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Review

Plant Extraction in Water: Towards Highly Efficient Industrial Applications

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Abstract: Since the beginning of this century, the world has experienced a growing need for enabling techniques and more environmentally friendly protocols that can facilitate more rational industrial production. Scientists are faced with the major challenges of global warming and safeguarding water and food quality. Organic solvents are still widely used and seem to be hard to replace, despite their enormous environmental and toxicological impact. The development of water-based strategies for the extraction of primary and secondary metabolites from plants on a laboratory scale is well documented, with several intensified processes being able to maximize the extraction power of water. Technologies, such as ultrasound, hydrodynamic cavitation, microwaves and pressurized reactors that achieve subcritical water conditions can dramatically increase extraction rates and yields. In addition, significant synergistic effects have been observed when using combined techniques. Due to the limited penetration depth of microwaves and ultrasonic waves, scaling up entails changes to reactor design. Nevertheless, the rich academic literature from laboratory-scale investigations may contribute to the engineering work involved in maximizing mass/energy transfer. In this article, we provide an overview of current and innovative techniques for solid-liquid extraction in water for industrial applications, where continuous and semi-continuous processes can meet the high demands for productivity, profitability and quality.

Keywords: extraction in water; enabling technologies; ultrasound; microwaves; hydrodynamic cavitation; subcritical water

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1. Introduction

Conventional hydroalcoholic extraction systems based on maceration and percolation are usually time- and energy-consuming. In addition, the rising price of ethanol has led to an increase in production costs. Other organic solvents are commonly used in the chemical and pharmaceutical industries for extraction processes but do not meet the requirements for the environmental and economic sustainability of the ecological transition. The need to improve process safety and product quality and the growing interest in preserving the environment have led R&D groups to explore more environmentally friendly methods [1]. While the literature is replete with excellent results achieved with new technologies on a laboratory scale [2,3], industrial implementation remains a complicated task [4]. The possibilities of saving energy (e.g., reducing extraction time) and avoiding toxic and flammable solvents have led researchers to develop new technological solutions. In addition, cold extraction using techniques such as cavitation reactors and pulsed electric fields (PEF) allows heat-sensitive compounds to be preserved from degradation. Our consolidated experience in the application of extraction technologies, such as microwaves (MW), ultrasound (US), hydrodynamic cavitation (HC), pressurized reactors for subcritical water extractions (SWE), PEF and enzymatic treatments, have paved the way for the expansion of studies and the development of new pilot and semi-industrial reactors.

2. Plant Material Pretreatment

Due to the influence of particle size, suitable milling and controlled size screening processes are the first steps toward efficient extraction [5]. The parts of a plant differ in hardness, fiber structure and chemical composition. The particle fractions isolated by sieving and cyclone treatment have different physicochemical properties in addition to their size [6]. In the extraction of vegetal matrices, a partially-damaged cell wall significantly improves solvent accessibility for both intracellular solutes and cell-wall constituents [7]. Hydrolytic enzymes, often used as a mixture, can selectively depolymerize and degrade certain cell wall components [8,9]. Enzymatic pretreatment induces hydrolysis of cell-wall biopolymers (pectin, cellulose and hemicellulose), resulting in structural disruption of the cell wall that facilitates mass transfer and matrix dissolution. This means that several enzymes (cellulases, hemicellulose, pectinases, etc.) are currently used in industrial extractions. In water-based extraction processes, the incubation with the enzymes can take place simultaneously with the mixing phase. The mild operating conditions favor the recovery of thermolabile products, and further processing of the solid fraction can be proposed as part of a biorefinery concept for the integral use of biomass. The enzymatic pre-treatment leads to a reduction in solvent consumption and extraction time. The effectiveness of enzymatic pretreatment and enzyme-assisted extraction has been extensively documented in the literature [10,11]. Pretreatment under intense cavitation using US (Figure 1) and hydrodynamic rotor/stator units or with high cutting power in high-shear homogenizers strongly promotes rehydration and solvation of the plants [12], (Figure 2).

Figure 1. Semi-industrial US flow reactor (Weber Ultrasonics AG) for biomass pretreatment.

