and coupled green extraction technologies, together with an outline of future challenges to be overcome and the opportunities to be seized on an economic and ecological scale for industrial deployment.

2. Green extraction technologies: a quick view

Green extraction technologies are a precious way to enhance the recovery of valuable compounds in a sustainable and cost-effective manner. Each technique is characterized by peculiar features that can be either advantages or weak points. It therefore becomes necessary to design appropriate combinations (sequential or simultaneous) that can generate synergic effects to overcome each technique's shortcomings and further push process enhancement forward. To contextualize the following sections, the green technologies considered across this review are briefly depicted in Table 1, with their principles, modifiable parameters, pros and cons.

3. Non-simultaneous combination of green extraction technologies - innovative pretreatment for efficient extraction

An effective strategy for increasing the efficiency of vegetal-matrix extraction processes may be the implementation of a sample pretreatment step. The key role of this operation is the disruption of matrix structure to enhance solvent permeation and expand surface area. These structural changes favour the desorption of target compounds within the solution, boosting the solvent's extractive capacity. Several characteristic aspects of the raw material must be considered for the correct choice of pretreatment procedure as a poor choice can affect the quality of the main extractable compounds, leading to irreversible chemical changes, such as oxidation, hydrolysis and the condensation of the components (Krakowska-Sieprawska et al., 2022). There are several strategies to achieve effective matrix pretreatment, and these include mechanical and shear forces, microwaves, ultrasound and biological approach with enzymes (Table 2).

3.1. Extrusion

Extrusion technology is a continuous and low-cost thermomechanical process, commonly used in metallurgical, food, chemical and polymer sectors (Guiao et al., 2022a). Extrusion apparatus consists of a heated chamber (or orifice) inside which the biomass is squeezed by one or more screws (Prabha et al., 2021). (Fig. 2).

The shear forces and high temperatures generated deconstruct the inner structure of the matrix compressing it into a pellet-shaped material. The product is more porous than the starting material and allows for better solvent penetration. Although literature has reported some examples of extruded non-lignocellulosic biomass (Fadel et al., 2018; Perez-Pirotto et al., 2022), this technology is mostly applied to highly recalcitrant lignocellulosic material that is derived directly from forestry- and agricultural-industry residues and most of which is currently burned as an energy source (Guiao et al., 2022b). However, these matrixes are a significant feedstock as a highly available raw material for cellulose, hemicellulose and lignin. In particular, whilst lignin is considered to be an important source of polyphenols, cellulose and hemicellulose can be used for bioethanol production, (Tribot et al., 2019) and, following the extrusion procedure, these two

Table 2
Summary of non-simultaneous combinations of green extraction technologies.

Technologies	*Source*	*Scientific name*	*Target compounds*	*Pretreatment effect*	*Ref.*
Ext + US	Rice hulls	*Oryza sativa* L.	Fermentable sugars	Enhance enzymatic hydrolysis, higher yield	(Zhang et al., 2020a)
Ext + US	Wheat bran	*Triticum aestivum*	Phenols	Higher yield with the same phenols profile	(Fadel et al., 2018)
Ext + $ScCO_2$	Red pepper	*Capsicum annuum* L.	Carotenoids	Substrate densification	(Uquiche et al., 2022)
HSH /PEF + WE	Green spirulina powder	*Arthrospira platensis*	Soluble protein, carbohydrates, and C-phycocyanin	Higher purity of extracts and lower energy consumption, similar results for HSH and PEF as a pretreatment	(Carullo et al., 2021)
HSH + UAE	Seafood by-products and green seaweed	*Clupea harengus,* *Vaccinum vitis-idaea* *Ulva fenestrata*	Proteins	– –	(Zhang et al., 2022)
PEF + SLE	Brewer spent grain	–	Polyphenols	–	(Martín-García et al., 2020)
PEF + water infusion	Prickly pears (pulp and peels)	*Opuntia ficus-indica* L.	Betanin and isobetanin	Higher yield	(Koubaa et al., 2016)
PEF + SWE	Green tea	*Camellia sinensis* L.	Catechins	Higher yield Lower temperature (SWE)	(Hwang et al., 2021)
PEF + SWE	Satsuma mandarin peels	*Citrus unshiu*	Hesperidin and narutin	Higher yield	(Hwang et al., 2021)
PEF + SWE	Onion skins	*Allium cepa* L.	Flavonoids	Higher quercetin recovery Lower temperature (SWE)	(Kim et al., 2022)
PEF + PLE	Green spirulina powder	*Arthrospira platensis*	Antioxidant compounds (protein, polyphenols, chlorophyll a, chlorophyll b, and carotenoids)	Higher quantity of phenolic compounds, antioxidant activity, increasing variety of recovered metabolites	(Zhou et al., 2022a)
PEF + UAE	Rosemary and thyme	*Rosmarinus officinalis* L. *Thymus vulgaris* L.	Phenolic fraction	Reducing operating time and energy input required	(Tzima et al., 2021)
PEF + UAE	Chenpi	*Pericarpium Citri Reticulatae*	Flavonoids	Highest content of hesperidin, nobiletin and tangeretin	(Zhan et al., 2022)
MAE + PEF + Ext	Niger seeds	*Guizotia abyssinica* (L.f.) *Cass.*	Oil	Slight increment in quality and yield	(Mohseni et al., 2020)
PEF + MAE	Coffee pulp	*Coffea arabica* L.	Antioxidant compounds	Higher phenolics extraction yield	(Macías-Garbett et al., 2022)
PEF + MAE	Jackfruit	*Artocarpus heterophyllus* Lam.	Pectin	Increase pectin yield and quality, reducing energy intake	(Lal et al., 2021)
PEF + mech. pressing	Grass and clover juice	*Lolium perenne* L. *Trifolium repens* L.	Proteins	Higher yields (+25 %)	(Guo et al., 2022)
PEF + wine production	Grapes	*Vitis vinifera* L.	Esters and terpenes	Higher extraction performance due to cell permeabilization Reducing fermentation time Increased turbidity and lower polyphenols content	(Fauster et al., 2020)
OH + water infusion	Grape skins	*Vitis vinifera* L.	Anthocyanins	Double yield compared to untreated sample Reduced energy consumption Major yields if thermal and non-thermal effects are combined	(Pereira et al., 2020)
OH + US	Cherries	*Cornus mas* L.	Polyphenols	Increasing in TPC yields proportionally to the applied electric field Reduction of extraction time up to 34 %	(Kutlu et al., 2021a)
OH + MAE	Cherries	*Cornus mas* L.	Polyphenols (anthocyanins)	Enhanced TPC yield and antioxidant activity, anthocyanins preservation Reduced process time Unaltered extraction yield according to different applied electric fields	(Kutlu et al., 2021b)[.]
OH + water extraction	Curcuma	*Curcuma longa* L.	Curcuminoids	Higher increase in curcuminoids yield than US pretreatment	(Le-Tan et al., 2022)
OH + water extraction	Artichoke	*Helianthus tuberosus* L.	Inulin	Low electric field strength led not to a significant increasing in inulin yield	(Khuenpet et al., 2017)[,]
IPL + SWE	Onion skins	*Allium cepa* L.	Quercetin and other phenolics	Major presence of quercetin on matrix surface, released from inside of the cell Long exposition can lead to sensible-compounds degradation	(Kim et al., 2019)
IPL + SWE	Green tea leaves	*Camellia sinensis* L.	Catechins	Higher recovery Possible degradation caused by stronger treatments	(Hwang et al., 2021a)
UAE + SWE MAE + SWE	Spent coffee ground	*Coffea arabica* L.	Polyphenols, sugars, proteins and antimicrobial power	MAE gives better extraction yields than the US	(Getachew, Chun, 2017)
UAE + SWE MAE + SWE $ScCO_2$ + SWE			Antioxidant polysaccharides	US results as the best pretreatment	(Getachew et al., 2018)
UAE + SWE	Tartary buckwheat	*Fagopyrum tartaricum* L. Gaerth	Polyphenols	Lower TPC yield, lower antioxidant capacity	(Dzah et al., 2020)

(*continued on next page*)

Table 2 (*continued*)

Technologies	*Source*	*Scientific name*	*Target compounds*	*Pretreatment effect*	*Ref.*
UAE + $ScCO_2$	Candytuft	*Iberis amara* L.	Oil	Higher yields (+28 %) respect US untreated sample	(Liu et al., 2020a)
USMAEIND	Olives	*Olea europaea* L.	Olive oil	The US allows breaking down the biological cell walls, facilitating the release of minor compounds, reducing time and energy cost	(Tamborrino et al., 2019)
DIC + UAE	Orange peels	*Citrus sinensis* L.	Essential oils and polyphenols	Higher yield and antioxidant activity	(Allaf et al., 2013)
SWE + bioethanol conversion	Wheat straw	–	–	Increase in solid hydrolysis and better outcomes of the process	(Chen et al., 2021)
SWE + extraction	Sesame hull	*Sesamum indicum* L.	Cellulose	Greener method than alkali treatment	(Zhang et al., 2021)
SWE + extraction	Olive pomace	*Olea europaea* L.	Residual oil, sterol glucosides (β-sitosterol) and phenols,	Higher yield, working at high temperature	(Seçmeler et al., 2018)
$ScCO_2$ + SWE	Soursop seeds	*Annona muricate* L.	Polar and non-polar antioxidant molecules	Prior lipids removal and cell wall rupture. obtaining two separated fraction (non-polar with $ScCO_2$, and polar with SWE)	(Mesquita et al., 2021)
Enzyme + UAE Hemicellulases + cellulases, pectinases	Liquorice roots	*Glycyrrhiza glabra* L.	Glycyrrhizic acid	Higher yields with hemicellulases and cellulases, no difference with pectinases.	(Giahi et al., 2021)
Enzyme + UAE	Camphor seeds	*Cinnamomum camphora* L.	Oil recovery	Higher yields and nutritional values. Best efficiency with neutral protease.	(Wei et al., 2022)
Enzyme + MAE Hemicellulases + cellulases	Leaves	*Cinnamon burmanii*	Essential oil	Higher mass transfer rate, higher yields and a major presence of oxygenated compounds	(Liu et al., 2021)
Enzyme + MAHD Cellulase	Rose geranium fresh leaves	*Pelargonium graveolens* L. Herit	Essential oil	Higher yields and oil quality (TPC, total flavonoid content, antioxidant activity, acetylcholinesterase inhibition and antimicrobial activity)	(Wei et al., 2022)
Enzyme + HD	Pineapple peels	*Ananas comosus* L. Merr.	Essential oil	Higher yield than the untreated sample but much lower than $ScCO_2$ pretreated	(Mohamad et al., 2019)
Enzyme + MAE Viscozyme	Pomegranate peels	*Punica granatum* L.	Phenolic compounds	Higher yield and superior antioxidant power	(Kumar et al., 2020)
Enzyme + $ScCO_2$	Black tea leftover	*Camellia sinensis* L.	Phenolic compounds	5-times higher extract yield	(Mushtaq et al., 2017)
Enzyme + SFE Kenzyme	Alfalfa leaves	*Medicago sativa* L.	Phenolic compounds	2- and 3.4-times higher yields	(Krakowska-Sieprawska et al., 2021)

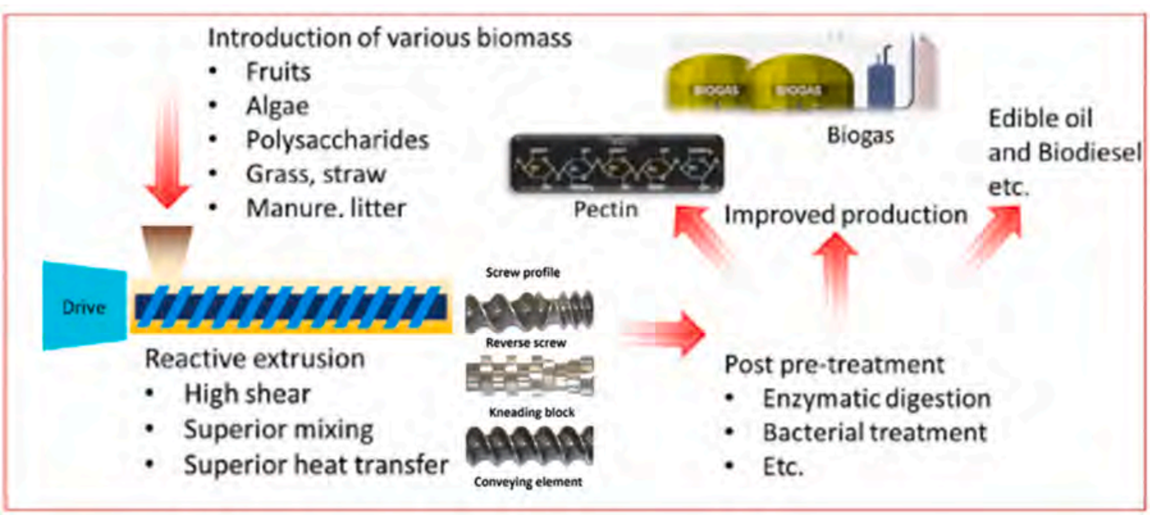


Fig. 2. Typical reactive extrusion pretreatment process of non-lignocellulosic biomasses and its subsequent valorisation in various applications. Reprinted with permission from Elsevier. (Guiao et al., 2022a).

polysaccharides can be obtained from the disrupted lignocellulosic material, thus becoming the substrate for a subsequent enzymatic hydrolysis to obtain fermentable sugars. Fractionation operations commonly follow pretreatment to conclude the separation of the de-structured components (Khadhraoui et al., 2021). A study conducted by Zhang et al. (2020b), tested the effectiveness of a dual pretreatment, which combined mechanical extrusion and ultrasound, for rice hulls with an enhancement in the subsequent enzymatic hydrolysis being observed; a 38.2 % increase in yield of fermentable sugars was obtained compared to the use of extrusion alone. Furthermore, the intensification of phenol extraction from wheat bran has been achieved by coupling extrusion pretreatment with UAE. The most relevant data in the increase of 14.10 % in the total polyphenol content (TPC) concern caffeic acid and rutin as they were not quantified, via UPLC analysis, in the raw wheat bran, but gave 61 and 184 μg/g, respectively, in the pre-extruded sample. In parallel to the increase in TPC values, the antioxidant activity, expressed in Trolox eq., also increased when the extrusion pretreatment was applied (Jin et al., 2020).

Extrusion pretreatment has also been used for the intensification of the carotenoid extraction process with $ScCO_2$ from red pepper flakes (Uquiche et al., 2022). The main advantage of combining these two techniques was the densification of the substrate, which allowed more material to be processed in the same amount of time and space, taking full advantage of the volumetric capacity of the $ScCO_2$ equipment. Specifically, it was reported that the bulk density of the ground red bell pepper flakes increased by 35 %, from 480 kg/m^3 to 650 kg/m^3 because of the extrusion process. However, due to thermal and shear damage caused by the mechanical pretreatment, the oleoresin showed a loss of colour and carotenoid content, as well as a lower total extraction yield, thus revealing the need for further studies to optimize the operative conditions and minimize undesirable chemical changes to labile and bioactive compounds in carotenoid-rich extracts.

Kupryaniuk et al. (2020), have stated that the energy input of an extrusion process, which is a key factor for industrial applications, is correlated with screw-rotation speed, and also strongly dependent on the type of matrix treated. The results obtained for grass hay, in terms of both efficiency and energy intensity, were closely correlated with rotational speed, as above mentioned, but not influenced by the raw material's moisture content. On the other hand, the extrusion of maize bran showed that a reduction in energy input occurs as the moisture content increases, i.e. from 20 % to 30 %, while energy expenditure increases with screw rotation intensity, as expected. The same authors reported that, in the case of raw lignocellulosic material, extrusion is an extremely energy-intensive process, particularly for hardwood tree species.

An example of an efficient pretreatment strategy for cellulosic bioethanol production from hardwoods, consisting of a coupled treatment using liquid extraction with hot water followed by an extrusion process, has been reported by Tian et al. (2019). The thermomechanical process before extrusion softened the matrix and reduced the energy expenditure caused by screw shear stress. Furthermore, the thermomechanical treatment before extrusion resulted in a raw material with more accessible cellulose, leading to free fermentable simple sugars with limited lignin condensation, which is a possible side reaction.

Compared to the direct use of biomass as a fuel, the introduction of the reported coupled technologies as pre-combustion steps will potentially lead to the more efficient use of lignocellulosic residues in a circular biorefinery system.

3.2. High-shear homogenization

Conventional disruption methods, such as high-pressure homogenization (HPH), bead milling (BM), and freeze/thawing cycles, are highly efficient and create a high degree of cell rupture, while presenting some disadvantages. Indeed, these techniques are typically energy-intensive and can have too high an impact on the inner structure of a matrix, and thus run the risk of damaging the most susceptible compounds and generating a huge amount of fine debris (Poojary et al., 2016). In response to these drawbacks, less invasive and low-energy-consumption techniques have been proposed. A study by Carullo et al. (2021) has evaluated the possibility of replacing conventional HPH with a combined approach of high-shear homogenization (HSH) and PEF, followed by classical water extraction, to increase the recovery efficiency of bioactive compounds from *Arthrospira platensis*. The effectiveness of each individual pretreatment (HSH and PEF alone) and of their combination was determined by comparing the obtained extraction yields with those derived from water extraction alone. The synergistic use of HSH and PEF increased the recovery of soluble proteins, carbohydrates and C-phycocyanin, when compared with the two individual techniques. In addition, the coupled technique shows similar yields to HPH for the different extracted bioactives, with the main advantage of achieving higher extract purity and significantly lower energy consumption. HSH has also been combined with UAE for the recovery of protein from seafood by-products and green seaweed (Zhang et al., 2022). No control tests using UAE alone were reported in this study, meaning that the positive effect of HSH pretreatment cannot be properly verified. However, the influence of an additional processing step, using UAE, that can increase protein solubilization and total yields for herring by-products, lingonberry cake and green seaweed was assessed. On the other hand, the application of US on shrimp shells reduced the overall protein solubilization rate, most probably due to $CaCO_3$ release, promoted by US, which induced protein precipitation.

3.3. Pulsed electric field (PEF)

PEF, currently used as a non-thermal sterilization method for liquids in the food industry, is finding new applications in the field of extraction. This technology can be applied as part of extraction processes either alone, as a pretreatment method, or in simultaneous combination with other techniques, and, as will be discussed in this section, advances have recently been made both on the laboratory and industrial scales. The effectiveness of PEF lies in the effects that it induces on the matrix via the electroporation phenomenon, which is achieved using electrodes that emit high-voltage electrical pulses for a few seconds. As a consequence of electroporation, the cell walls become more permeable to the solvent, facilitating its penetration into the biomass and the subsequent elution of the target compounds.

Preliminary studies to assess the potential of PEF as a pretreatment for polyphenol recovery from spent beer grain have been conducted, with successful results being achieved when it was coupled with classic solid-liquid extraction using a water EtOH 4:1 v/v solution (Martín--García et al., 2020). A study by Koubaa et al. (2016), tested the effects of separate PEF and UAE pretreatments for the extraction of colorant molecules, such as betanin and isobetanin, from the pulp and peel of prickle pears using water infusion extraction (Koubaa et al., 2016). The colorant yield was twice as high in the peel than in the fruit, and the sample treated with PEF and US gave a yield that was significantly higher than that of the control benchmark. In comparison, PEF and UAE gave similar results in terms of extracted betanin, although PEF was found to be more economical and less energy consuming than US (27 kJ/kg vs 800 kJ/kg). In addition, the ability of PEFs to induce cell-wall permeabilization was observed in SEM images. In this way, PEF may provide a more selective extract of valuable intracellular compounds than UAE as it does not disintegrate cell tissue.