This effect has been exploited in biomass conversion, where the delignification process is promoted by cavitation pretreatment, leading to higher conversion rates in the subsequent fermentation and extraction processes [13,14]. Cavitation pretreatments also affect the matrix surface and increase the surface area of the material [15]. PEF can be used both as pretreatment and directly for extraction as it can cause electroporation, which opens pores in cell membranes, and cell rupture, both of which mechanisms greatly enhance the recovery of intracellular compounds [16,17]. PEF extraction and pretreatment processes are mainly studied on a laboratory scale [18,19]. Their application on the pilot and semi-industrial scales is documented in Figure 3, with a sequential process in which PEF follows US treatment.

Figure 2. Rotor/stator HC reactor (EPIC Srl) at DSTF—University of Turin.

Figure 3. Continuous-flow extraction under combined US (C2FUT Srl) and PEF (Energy Pulse Systems), DSTF—University of Turin.

3. Ultrasound-Assisted Extraction

Cavitation is the formation, growth and decay of gaseous bubbles in a liquid. This process can be triggered by acoustic or mechanical waves that create a cycle of compression and expansion. When the negative pressure created in the expansion phase is sufficient to overcome the intramolecular forces of the liquid, a cavity, or bubble, forms. Through the cycles of expansion and compression, a bubble can reach its critical size and then collapse, releasing a large amount of energy and creating a microenvironment that reaches up to 5000 K and 1000 bar [20,21].

The parameters affecting the cavitation process and its mechanisms of action have been extensively discussed over the years. Cavitation affects the matrix and improves extraction yield and recovery of metabolites. The mechanisms of action are erosion, fragmentation, sonoporation and the so-called ultrasonic capillary effect. The erosion and fragmentation of the matrix are phenomena resulting from the mechanical effect of cavitation, as the jet streams and shear forces generated by the bubble collapse have a destructive effect on the matrix. These effects greatly increase the contact area with the solvent, which enables better mass transfer. Also worth mentioning is the effect on oil glands and similar structures, which can be ruptured so that their contents pass directly into the extraction medium. As shown in the literature, these changes can be detected by SEM analysis [22,23]. Reduction in particle size is another effect that cavitation has on a matrix, and this effect can be observed both for larger fragments visible to the eye and for smaller, micrometric

fragments. Again, the main benefit of reducing particle size is to increase the surface area of the matrix and its exposure to the extraction solvent [24,25]. Sonoporation is the formation of pores in cell membranes and can be reversible or irreversible [26–28]. This process has been studied using various methods, such as fluorescence imaging, the voltage clamp technique and optical observations of cells exposed to sonoporation [29–31]. The pores formed by sonoporation allow solutes to penetrate the membrane and enhance mass transfer by removing a physical barrier. This phenomenon also has applications in various other fields, such as gene transfer and drug delivery [32–36]. The mechanism behind the ultrasonic-capillary effect is still unclear. It consists in increasing the penetration of fluids into the channels and pores of a matrix, both in-depth and at high speed [37]. This mechanism promotes the swelling of the matrix and improves mass transfer through enhanced diffusion [38]. The choice of solvent is influenced by its physical properties, e.g., viscosity and vapor pressure, and its suitability for the particular extraction process based on the target molecules and their solubility in the chosen medium. To trigger cavitation, the intramolecular forces in the liquid must be overcome by the negative pressure generated during the expansion phase [39]. Water is the preferred solvent for cavitation because it generates a high acoustic pressure, which leads to a stronger collapse of the bubbles [40]. Vapour pressure affects the cavitation process by changing the composition of the bubbles, where an increase in vapor pressure means that more solvent vapor enters the bubble cavities, which are more stable and collapse less violently [37]. The vapor pressure in a given solvent is mainly influenced by temperature, and an increase in temperature is reflected in an increase in vapor pressure; when using water, optimal cavitation can be achieved in a temperature range between 20 and 35 °C [41,42]. Cavitation can be generated by ultrasonic (US) or hydrodynamic (HC) reactors. US reactors can be either bath or probe systems (horn and cup horn). Probe systems (also called horn or sonotrode systems) usually have higher power because they emit US intensity only over the small surface area of their tips, while bath systems emit less powerful and less uniform acoustic waves because of their design (since their transducers are embedded in the bath walls) [23,43]. HC devices produce cavitation either by forcing a fluid through a constricted channel (orifices or Venturi tubes) or by exerting a force on the fluid using fast-moving parts, such as rotor-stator cavitators [44].