Several studies have evaluated the pretreatment efficacy of PEF in pressurized liquid extraction (PLE) with interesting results. Hwang et al. (2021a), have coupled PEF with SWE in a study with the aim of improving the extraction of catechins from green tea. The results highlighted the enhancement in extraction yield, while also lowering the SWE temperature at the same time. Optimized conditions (PEF 2 kV/cm, 60 s followed by SWE treatment of 5 min at 130 °C), achieved extraction yields that were 15.4 % higher than those of the optimized control procedure with no PEF at 150 °C. In another work, the same authors also tested a combination of PEF and SWE to improve flavonoid extraction from *Citrus unshiu* peels (Hwang et al., 2021b). The optimal conditions for the extraction of hesperidin were PEF pretreatment at a strength of 3 kV/cm and subsequent SWE at 150 °C for 15 min, which increased total yields by 22.1 %, compared to SWE alone. A 33.6 % improvement in narirutin extraction was achieved by employing the same PEF treatment coupled with SWE at 190 °C for 5 min. Continuing with flavonoid extraction, Kim et al. (2022), have tested the efficacy of PEF pretreatment prior to the SWE of onion skins. The obtained results showed an increase in quercetin yield of 33.22 % in the PEF pre-treated sample (2.5 kV/cm for 15 s), compared to the untreated one, and a lowering of the temperature required for SWE, from 165 °C to 145 °C with respect to SWE alone. The effectiveness and versatility of the combined use of PEF and PLE on green spirulina powder extraction using a solution of water and 50 % DMSO has been investigated (Zhou et al., 2022a). The yield of the obtained biomolecules (proteins, polyphenols, chlorophyll a and β-carotene) revealed that the coupled procedure has a synergistic effect

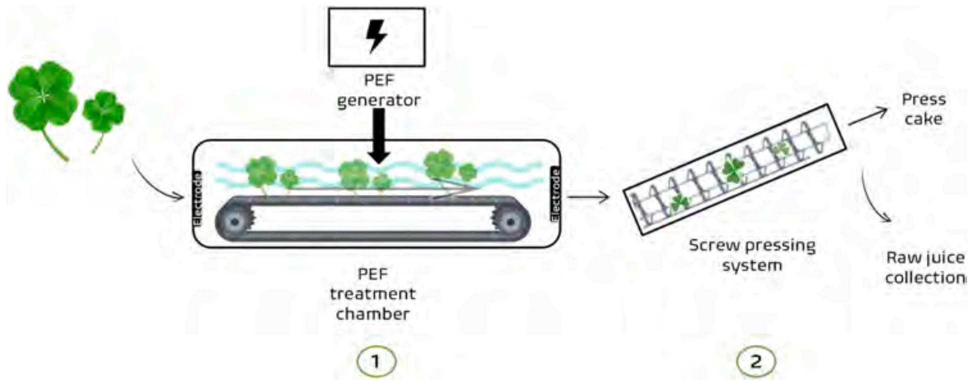


Fig. 3. Pilot plant for protein recovery from clover, exploiting PEF pretreatment, followed by extrusion. Image inspired by Guo et al. (2022).

that not only increased the quantity of phenolic compounds and the antioxidant activity of the product, in comparison with the single techniques alone, but also the variety of the extracted metabolites. However, while initial investigations are encouraging, the energy consumption of the process must be analysed before the development of an industrial-scale application.

Interesting results on the recovery of the phenolic fraction from fresh rosemary and thyme by-products, using a hydroalcoholic solution at 55.19 % v/v, have been obtained using a combination of PEF and US by Tzima et al. (2021). Despite the reduction in the extracted amounts of some of the most sensitive metabolites, such as carnosol, the electroporation resulted in an enhanced concentration of the main phenolic components of rosemary and thyme. In the PEF pre-treated samples, the sequential increase in sonication time from 4.16 to 12.48 min only slightly increased the TPC, FRAP and DPPH values of the product, thus demonstrating PEF's efficiency in reducing operating time and energy input required, which offers interesting perspectives for possible process scale-up. Four experimental conditions - named hot water extraction (HWE), US alone, PEF alone and PEF+US) - were tested during *Pericarpium Citri Reticulatae* extraction with 60 % ethanol (v/v) at 60 °C and compared in terms of total flavonoid yields (Zhan et al., 2022). The extracts were purified using microporous resins, and higher flavonoid values and higher contents of hesperidin, nobiletin and tangeretin were obtained using the sequential PEF+US treatment.

The importance of MAE as a green extraction technology has been demonstrated in numerous publications on its effectiveness in recovering metabolites, especially from plant matrices. A double pretreatment method, which applies MAE and PEF before the final extraction through a screw press for oil recovery from Niger seeds (Mohseni et al., 2020), was found to be effective under optimized equipment conditions: microwave irradiation 900 W; process time of 156.34 s, PEF intensity of 1.18 kV/cm and screw-press rotational speed of 20 rpm. It is worth noting that longer MAE treatment and more intense PEF reduced the quality and the yields of the oil. Although sample pretreatment led to an increase in yield and quality (35 % extraction efficiency, free acidity 1.94 %, 2.79 % increase in tocopherol and 410 ppm TPC), the proposed combined technology has high-energy consumption, which would be even higher in a large-scale process, meaning that further studies on the evaluation of possible process scalability are required.

In the recovery of bioactive compounds from a coffee-chain by-product, the pulp of the coffee fruit, PEF pretreatment prior to MAE in water resulted in an enhancement in the phenolic-compound extraction yield compared to MAE alone, thus confirming the potential of the PEF+MAE extraction process (Macías-Garbett et al., 2022). Another example of the PEF+MAE combination has been applied in a study by Lal et al. (2021), on the recovery of pectin from jackfruit. In this case, the tests compared the efficiency of the coupled procedure to a conventional method involving boiling water acidified with citric acid. The optimized conditions (PEF strength 11.99 kV/cm, 5.47 min, followed by MAE at a power density of 647.30 W/g and 5.00 min of time of exposure) for the combined process led to an increase in extract quality and pectin yields, by 18.24 %, while also reducing energy intake. In fact, during the process, PEF and MAE convey power directly into the treated sample volume, unlike conventional heating, in which heat is transferred via conduction to the vessel walls and then to the matrix via convection and conduction through the reaction solvent, resulting in higher total energy consumption.

As processes involving a combination of technologies represent a relatively recent field of study, research into their pilot-scale implementation are still in the early stages. Guo et al. (2022) have very recently tested the efficiency of protein extraction from a juice blend of grass and clover in a pilot-pressing facility that incorporated PEF pretreatment on a large-scale level (Fig. 3). The industrial PEF equipment operated at 1.1 kV/cm and 305 Hz, in continuous mode, and processed 100 kg of freshly harvested weeds in a water bath for 30 min, with a mass flow rate of 200 kg/h and a residence time of 2–3 s

After PEF pretreatment, the grass and clover mixture were transported to the screw press via an upwardly inclined screw. Protein recovery was performed using acid precipitation with HCl, followed by separation from plant debris through a disk separator. PEF pretreatment increased the yield of the juice blend by approximately 25 % compared to control samples (no PEF), as it released 31 % of the crude protein. The authors report that this first attempt still has plenty of room for the optimization of parameters such as pH, temperature and different strategies for protein isolation. Furthermore, as reported by Fauster et al. (2020), PEF pretreatment technology may also have future applications in the wine industry as a means to reduce fermentation time and improve the organoleptic quality of the final product. The study was conducted on a pilot level starting from two grape lots of 30 kg each that were destemmed and crushed. Each of the two mashes, from two different varieties, Traminer and Grüner Veltliner, was treated with PEF (3 and 10 kJ/kg) using a continuous co-linear treatment chamber (50 mm diameter). Immediately after PEF, pectinases were added and maceration was carried out for 4 and 24 h. Despite the lack of beneficial effects on juice extraction yields, the results showed that selected primary aroma compounds, specifically terpenes and esters (e.g., ethyl butyrate, 2- and 3-methylbutyl ester of acetic acid), can be extracted more efficiently thanks to the electroporation effects (Fig. 4).

In addition, a reduction in mean fermentation times up to 20 %, depending on the grape variety (only with Traminer), was found in the fermentation stage when PEF pretreatment was applied. On the other hand, negative outcomes, including increased turbidity and polyphenol content have been reported, which are undesirable, especially for white wine. However, the authors state that the treatment is highly dependent on grape cultivar and that the application of PEF in the wine industry is very promising.

Another reported use of PEF as a pretreatment is in its combination with supercritical CO_2 ($ScCO_2$). This coupling strategy has not yet been

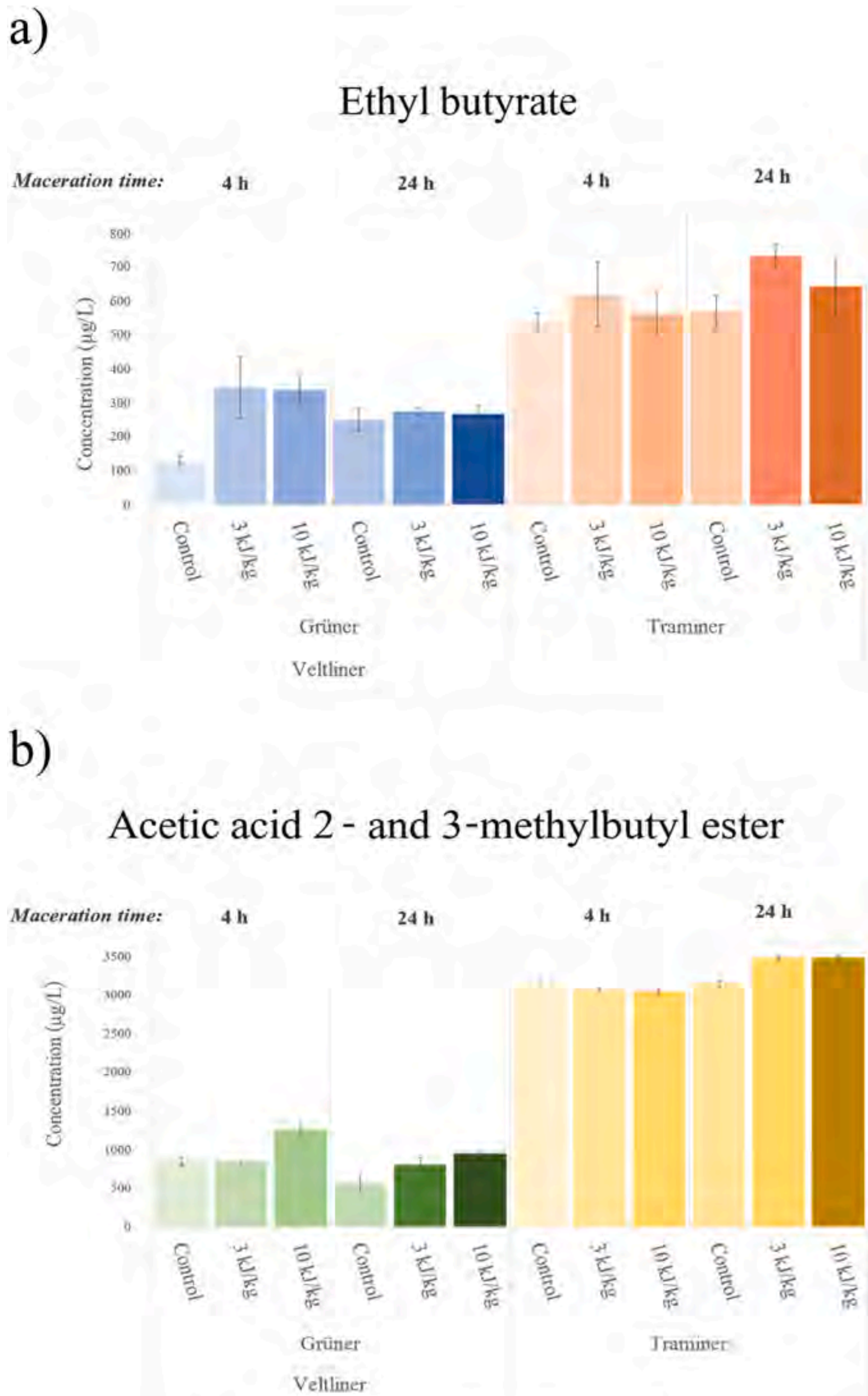


Fig. 4. Ethyl butyrate and acetic acid 2- and 3-methylbutyl ester extraction from Gruner Veltliner and Traminer variety, enhanced by the synergistic effect of PEF pretreatment (Fauster et al., 2020).

studied to the same extent as PEF+US and PEF+MAE, but the few published results on it are encouraging. One of the earliest articles dates back to 2019, and concerns the feasibility of PEF pretreatment in the extraction of pigments (total carotenes, chlorophyll a) from the microalga *Nannochloropsis oceanica* via $ScCO_2$ (Pataro et al., 2019). The extraction solution (1 % w/w aqueous matrix suspension, with 10 % ethanol added) was treated in a bench-scale continuous flow unit to be processed by applying PEF and $ScCO_2$ treatments alone, or with PEF pretreatment followed by the $ScCO_2$ protocol. The single treatment with $ScCO_2$ yielded 6.3- and 9.4-fold more total carotenes and chlorophyll, respectively, than the single treatment with PEF. The combined process showed a synergistic effect by exceeding the extraction yield of total carotenes (36 %) and chlorophyll a (52 %) compared with the single extraction process with $ScCO_2$. In a later published study, PEF was used with the dual purpose of pre-treating the solid matrix while separating the most water-soluble parts prior to extraction with $ScCO_2$. This study, conducted by Salgado-Ramos et al. (2022), was focused on the extraction of bioactive compounds from almond husks. An LC-MS analysis of the filtered solution after PEF action showed several classes of polyphenols and the NMR analysis confirmed the presence of freely accessible carbohydrates (glucose, fructose or sucrose), which are useful starting materials for platform chemicals (levulinic acid or 5-

hydroxymethylfurfural), or as additives in many foodstuffs (Jäger and Büchs, 2012). The solid residue that was pre-treated with PEF and subjected to $ScCO_2$ provides interesting lipid fractions and also a polyphenol fraction, although in smaller amounts. Therefore, this dual approach may provide the basis for an excellent process of almond hull valorisation via the recovery of carbohydrates and polyphenols by PEF and lipids and polyphenols by $ScCO_2$.

3.4. Ohmic heating (OH)

Ohmic heating (OH) by the Joule effect is an innovative technology with great potential for rapid and uniform volumetric heating. Currently, numerous researchers have investigated its industrial applications, including aseptic processing and food sterilisation, pasteurisation and drying (Kaur, Singh, 2016; Gavahiana, Farahnaky, 2018). As indicated by several publications, there is an increasing trend towards the application of this technology in the extraction of bioactive compounds from plant and algal biomass (Pereira et al., 2023) such as polyphenols (Sánchez et al., 2023), oils (Aamir, Jittanit, 2017) and pectins (Sharifi et al., 2022). OH technology is of particular importance in hydrodistillation for the extraction of essential oils (Gavahian et al., 2020). In addition to its thermal effect, this technology also contributes to the intensification of the extraction process, thanks to its non-thermal effect on the biomass, such as the electrical breakdown, electroporation and electropermeabilisation (Gavahian et al., 2020). In addition, there is already a pilot-scale study with a five liters prototype that reports on the effective scalability potential in the extraction of polyphenols (Al-Hilphya et al., 2020). A side effect of the industrial application of OH is corrosion of the electrodes and consequently an increase in maintenance costs (Samaranayake, Sastry, 2005).

The effectiveness of OH as a pretreatment step lies in the combination of thermal and non-thermal effects on the biomass (electrical degradation, electroporation and electropermeabilisation). These phenomena lead to greater permeability of the solvent within the matrix cell wall and promote the leakage of intracellular bioactive compounds. One study reported the efficacy of OH as a pretreatment for the recovery of anthocyanins from grape skins, demonstrating a double yield compared to an untreated sample and lower energy consumption compared to conventional heating in a water bath (Pereira et al., 2021). In the same study, a distinction was made between thermal and non-thermal effects induced by OH at two different electric field strengths (16 and 80 V/cm) and the same electric frequency of 25 kHz. Pretreatment at 40 °C for 20 min at 16 V/cm resulted in higher recovery of anthocyanins compared to the conventional preheating method and the untreated sample, demonstrating the non-thermal effects of cell permeabilisation. Subsequently, higher yields (+120 % anthocyanin and +60 % TPC) were obtained by combining thermal and non-thermal OH effects in the sample by treating at 100 °C for 1 min with an electric field strength of 80 V/cm. In another sequential combination strategy, the OH effect was applied prior to US extraction to obtain polyphenols from cherries (Kutlu et al., 2021a) at electric field strengths of 20, 30 and 40 V/cm. The coupling strategy increased the TPC yield proportionally to the applied electric field and, at its best, exceeded the 13 % achieved by the sample with US alone. Furthermore, the optimal OH parameter enabled a reduction in extraction time of about 34 %. The same research group also investigated OH pretreatment before MAE, keeping the parameters investigated: electric field strength 20, 30 and 40 V/cm (Kutlu et al., 2021b). The application of OH before MAE increased TPC yield, anthocyanin preservation and antioxidant activity compared to untreated samples, with the additional advantage of reducing process time. However, in this case, the electric field modulation did not affect the extraction yields of MAE as observed for US in the previous work. OH pretreatment was also explored for implementing the recovery of lipophilic molecule in aqueous media, comparing the results with PEF, US and high pressure processing (Le-Tan et al., 2022). In this case, the applied electric field strength was much higher respect to the previously reported articles, reaching 1000 V/cm. The results showed that the pretreatment step increased the recovery of curcumin by 3.39-, 3.13-, and 1.24-fold for PEF, HPP and OH, respectively. Unexpectedly, the US pretreatment showed less efficacy than other methods due to the strong local thermal effect of the technology, which degrades curcuminoids. However, the effectiveness of this technique was particularly poor when applied at relatively low electric field strengths. For example, in a study conducted on artichokes by Khuenpet et al. (2017), the application of OH at a frequency of 20 kHz and an electric strength of 15 and 20 V/cm did not significantly increase the yield of inulin extraction compared to conventional heating. Consequently, it is advisable to explore stronger field strengths to facilitate permeabilisation of cell walls. In summary, OH technology has demonstrated its versatility and efficiency in terms of time and energy expenditure and is suitable for the pretreatment of various biomass materials. This efficiency is particularly pronounced at high electric field strengths and promises promising applications in the extraction and recovery of thermolabile bioactive compounds.

3.5. Intense pulsed light (IPL)

Born as a technique to inactivate microorganisms on food surfaces, IPL is now also emerging as a novel non-thermal technology for the extraction of bioactive compounds from vegetal biomass (Hossain et al., 2015). The intermittent bursts are usually provided by a xenon lamp, which emits light at wavelengths from 200 to 1100 nm (Jo et al., 2019). More recently, IPL pretreatment processes combined with green extraction technologies have been reported. Kim et al. (2019), have demonstrated the effectiveness of the combined treatment of IPL (power:800,100,1200 V; time: 60, 120 s) and SWE (145 °C for 15 min) on onion skins for the recovery of quercetin and other phenolics (Kim et al., 2019). The highest quercetin value, achieved on the lab scale by applying 1200 V for 60 s IPL, was 17.32 ± 1.12 mg/g onion skin, while the control sample, i.e. no IPL treatment, showed a quercetin extraction yield of 15.19 ± 1.12 mg/g onion skin. The efficacy of the combined procedure was also demonstrated using fluorescence microscopy analysis on sample tissues, with the analyses highlighting the considerable release of the bioactive compound, from the inside to the outside of the cell, on the pre-treated matrix surface. On the other hand, extending the irradiation to 120 s resulted in a decrease in quercetin yields. It was thus surmised that long exposition to IPL can lead to secondary-metabolite degradation. Furthermore, the scalability potential of the process was demonstrated in a SWE plant (8 L scale) under the following conditions: 1.1 L solvent, 145 °C for 10 min in combination with an IPL pretreatment at 1200 V for 60 s. In this case, the quercetin yield was 22.16 ± 1.61 mg/g onion peel.

The same combination has been applied by Hwang et al. (2021a), to intensify the recovery process of catechins from green tea leaves. The optimal conditions, IPL 800 V for 60 s followed by SWE at 130 °C for 5 min, led to an increase in the total catechins content in the extract of 25.09 % compared to the control sample, i.e. SWE performed at 150 °C. Interestingly, the optimized IPL pretreatment showed a greater yield increase than that achieved in the PEF-treated sample (15.43 %). Stronger IPL treatments (1000 and 1200 V) led to decreases in the final catechin amounts, thus confirming the possible degradation of the phenolic metabolites over extended irradiation.

Several economic advantages can be ascribed to this kind of combined extraction protocol. Firstly, SWE technology exploits water, which is a cheap, safe and environmentally friendly solvent. Secondly, the application of IPL reduces extraction time while saving energy. Both the reported studies contain useful information for the future realization of a pilot-scale model of SWE equipment combined with non-thermal pretreatment technology.

3.6. Ultrasound pretreatment

Ultrasound is a particular kind of sound wave with a characteristic frequency of between 20 kHz and 100 MHz. High-frequency ultrasound is used principally for diagnostic purposes thanks to its ability to non-invasively penetrating organic material (Izzetti et al., 2021). Conversely, low-frequency ultrasound (20–100 kHz), usually exploited at high intensity (10–1000 W/cm^2), finds several applications in the food industry because of its well-known cavitation effects, which consist of the formation and sequential collapse of micro-bubbles inside a liquid medium (Khadhraoui et al., 2021). Due to these characteristics, the application of US in pretreatment processes prepares the biomass for the extraction, increasing process yield and reducing overall process time and energy consumption.

Getachew, Chun (2017), have published two subsequent studies on the efficiency of different pretreatments used prior to SWE on spent coffee grounds. In the first, better extraction yields in polyphenols, protein and sugars were achieved when using MAE (800 W for 5 min) rather than US pretreatments (400 W, and 60 kHz for 30 min) (Getachew, Chun, 2017) before SWE extraction (220 °C for 10 min). In the second study, modified US conditions (750 W and 20 kHz for 30 min) gave a higher polysaccharide yield than MAE and $ScCO_2$ pretreatments before SWE at 178.85 °C for 5 min (Getachew et al., 2018). These final results confirmed that increasing the intensity and reducing the frequency of US results in more effective matrix pretreatment. Future studies should address the scalability of these results to industrial-plant scale and consider the economic value of the process, including energy consumption costs and the possibility of reproducing the US and MAE pretreatment effects. In the case of the extraction of polyphenols from *Tartary buckwheat*, 20 min of pretreatment with US prior to SWE (60 min at 220 °C) (Dzah et al., 2020) led to lower total polyphenolic yields than the use of SWE application alone. A decrease in antioxidant activity and a variability in the phenolic species extracted by the two protocols were also observed. The possible co-extraction of non-active compounds and the degradation of active metabolites may explain this outcome.

Fig. 5. Semi-industrial plant for US-assisted wheat-straw delignification. Reprinted with permission from Elsevier (Calcio Gaudino et al., 2022).

Liu et al. (2020a), have investigated the process intensification of oil extraction from *Iberis amara* seeds using the pairing of US pretreatment with $ScCO_2$ extraction. The yield obtained with the coupled technology was 25 % higher than that of the $ScCO_2$ process alone. It was also reported that the achieved values are comparable to those of Soxhlet extraction with n-hexane, while achieving higher selectivity in mono-unsaturated fatty acid composition as well as better physicochemical properties and antioxidant activities.

There is still a lack of literature on scaled-up hybrid MAE+US processes for the extraction of bioactive components from biomass. Nevertheless, a concrete scale-up attempt has been pursued for the optimization of an olive-oil extraction process by the group of Tamborrino et al. (2019). The industrial hybrid plant combines a low-frequency ultrasound device, microwave apparatus and a heat exchanger, and was investigated as means of introducing these innovative technologies into the current olive-oil production process with the aim of aiding malaxation, while increasing oil yield and quality. Different set-up combinations were evaluated in the study: a spiral-coil heat exchanger (SCHE); SCHE+ malaxer machine (MM); SCHE+MAE; SCHE+US+MAE; and a set up with only traditional MM, used as a control. The designed apparatus had the capacity of process 1300 kg/h of olive paste under combined MAE at 5.8 kW and 2.45 GHz and US at frequency of 20 kHz and 2700 W. A comparison showed that the different set-ups provided no statistically significative differences in terms of oil yield compared to control (common malaxation). However, the combination of SCHE+MAE allows the olive paste to undergo continuous conditioning in only a few minutes of treatment, but with a loss of phenolic compounds. The introduction of the ultrasonic phase contributed to breaking down the biological cell walls, which facilitated the release of minor compounds. Consequently, considering the presence of phenolic compounds, the oil obtained using SCHE+US+MAE is comparable in terms of quality to that obtained with the traditional malaxing process, with the added advantage of highly reduced time and energy costs.

Calcio Gaudino et al. (2022), have investigated the scalability of US pretreatment in a US-assisted delignification of wheat straw in alkali solution, followed by enzymatic hydrolysis (EH) for the production of platform sugars for bioethanol conversion. The semi-industrial US plant consisted of two parallel tubular ultrasonic reactors with an effective volume of 15 L each in which, under optimum condition, 40.35 kg of dry biomass were sonicated at 3000 W and 25 kHz for 5.15 h (Fig. 5).

After sonication, the solution was filtered using a decanter centrifuge and the solid was washed with a 33 % HCl solution. The washed solid was loaded into a 250 L reactor for enzymatic hydrolysis with a proprietary enzyme blend at pH 5 with Na-acetate buffer. Furthermore, the hydrolysed solution was subjected to nanomembrane fractionation in order to augment the sugar concentration. The results obtained with the combination of the US semi-industrial plant and enzyme were compared with steam-explosion, which is a widespread method for the pretreatment of lignocellulosic biomass, and a Life Cycle Analysis (LCA) was performed. The final outcome highlighted the scalability of the US pretreatment process and the fact that the enzymes had more efficient accessibility to the biomass than under steam explosion. Additionally, the LCA analysis highlighted the significant advantages that US pretreatment has over steam explosion in terms of environmental impact. Specifically, 12 % less energy consumption, 36 % less fossil-fuel consumption and 32 % less global-warming potential.

3.7. Instant controlled pressure drop (DIC)

DIC is a flexible technique that is based on the thermal effects induced by saturated steam, followed by a pressure drop to vacuum for a short period. This treatment induces a change in the structure of a material, making it more porous and permeable to a solvent. It has been described as a pretreatment strategy to increase the extraction rate of various bioactive compounds, such as polyphenols, essential oils and oligosaccharides (Zhou et al., 2022b). Allaf et al. (2013), have reported a strategy for the valorisation of dried orange peels that couples DIC with US extraction. The in-line process provides two separated products: an essential oil and polyphenols. The DIC pretreatment + US was compared to: i) DIC + Soxhlet; ii) hydrodistillation (HD) + US; iii) HD + Soxhlet; and, iv) HD alone. The DIC optimized protocol (total heating time of 120 s, and six DIC cycles) recovered 16.57 mg/g of essential oil in just 2 min, while the hydrodistillation treatment have 1.97 mg/g after 4 h. In addition, extracts obtained from the combination of DIC and 1 h US had higher antioxidant activity than DIC + Soxhlet; $0.825{\cdot}10^{-2}$ g/g dry material (dm) for hesperidin and $6.45{\cdot}10^{-2}$ g/g dm for naringin (DIC+US) compared to $0.64{\cdot}10^{-2}$ g/g dm and $5.7{\cdot}10^{-2}$ g/g dm (DIC+SE). Although a limited number of literature studies have been published and no scale-up possibilities have been investigated, the preliminary outcomes of the use of DIC technology as an extraction pretreatment have shown some very interesting features.

3.8. Subcritical water

SWE has recently emerged as a powerful technique that can replace conventional extraction methods using simple water as the solvent. It is an environmentally friendly approach that combines temperatures between 100 and 374 °C and high pressures to bring water into its subcritical phase. Some studies have also reported the efficacy of this technique as a pretreatment method to disrupt the structure of highly recalcitrant biomass, such as lignocellulosic matrices. Chen et al. (2021) have attested that the efficiency of bioethanol conversion from wheat straws was improved by SWE pretreatment, with a corresponding increase in solid hydrolysis. Due to the higher concentration of hydroxide and hydronium ions in subcritical water compared to ordinary water, the technique can be applied as a substitute for the alkaline pretreatment of lignocellulosic biomass. Indeed, high temperatures, of around 180 °C, initiate the hydrolysis of hemicellulose and the depolymerization of lignin into low-molecular-weight phenolic compounds, while cellulose is preserved intact up to 220 °C. As a result, pure cellulose fibres were obtained through the removal of hemicelluloses and lignin. SWE has also been employed in sesame-hull pretreatment for cellulose recovery with the potential of subcritical water as a greener alternative method than conventional alkali treatment through sodium hydroxide indeed being demonstrated in this context (Zhang et al., 2021). The yields from the two processes did not differ significantly, although the sample extracted using SWE demonstrated the advantage of exhibiting higher crystallinity, having lower diameter particles and better thermal stability, leading to cellulose of higher quality.

The efficacy of steam explosion and subcritical water pretreatment in olive-pomace valorisation, compared to the conventional method of acid hydrolysis, has been investigated by Seçmeler et al. (2018), who investigated the recovery of residual oil, sterol glucosides and phenols before further utilization for biofuel production. Similar results were obtained, in terms of oil yield, when acid hydrolysis and green hydrothermal pretreatments were compared, although lower β-sitosterol recovery was observed with the latter technique. It was also reported that the steam explosion procedure was more efficient than subcritical water at lower temperatures (150 °C). However, subcritical water showed a tendency to increase oil yield at higher temperatures (200 °C). Furthermore, it was hypothesized that increasing the temperature of subcritical water to 210–230 °C would result in the complete destruction of cell walls, thus promoting the recovery of β-sitosterols while producing a biomass suitable for biofuel production.

3.9. Supercritical CO_2 ($ScCO_2$)

A substance is considered to be a supercritical fluid when it is above its critical temperature and pressure. It is an environmentally friendly alternative to conventional organic solvents in the recovery of non-polar compounds thanks to several advantages, such as the lack of solvent residue in the final product and the better retention of aromatic compounds. The solvent that is most commonly used in the form of a supercritical fluid is currently CO_2 (Sahena et al., 2009). To the best of our knowledge, there is only a single paper that reports the combined application of $ScCO_2$ and SWE, and that is for the recovery of non-polar and polar molecules with antioxidant activity from soursop seeds (Mesquita et al., 2021). This study demonstrated how $ScCO_2$ can be considered a possible pretreatment in the removal of lipids and in preliminary cell wall rupture, as demonstrated by SEM analyses. The combination of these two high-pressure technologies in a sequential operating process produced two separate fractions; one that was non-polar and the second that was rich in polar compounds. It is worth noting that both contained phenolic and antioxidant metabolites, which is interesting for the formulation of bioactive cosmetic, pharmaceutical and food products.

3.10. Enzymatic pretreatments

Enzymatic pretreatment methods employ different types of enzymes to reduce matrix recalcitrance to solvent permeation during extraction protocols, e.g. via depolymerization (mainly pectin, cellulose and hemicellulose), which leads to decreased solvent consumption and extraction times. The optimization of these treatments must be pursued by modulating the incubation conditions, such as time, temperature, pH, enzyme concentration and substrate particle size. Mild operating conditions favour the recovery of thermolabile compounds, and the further treatment of the solid fraction can be performed with a view to a biorefinery approach to the complete exploitation of biomass. This technology has applications in both pretreatment and extraction procedures and, as extensively documented in the literature, is gaining enormous interest as an efficient, environmentally friendly and sustainable approach.

As reported by Giahi et al. (2021), the efficiency of an enzyme cocktail depends largely on its specific enzyme composition, activity and the type of biomass being treated. The incubation parameters were set (temperature 45 °C, time 1 h and pH 5) to evaluate the efficiency of the different enzyme mixtures as pretreatment for the extraction of glycyrrhizic acid from liquorice roots under US. The authors also compared extraction yields from US and Heat reflux. The protocol that used cellulases and hemicellulases was observed to be more effective at improving yields than that used on the control sample (no enzyme pretreatment), while no significant differences were reported when pectinases were used. The highest total-extraction and glycyrrhizic-acid yields, of 44.95 % and 2.22 % respectively, were achieved when 3 % cellulase was used prior to US extraction, whereas the thermal reflux extraction with the same pretreatment method, 42.77 % and 1.94 %, and the control sample, 23.77 % and 1.93 % provided lower yields.

An attractive application of this combination of enzymatic incubation and US can be found in a report by Wei et al. (2022a), who performed the extraction of *Cinnamomum camphora* seed oil in water, replacing organic solvents. The optimized parameters (4 h of incubation time with neutral protease, 400 rpm agitation rate, 10 mL/g of water-to-solid ratio, 2 % of enzyme amount, 1.4 h of sonication time and 220 W of ultrasound power) resulted in similar product yields, 80.12 %, to those achieved in the conventional Soxhlet protocol with hexane (SE) (Wei et al., 2022a). Furthermore, the aqueous extract was reported to contain a significantly higher amount of monounsaturated fatty acids, a lower acid value and a higher nutritive value, as measured by the therapeutic index, the thrombogenic index and the hypocholesterolemic/ hypercholesterolemic ratio.

A combination of hemicellulase and cellulase enzymes has been applied by Liu et al. (2021), to pre-treat *Cinnamomum burmannii* leaves before MAE to obtain the essential oil. The combined method enhanced the mass-transfer rate, thus giving higher yields, while the recovered

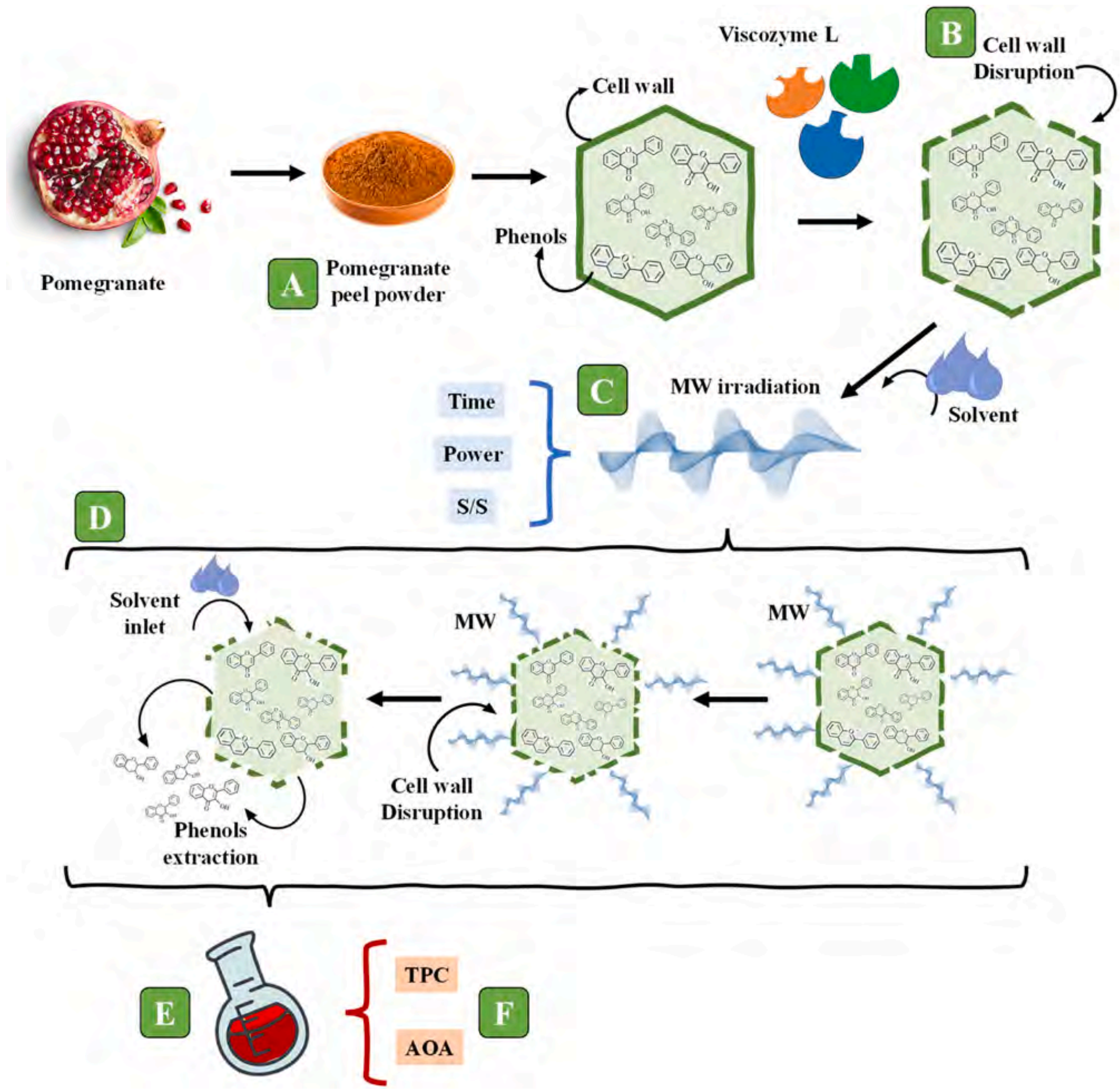


Fig. 6. Microwave-assisted extraction of Punica granatum peels with enzyme pretreatment. A) Peels comminution; B) Enzymatic pretreatment with Viscozyme L; C) MAE screening; D) Enzyme+MAE mechanism; E) Phenolic extract recovery; F) Antioxidant activity (AOA) and total phenolic content (TPC) determination. Figure inspired by Kumar et al. (2020).

product contained a higher proportion of oxygenated compounds than the products of simple MAE and conventional hydrodistillation. Wei et al. (2022b), has published a similar study on the efficiency of pretreatment using the cellulase enzyme prior to solvent-free hydrodistillation under MW for the recovery of essential oil from *Pelargonium graveolens* L'Herit. Several aspects such as yields and oil quality, including total phenolic content, total flavonoid content, antioxidant activity, acetylcholinesterase inhibition and antimicrobial activity, were significantly enhanced when using the enzyme rather than the simple hydrodistillation method.

The efficiency of enzyme pretreatment prior to hydrodistillation has been compared to that of $ScCO_2$ extraction in essential oil recovery from pineapple peels (Mohamad et al., 2019). The enzyme-treated sample gave a higher yield than the untreated sample after hydrodistillation, although it was limited to only producing a pineapple-flavoured hydrosol. On the other hand, the $ScCO_2$ sample led to a 0.17 % yield in essential oil. SEM analyses revealed that only the latter technique lead to the breakage of the oil gland in the matrix peels, confirming the obtained results. Kumar et al. (2020), have combined a MAE extraction in 30 % ethanol solvent with an enzyme pretreatment with Viscozyme®, a cellulolytic mixture, to obtain phenolic compounds from the *Punica granatum* peels (Fig. 6).

The final extract displayed higher quantities of phenolic compounds and had higher antioxidant power than the extracts obtained from Soxhlet extraction and the individual use of MAE or an enzyme mixture. Hydrolysis using different enzymes has also been proven to be successful when applied before $ScCO_2$ extraction (co-solvent: ethanol) on black tea that was leftover for the recovery of phenolic compounds (Mushtaq et al., 2017). Kemzyme™, whose extract yield was 5-times higher than that of the untreated sample, was found to be the ideal enzyme, proving the efficacy of the biological treatment. Another study has been conducted by Krakowska-Sieprawska et al. (2021), who investigated the

Table 3
Summary of hybrid extractions technologies.

Technologies	*Source*	*Scientific name*	*Target compounds*	*Advantages from hybridization*	*Ref.*
PEFxmech. pressing	Apple, sugar beet, carrot and spinach	–	Juice extraction	Higher yields and in case of sugar beet less colour, higher purity and sugar concentration	(Vorobiev and Lebovka, 2006)
PEFxmech. pressing	Waste of chicken meat	–	Protein-rich liquid	The electroporation effect enhances the pressing yield	(Ghosh et al., 2019)
PEF×OH	Chicory leaf	*Cichorium intybus* L.	Inulin	Higher ohmic heating treatments led to higher level of tissues damage, increasing the extraction rate	(Zhu et al., 2015)
USxMAE (USMAE)	“Ataulfo” mango peels	*Mangifera indica* L.	Polyphenols		(Ordoñez-Torres et al., 2021)
USMAE	Mexican Rambutan	*Nephelium lappaceum* L.	Geranin and other polyphenols	Best metabolites recovery (in absence of alcohol)	(Hernández-Hernández et al., 2020)
USMAE	Mexican Rambutan peels	*Nephelium lappaceum* L.	Ellagitannins (geraniin, corilagin, ellagic acid)	Relevant increase in TPC yield	(Estrada-Gil et al., 2022)
USMAE	Bark	*Larix decidua* Mill.	Antioxidant phenolic compounds	Higher yield (x2), enhancement of the antioxidant capacity, reducing the extraction time of 47-fold,	(Sillero et al., 2018)
Enzyme + USMAE Pectinases	Chinese water chestnut	*Eleocharis dulcis* Trin.	Flavonoids	Higher yield	(Xu et al., 2018a)
USMAE + NADES extraction	Grape pomace	*Vitis vinifera* L.	Anthocyanin	Double yields	(Panić et al., 2019)
USMAE	Happy tree	*Camptotheca acuminate* Decne.	Sugars and polysaccharides (antitumoral bioactives)	Higher yield in short time	(Sun et al., 2019)
Enzyme + USMAE α-amylase	Potato peels	*Solanum tuberosum* L.	Pectins	Increased pectins extraction efficiency	(Yang et al., 2019)
USMAE	Jackfruit peels	*Artocarpus heterophyllus* Lam.	Pectins	Higher yield and greener addhitives (citric acid vs. mineral acid)	(Xu et al., 2018b)
USMAE	Brown algae	–	Fucose-sulphated polysaccharides (FSPs)	Major yields in FSPs, total soluble carbohydrates and phenolics	(Garcia-Vaquero et al., 2020)
US×ScCO_2	Ginger roots	*Zingiber officinale* L.	Gingerol	Higher yield (+30 %) due to cellular damage	(Balachandran et al., 2006)
US×ScCO_2	Candytuft seeds	*Iberis amara* L.	Cucurbitacin E (CuE)	Higher yield (*approx.* +26 %), reduced use of CO_2 (*approx. −50* %), reduced time.	(Liu et al., 2020b)
US×ScCO_2	Barbed skullcap	*Scutellaria barbata* D. Don	Oleanolic acid (OA) and ursolic acid (UA)	Higher yield (*approx.* +25/30 %), reducing time and temperature required, saving energy	(Yang et al., 2013)
US×ScCO_2	Barbed skullcap	*Scutellaria barbata* D. Don	Apigenin	Higher yield (*approx.* +20 %)	(Yang, Wei, 2018)
US×ScCO_2	Flat-top mille graines	*Hedyotis corymbose* L.	Oleanolic acid (OA) and ursolic acid (UA)	Higher yield, saving time and energy	(Yang, Wei, 2016)
US×ScCO_2	Rice bran	*Oryza sativa* L.	Rice oil, oryzanol and tocopherols	Higher yield (*approx.* +27 %),	(Soares et al., 2018)
US×ScCO_2Scale up	Ground almonds	*Prunus amygdalus* Batsch.	Oil	Higher yield (*approx.* +90 %),	(Riera et al., 2004)
US×ScCO_2Scale up	Cocoa cake	*Theobroma cacao* L.	Oil	Higher yield (*approx.* +43 %),	(Riera et al., 2010)
USxPEF Scale up	Olives	*Olea europaea* L.	EVOO production	Higher yield and quality, tocopherol and tocotrienol enrichment (*approx.* +17 %), without affecting free acidity and peroxide values, reducing water used and energy costs.	(Grillo et al., 2022)
US×OH	Citronella	*Cymbopogon citratus*	Essential oil	Higher essential oil extraction yield and energy-saving in comparison to water distillation	(Zhang et al., 2022)
USxEnzyme (USAEE) Tannase+cellulase+pectinase	Crowberry	*Empetrum nigrum*	Phenolics	Higher yields	(Gao et al., 2022)
USAEE	–	*Cyclocarya paliurus* (Batal) Iljinskaja	Flavonoids	–	(Lei-Xiong et al., 2019)
USAEE + UAE	Roots	*Scutellaria baicalensis*	Baicalein and wogonin	Higher yields (double)	(Yun et al., 2022)
USAEE	Oak nut	–	Tannins	High yield but strong conditions may inactivate enzymes	(Luo et al., 2019)
USAEE	Red algae	*Gelidium sesquipedale*	Agar	Higher extraction (x6), reducing time	(Li et al., 2021)
USAEE	Sesame bran	*Sesamum indicum* L.	Protein and antioxidant compounds	Higher proteins yields, lower TPC yields (maybe for the US degradation on polyphenols)	(Görgüç et al., 2019)
USAEE	Bovine bone	–	Low molecular weight peptides	Higher yields	(Yang et al., 2021)
USAEE, AMNPs Pectinase, cellulase	Tomato peels	*Solanum lycopersicum* L.	Lycopene extraction	Higher yield, high US power and longer incubation times show a decrease in the carotenoid recovery	(Ladole et al., 2018)