While acoustic cavitation has a localized cavitation effect due to the poor penetration of the pressure wave through solid-liquid mixtures, HC cavitation affects the entire mixture. Both technologies are already partly used on a pilot and industrial scale. The up-scaling of these two technologies is associated with different difficulties and technical considerations [44,45]. The main issues in HC scaling-up are related to the distance between the rotor and stator, their surface shapes, dimension and optimal rotation speed aiming to limit maintenance stops. US scaling-up efforts are focused on the uniform distribution and intensity of cavitation bubbles. The main strategy is moving from batch to flow and deploying more transducers or wide areas of cavitation to ensure optimal mass transfer and uniform bubble generation [46] (Figure 4).

Several studies have been published on laboratory-scale extractions with acoustic and hydrodynamic cavitations [37,38,48,49]. However, there are currently few papers reporting on the application of these technologies on a pilot or semi-industrial scale. Only a few studies address the up-scaling of these techniques, which are listed in Table 1 below.

As can be seen from Table 1, there are few published studies on cavitation-assisted extraction on a pilot or semi-industrial scale. Moreover, few of the published studies have thoroughly analyzed the effects of various parameters on the extraction process. Further research on cavitation processes is therefore essential if this promising technology is to be effectively applied in real industrial processes.

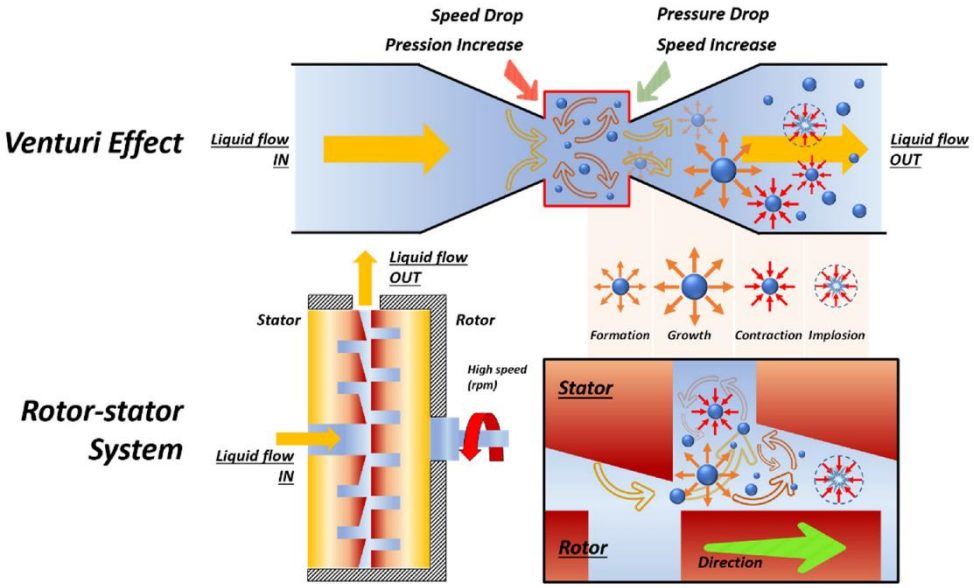

Figure 4. Hydrodynamic cavitation phenomenon in a fluid: Venturi effect and rotor-stator system [47].

Table 1. Pilot and semi-industrial scale US extraction studies, from 2017 to 2022.