(*continued on next page*)

Table 3 (*continued*)

Technologies	*Source*	*Scientific name*	*Target compounds*	*Advantages from hybridization*	*Ref.*
USAEE	–	*Pinus pumila* Pall. Regel	Kernel oil	Lower yield but high-quality product (fatty acid composition and physico-chemicals property)	(Chen et al., 2016)
USAEE (LIU)	Sludge	–	Proteins	Better enzymatic efficiency under LIU than HIU, with high amount of free aminoacids obtained, saving energy and the quantity of enzyme required	(Yan et al., 2022)
MAE×Enzyme (EMAE)	Olive pomace	*Olea europaea* L.	Phenolics	Higher TPC with tannase, compared with cellulase or pectinase. Higher extraction yield compared to simple MAE, reduction in time and use of water as solvent	(Macedo et al., 2021)
EMASOE	Grape seed	*Vitis vinifera* L.	Phenolics	Higher yield and diversity of monophenols	(Jia et al., 2021)
EnzymexMAExATPS	Purple heart radish	*Raphanus sativus. L*	Antioxidant polysaccharides	Reduced process time and promising antioxidant activity	(Lin et al., 2022)
EMAE Xylanase	Hypericum	*Hypericum perforatum* L.	Hypericin	Higher yield for the EAE+MAE, instead of EMAE, because of the possible enzyme inactivation caused by MW	(Zhang et al., 2019)
EMAE + MAHD + L/Lseparator	–	*Polygonum viscosum* Buch-ham	Essential oil	Higher yields in shorter times	(Chen et al., 2018)
OH×Enzymes	Spirulina algae	*Arthrospira platensis*	Phycobiliprotein	Facilitate following extraction, caused by cell wall disruption	(Ferreira-Santos et al., 2021)

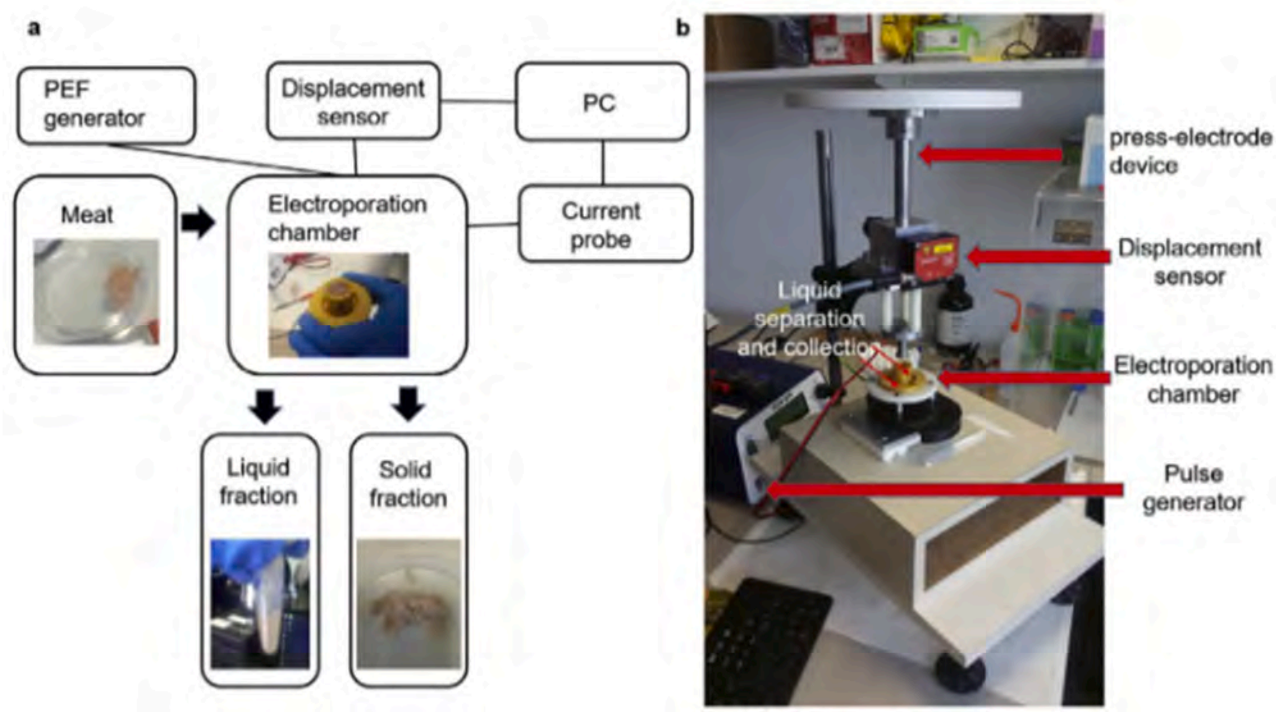


Fig. 7. Self-designed PEF x mechanical pressing machine for waste meat biomass fractionation. a) schematic diagram of the process. b) digital imagine of the custom-made gravitation drive press electrode device. Reprinted with permission from Elsevier (Ghosh et al., 2019).

same combination in the recovery of phenolic metabolites from *Medicago sativa* leaves. The optimized protocol combined Kemzyme™ treatment (pH 6, 2.96 % enzyme, 58 min at 38 °C) and $ScCO_2$ extraction (50 °C, 216 bar, 19.4 % ethanol as co-solvent, running time 40 min) resulting in 2- and 3.4-fold higher yields compared to the control (no enzymes) and simple maceration (ethanol soaking, 24 h at 50 °C), respectively. Thus, the overall process is reduced from 24 h to an average of 100 min

4. Hybrid extraction technologies

Green extraction technologies are an advantageous opportunity for the production chain of pharmaceutical, nutraceutical and cosmetic bio products, although each technique still has room for improvement. In a green extraction approach, the combination of two techniques can benefit the whole process by overcoming the weaknesses of each individual method. The simultaneous application of two or more technologies allows their respective advantages to be merged into a single device, reducing the space, time and energy required for treatment, compared to an "in-series" connection. In addition, the development of synergistic effects has been observed, since several articles also report an improvement in extraction yield and selectivity. However, if not well balanced, the application of hybrid systems may lead to poorer results due to possible interference between the techniques. In addition, combinations of multiple technologies require higher initial investment and potentially higher energy requirements, meaning that these systems must be highly productive and have significant advantages to offset these factors. There are currently numerous literature studies that present the development of hybrid processes with different combinations of MAE, US, $ScCO_2$, PEF and enzymes (Table 3). It must be specified that scaled-up applications are still on a very low technology readiness level. The state of the art of hybrid extraction strategies is presented in the following paragraphs, together with a description of the operating devices, cost/energy-efficiency balance and relative process-scalability outlook.

4.1. Combinations of physico-chemical methods

4.1.1. PEF×mechanical pressing

The application of PEF in combination with mechanical pressing can increase the squeezed volume due to the electroporation effect that is induced by electric fields. The coupling of PEF treatment with pressing for the extraction of juices from apples, sugar beets, carrots and spinach has been described by Vorobiev and Lebovka (2006). An increase in sugar-beet-juice yield was noted if compared to simple pressing (10 bars), both under i) PEF pretreatment and ii) simultaneous combination PEF+pressing, by i) 62.3 % and ii) 82.4 %, respectively. In addition, the quality of the obtained juices was higher in terms of color (less colored), purity (no pectin contamination) and sugar concentration (higher). Similar improvements were observed for carrot and spinach juice, where, through the combined treatment, total yields were enhanced by about 33 % and 50 %, respectively.

Ghosh et al. (2019), have designed a lab-scale combined apparatus for the extraction of protein-rich liquid from the waste from chicken meat production chains. The custom-made device consisted of a pulse generator and an electroporation cell combined with a gravitation-press electrode device (Fig. 7).

The authors highlight how electroporation contributed to deconstructing the cell lipidic bilayer, thus favouring the release of the cytoplasmatic solution during pressing. The designed valorisation strategy gave a liquid fraction that was 12 % of the initial weight and that had a protein content of 78 mg/g with an energy investment of 38.4 J/g. Furthermore, a gene ontology analysis of the extract indicated that several functional proteins, suitable for market application in several sectors, were present. The reported strategy represents a possible new source of income in the meat-food chain, and can also act as a means to reduce waste and waste-related environmental pollution (Ghosh et al., 2019).

4.1.2. PEF×OH

According to information in the literature, the hybrid technique of PEF and OH is not yet very widespread. The only attempt of simultaneous combination of these technologies was used for pretreatment of chicory leaves for water extraction of inulin (Zhu et al., 2015). Moderate (400–1000 V/cm) and high (10,000 V/cm) PEF treatments were combined with OH, increasing the number of pulses and thus the induced temperature. The results showed that treatments with higher Joule heating treatments the chicory tissue was more damaged, increasing the extraction rate; instead, there were no significant differences between the two electric field strengths tested for PEF. This combination of technologies could enable new protocols for effective extraction of thermolabile compounds due to the fast and effective heating profile of OH while enhancing the electroporation phenomenon with PEF.

4.1.3. US×MAE (USMAE)

The hybrid combination of US and MAE (USMAE) has demonstrated synergistic effects, as demonstrated by theoretical and experimental knowledge. This technology has several applications in the food industry, such as preservation treatments (such as drying or frying), the extraction of high-value compounds and the enzymatic hydrolysis of proteins (Muñoz-Almagro et al., 2021). USMAE is a non-thermal treatment with moderate energy consumption and low-costs, and that presents the synergistic advantages of the two separate techniques. The cavitational effect provided by US leads to the destruction of the matrix structure, enhancing solvent penetration, while MW provides rapid and effective heat exchange with high energy efficiency, and augments mass transfer (Wen et al., 2020). The in-situ combination of this technology had some difficulties, such as the low propagation of US waves in a hot medium and interference between MW and metallic sonotrodes. It is possible to use US horns typically made of a ceramic material, quartz, pyrex® or special PEEK® containing glass fibres, to overcome these issues. The use of a commercially available set up that combines the MW processor with an US bath, as reported by K. Shikha Ojha et al. (2020) is another potential solution (Fig. 8).

The temperature drawbacks can be addressed either by working in intermittent mode, (US or MW irradiation stops for an interval during extraction), or with the aid of a cooling system (Fauster et al., 2020; Martina et al., 2016). One further solution is that of separating the MW and the US in two sequential chambers in order to avoid direct contact between the two devices.

The following section focuses on the application of USMAE as an enabling hybrid technology for green extraction. As already reported, polyphenols are one of the most valuable extractable components from

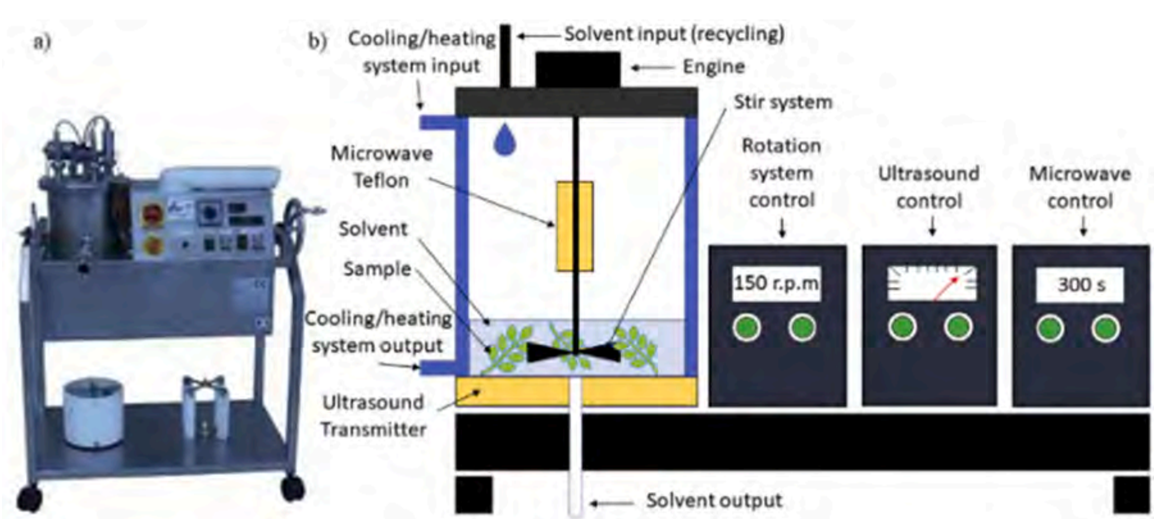


Fig. 8. Hybrid system for the in-situ combination of microwave and an US bath reactor. Reprinted with permission from Elsevier (Ojha et al., 2020).

vegetable biomass, thanks to their well-known beneficial bioactivities. The effectiveness of this hybrid technique has been demonstrated in various plant matrices, thus representing a possible future pathway for the industrial optimization of first-generation feedstocks and residual biomass valorisation. Ordoñez-Torres et al. (2021), have investigated the combined effect of USMAE for the extraction of these secondary metabolites from Mexican "Ataulfo" mango peels. The experimental extraction designs were performed at a fixed MW frequency of 2450 MHz and fixed US frequency of 25 kHz, while different $EtOH_{aq}$ solvent concentrations, solid-liquid ratios and extraction times were compared. The best conditions, which gave a total polyphenol yield of 54.14 mg/g of dry matter, were 1:5 g/mL ($EtOH_{aq.}$ 50 %) as the solid/liquid ratio and 10 min of extraction time. The polyphenol extracts showed potent inhibitory activity against free radicals (DPPH inhibition 94.00 ± 3.08 %, ABTS inhibition 98.52 ± 2.05 %), which are interesting results for their future application as natural ingredients in the pharmaceutical and functional-food industries.

The applications of USMAE technology in polyphenol recovery from Mexican Rambutan (*Nephelium lappaceum* L.) has been reported (Hernández-Hernández et al., 2020). Several hydroalcoholic solutions (ethanol-based) were tested during a screening, revealing that the complete absence of alcohol grants the best metabolite recovery; 307.57 and 26.53 mg/g dry matter for soluble and bond polyphenols, respectively. The removal of ethanol from an industrial process has several advantages, such as reduced solvent costs, the absence of flammable substances and, consequently, the lack of a need for specialized ATEX equipment. The team of Estrada-Gil et al. worked in a similar manner on *Nephelium lappaceum* L., and compared the extraction efficiency of MAE, US and their hybrid combination in the recovery of bioactive ellagitannins from the peels (Estrada-Gil et al., 2022). The hybrid approach led to a considerable increase in extraction outcomes, with a yield in total phenolic content of 176.38 mg/g, which is significantly higher than those of UAE (24.90 mg/g) and MAE (12.56 mg/g). The HPLC/MS/ESI analysis revealed three main components; geraniin, corilagin and ellagic acid. Sillero et al. (2020), have applied a simultaneous combination of MAE and US for the extraction of bioactive compounds from *Larix decidua* bark. Extract characterization noted the presence of phenolic compounds with antioxidant activity. The adoption of USMAE doubled the extraction yield with a concomitant enhancement in antioxidant capacity, reducing the extraction time 47-fold compared to conventional method (Sillero et al., 2018). Xu et al. (2018a) have coupled enzyme pretreatment, pectinases and USMAE for the recovery of flavonoids from Chinese water chestnut (*Eleocharis dulcis*). A MW power of 10–800 W (2450 MHz) and a US transducer with fixed power and frequency (50 W, 40 kHz) were tested. A comparison with standard organic solvent extraction, MAE and US alone were promising, with yield increases of 26.50 %, 22.31 % and 12.98 %, respectively. The simultaneous application of US and MAE has also been tested as a pretreatment method to enhance anthocyanin extraction with natural deep eutectic solvents (NADES) from grape pomace (Panić et al., 2019). The optimal conditions (MW power at 300 W, US power 50 W, for 10 min with 30 % (v/v) of water) gave 1.77 mg g_{dw}^{-1} of anthocyanins, which is almost double that provided by MAE and US alone. The optimal NADES was a combination of choline chloride/citric acid, which was selected because of its cost, physicochemical properties, anthocyanin recovery and stability. While the authors attempted to scale-up the process to a half-liter batch, the lack of dedicated hybrid apparatus led to the choice of a sequential US+MAE approach. However, the yields of the scaled-up system were similar to the lab-scale results, indicating that this technology combination is promising for extraction on larger scales.

USMAE has also been applied for the effective extraction of sugars from various matrices, including plant food by-products, algae and other plant biomasses. For example, Sun et al. (2019), have developed an optimized procedure for the recovery of antitumoral bioactives from the fruit of *Camptotheca acuminate*. Under optimal conditions (solid/liquid ratio of 1:30 g/mL, MW power 570 W, US irradiation 50 W for 20 min), satisfactory yields (6.81 %) were obtained, with a slightly higher yield (1.04-fold increase) being achieved in a shorter time (6-fold decrease) than that of conventional hot water extraction. Yang et al. (2019), have optimized an USMAE extraction protocol for the recovery of pectin from potato peels. The sample was preliminary treated with thermostable α-amylase for starch hydrolysis, and the optimized conditions (93 °C, pH 2.0, 50 min) gave a final outcome of 22.86 % (Yang et al., 2019). Xu et al. (2018b), have developed a safe and efficient protocol for pectin extraction from jackfruit (*Artocarpus heterophyllus* Lam.) peels exploiting USMAE. The optimized protocol (86 °C, 29 min, solid/liquid ratio 1:48 g/mL) recovered up to 21.5 % of pectin, more than the conventional heating approach. Furthermore, this result was obtained with mineral acids (hydrochloric, sulfuric and nitric acid) being replacing with citric acid, strongly reducing the environmental impact of the whole process. Garcia-Vaquero et al. (2020), have also explored the application of a USMAE hybrid system for the extraction of fucose-sulfated polysaccharides (FSPs) and antioxidants from brown algae (Fig. 9).

The combined protocol gave higher yields than the control ones (MAE and US alone), with 3533.75 ± 55.81 mg fucose/100 g_{dm} of FSPs, 10,408.72 ± 229.11 mg glucose equivalents/100 g_{dm} of total soluble carbohydrates, and 2605.89 ± 192.97 mg gallic acid equivalents/100 g_{dm} of phenolic compounds being recovered. However, antioxidant power failed to demonstrate a regular trend and further studies are needed to assess the efficacy of this approach on macroalgae.

4.1.4. US×$ScCO_2$

As mentioned above (Section 3.8), combining US with $ScCO_2$ increases mechanical damage to cells, improving solvent penetration and mass transfer. The current literature on their simultaneous application diverges into two possible hybrid systems (Dias et al., 2021; Kumari et al., 2018). One option is to place the transducer outside of the $ScCO_2$ extraction vessel, with the major disadvantage of reducing the ultrasonic power delivered to the sample. The second possibility is that of placing

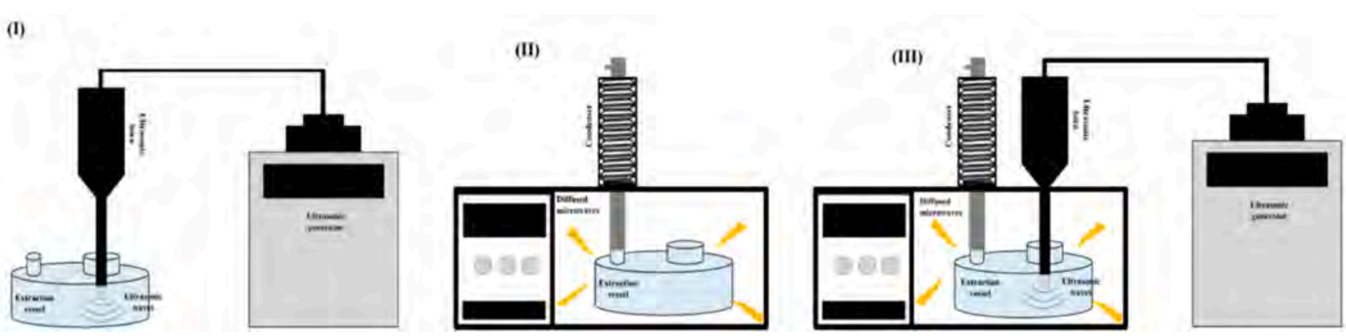


Fig. 9. Schemes showing the technological designs for: (I) ultrasound-assisted extraction (UAE); (II) microwave-assisted extraction (MAE); and, (III) ultrasound horn x microwave-assisted extraction (UMAE) used to generate macroalgal extracts (Garcia-Vaquero et al., 2020).