Matrix	Technology	Scale	Other Parameters	Results	Ref.
Grape stalks	US; recirculating flow	2 kg/60 L	Water; 1 h; S/L 1:30; 30 L/min; RT; 29 kHz; 2 kW	Efficient process scaling-up in terms of extraction yield and antioxidant power of the extract	[50]
Olives	US; continuous flow	2 tons/h	RT; 20 kHz; 2.8 kW	22.7% increase in oil yield and 10.1% increase in oil phenolic compounds	[51]
Spirulina	US; recirculating flow	1.5 kg/30 L	Water (Phosphate buffer); 40 min; S/L 1:20; 20 kHz	127% increase in protein yield compared to the conventional method	[26]
Undaria pinnatifida	US; recirculating flow	200 g/20 L	Water; 3 h; S/L 1:100; 30 °C; 20 kHz; 960 W	111% increase in extraction yield compared to the conventional method	[52]
Sesame oil cake	US; recirculating flow	33 kg/1000 L	Water; 4 h; S/L 1:30; 25 °C; 20 kHz; 900 W	193% increase in extraction yield compared to conventional method	[53]
Soybean	HC; high-pressure homogenizer	9 L/h	Water; S/L 1:7; 100 MPa	Protein yield increase of 82%; a single step was optimal	[54]
Orange peel	HC (reactor with Venturi cross-section); recirculating flow	42 kg/120 L (1° test) 6.38 kg/147 L (2° test)	Water; S/L 1:2.85; 330 L/min; 0.62 kWh/kg matrix (1° test) Water; S/L 1:23.04; 330 L/min; 2.20 kWh/kg matrix (2° test)	No optimal extraction parameters are given. Efficient and rapid extraction of flavones and monoterpenes; isolation of high-quality pectin.	[55]
Silver fir needles	HC (reactor with Venturi cross-section); recirculating flow	0.529 kg/120 L	Water; S/L 1:227; 330 L/min; 4.8 kWh	No optimal extraction parameters are given. The ORAC/ TPC ratio increased as a function of cavitation time, reaching a maximum at 60 min of extraction.	[56]

The studies listed in Table 1 show how US can be effectively applied in flow processes such as olive oil extraction, resulting in higher recovery of oil richer in phenolic compounds [51]. Grillo et al. [50] developed a method to intensify the water extraction of grape stems using a US system with recirculating flow (Figure 5). The extract was subjected to nanofiltration after centrifugation to obtain a concentrated final product with strong antioxidant activity.

Similar systems were used for the extraction of several other matrices, such as Spirulina [26], *Undaria pinnatifida* [52], and sesame oil cake [53]. Extraction of Spirulina with US resulted in increased protein yield. In addition, microscopic observations showed the effects of acoustic cavitation on Spirulina filaments caused by fragmentation, sonoporation and deconstruction mechanisms. This effect facilitates the extraction, release and solubilization of bioactive Spirulina compounds [26].

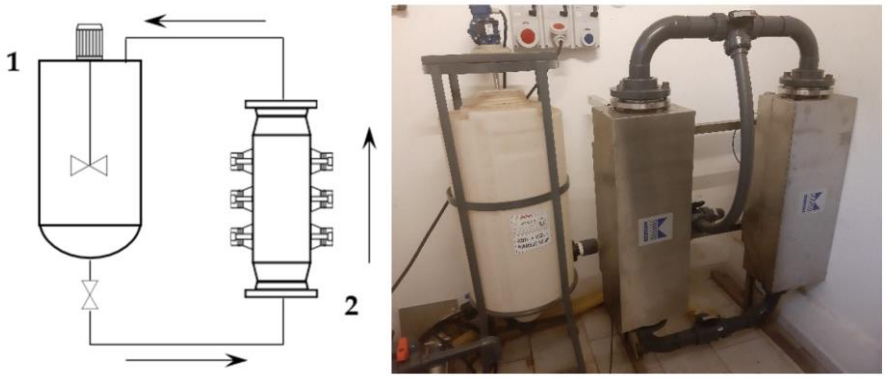

Figure 5. Recirculating flow system used in Grillo et al. [50]. Left: set-up scheme (1: mixed tank; 2: US flow-through cell); and right: facility picture.

Extraction with US of *Undaria pinnatifida* and sesame oil cake showed an increase in extraction yield of 111% and 193% compared to the corresponding conventional methods. Moreover, high-intensity US can produce a fine emulsion of lipids in water, as reported in the case of solvent-free extraction of fatty alcohols from rice bran [57].