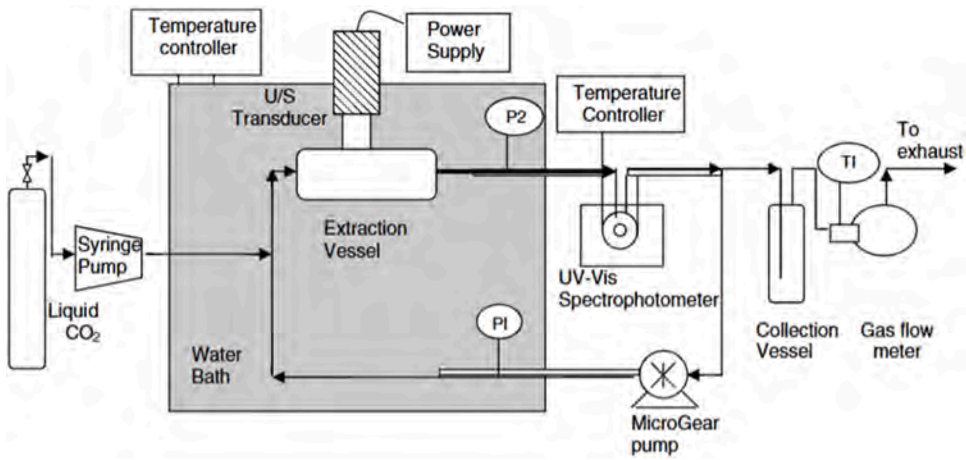


Fig. 10. Process flow for supercritical CO_2 coupled with an US bath system for the extraction of gingerol. Reprinted with permission from Elsevier (Balachandran et al., 2006).

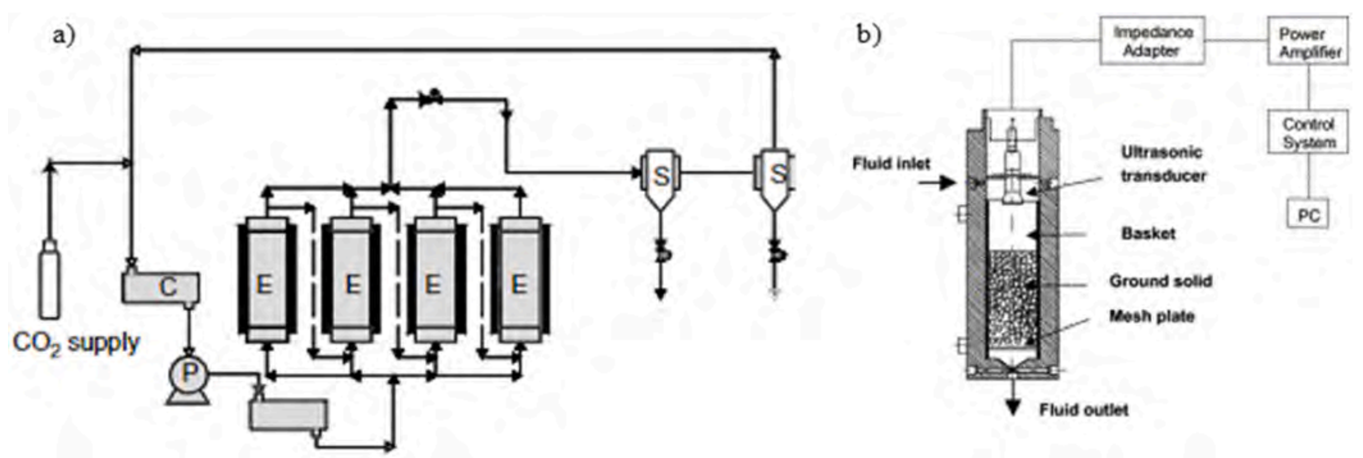
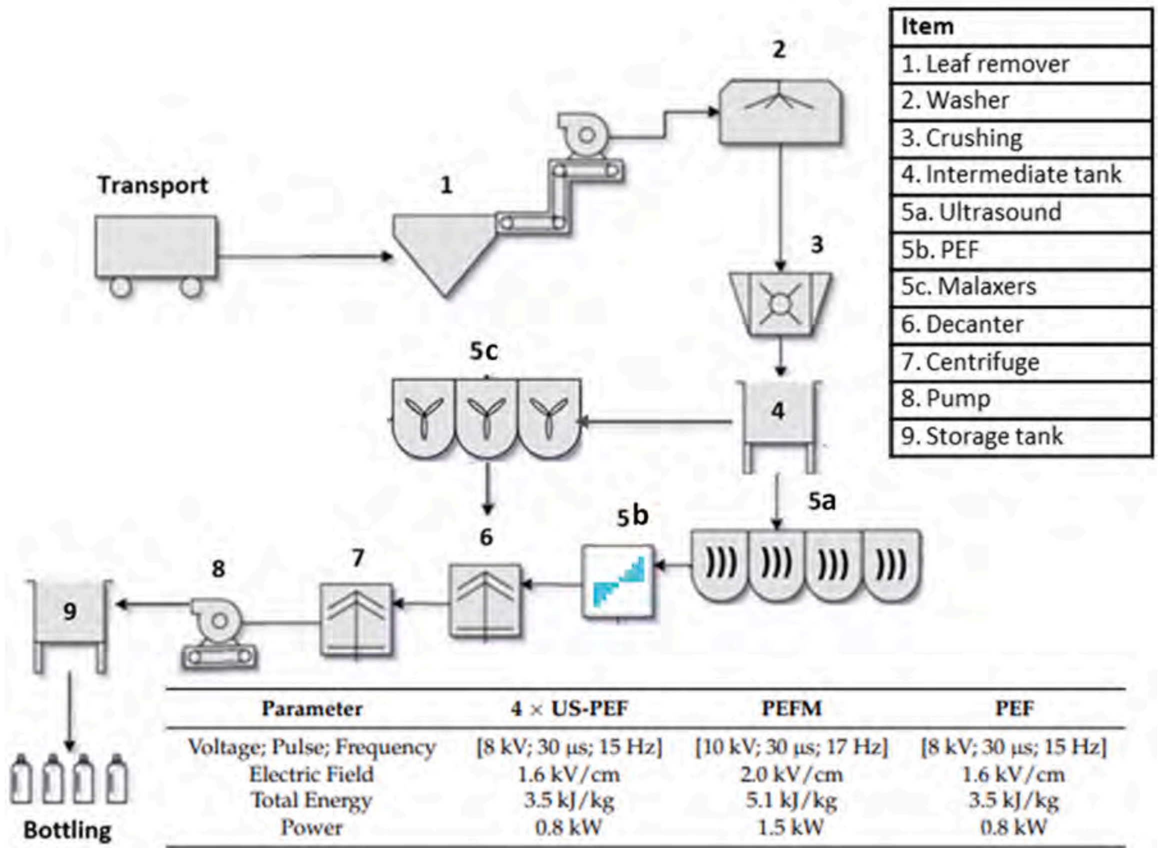


Fig. 11. a) Scheme of the basic experimental set-up used for SFE assisted by power ultrasound. (E) Extractors; (S) Separators; (C) Coller and (P) High Pressure Pump; b) Scheme of the installation of the ultrasonic transducer inside the extractor. Reprinted with permission from Elsevier (Riera et al., 2004).

the transducer inside of $ScCO_2$ extraction vessel and directly in contact with the sample. In this last case, the major disadvantage is eventual probe erosion, and here the use of titanium sonotrodes is recommended, due to its corrosion resistance, to mitigate wear (Cravotto, Binello, 2016). Balachandran et al. (2006), have designed a combined unit that includes a 150 mL extractor for $ScCO_2$ coupled to an external 20 kHz transducer within a water bath that is used to maintain constant temperature (Fig. 10). This set-up has been applied for the extraction of gingerol from ginger (*Zingiber officinale* L.) root showing that the combined effect of US+$ScCO_2$ enhanced the total yield by 30 %, compared extraction without US.

The authors concluded that the yield increase can be attributed to the cellular damage induced by cavitational collapse and to the rapid changes in density associated with the pressure fluctuations induced by the ultrasonic wave (Giahi et al., 2021). The efficiency of a US+$ScCO_2$ (co-solvent: ethanol) hybrid treatment for cucurbitacin E (CuE) extraction from *Iberis amara* seeds has been investigated using a system consisting of an extraction vessel submerged in an ultrasonic bath (power 300 W, 40 kHz) (Liu et al., 2020b). The results revealed how the hybrid approach improved the CuE yield by 26.1 %, while decreasing extraction duration by 34.8 %, as well as reducing CO_2 usage by 52 %, compared with the simple $ScCO_2$ procedure. A similar investigation has been performed using self-designed US+$ScCO_2$ apparatus for the recovery of oleanolic acid (OA) and ursolic acid (UA) from *Scutellaria barbata* D. Don (Yang et al., 2013). After procedure optimization with 80 % (v/v) aqueous ethanol, the extract yields obtained with the synergistic apparatus were found to be 26.8–27.7 % higher, in OA and UA respectively, than that of the conventional reflux protocol, with the additional value of reducing time and temperature required, thereby saving energy. In addition, US+$ScCO_2$ and $ScCO_2$ have similar yields, with a small increase for the hybrid system, but with the main advantage of reducing the extraction time from 140 min to 65. The authors also investigated the same apparatus in the extraction of apigenin from *S. barbata* D. Don (Yang, Wei, 2018). The hybrid technology reached an overall yield of 20.1 %, with 31.6 % more apigenin and a reduction in process time, compared to simple $ScCO_2$ and reflux protocols (1.9 and 2.4 times, respectively). Again, the time and energy efficiency of this hybrid system have been confirmed, in this case in the extraction of oleanolic and ursolic acids from *Hedyotis corymbosa*, and higher obtained yields, with respect to conventional $ScCO_2$ and reflux extraction, have been achieved (Yang, Wei, 2016). Soares et al. (2018), have used hybrid US+$ScCO_2$ apparatus for the extraction of rice oil, which contained a high percentage of oryzanol and tocopherols, from rice bran. The lab extraction unit merged the $ScCO_2$ extraction vessel with an ultrasonic probe. The best condition (160 W / 40 min) resulted in a 12.65 % extraction yield, over initial biomass. When US was not applied, the global outcome dropped to 9.94 %, representing a decrease of 21.4 % in global production rate. Although published back in 2010, the work of Riera et al. (2010), remains the only piece of literature to report an attempt to scale up the hybrid US+$ScCO_2$ technology combination. The described pilot



Parameter	4 × US-PEF	PEFM	PEF
Voltage; Pulse; Frequency	[8 kV; 30 µs; 15 Hz]	[10 kV; 30 µs; 17 Hz]	[8 kV; 30 µs; 15 Hz]
Electric Field	1.6 kV/cm	2.0 kV/cm	1.6 kV/cm
Total Energy	3.5 kJ/kg	5.1 kJ/kg	3.5 kJ/kg
Power	0.8 kW	1.5 kW	0.8 kW

Fig. 12. Flow diagram of US×PEF system, adapted from Grillo et al. (2022).

reactor was designed, built, tested and validated by the research group on the basis of an earlier prototype from previous work (Riera et al., 2004). In the former prototype, some instabilities were detected in the US transducer, caused by observed changes in the acoustic impedance of the medium under supercritical conditions during the process. In the new system, these interactions were eliminated by separating the resonance frequencies of the basket and the transducer to a value of approximately 1 kHz. In addition, the automatic control of the operating conditions was included in the new device features. The prototype was designed with four extraction vessels of 5 L capacity each and a transducer, inserted into the upper part of the vessel, working at 19 kHz with a maximum power of approx. 110 W. Specifically, two experimental trials were performed using ground almonds (*Prunus amygdalus* Batsch.) and cocoa (*Theobroma cacao* L.) cake (Fig. 11).

The system treated 1500 g of each biomass at a time, for 3.5–4 h with an US power of 85 W. The results confirmed yield increases, compared to the control sample without US, of 90 % and 43 % for almond and cocoa cakes, respectively.

4.1.5. US×PEF

The combination of PEF and US in pretreatment processes for the hydroalcoholic extraction of polyphenols, ascorbic acid, hesperidin and

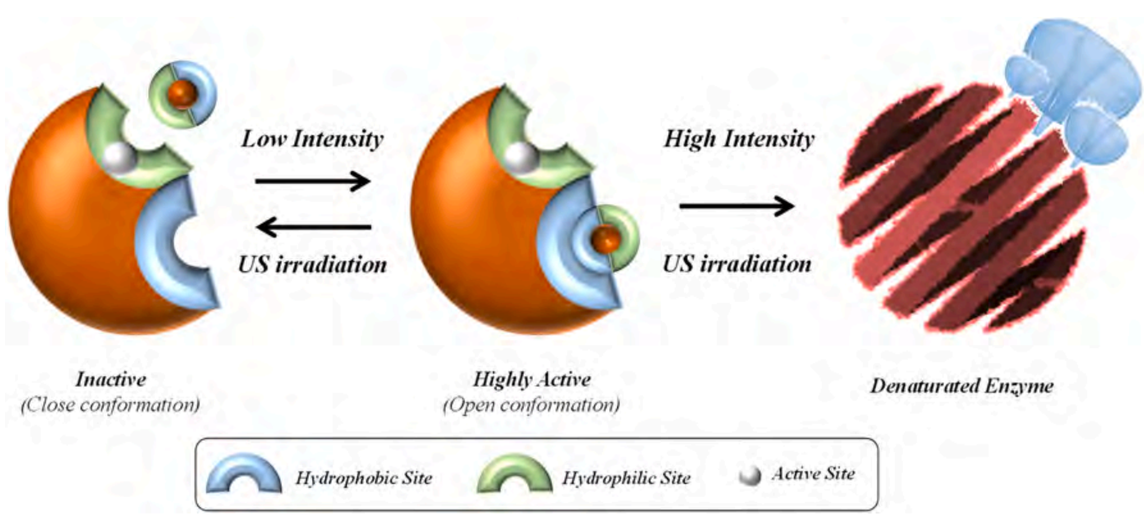


Fig. 13. Schematic representation of ultrasound effect of enzyme structural changes. Figure inspired by Nadar, Rathod (2017).

carotenoids from orange peels has been carried out by Athanasiadis et al. (2022). The recorded results underline that the combined PEF+US treatment produced the extract with the highest TPC and antioxidant activity of the other conditions tested. The possible application of non-thermal US and PEF in a single continuous flow process has been investigated by Grillo et al. (2022) for the production of virgin olive oil (VOO) on an industrial scale (Fig. 12). The customized production line replaced the malaxing process with a multi-reactor US device followed by a PEF chamber.

Besides a control using a conventional malaxer, three different combinations (US only, US+PEF and PEF+malaxation) were tested. The employed US device was made up of four flow reactors (internal volume of 330 mL) operating in series at 22 kHz and 600 W, equipped with a CAA-GP-1 generator (maximum power 1000 W), while an EPULSUS®-PM2–15 device was used for PEF (maximum voltage 15 kV, maximum power 6 kW). The results, obtained upon treating 1800 kg of olives (of the Coratina and Taggiasca cultivars) in each trial at a workflow of 900 kg/h, attested the efficiency of both technologies for VOO extraction, especially when the hybrid set-up without malaxing was applied. In the latter case, the increase in oil yield was 18.1 %, and a higher quality was achieved, as evidenced by the overall tocopherol and tocotrienol enrichment (+17.0 %) and the comparable phenolic concentration (+3.8 %), without affecting the free acidity or peroxide values. The sustainability of the process was also enhanced, saving up to 1512 L of water in the decanting phase per working day (about 7200 kg vs 4120 kg of olive paste) and reducing the energy consumed (0.964 kW/kg of oil vs. 0.500 kW/kg), mainly due to the low operating costs and the elimination of the malaxation phase (Angiolillo et al., 2015).

4.1.6. US×OH

The combination of OH and US in a single prototype serves to increase the extraction potential of these technologies when used individually. This synergy utilises electroporation induced by OH in tandem with the shear forces generated by US cavitation. The combined effect facilitates the release of bioactive constituents trapped within plant cells, overcoming the inherent barriers created by cell walls. The simultaneous application of OH×US has been tested in the hydrodistillation of Citronella essential oil (*Cymbopogon citratus*), which provides a more efficient process in terms of yield and energy saving compared to conventional water distillation (Zhang et al., 2022). A clear positive correlation is observed between the increase in electric current and US power and the increase in essential oil yield. In addition, it is found that increasing the solid to liquid ratio decreases the thermal conductivity of the OH, which requires a longer processing time to reach the target temperature. With the optimised process conditions (40 min, US power of 180 W, liquid:solid ratio of 7, ohmic current of 5 A), an essential oil extraction rate of 22.91 ± 0.13 mL/kgDW.

4.2. Enzyme-based hybrids

The technologies already addressed, namely US, PEF and MAE, can enhance enzyme activity by improving their access to the substrate, changing the medium's characteristics and promoting system kinetics. As reported in the following sections, the simultaneous combination of enzyme-assisted extraction (EAE) and physical methods must be performed in view of the labile stability of enzyme structure, which can be affected by harsh conditions, leading to a lack of activity. With these cautions in mind, the simultaneous integration of enzymes into extraction procedures can lead to increases in efficiency, representing an interesting opportunity for process intensification.

4.2.1. US×Enzyme (USAEE)

The focus on this type of coupling is found in the numerous research papers in the current literature that report many results from the simultaneous combination of enzymes and US on different natural matrices. This hybrid technology can increase process yields due to the shear forces exerted by cavitation, which enhance the surface area of the material and promote mass transfer, making it more accessible by the enzymes. Moreover, when the enzyme is irradiated with an optimal US frequency at the right power/intensity, it can undergo favourable conformational changes, resulting in an increase in its activity (Huang et al., 2017). As a consequence, it is possible to decrease the required enzyme concentration for the extraction, with a final cost reduction. Despite these advantages, the energy consumption of the process and the effect of US on enzyme structure, which directly affects bio-catalytic activity, must be considered (Fig. 13). In fact, high-intensity cavitation consumes a considerable amount of energy and has a strong impact on the matrix, with the risk of damaging enzymatic proteins through mechanical forces or the generation of free radicals. Tolerance towards US might depend on the physiological properties of enzyme as well as on operational parameters, such as power/intensity and frequency (Nadar, Rathod, 2017).

Gao et al. (2022) have investigated a green US-assisted enzymatic method (USAEE) for the efficient extraction of phenolic metabolites from *Empetrum nigrum*. The optimal USAEE conditions (incubation temperature 38.39 °C, incubation time 3.39 h, tannase concentration 386.53 U/g, cellulase concentration 224.42 U/g followed by sonication in an ultrasonic bath at 42 °C, 250 W for 22 min) led to an increment in the total phenolic content of 1.62 and 1.73 times respect to those of the single UAE and AEE, respectively. Lei-Xiong et al. (2019) have obtained promising results in the USAEE of flavonoid from *Cyclocarya paliurus* (Batal) Iljinskaja. To maximize the yield and biological activity of the extracts, four main parameters were optimized; temperature, pH, enzyme concentration and US power. The cavitation treatment was carried out in an ultrasonic bath at 90 W for 30 min. It is worth noting that an increase in these two parameters resulted in accelerating the degradation of flavonoids. The enzyme working temperature and its concentration, in this case 50 °C and 3 % w/w respectively, are essential in optimizing the whole process. For both parameters, exceeding the optimum values not only wastes resources, but also reduces extraction efficiency. Purified extracts rich in polyphenols could be used for their antioxidant properties in pharmaceuticals, functional foods and natural cosmetics. Yun et al. (2022) have applied USAEE as a pretreatment method on *Scutellaria baicalensis* roots for the extraction (performed in hydroalcoholic solution under US irradiation) of baicalein and wogonin. The optimized conditions (cellulase 1.1 % w/v, pH 5.5, 39.4 min, US power 200 W and frequency 40 kHz) resulted in an almost two-fold increase in the recovery of the two metabolites from the matrix, demonstrating that cavitation significantly accelerates enzyme-assisted hydrolysis. This pretreatment method represents a simple and environmentally friendly process that can be combined with several main extraction procedures, such as UAE, MAE, *etc*. Luo et al. (2019) have investigated USAEE for tannin extraction from oak nut, exploiting cellulases. US was found to positively affect enzyme activity, with a maximum yield of 61.22 %. However, times above 2.5 h, power higher than 100 W and temperature above 38 °C drastically dropped the recovery of tannins, partially attributable to enzymes deactivation. Li et al. (2021) have developed a protocol for agar extraction from *Gelidium sesquipedale* using an enzyme-assisted method combined with US under controlled alkaline pretreatment. The optimal protocol using Viscozym® increases the agar extraction yield 6-fold, compared to the non-enzymatic approach. In addition, the hybrid solution shortens the process time from 3 h to 1 h, which is further reduced to 30 min when coupled with alkaline pretreatment. A synergetic effect due to the combination of alkalase enzyme with US was demonstrated in protein and antioxidant-compound extraction from sesame bran (Görgüç et al., 2019). However, the highest total polyphenol content was given by the extraction performed by simple enzymatic treatment, this result could be related to the negative effect of high-power US (700 W) on polyphenols. USAEE has also been reported for protein recovery on animal matrices. Yang et al. (2021) have investigated the extraction efficiency of low-molecular-weight peptides from bovine bone using US-assisted

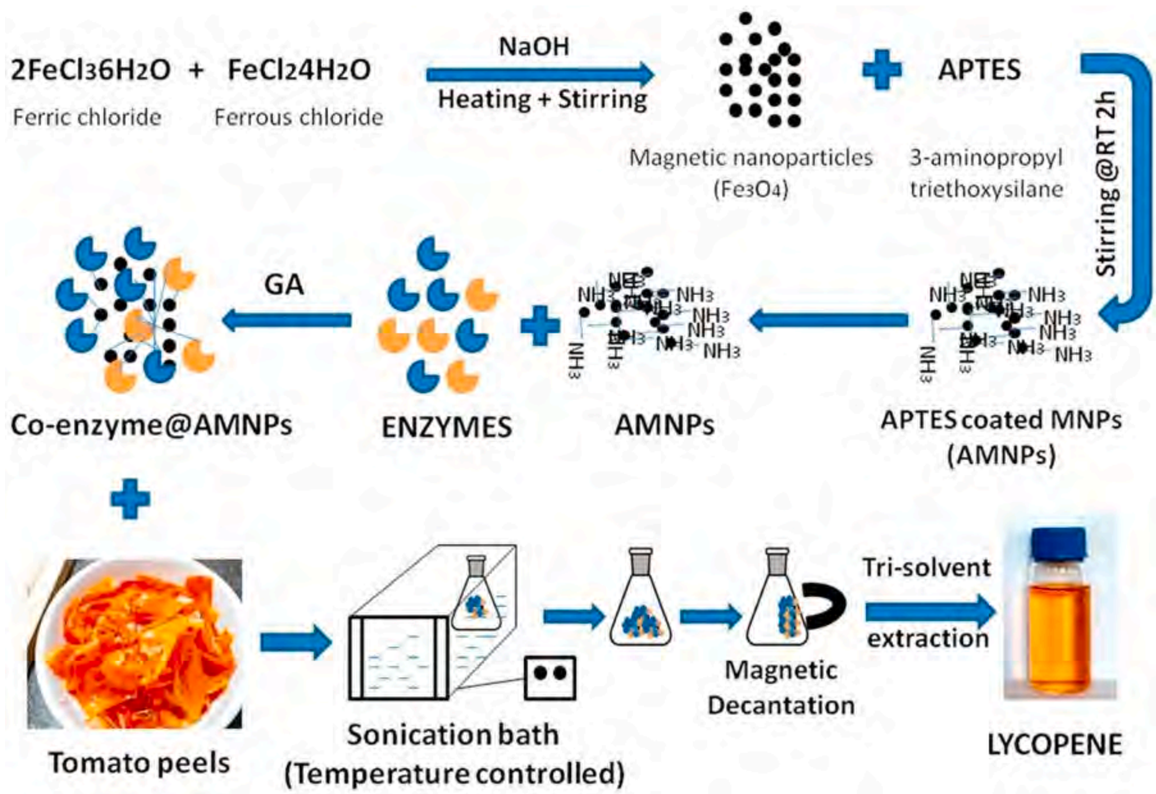


Fig. 14. Schematic of Lycopene extraction via the synergistic effect of co-immobilized enzyme and sonication. Reprinted with permission from Elsevier (Ladole et al., 2018).

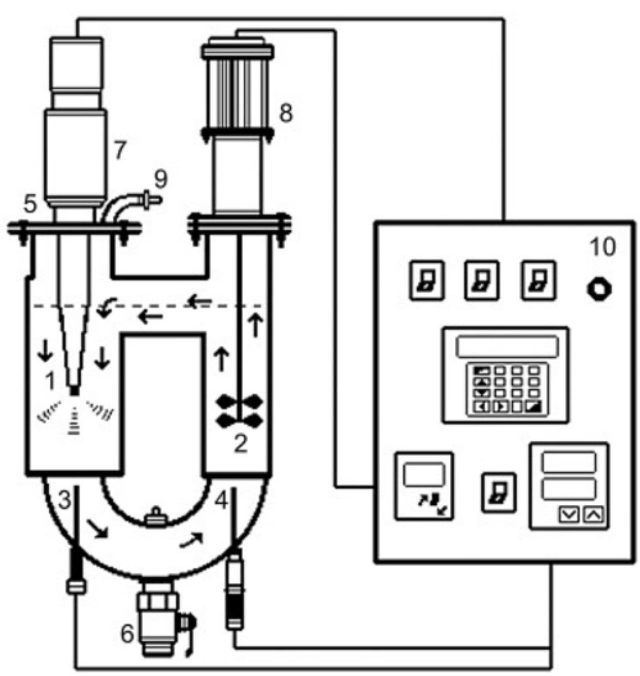


Fig. 15. Schematic diagram of ultrasonic circulating extraction equipment. (1) Transducer, (2) agitator, (3) heater, (4) temperature sensor, (5) inlet, (6) outlet, (7) ultrasonic generator, (8) reflux-circulating controller, (9) vent, (10) control system. Reprinted with permission from Elsevier (Chen et al., 2016).

double enzyme hydrolysis. The protocol was divided into two main steps; a neutral enzymatic hydrolysis of the bovine leg bone powder followed by an US-assisted alkaline enzymatic hydrolysis. The optimal experimental conditions (US power 400 W/L, pH 9, 2 h) resulted in a 4.33 % higher extraction yield than that provided by the simple application of enzymes.

One of the most common approaches to applying enzymes in extraction procedures consists in a simple suspension inside the treatment mixture. Recently, the immobilization of bio-catalysts on a solid substrate has gained relevance, and this kind of approach is attracting much interest due to the possibility of recovering the protein after the process and recycling it. It has been reported that US has the ability to hyperactivate the enzyme supported on magnetic nanoparticles (MNPs) (Ladole et al., 2017). Ladole et al. (2018) have verified that under US cavitation at 24 kHz, 6 W and 6 min of incubation time, immobilized cellulase increased activity almost 3.6-fold, compared to the control (without US). The authors also pre-treated tomato peels exploiting USAEE (with a combination of pectinase and cellulase) immobilized on MNPs to increase lycopene release (Fig. 14). The optimal conditions (pH 5, 50 °C, 3 % (w/w) MNPs co-immobilized enzyme and tomato peels, US at 10 W and 20 kHz, 20 min incubation) gave a higher lycopene yield than the two systems alone, demonstrating a synergistic effect.

Increased US power and longer incubation times lead to lower carotenoid recovery, probably due to enzyme deactivation and metabolite denaturation. Furthermore, the supported biocatalyst showed excellent reusability, with a product yield of more than 50 % even after 6 cycles. Chen et al. (2016) have used, for the extraction of kernel oil from *Pinus pumila*, a homogenate-circulating US apparatus in combination with an enzyme mixture consisting of cellulase, pectinase and hemicellulase (1:1:1, w/w/w) (Fig. 15). The extraction system is designed to improve solution mixing and promote deeper and the more uniform penetration of the mechanical wave into the extracting mixtures. Thus, solvent extraction capacity is enhanced, together with cavitation homogeneity and enzyme distribution, thus increasing process efficiency.

The applied optimal conditions, involving 1.7 h total extraction time with 2.5 % weight percentage of a mixed enzyme solution of cellulase, pectinase and hemicellulose (1:1:1 w/w/w) and 600-W ultrasound irradiation power, yielded 31.89 % of oil, compared to the 40.26 % of conventional Soxhlet method with n-hexane. Despite the lower yield, the fatty acid composition and physico-chemical properties of the oils were very similar between the two procedures, highlighting the efficiency of the hybrid system in obtaining a high-quality oil for pharmaceuticals and health-care applications. The application of US in an extraction process is usually carried out using high-intensity US to promote the shear forces, exerting a disrupting effect on the matrix. However, as already mentioned, high-intensity has the main drawbacks of consuming a considerable amount of energy and the risk of causing degradation of enzymes or extractants. Hence, reducing the power of the transducers reduces the energy consumed, but can also reduce the treatment effect. A different approach to USAEE extraction, using low-intensity US (LIU) instead of high-intensity US (HIU), has been reported by Yan et al. (2022) with the aim of enhancing enzyme activity rather than destroying the cell walls of the matrix. This study revealed that LIU, when combined with an alkaline protease for the extraction of protein from sludge (deriving from wastewater management), can compensate the limited matrix-destroying effects with an improved extraction efficiency, being at the same time more economical than HIU. Specifically, it was reported that, although HIU achieved a 13 % higher protein yield, the amount of free amino acids generated with LIU was the same, thus showing its better enzymatic efficiency. Moreover, if compared with an untreated sample, the protein extraction rate with LIU result was found to be 44.1 % higher, with 30.8 % lower enzyme consumption. In addition, the dehydration performance of LIU was approximately 12.1 % higher than that of HIU, benefiting the subsequent separation of the protein solution and helping to reduce the overall cost of the procedure. Finally, LIU consumed 19.8 % less energy than HIU, which combined with the other outcome, makes the proposed approach more economical and reasonable in terms of enzyme dosage and energy consumption.

4.2.2. MAE×Enzyme (EMAE)

The simultaneous combination of enzyme-assisted extraction and MW-assisted extraction (EMAE) has been recognized as a valuable technique for the obtainment of bioactive components from different biomasses. This is due to several advantages, such as environmental compatibility, high extraction yields, time efficiency and low solvent consumption (Nadar et al., 2018). The application of MAE in the extraction of bioactive compounds presents the peculiarity of higher efficiency and thermal selectivity, shorter extraction times, lower solvent usage and lower energy consumption compared with conventional heating methods. Thanks to their biological performance, the addition of suitable types of enzymes into the matrix during MAE extraction can boost the thermal selectivity and shorter process times of the dielectric heating. Macedo et al. (2021) have evaluated the EMAE efficiency for the extraction of phenolic compounds from olive pomace. The use of tannase increased the total phenolic content, when compared to EMAE with cellulase or pectinase. Moreover, when the applied temperature was 60 °C (optimal for the biocatalyst), the enzyme addition resulted in higher extraction yields, with respect to simple MAE. The advantages of adopting EMAE with water as an alternative to conventional ethanol-based solutions have therefore been demonstrated, also involving time and solid-liquid ratio reduction. Similarly, Jia et al. (2021) enhanced the recovery efficiency of phenolic compounds from grape seed via enzyme and MW co-assisted salting-out extraction (EMASOE). The efficiency of this technique was compared with those of enzyme-assisted salting-out extraction (EASOE), microwave-assisted salting-out extraction (MASOE), salting-out extraction and Soxhlet extraction (80 % ethanol, 90 °C, 6 h). Under optimal conditions, EMASOE (25 % w/w ethanol/20 % w/w ammonium sulfate, pectinase 540 U/g, enzyme pH 4.5, MW 180 W) achieved the highest polyphenol extraction yield (125.32 mg/g) of all the methods, also demonstrating the recovery of a greater diversity of monophenols in the product composition. Lin et al. (2022) have integrated the use of enzymes (papain) with MAE aiming to polysaccharides with antioxidant activities from Purple-heart Radish, using an aqueous two-phase system (ATPS) involving a mixture of ethanol-$(NH_4)_2SO_4$ as the biphasic extractant. The use of ATPS allowed two different polysaccharide classes, retained in the polar and non-polar phases, to be simultaneously extracted. Different extraction methods, such as hot maceration and reflux extraction, together with EAE, MAE, UAE were utilized for the comparative study. The hybrid methodology showed the best extraction performance with a total polysaccharide yield of 41.61 %, in addition to reduced process times and promising antioxidant activity. The high extraction efficiency was mainly attributed to the synergistic effect of MW irradiation, which cleaved the plant tissues, promoting mass transfer and the enzymatic hydrolysis of glycoproteins, which promoted polysaccharide dissolution. Different MW and xylanase coupling strategies were investigated to assist the extraction of hypericin from *Hypericum perforatum* (MAE before/after enzyme extraction and EMAE, with simultaneous application), with best results were achieved by applying MAE after xylanase addition (Zhang et al., 2019). In the optimized protocol, the extraction yield of hypericin (0.315 ± 0.002 mg/g) increased by 33.5 %, compared to xylanase-assisted extraction alone, by 11.4 % compared to MAE alone and by 209.7 % compared with the conventional Soxhlet protocol with ethanol. It has been observed that the simultaneous use of enzymes and MW irradiation can affect the structure of the former due to thermal unfolding effects, which lower activity and, consequently, reduce the overall extraction yield. A study conducted by Chen et al. (2018) has reported the use of EMAE as pretreatment followed by MW-assisted hydrodistillation concatenated with a liquid-liquid separator to extract the essential oil from *Polygonum viscosum* Buch-ham. Enzyme hydrolysis was applied with a mixture of cellulases, pectinases and hemicellulases to release the intracellular components and improve the extraction of essential oils. The combined treatment resulted in higher yields in shorter times, presenting a promising strategy in the isolation of essential oils from a plant matrix.

4.2.3. OH×Enzyme

The process intensification of enzyme pretreatments with the simultaneous combination of OH has been reported in a multistage extraction from *Spirulina platensis* (Ferreira-Santos et al., 2021). The combined pretreatment was compared with the single technology and an aqueous extraction at 37 °C as a control. The pretreatment had the double role of phycobiliprotein recovery in the aqueous phase and facilitate the cell wall disruption, increasing the extraction in the next stages (intracellular compounds). After the pretreatment, the biomass was subject to a second extraction for polyphenols recover and a third for lipids. The simultaneous combination of the two technologies resulted in an enhanced yield of phycobiliprotein, doubled when compared with the control pretreatment. Furthermore, the recovery of lipophilic bioactives, promoted by the combined techniques, was upgraded compared to the other tested protocols.

5. Conclusions

The increasing urge towards greener and sustainable approaches merges with the circular economy concepts, which aim towards waste reduction, efficiency maximization and pollution prevention. The chance to fulfil these conditions can strongly rely on so-called enabling technologies, leading to disruptive process intensification. It follows that the implementation of these techniques has become widespread in several processes over recent decades, enhancing heat/mass transfer together with reducing energy and time consumptions. On the other hand, the valorisation of wastes and the “benign-by-design” approach are becoming more and more compulsory, requiring even further efforts. Hence, to meet this rising demand, new solutions are required. In this framework, two or even more enabling technologies can be hyphenated in sequential or simultaneous systems, dramatically boosting process intensifications and applicability by, for example, improving mixing and heating protocols, promoting biomass pretreatment and metabolite dissolution. In this literature review, both hybrid and coupled green-extraction technologies have been surveyed over a time-span of 5 years, highlighting differences between these two strategies, with particular attention toward achievements and TRL (Technology Readiness Level). The aim is to shed light on the potential offered by the synergic combination of multiple green methods in the recovery of bioactive compounds from biomass. The collected information can provide an outlook of the state-of-the-art, helping the reader to focus on challenges to overcome and opportunities to seize. One major aspect worthy of attention is the imperative role of pilot and pre-industrial systems, given that just a few examples can be found in literature, and in general with contained up-scaling dimensions. To ensure the effective exploitability of the proof-of-concept procedures realized on small/medium scales, an increase in the overall TRL to meet industrial demand will lead to evolution towards real case-studies. This role is commonly filled by "intermediate" readiness levels, able to bridge between "gram-scale" academia and "ton-scale" industry. Those TRLs could be suitably developed taking the lead from already mature single technologies (i.e. US, PEF, HSH, …), that could act as a core around which evolve several sequential or hybrid upgrades. In this framework, another under-focused topic, strictly related to industrial applications, are the cost and energy analyses, which cover a key-role for real applications. These essential evaluations are the foundation on which build a proper sustainability assessment by means of Green Metrics (i.e. E-Factor, Process Mass Intensity, …) and Life Cycle Analysis (i.e. cradle-to-cradle or creed-to-grave approach). The lack of these aspects strongly limits the appropriate development of hybrid and sequential green technologies.

It is not necessary to underline that green chemistry and circular economy concepts have an undeniable contribution to shaping the next generation of the chemical industry, focusing on innovation and more sustainable production/valorisation models of food, clean water and energy.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

No data was used for the research described in the article.

References

Aamir, M., Jittanit, W., 2017. Ohmic heating treatment for gac aril oil extraction: effects on extraction efficiency, physical properties and some bioactive compounds. Innov. Food Sci. Emerg. 41, 224–234. https://doi.org/10.1016/j.ifset.2017.03.013.

Abad, V., Alejandre, M., Hernández-Fernández, E., Raso, J., Cebrián, G., Álvarez-Lanzarote, I., 2023. Evaluation of pulsed electric fields (PEF) parameters in the inactivation of Anisakis Larvae in saline solution and hake meat. Foods 12 (2). https://doi.org/10.3390/foods12020264.

Al-Hilphya, A.R., Al-Musaferb, A.M., Gavahian, M., 2020. Pilot-scale ohmic heating-assisted extraction of wheat bran bioactive compounds: effects of the extract on corn oil stability. Food Res. Int. 137, 109649 https://doi.org/10.1016/j.foodres.2020.109649.

Allaf, T., Tomao, V., Ruiz, K., Chemat, F., 2013. Instant controlled pressure drop technology and ultrasound assisted extraction for sequential extraction of essential oil and antioxidants. Ultrason Sonochem. 20 (1), 239–246. https://doi.org/10.1016/j.ultsonch.2012.05.013.

Anastas, Paul A., 1995. Benign by design: alternative synthetic design for pollution prevention, 296A-296A Environ. Sci. Technol. 29 (7). https://doi.org/10.1021/es00007a708.

Angiolillo, L., Del Nobile, M.A., Conte, A., 2015. The extraction of bioactive compounds from food residues using microwaves. Curr. Opin. Food Sci. 5, 93–98. https://doi.org/10.1016/j.cofs.2015.10.001.

Athanasiadis, V., Chatzimitakos, T., Kotsou, K., Palaiogiannis, D., Bozinou, E., Lalas, S.I., 2022. Optimization of the extraction parameters for the isolation of bioactive compounds from orange peel waste. Sustain. (Switz.) 14 (21). https://doi.org/10.3390/su142113926.

Balachandran, S., Kentish, S.E., Mawson, R., Ashokkumar, M., 2006. Ultrasonic enhancement of the supercritical extraction from ginger. Ultrason Sonochem. 13 (6), 471–479. https://doi.org/10.1016/j.ultsonch.2005.11.006.

Calcio Gaudino, E., Grillo, G., Tabasso, S., Stevanato, L., Cravotto, G., Marjamaa, K., Pihlajaniemi, V., Koivula, A., Aro, N., Uusitalo, J., Ropponen, J., Kuutti, L., Kivinen, P., Kanerva, H., Arshanitsa, A., Jashina, L., Jurkjane, V., Andersone, A., Dreyer, T., Schories, G., 2022. Optimization of ultrasound pretreatment and enzymatic hydrolysis of wheat straw: from lab to semi-industrial scale. J. Clean. Prod. 380, 134897 https://doi.org/10.1016/j.jclepro.2022.134897.

Carullo, D., Donsì, F., Ferrari, G., Pataro, G., 2021. Extraction improvement of water-soluble compounds from Arthrospira platensis through the combination of high-shear homogenization and pulsed electric fields. Algal Res. 57 https://doi.org/10.1016/j.algal.2021.102341.

Chemat, F., Vian, M.A., Cravotto, G., 2012. Green extraction of natural products: concept and principles. Int J. Mol. Sci. 13 (7), 8615–8627. https://doi.org/10.3390/ijms13078615.

Chemat, F., Vian, M.A., Ravi, H.K., 2021. Toward petroleum-free with plant-based chemistry. Curr. Opin. Green. Sustain Chem. 28 https://doi.org/10.1016/j.cogsc.2021.100450.

Chen, F., Zhang, Q., Gu, H., Yang, L., 2016. An approach for extraction of kernel oil from Pinus pumila using homogenate-circulating ultrasound in combination with an aqueous enzymatic process and evaluation of its antioxidant activity. J. Chromatogr. A 1471, 68–79. https://doi.org/10.1016/j.chroma.2016.10.037.

Chen, F., Jia, J., Zhang, Q., Yang, L., Gu, H., 2018. Isolation of essential oil from the leaves of Polygonum viscosum Buch-ham. using microwave-assisted enzyme pretreatment followed by microwave hydrodistillation concatenated with liquid–liquid extraction. Ind. Crops Prod. 112, 327–341. https://doi.org/10.1016/j.indcrop.2017.12.024.