Encouraging results have been obtained by using pilot-scale HC cavitation systems with recirculating flow, for example for orange peels [55] and silver fir needles [56].

The truly new paradigm in extraction is the shift from batch to continuous flow processes. Suspensions of plant material in water or aqueous-alcoholic mixtures can be rapidly processed in high-intensity multi-transducer ultrasonic units in flow-through treatments and/or rotor/stator hydrodynamic cavitation reactors. These processes are at the forefront of extraction systems due to a significant increase in extraction efficiency and the potential application on an industrial scale.

4. Microwave-Assisted Extraction

Microwaves (MWs) are electromagnetic waves ranging from 300 to 300,000 MHz. The energy associated with MWs is not enough to break chemical bonds (from 0.004 to 0.4 meV, while bond strengths are usually a few eV), classifying them as non-ionizing [58].

MWs are mainly used for selective and rapid heating and provide reduced energy consumption compared to conventional technologies [59–61]. Based on the interactions with MW, materials can be classified as reflective, transparent, or absorptive [62].

Reflective materials, such as metals, interact with the MWs by reflecting them. Transparent materials transmit MWs, which pass through them with little to no interaction: examples of transparent materials include but are not limited to quartz, PTFE and PFA. Absorptive materials interact with the MWs and absorb them partially or entirely, depending on the material characteristics; polar liquids are an example of such materials.

Two main processes determine the absorption of MWs in a liquid medium: ionic conduction and dipole rotation. The ionic conduction mechanism is based on the interaction between ions and the oscillating electric field generated by MWs, resulting in an ion flow. Localized heating is then produced by the resistance opposing ion movement inside the liquid. Dipole rotation is a phenomenon generated by the oscillating electric field and affects dipolar molecules. These molecules will align themselves with the electric field and then return to a disordered state billions of times per second (ca. 5 billion times per second for an instrument working at 2450 MHz).

While there is no lack of studies on microwave-assisted extraction (MAE) on a laboratory scale [63–67], process scalability is limited by a need for technological advancement. Nevertheless, MW reactors are used in various industrial processes [66–68], despite extraction-system scalability still being a major challenge; the problems in up-scaling are not only technical but are also related to the nature of the biomass to be processed. MWs have limited penetration depth, which limits the volume that can be processed in batch reactors [68]. Switching to continuous systems can overcome the disadvantages of batch

processes, but the design of MW flow reactors raises other issues that need to be addressed. Moreover, the addition of carbonaceous material (such as biochar) can directly improve MW energy transmission in batch reactors, while moving from single-mode to multimode systems can provide an indirect way to overcome this challenge [4,69].

An efficient process is microwave-assisted hydrodistillation (MAHD), which is used for the extraction of essential oils (EO). The principles of this process are similar to those of conventional hydrodistillation, but with improved heating and distillation efficiency. These aspects enable a reduction in energy consumption and operating time (Figure 6). In addition, microwave hydrodiffusion and gravity (MHG) has shown promise as an extraction technique for the simultaneous recovery of EO and other compounds such as pec-tins, pigments and polyphenols [68].

Figure 6. Ethos XL and Ethos X (Milestone Srl), DSTF—University of Turin.

As shown in Table 2, most of the few available results on MW-assisted extraction on a pilot scale are hydrodistillation processes.

Table 2. Studies on pilot scale MAE, from 2017 to 2022.