Chen, J., Wang, X., Zhang, B., Yang, Y., Song, Y., Zhang, F., Liu, B., Zhou, Y., Yi, Y., Shan, Y., Lü, X., 2021. Integrating enzymatic hydrolysis into subcritical water pretreatment optimization for bioethanol production from wheat straw. Sci. Total Environ. 770 https://doi.org/10.1016/j.scitotenv.2021.145321.

Cravotto, C., Grillo, G., Binello, A., Gallina, L., Olivares-Vicente, M., Herranz-López, M., Micol, V., Barrajón-Catalán, E., Cravotto, G., 2022. Bioactive antioxidant compounds from chestnut peels through semi-industrial subcritical water extraction. Antioxidants 11 (5). https://doi.org/10.3390/antiox11050988.

Cravotto, G., Binello, A., 2016. Low-frequency, high-power ultrasound-assisted food component extraction. : Innovative Food Processing Technologies: Extraction, Separation, Component Modification and Process Intensification. Elsevier Inc,, pp. 3–29. https://doi.org/10.1016/B978-0-08-100294-0.00001-8.

Dias, A.L.B., de Aguiar, A.C., Rostagno, M.A., 2021. Extraction of natural products using supercritical fluids and pressurized liquids assisted by ultrasound: current status and trends. Ultrason Sonochem. 74, 105584 https://doi.org/10.1016/j.ultsonch.2021.105584.

Dzah, C.S., Duan, Y., Zhang, H., Authur, D.A., Ma, H., 2020. Ultrasound-, subcritical water- and ultrasound assisted subcritical water-derived Tartary buckwheat polyphenols show superior antioxidant activity and cytotoxicity in human liver carcinoma cells. Food Res. Int. 137 https://doi.org/10.1016/j.foodres.2020.109598.

Esposito, B., Sessa, M.R., Sica, D., Malandrino, O., 2020. Towards circular economy in the agri-food sector. A systematic literature review. Sustain. (Switz.) 12 (18). https://doi.org/10.3390/SU12187401.

Estrada-Gil, L., Contreras-Esquivel, J.C., Flores-Gallegos, C., Zugasti-Cruz, A., Govea-Salas, M., Mata-Gómez, M., Rodríguez-Herrera, R., Ascacio-Valdés, J., 2022. Recovery of bioactive ellagitannins by ultrasound/microwave-assisted extraction from Mexican Rambutan Peel (Nephelium lappaceum L.). Molecules 27 (5). https://doi.org/10.3390/molecules27051592.

Fadel, A., Ashworth, J., Plunkett, A., Mahmoud, A.M., Ranneh, Y., Li, W., 2018. Improving the extractability of arabinoxylans and the molecular weight of wheat endosperm using extrusion processing. J. Cereal Sci. 84, 55–61. https://doi.org/10.1016/j.jcs.2018.09.009.

Fauster, T., Philipp, C., Hanz, K., Scheibelberger, R., Teufl, T., Nauer, S., Scheiblhofer, H., Jaeger, H., 2020. Impact of a combined pulsed electric field (PEF) and enzymatic mash treatment on yield, fermentation behaviour and composition of white wine. Eur. Food Res. Technol. 246 (3), 609–620. https://doi.org/10.1007/s00217-020-03427-w.

Ferreira-Santos, P., Miranda, S.M., Belo, I., Spigno, I., Teixeira, J.A., Rocha, C.M.R., 2021. Sequential multi-stage extraction of biocompounds from Spirulina platensis: combined effect of ohmic heating and enzymatic treatment. IFSET 71, 102707. https://doi.org/10.1016/j.ifset.2021.102707.

Gao, Y., Shi, Y., Miao, N., Xing, W., Yun, C., Wang, S., Wang, W., Wang, H., 2022. A green ultrasound-assisted enzymatic extraction method for efficient extraction of total polyphenols from Empetrum nigrum and determination of its bioactivities. J. Ind. Eng. Chem. 109, 559–567. https://doi.org/10.1016/j.jiec.2022.02.041.

Garcia-Vaquero, M., Ummat, V., Tiwari, B., Rajauria, G., 2020. Exploring ultrasound, microwave and ultrasound-microwave assisted extraction technologies to increase the extraction of bioactive compounds and antioxidants from brown macroalgae. Mar. Drugs 18 (3). https://doi.org/10.3390/md18030172.

Gaudino, E.C., Grillo, G., Manzoli, M., Tabasso, S., Maccagnan, S., Cravotto, G., 2022. Mechanochemical applications of reactive extrusion from organic synthesis to catalytic and active materials. Molecules 27 (2). https://doi.org/10.3390/molecules27020449.

Gavahian, M., Sastry, S., Farhoosh, R., Farahnaky, A., 2020. Chapter six - Ohmic heating as a promising technique for extraction of herbal essential oils: understanding mechanisms, recent findings, and associated challenges. Adv. Nutr. 91, 227–273. https://doi.org/10.1016/bs.afnr.2019.09.001.

Gavahiana, M., Farahnaky, A., 2018. Ohmic-assisted hydrodistillation technology: a review. Trends Food Sci. Tech. 72, 153–161. https://doi.org/10.1016/j.tifs.2017.12.014.

Getachew, A.T., Chun, B.S., 2017. Influence of pretreatment and modifiers on subcritical water liquefaction of spent coffee grounds: a green waste valorization approach. J. Clean. Prod. 142, 3719–3727. https://doi.org/10.1016/j.jclepro.2016.10.096.

Getachew, A.T., Cho, Y.J., Chun, B.S., 2018. Effect of pretreatments on isolation of bioactive polysaccharides from spent coffee grounds using subcritical water. Int J. Biol. Macromol. 109, 711–719. https://doi.org/10.1016/j.ijbiomac.2017.12.120.

Ghosh, S., Gillis, A., Sheviryov, J., Levkov, K., Golberg, A., 2019. Towards waste meat biorefinery: Extraction of proteins from waste chicken meat with non-thermal pulsed electric fields and mechanical pressing. J. Clean. Prod. 208, 220–231. https://doi.org/10.1016/j.jclepro.2018.10.037.

Giahi, E., Jahadi, M., Khosravi-Darani, K., 2021. Enzyme-assisted extraction of glycyrrhizic acid from licorice roots using heat reflux and ultrasound methods. Biocatal. Agric. Biotechnol. 33 https://doi.org/10.1016/j.bcab.2021.101953.

Görgüç, A., Bircan, C., Yılmaz, F.M., 2019. Sesame bran as an unexploited by-product: effect of enzyme and ultrasound-assisted extraction on the recovery of protein and antioxidant compounds. Food Chem. 283, 637–645. https://doi.org/10.1016/j.foodchem.2019.01.077.

Grillo, G., Boffa, L., Talarico, S., Solarino, R., Binello, A., Cavaglià, G., Bensaid, S., Telysheva, G., Cravotto, G., 2020. Batch and flow ultrasound-assisted extraction of grape stalks: process intensification design up to a multi-kilo scale. Antioxidants 9 (8), 1–30. https://doi.org/10.3390/antiox9080730.

Grillo, G., Boffa, L., Calcio Gaudino, E., Cravotto, G., 2022. Combined ultrasound and pulsed electric fields in continuous-flow industrial olive-oil production. Foods 11 (21), 3419. https://doi.org/10.3390/foods11213419.

Guiao, K.S., Gupta, A., Tzoganakis, C., Mekonnen, T.H., 2022a. Reactive extrusion as a sustainable alternative for the processing and valorization of biomass components. J. Clean. Prod. 355 https://doi.org/10.1016/j.jclepro.2022.131840.

Guiao, K.S., Tzoganakis, C., Mekonnen, T.H., 2022b. Green mechano-chemical processing of lignocellulosic biomass for lignin recovery. Chemosphere 293. https://doi.org/10.1016/j.chemosphere.2022.133647.

Guo, X., Aganovic, K., Bindrich, U., Juadjur, A., Hertel, C., Ebert, E., Macke, J., Geil, C., Heinz, V., 2022. Extraction of protein from juice blend of grass and clover pressed by a pilot pressing facility combined with a pulsed electric field treatment. Future Foods 6, 100173. https://doi.org/10.1016/j.fufo.2022.100173.

Hashemi, B., Shiri, F., Švec, F., Nováková, L., 2022. Green solvents and approaches recently applied for extraction of natural bioactive compounds. TrAC - Trends Anal. Chem. 157 https://doi.org/10.1016/j.trac.2022.116732.

Hernández-Hernández, C., Aguilar, C.N., Flores-Gallegos, A.C., Sepúlveda, L., Rodríguez-Herrera, R., Morlett-Chávez, J., Govea-Salas, M., Ascacio-Valdés, J., 2020. Preliminary testing of ultrasound/microwave-assisted extraction (U/M-AE) for the isolation of Geraniin from Nephelium lappaceum L. (Mexican variety) peel. Processes 8 (5). https://doi.org/10.3390/PR8050572.

Hossain, M.B., Aguiló-Aguayo, I., Lyng, J.G., Brunton, N.P., Rai, D.K., 2015. Effect of pulsed electric field and pulsed light pretreatment on the extraction of steroidal alkaloids from potato peels. Innov. Food Sci. Emerg. Technol. 29, 9–14. https://doi.org/10.1016/j.ifset.2014.10.014.

Huang, G., Chen, S., Dai, C., Sun, L., Sun, W., Tang, Y., Xiong, F., He, R., Ma, H., 2017. Effects of ultrasound on microbial growth and enzyme activity. Ultrason Sonochem. 37, 144–149. https://doi.org/10.1016/j.ultsonch.2016.12.018.

Hwang, H.J., Kim, Y.G., Chung, M.S., 2021a. Improving the extraction of catechins of green tea (Camellia sinensis) by subcritical water extraction (SWE) combined with pulsed electric field (PEF) or intense pulsed light (IPL) pretreatment. Foods 10 (12), 3092. https://doi.org/10.3390/foods10123092.

Hwang, H.J., Kim, H.J., Ko, M.J., Chung, M.S., 2021b. Recovery of hesperidin and narirutin from waste Citrus unshiu peel using subcritical water extraction aided by pulsed electric field treatment. Food Sci. Biotechnol. 30 (2), 217–226. https://doi.org/10.1007/s10068-020-00862-z.

Izzetti, R., Vitali, S., Aringhieri, G., Nisi, M., Oranges, T., Dini, V., Ferro, F., Baldini, C., Romanelli, M., Caramella, D., Gabriele, M., 2021. Ultra-high frequency ultrasound, a promising diagnostic technique: review of the literature and single-center experience. Can. Assoc. Radiol. J. 72 (3), 418–431. https://doi.org/10.1177/0846537120940684.

Jäger, G., Büchs, J., 2012. Biocatalytic conversion of lignocellulose to platform chemicals. Biotechnol. J. 7, 1122–1136. https://doi.org/10.1002/biot.201200033.

Jia, M.Z., Fu, X.Q., Deng, L., Li, Z.L., Dang, Y.Y., 2021. Phenolic extraction from grape (Vitis vinifera) seed via enzyme and microwave co-assisted salting-out extraction. Food Biosci. 40 https://doi.org/10.1016/j.fbio.2021.100919.

Jin, Z., Wang, M., Wu, F., Cai, H.Y., Jin, W.P., Sun, W., Chen, X., Li, F., Wang, Z., Shen, W.Y., 2020. Effect of extrusion on the total antioxidant capacity and free phenolic compounds of wheat bran by response surface methodology. Ital. J. Food Sci. 32, 386–398. https://doi.org/10.14674/IJFS-1626.

Jo, H.L., Hwang, H.J., Chung, M.S., 2019. Inactivation of Bacillus subtilis spores at various germination and outgrowth stages using intense pulsed light. Food Microbiol 82, 409–415. https://doi.org/10.1016/j.fm.2019.03.013.

Kamble, M.G., Singh, A., Prabhakar, P.K., Meghwal, M., Singh, S.V., Chinchkar, A.V., 2022. Effect of high shear homogenization on quality characteristics of bael fruit pulp. Food Qual. Saf. 6 https://doi.org/10.1093/fqsafe/fyac011.

Kaur, N., Singh, A.K., 2016. Ohmic heating: concept and applications—a review. Crit. Rev. Food Sci. Nutr. 56 (14), 2338–2351. https://doi.org/10.1080/10408398.2013.835303.

Khadhraoui, B., Ummat, V., Tiwari, B.K., Fabiano-Tixier, A.S., Chemat, F., 2021. Review of ultrasound combinations with hybrid and innovative techniques for extraction and processing of food and natural products. Ultrason. Sonochem. 76 https://doi.org/10.1016/j.ultsonch.2021.105625.

Khuenpet, K., Fukuoka, M., Jittanit, W., Sirisansaneeyakul, S., 2017. Spray drying of inulin component extracted from Jerusalem artichoke tuber powder using conventional and ohmic-ultrasonic heating for extraction process. J. Food Eng. 194, 67–78. https://doi.org/10.1016/j.jfoodeng.2016.09.009.

Kim, H.S., Ko, M.J., Park, C.H., Chung, M.S., 2022. Application of pulsed electric field as a pretreatment for subcritical water extraction of quercetin from onion skin. Foods 11 (8). https://doi.org/10.3390/foods11081069.

Kim, S.W., Ko, M.J., Chung, M.S., 2019. Extraction of the flavonol quercetin from onion waste by combined treatment with intense pulsed light and subcritical water extraction. J. Clean. Prod. 231, 1192–1199. https://doi.org/10.1016/j.jclepro.2019.05.280.

Koubaa, M., Barba, F.J., Grimi, N., Mhemdi, H., Koubaa, W., Boussetta, N., Vorobiev, E., 2016. Recovery of colorants from red prickly pear peels and pulps enhanced by pulsed electric field and ultrasound. Innov. Food Sci. Emerg. Technol. 37, 336–344. https://doi.org/10.1016/j.ifset.2016.04.015.

Krakowska-Sieprawska, A., Rafińska, K., Walczak-Skierska, J., Kiełbasa, A., Buszewski, B., 2021. Promising green technology in obtaining functional plant preparations: combined enzyme-assisted supercritical fluid extraction of flavonoids isolation from Medicago sativa leaves. Materials 14 (11). https://doi.org/10.3390/ma14112724.

Krakowska-Sieprawska, A., Kiełbasa, A., Rafińska, K., Ligor, M., Buszewski, B., 2022. Modern methods of pretreatment of plant material for the extraction of bioactive compounds. Molecules 27 (3). https://doi.org/10.3390/molecules27030730.

Kumar, M., Tomar, M., Punia, S., Amarowicz, R., Kaur, C., 2020. Evaluation of cellulolytic enzyme-assisted microwave extraction of Punica granatum peel phenolics and antioxidant activity. Plant Foods Hum. Nutr. 75 (4), 614–620. https://doi.org/10.1007/s11130-020-00859-3.

Kumari, B., Tiwari, B.K., Hossain, M.B., Brunton, N.P., Rai, D.K., 2018. Recent advances on application of ultrasound and pulsed electric field technologies in the extraction of bioactives from agro-industrial by-products. Food Bioproc Tech. 11 (2), 223–241. https://doi.org/10.1007/s11947-017-1961-9.

Kupryaniuk, K., Oniszczuk, T., Combrzyński, M., Dach, J., Czekała, W., 2020. Process efficiency and energy consumption during the extrusion of lignocellulosic materials. In: IOP Conference Series: Earth and Environmental Science, Vol 505. Institute of Physics Publishing,. https://doi.org/10.1088/1755-1315/505/1/012040.

Kutlu, N., Isci, A., Sakiyan, O., Egemen Yilmaz, A., 2021a. Effect of ohmic heating on ultrasound extraction of phenolic compounds from cornelian cherry (Cornus mas). J. Food Process. Preserv. 45 (10), e15818 https://doi.org/10.1111/jfpp.15818.

Kutlu, N., Isci, A., Sakiyan, O., Egemen Yilmaz, A., 2021b. Extraction of phenolic compounds from Cornelian Cherry (Cornus mas L.) using microwave and Ohmic heating assisted microwave methods. Food Bioprocess Tech. 14, 650–664. https://doi.org/10.1007/s11947-021-02588-0.

Ladole, M.R., Mevada, J.S., Pandit, A.B., 2017. Ultrasonic hyperactivation of cellulase immobilized on magnetic nanoparticles. Bioresour. Technol. 239, 117–126. https://doi.org/10.1016/j.biortech.2017.04.096.

Ladole, M.R., Nair, R.R., Bhutada, Y.D., Amritkar, V.D., Pandit, A.B., 2018. Synergistic effect of ultrasonication and co-immobilized enzymes on tomato peels for lycopene extraction. Ultrason Sonochem. 48, 453–462. https://doi.org/10.1016/j.ultsonch.2018.06.013.

Lal, A.M.N., Prince, M.V., Kothakota, A., Pandiselvam, R., Thirumdas, R., Mahanti, N.K., Sreeja, R., 2021. Pulsed electric field combined with microwave-assisted extraction of pectin polysaccharide from jackfruit waste. Innov. Food Sci. Emerg. Technol. 74 https://doi.org/10.1016/j.ifset.2021.102844.

Lee, A.K., Lewis, D.M., Ashman, P.J., 2012. Disruption of microalgal cells for the extraction of lipids for biofuels: processes and specific energy requirements. Biomass-.-. Bioenergy 46, 89–101. https://doi.org/10.1016/j.biombioe.2012.06.034.

Lei-Xiong, Hu, W.B., Yang, Z.W., Chen, H., Ning, W., Xin, L., Jun, W.W., 2019. Enzymolysis-ultrasonic assisted extraction of flavanoid from Cyclocarya paliurus (Batal) Iljinskaja:HPLC profile, antimicrobial and antioxidant activity. Ind. Crops Prod. 130, 615–626. https://doi.org/10.1016/j.indcrop.2019.01.027.

Le-Tan, H., Fauster, T., Haas, K., Jaeger, H., 2022. Aqueous extraction of curcuminoids from Curcuma longa: effect of cell disintegration pre-treatment and extraction condition. Food Bioproc. Tech. 15, 1359–1373. https://doi.org/10.1007/s11947-022-02820-5.

Li, Y., Zhao, M., Gomez, L.P., Senthamaraikannan, R., Padamati, R.B., O'Donnell, C.P., Tiwari, B.K., 2021. Investigation of enzyme-assisted methods combined with ultrasonication under a controlled alkali pretreatment for agar extraction from Gelidium sesquipedale. Food Hydrocoll. 120 https://doi.org/10.1016/j.foodhyd.2021.106905.

Lin, Y., Pi, J., Jin, P., Liu, Y., Mai, X., Li, P., Fan, H., 2022. Enzyme and microwave co-assisted extraction, structural characterization and antioxidant activity of polysaccharides from Purple-heart Radish. Food Chem. 372 https://doi.org/10.1016/j.foodchem.2021.131274.

Liu, X., Ou, H., Gregersen, H., 2020. Ultrasound-assisted supercritical CO_2 extraction of cucurbitacin E from Iberis amara seeds. Ind. Crops Prod. 145. https://doi.org/10.1016/j.indcrop.2020b.112093.

Liu, X., Ou, H., Xiang, Z., Gregersen, H., 2020a. Ultrasound pretreatment combined with supercritical CO_2 extraction of Iberis amara seed oil. J. Appl. Res. Med. Aromat. Plants 18. https://doi.org/10.1016/j.jarmap.2020a.100265.

Liu, X., Li, Y., Zeng, L., Li, X., Chen, N., Bai, S., He, H., Wang, Q., Zhang, C., 2022. A review on mechanochemistry: approaching advanced energy materials with greener force. Adv. Mater. 34 (46) https://doi.org/10.1002/adma.202108327.

Liu, Z., Li, H., Cui, G., Wei, M., Zou, Z., Ni, H., 2021. Efficient extraction of essential oil from Cinnamomum burmannii leaves using enzymolysis pretreatment and followed by microwave-assisted method. LWT 147. https://doi.org/10.1016/j.lwt.2021.111497.

Luo, X., Bai, R., Zhen, D., Yang, Z., Huang, D., Mao, D., Li, X., Zou, H., Xiang, Y., Liu, K., Wen, Z., Fu, C., 2019. Response surface optimization of the enzyme-based ultrasound-assisted extraction of acorn tannins and their corrosion inhibition properties. Ind. Crops Prod. 129, 405–413. https://doi.org/10.1016/j.indcrop.2018.12.029.

Macedo, G.A., Santana, Á.L., Crawford, L.M., Wang, S.C., Dias, F.F.G., de Mour Bell, J.M.L.N., 2021. Integrated microwave- and enzyme-assisted extraction of phenolic compounds from olive pomace. LWT 138. https://doi.org/10.1016/j.lwt.2020.110621.

Macías-Garbett R., Sosa-Hernández J.E., Iqbal H.M.N., Contreras-Esquivel JC, Chen WN, Melchor-Martínez EM, Parra-Saldívar R. Combined Pulsed Electric Field and Microwave-Assisted Extraction as a Green Method for the Recovery of Antioxidant Compounds with Electroactive Potential from Coffee Agro-Waste. Published online 2022. doi:10.3390/plants.

Martina, K., Tagliapietra, S., Barge, A., Cravotto, G., 2016. Combined microwaves/ultrasound, a hybrid technology. Top. Curr. Chem. 374 (6) https://doi.org/10.1007/s41061-016-0082-7.

Martín-García, B., Tylewicz, U., Verardo, V., Pasini, F., Gómez-Caravaca, A.M., Caboni, M.F., Della Rosa, M., 2020. Pulsed electric field (PEF) as pretreatment to improve the phenolic compounds recovery from brewers' spent grains. Innov. Food Sci. Emerg. Technol. 64 https://doi.org/10.1016/j.ifset.2020.102402.

Mesquita, P.C., Rodrigues, L.G.G., Mazzutti, S., da Silva, M., Vitali, L., Lanza, M., 2021. Intensified green-based extraction process as a circular economy approach to recover bioactive compounds from soursop seeds (Annona muricata L.). Food Chem. X 12. https://doi.org/10.1016/j.fochx.2021.100164.

Mohamad, N., Ramli, N., Abd-Aziz, S., Ibrahim, M.F., 2019. Comparison of hydro-distillation, hydro-distillation with enzyme-assisted and supercritical fluid for the extraction of essential oil from pineapple peels. 3 Biotech 9 (6). https://doi.org/10.1007/s13205-019-1767-8.

Mohseni, N.M., Mirzaei, H., Moghimi, M., 2020. Optimized extraction and quality evaluation of Niger seed oil via microwave-pulsed electric field pretreatments. Food Sci. Nutr. 8 (3), 1383–1393. https://doi.org/10.1002/fsn3.1396.

More, P.R., Jambrak, A.R., Arya, S.S., 2022. Green, environment-friendly and sustainable techniques for extraction of food bioactive compounds and waste valorization. Trends Food Sci. Technol. 128, 296–315. https://doi.org/10.1016/j.tifs.2022.08.016.

Muñoz-Almagro, N., Morales-Soriano, E., Villamiel, M., Condezo-Hoyos, L., 2021. Hybrid high-intensity ultrasound and microwave treatment: a review on its effect on quality and bioactivity of foods. Ultrason Sonochem. 80 https://doi.org/10.1016/j.ultsonch.2021.105835.

Mushtaq, M., Sultana, B., Akram, S., Anwar, F., Adnan, A., Rizvi, S.S.H., 2017. Enzyme-assisted supercritical fluid extraction: an alternative and green technology for non-extractable polyphenols. Anal. Bioanal. Chem. 409 (14), 3645–3655. https://doi.org/10.1007/s00216-017-0309-7.

Nadar, S.S., Rathod, V.K., 2017. Ultrasound assisted intensification of enzyme activity and its properties: a mini-review. World J. Microbiol Biotechnol. 33 (9) https://doi.org/10.1007/s11274-017-2322-6.

Nadar, S.S., Rao, P., Rathod, V.K., 2018. Enzyme assisted extraction of biomolecules as an approach to novel extraction technology: a review. Food Res. Int. 108, 309–330. https://doi.org/10.1016/j.foodres.2018.03.006.

Naliyadhara, N., Kumar, A., Girisa, S., Daimary, U.D., Hegde, M., Kunnumakkara, A.B., 2022. Pulsed electric field (PEF): Avant-garde extraction escalation technology in food industry. Trends Food Sci. Technol. 122, 238–255. https://doi.org/10.1016/j.tifs.2022.02.019.

Ojha, K.S., Aznar, R., O'Donnell, C., Tiwari, B.K., 2020. Ultrasound technology for the extraction of biologically active molecules from plant, animal and marine sources. TrAC - Trends Anal. Chem. 122 https://doi.org/10.1016/j.trac.2019.115663.

Ordoñez-Torres, A., Torres-León, C., Hernández-Almanza, A., Flores-Guía, T., Luque-Contreras, D., Aguilar, C.N., Ascacio-Valdés, J., 2021. Ultrasound-microwave-assisted extraction of polyphenolic compounds from Mexican "Ataulfo" mango peels: antioxidant potential and identification by HPLC/ESI/MS. Phytochem. Anal. 32 (4), 495–502. https://doi.org/10.1002/pca.2997.

Panić, M., Gunjević, V., Cravotto, G., Radojčić Redovniković, I., 2019. Enabling technologies for the extraction of grape-pomace anthocyanins using natural deep eutectic solvents in up-to-half-litre batches extraction of grape-pomace anthocyanins using NADES. Food Chem. 300 https://doi.org/10.1016/j.foodchem.2019.125185.

Pataro, G., Carullo, D., Ferrari, G., 2019. PEF-assisted supercritical CO_2 extraction of pigments from microalgae nannochloropsis oceanica in a continuous flow system. Chem. Eng. Trans. 74, 97–102. https://doi.org/10.3303/CET1974017.

Pereira, R.N., Coelho, M.I., Genisheva, Z., Fernandes, J.M., Vicente, A.A., Pintado, M.E., Teixeira, J.A., 2021. Using Ohmic heating effect on grape skins as a pretreatment for anthocyanins extraction. Food Bioprod. Process 124, 320–328. https://doi.org/10.1016/j.fbp.2020.09.009.

Pereira, Sara G., Pereira, Ricardo N., Rocha, Cristina M.R., Teixeira, José A., 2023. Electric fields as a promising technology for the recovery of valuable bio compounds from algae: novel and sustainable approaches. Bioresour Technol. Rep. 22, 101420 https://doi.org/10.1016/j.biteb.2023.101420.

Perez-Pirotto, C., Cozzano, S., Hernando, I., Arcia, P., 2022. Different green extraction technologies for soluble dietary fibre extraction from orange by-product (Published online). Int. J. Food Sci. Technol.. https://doi.org/10.1111/ijfs.15756.

Poojary, M.M., Barba, F.J., Aliakbarian, B., Donsì, F., Pataro, G., Dias, D.A., Juliano, P., 2016. Innovative alternative technologies to extract carotenoids from microalgae and seaweeds. Mar. Drugs 14 (11). https://doi.org/10.3390/md14110214.

Prabha, K., Ghosh, P., S.A., Joseph, R.M., Krishnan, R., Rana, S.S., Pradhan, R.C., 2021. Recent development, challenges, and prospects of extrusion technology. Future Foods 3. https://doi.org/10.1016/j.fufo.2021.100019.

Riera, E., Golás, Y., Blanco, A., Gallego, J.A., Blasco, M., Mulet, A., 2004. Mass transfer enhancement in supercritical fluids extraction by means of power ultrasound. In: Ultrasonics Sonochemistry, vol 11. Elsevier,, pp. 241–244. https://doi.org/10.1016/j.ultsonch.2004.01.019.

Riera, E., Blanco, A., García, J., Benedito, J., Mulet, A., Gallego-Juárez, J.A., Blasco, M., 2010. High-power ultrasonic system for the enhancement of mass transfer in supercritical CO_2 extraction processes. Ultrasonics 50 (2), 306–309. https://doi.org/10.1016/j.ultras.2009.09.015.

Sahena, F., Zaidul, I.S.M., Jinap, S., Karim, A.A., Abbas, K.A., Norulaini, N.A.N., Omar, A.K.M., 2009. Application of supercritical CO_2 in lipid extraction - a review. J. Food Eng. 95 (2), 240–253. https://doi.org/10.1016/j.jfoodeng.2009.06.026.

Salgado-Ramos, M., Martí -Quijal, F.J., Huertas-Alonso, A.J., Prado, M., Verdú, S., Cravotto, G., Moreno, A., Barba, F.J., 2022. Multistep process extraction of almond hull biomass with pulsed electric fields and supercritical CO_2 to recover carbohydrates and biologically active compounds. Biol Life Sci. Forum 2022. https://doi.org/10.3390/xxxxx.

Samaranayake, C.P., Sastry, S.K., 2005. Electrode and pH effects on electrochemical reactions during ohmic heating. J. Electroanal. Chem. 577, 125–135. https://doi.org/10.1016/j.jelechem.2004.11.026.

Sánchez, M., Ferreira-Santos, P., Gomes-Dias J, S., Botelho, C., Laca, A., M.R., Rocha, C., 2023. Ohmic heating-based extraction of biocompounds from cocoa bean shell. Food Biosci. 54, 102886 https://doi.org/10.1016/j.fbio.2023.102886.

Seçmeler, Ö., Güçlü Üstündağ, Ö., Fernández-Bolaños, J., Rodríguez-Gutiérrez, G., 2018. Effect of subcritical water and steam explosion pretreatments on the recovery of sterols, phenols and oil from olive pomace. Food Chem. 265, 298–307. https://doi.org/10.1016/j.foodchem.2018.05.088.

Sharifi, A., Hamidi-Esfahani, Z., Ahmadi Gavlighi, H., Saberian, H., 2022. Assisted ohmic heating extraction of pectin from pomegranate peel. Chem. Eng. Process 172, 108760. https://doi.org/10.1016/j.cep.2021.108760.

Sillero, L., Prado, R., Labidi, J., 2018. Optimization of different extraction methods to obtaining bioactive compounds from Larix decidua bark. Chem. Eng. Trans. 70, 1369–1374. https://doi.org/10.3303/CET1870229.

Sillero, L., Prado, R., Labidi, J., 2020. Simultaneous microwave-ultrasound assisted extraction of bioactive compounds from bark. Chem. Eng. Process. - Process. Intensif. 156 https://doi.org/10.1016/j.cep.2020.108100.

Soares, J.F., Prá, V.D., Barrales, F.M., Lunelli, F.C., Abaide, E., Silva, R.F., Kuhn, R.C., Martínez, J., Mazutti, M.A., 2018. Extraction of rice bran oil using supercritical CO_2 combined with ultrasound. Braz. J. Chem. Eng. 35 (2), 785–794. https://doi.org/10.1590/0104-6632.20180352s20160447.

Soquetta, M.B., Terra, L. de M., Bastos, C.P., 2018. Green technologies for the extraction of bioactive compounds in fruits and vegetables. CYTA - J. Food 16 (1), 400–412. https://doi.org/10.1080/19476337.2017.1411978.

Sun, H., Li, C., Ni, Y., Yao, L., Jiang, H., Ren, X., Fu, J., Zhao, C., 2019. Ultrasonic/microwave-assisted extraction of polysaccharides from Camptotheca acuminata fruits and its antitumor activity. Carbohydr. Polym. 206, 557–564. https://doi.org/10.1016/j.carbpol.2018.11.010.

Taha, A., Casanova, F., Šimonis, P., Stankevič, V., Gomaa, M.A.E., Stirkė, A., 2022. Pulsed electric field: fundamentals and effects on the structural and techno-functional properties of dairy and plant proteins. Foods 11 (11). https://doi.org/10.3390/foods11111556.

Tamborrino, A., Romaniello, R., Caponio, F., Squeo, G., Leone, A., 2019. Combined industrial olive oil extraction plant using ultrasounds, microwave, and heat exchange: impact on olive oil quality and yield. J. Food Eng. 245, 124–130. https://doi.org/10.1016/j.jfoodeng.2018.10.019.

Teresa-Martínez, G.D., Cardador-Martínez, A., Téllez-Pérez, C., Allaf, K., Jiménez-Martínez, C., Alonzo-Macías, M., 2022. Effect of the instant controlled pressure drop technology in Cardamom (Elettaria cardamomum) essential oil extraction and antioxidant activity. Molecules 27 (11). https://doi.org/10.3390/molecules27113433.

Tian, D., Shen, F., Yang, G., Deng, S., Long, L., He, J., Zhang, J., Huang, C., Luo, L., 2019. Liquid hot water extraction followed by mechanical extrusion as a chemical-free pretreatment approach for cellulosic ethanol production from rigid hardwood. Fuel 252, 589–597. https://doi.org/10.1016/j.fuel.2019.04.155.

Tribot, A., Amer, G., Abdou Alio, M., Baynast, H., Delattre, C., Pons, A., Mathias, J.D., Callois, J.M., Vial, C., Michaud, P., Dussap, C.G., 2019. Wood-lignin: supply, extraction processes and use as bio-based material. Eur. Polym. J. 112, 228–240. https://doi.org/10.1016/j.eurpolymj.2019.01.007.

Tzima, K., Brunton, N.P., Lyng, J.G., Frontuto, D., Rai, D.K., 2021. The effect of pulsed electric field as a pretreatment step in ultrasound assisted extraction of phenolic compounds from fresh rosemary and thyme by-products. Innov. Food Sci. Emerg. Technol. 69 https://doi.org/10.1016/j.ifset.2021.102644.

Uquiche, E., Millao, S., del Valle, J.M., 2022. Extrusion affects supercritical CO_2 extraction of red pepper (Capsicum annuum L.) oleoresin. J. Food Eng. 316 https://doi.org/10.1016/j.jfoodeng.2021.110829.

Verdini, F., Gaudino, E.C., Grillo, G., Tabasso, S., Cravotto, G., 2021. Cellulose recovery from agri-food residues by effective cavitational treatments. Appl. Sci. (Switz.) 11 (10). https://doi.org/10.3390/app11104693.

Vidovic, S., Vladić, J., Nastic, N., Jokić, S., 2020. Subcritical and supercritical extraction in food by-product and food waste valorization. Innovative Food Processing

Technologies: A Comprehensive Review. Elsevier,, pp. 705–721. https://doi.org/10.1016/b978-0-08-100596-5.23014-x.
Vorobiev, E., Lebovka, N.I., 2006. Extraction of intercellular components by pulsed electric fields. In: Raso, J., Heinz, V. (Eds.), Pulsed Electric Fields Technology for the Food Industry. Food Engineering Series. Springer, Boston, MA. https://doi.org/10.1007/978-0-387-31122-7_6.
Web of Science, 2023b https://www.webofscience.com/wos/woscc/summary/2c679c25–7f5c-4ee6-aaf2-ab10aa147d34–8786b1a7/relevance/1. (Accessed on 5 May 2023).
Web of Science, 2023a, https://www.webofscience.com/wos/woscc/summary/1ec0358f-96df-48db-bb4b-94a8432f3d7a-87869376/relevance/1 (Accessed on 5 May 2023).
Wei, C., Xiao, K., Li, H., Qi, Y., Zou, Z., Liu, Z., 2022. Optimization of ultrasound assisted aqueous enzymatic extraction of oil from Cinnamomum camphora seeds. LWT 164, 113689. https://doi.org/10.1016/j.lwt.2022.113689.
Wei, L., Yang, H., Li, H., Zhu, M., Mi, S., Lu, Q., Liu, M., Zu, Y., 2022. Comparison of chemical composition and activities of essential oils from fresh leaves of Pelargonium graveolens L'Herit. extracted by hydrodistillation and enzymatic pretreatment combined with a solvent-free microwave extraction method. Ind. Crops Prod. 186 https://doi.org/10.1016/j.indcrop.2022.115204.
Wen, L., Zhang, Z., Sun, D.W., Sivagnanam, S.P., Tiwari, B.K., 2020. Combination of emerging technologies for the extraction of bioactive compounds. Crit. Rev. Food Sci. Nutr. 60 (11), 1826–1841. https://doi.org/10.1080/10408398.2019.1602823.
Xu, L., He, W., Lu, M., Yuan, B., Zeng, M., Tao, G., Qin, F., Chen, J., Guan, Y., He, Z., 2018a. Enzyme-assisted ultrasonic-microwave synergistic extraction and UPLC-QTOF-MS analysis of flavonoids from Chinese water chestnut peels. Ind. Crops Prod. 117, 179–186. https://doi.org/10.1016/j.indcrop.2018.03.012.
Xu, L., He, W., Lu, M., Yuan, B., Zeng, M., Tao, G., Qin, F., Chen, J., Guan, Y., He, Z., 2018b. Ultrasonic-microwave assisted extraction, characterization and biological activity of pectin from jackfruit peel. LWT 90, 577–582. https://doi.org/10.1016/j.lwt.2018.01.007.
Yan, Y., Liu, F., Gao, J., Wan, J., Ding, J., Li, T., 2022. Enhancing enzyme activity via low-intensity ultrasound for protein extraction from excess sludge. Chemosphere 303. https://doi.org/10.1016/j.chemosphere.2022.134936.
Yang, J.S., Mu, T.H., Ma, M.M., 2019. Optimization of ultrasound-microwave assisted acid extraction of pectin from potato pulp by response surface methodology and its characterization. Food Chem. 289, 351–359. https://doi.org/10.1016/j.foodchem.2019.03.027.
Yang, L., Guo, Z., Wei, J., Han, L., Yu, Q.L., Chen, H., Chen, Y., Zhang, W., 2021. Extraction of low molecular weight peptides from bovine bone using ultrasound-assisted double enzyme hydrolysis: Impact on the antioxidant activities of the extracted peptides. LWT 146. https://doi.org/10.1016/j.lwt.2021.111470.
Yang, Y.C., Wei, M.C., 2016. A combined procedure of ultrasound-assisted and supercritical carbon dioxide for extraction and quantitation oleanolic and ursolic acids from Hedyotis corymbosa. Ind. Crops Prod. 79, 7–17. https://doi.org/10.1016/j.indcrop.2015.10.038.
Yang, Y.C., Wei, M.C., 2018. Development and characterization of a green procedure for apigenin extraction from Scutellaria barbata D. Don. Food Chem. 252, 381–389. https://doi.org/10.1016/j.foodchem.2017.12.086.
Yang, Y.C., Wei, M.C., Hong, S.J., Huang, T.C., Lee, S.Z., 2013. Development/optimization of a green procedure with ultrasound-assisted improved supercritical carbon dioxide to produce extracts enriched in oleanolic acid and ursolic acid from Scutellaria barbata D. Don. Ind. Crops Prod. 49, 542–553. https://doi.org/10.1016/j.indcrop.2013.05.013.
Yun, C., Wang, S., Gao, Y., Zhao, Z., Miao, N., Shi, Y., Ri, I., Wang, W., Wang, H., 2022. Optimization of ultrasound-assisted enzymatic pretreatment for enhanced extraction of baicalein and wogonin from Scutellaria baicalensis roots. J. Chromatogr. B Anal. Technol. Biomed. Life Sci. 1188 https://doi.org/10.1016/j.jchromb.2021.123077.
Zhan, J., Liang, Z., Li, J., Zeng, X., Ou, G., Zhong, C., 2022. Pulsed electric field-ultrasonic assisted extraction combined with macroporous resin for the preparation of flavonoids from Pericarpium Citri Reticulatae (Published online). J. Food Process Preserv.. https://doi.org/10.1111/jfpp.16823.
Zhang, J., Ström, A., Bordes, R., Alminger, M., Undeland, I., Abdollahi, M., 2019. Radial discharge high shear homogenization and ultrasonication assisted pH-shift processing of herring co-products with antioxidant-rich materials for maximum protein yield and functionality. Published online August 2022: Food Chem., 133986 https://doi.org/10.1016/j.foodchem.2022.133986.
Zhang, J., Wen, C., Zhang, H., Duan, Y., Ma, H., 2020a. Recent advances in the extraction of bioactive compounds with subcritical water: a review. Trends Food Sci. Technol. 95, 183–195. https://doi.org/10.1016/j.tifs.2019.11.018.
Zhang, R.Y., Liu, H.M., Hou, J., Yao, Y.G., Ma, Y.X., Wang, X.De, 2021. Cellulose fibers extracted from sesame hull using subcritical water as a pretreatment. Arab. J. Chem. 14 (6) https://doi.org/10.1016/j.arabjc.2021.103178.
Zhang, X., Zhu, H., Wang, J., Li, F., Wang, J., Ma, X., Li, J., Huang, Y., Liu, Z., Zhang, L., Li, S., 2022. Anti-microbial activity of citronella (Cymbopogon citratus) essential oil separation by ultrasound assisted ohmic heating hydrodistillation. Ind. Crops Prod. 176, 114299 https://doi.org/10.1016/j.indcrop.2021.114299.
Zhang, Y., Li, T., Shen, Y., Wang, L., Zhang, H., Qian, H., Qi, X., 2020. Extrusion followed by ultrasound as a chemical-free pretreatment method to enhance enzymatic hydrolysis of rice hull for fermentable sugars production. Ind. Crops Prod. 149 https://doi.org/10.1016/j.indcrop.2020.112356.
Zhou, J., Wang, M., Berrada, H., Zhu, Z., Grimi, N., Barba, F.J., 2022a. Pulsed electric fields (PEF), pressurized liquid extraction (PLE) and combined PEF + PLE process evaluation: Effects on Spirulina microstructure, biomolecules recovery and Triple TOF-LC-MS-MS polyphenol composition. Innov. Food Sci. Emerg. Technol. 77 https://doi.org/10.1016/j.ifset.2022.102989.
Zhou, M., Bi, J., Li, C., Chen, J., 2022b. Enhancive effect of instant controlled pressure drop (DIC) pretreatment on pectin extractability from peach pomace. Food Hydrocoll. 130 https://doi.org/10.1016/j.foodhyd.2022.107696.
Zhu, Z., Bals, O., Grimi, N., Ding, L., Vorobiev, E., 2015. Better damage of chicory tissue by combined electroporation and ohmic heating for solute extraction. Food Bioprod. Process 94, 248–254. https://doi.org/10.1016/j.fbp.2014.03.003.