Matrix	Technology	Scale	Other Parameters	Results	Ref.
Lemon peel	MHG	20 kg	S/L 1:1.8; no fixed time	0.025% *w*/*w* EO	[70]
Cannabis	MAHD	2.6 kg	S/L 1:1; 1 h 50 min	0.35% *w*/*w* EO	[71]
Opuntia ficus-indica peel	MAHD; MHG	1–2 kg	S/L 1:0.067; 1 h; 1.5 kW (MAHD) S/L 1:0.121; 70 °C; 40 min; 1.2 kW (MHG)	About 128 mL extract/kg matrix (MAHD) About 342 mL extract/kg matrix (MHG)	[72]
Hops	MAHD	2–8 kg	S/L 2:1; 1 h 50 min	Increased extraction yield: 4 times higher (pellets), 2 times higher (dry matrix) than on a laboratory scale	[73]
Orange peel	MAE	3 kg	S/L 1:5; 1 h 30 min	64% increase in pectin yield	[74]

Table 2 shows that MAE requires further investigation on a pilot scale but has already shown promising results. Garcia-Garcia et al. [74] analyzed the life cycle assessment (LCA) of the pectin production process using MW and compared it to a conventional process. Their results showed a reduction in the environmental impact of about 75%, mainly due to the higher energy efficiency of the MW process. Research in this area requires further investigation for effective industrial implementation.

5. Subcritical Water Extraction

Water is the most environmentally friendly solvent as it is non-flammable, non-toxic and readily available. Although the dielectric properties of water mean that only polar products can be extracted from plant matrices, limiting its use, water can be used in a subcritical state to overcome these limitations. In the temperature range between 100 °C and its critical temperature (374 °C), the properties of water can be modified as long as it is kept in the liquid state by a suitable pressure. As the temperature rises, the dielectric constant of water decreases, reaching values similar to ethanol at 250 °C, allowing the extraction of less polar compounds. The viscosity also decreases, allowing better penetration into matrices, while the ionic product increases above 10^{-11}, favoring the depolymerization of complex structures.

By varying the temperature, these properties can be adjusted to optimize product yield while avoiding thermal degradation. Thermal degradation can also be avoided due to the shorter extraction times required under subcritical conditions compared to conventional extractions. Both temperature and water flow rate play an important role in subcritical water extraction (SWE) [75–78]. SWE of plants varies greatly depending on the technology and scale of use. Common laboratory-scale SWE uses high-pressure pumps for water supply and a heated cell loaded with biomass [2]. SWE can also be combined with MW heating to reduce operating times [66,79,80].

So far SWE scaling up is focusing on reactor dimensions and unit replication besides a higher water flow. Due to higher costs and technical complexity flow, SWE reactors still present technical issues that need to be addressed.

Only a few pilot-scale studies on SWE have been published, which are listed in Table 3.

Table 3. Studies on pilot scale SWE, from 2017 to 2022.

Matrix	Scale	Other Parameters	Yield %	Ref.
Miscanthus	150 kg/1200 L	160 °C; 2 h; S/L 1:8	33.05%	[81]
Zingiber zerumbet	0.25 kg/5 L	170 °C; 20 min; S/L 1:20	20.70%	[82]
Wheat bran	0.1 kg/1 L	160 °C; 60 min; S/L 1:10	15.82%	[83]
Chestnut peel	60 kg/180 L	150 °C; 30 min; S/L 1:3	39.42%	[84]

In the studies listed in Table 3, SWE was compared at laboratory and pilot scales, with promising results. Different reactor types were used for the extraction of bioactive compounds and all tests were performed in batch mode. Rudjito et al. [83] used a PEG-filled autoclave for the extraction of arabinoxylans from wheat bran pretreated by deamidation processes. Cravotto et al. [84] performed polyphenol extraction from chestnut hulls by moving from MAE-SWE on a laboratory scale to SWE on a pilot scale using a double vessel reactor with a total maximum capacity of 60 kg of plant material, and a maximum water flow rate of 1000 L/h (Figure 7). The extract was then rapidly concentrated by flash evaporation under a mild vacuum and steam condenser (Figure 8). Both dry extract and polyphenol yields were comparable to those obtained in the laboratory, demonstrating efficient up-scaling of the process.

The research group of Amir et al. [82] used a batch reactor for direct extraction of Zingiber zerumbet on a pilot scale and investigated the different parameters and their influence on the yield. Wang et al. [81] studied Miscanthus hot water extraction (at 160 °C) at three different scales: laboratory, intermediate and pilot scale. On a pilot scale, the authors extracted about 150 kg of biomass in a fermenter, with a yield loss of only 17% compared to laboratory-scale experiments.

The results described are promising and indicate the great potential and multiple applications of SWE on an industrial scale. However, much work is still needed to translate the numerous laboratory-scale studies into viable industrial applications.

Figure 7. Pressure-resistant reactors for SWE (C2FUT Srl.). **Left**: plant photo; **right**: reactor tank and loading vessel scheme, DSTF—University of Turin.

Figure 8. Flash evaporation unit (C2FUT Srl.) DSTF—University of Turin.

6. Pulsed Electric Field Extraction

Pulsed electric field (PEF) treatment consists of the application of an external electric field with very short pulses, favoring its permeabilization (Figure 9). Since the typical field intensity ranges from 10 to 80 kV cm^{-1} with pulse durations of micro- or milliseconds, PEF treatments have low energetic requirements that classify PEF as a green technology [85].

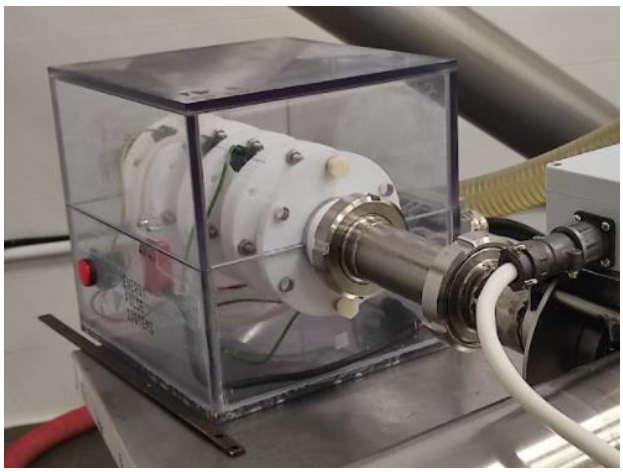

Figure 9. PEF unit by EnergyPulse Systems, Lisbon, Portugal.

Permeabilization of the cell occurs by polarization and reorientation of membrane components, inducing the formation of hydrophilic pores in the cellular membrane. Depending on the applied electric field intensity, the formation of pores can be reversible or irreversible, potentially leading to cell disruption [86].

Although PEF extraction treatments are widely explored at the lab scale, their use in scaled-up systems is limited, being more common in food processing. To the best of our knowledge, only two research papers on scaled-up PEF-assisted extraction of plant materials in water have been published between 2017 and 2022, and are reported in Table 4.

Table 4. Studies on pilot scale PEF, from 2017 to 2022.

Matrix	Technology	Scale	Other Parameters	Results	Ref.
Grapes	PEF, flow mode.	2500 kg/h	0.09 s residence time; 3.7 pulses of 4 kV cm^{-1}	Reduced maceration time, improved polyphenols concentration (up to 35% increase).	86
Grapes	PEF, flow mode.	200 L/h	0.09 s residence time; pulse duration of 8–16 µs	Increased varietal aroma precursors extraction, limited impact on wine color.	87

Andres Maza et al. [86] described the application of PEF to grape mass to reduce the maceration time in red wine making while improving polyphenols concentration under PEF treatment. Comuzzo et al. [87] also processed grapes for winemaking. Focusing on white wine production, authors could increase the number of varietal aroma precursors in wine, while avoiding undesired changes in the final product, such as a darker color.

Both studies validate a specific industrial application of PEF but the results also show the valuable effects on the extraction of natural products from plant materials.

7. Conclusions

This review highlighted the state of the art in the industrial scaling up of new technologies for water extraction of plant matrices. Despite the large gap between academia and industry, recent advances in the development of unconventional industrial reactors offer interesting new opportunities. Although extraction yields achieved in very small units for analytical purposes are hardly reproducible, the solid/liquid ratio can be dramatically improved on a larger scale, bringing remarkable benefits to the process downstream. The general trend of extractions in ultrasonic and hydrodynamic reactors is to work in continuous flow. Dielectric heating in microwave extraction and especially new reactors for subcritical water extraction will expand water extraction capabilities like never before. All of these technologies can greatly expand the use of water as a nearly universal solvent.

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