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# Recent advances in combined Avant-garde technologies (thermal-thermal, non-thermal-non-thermal, and thermal-non-thermal matrix) to extract polyphenols from agro byproducts

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## Abstract

Because food byproducts (waste) are rich in phytoconstituents, valorizing them is crucial for global food security. However, conventional extraction (CE), including decoction, maceration, Soxhlet, etc., for agro byproducts' polyphenol extraction are time-consuming and rely significantly on vast volumes of potentially aggressive solvents. Hence, Avant-garde extraction technologies, including non-thermal (high hydrostatic pressure (HHPE), pulsed-electric field (PEF), high voltage electrical discharges (HVED), etc.) and thermal extraction (supercritical fluid (SCF), subcritical water extraction (SWE), microwave-assisted extraction (MAE), etc.), as well as their thermal combinations (SCF-PLE, SCCO<sub>2</sub>-SWE, SCCO<sub>2</sub>-MAE, etc.), non-thermal combinations (HHPE + UAE, PEF + UAE, HVED + UAE, etc.) and combined thermal-non-thermal (MAE-UAE, etc.) are increasingly replacing CE. However, a review of combined Avant-garde extraction escalation technologies (non-thermal/thermal extraction matrix) for extracting polyphenols from agro-byproducts is limited. Hence, this manuscript reviewed Avant-garde extraction technologies (non-thermal/thermal extraction matrix) for extracting phenolics from agro-byproducts in the last 5 years. The key factors affecting polyphenols' extraction from the byproduct, the recent applications of Avant-garde technologies, and their principle were reviewed using databases from Web of Science and [Lens.org](https://www.lens.org). The results demonstrated that combined Avant-garde extraction escalation technologies increase extractability, resulting in polyphenols with higher extraction rates, fewer contaminants, and preservation of thermosensitive components. Therefore, combined Avant-garde extraction technologies should be explored over the next five years. Implementing an integrated process and the strategic sequencing of diverse Avant-garde extraction technologies are important. Thus, further investigation is required to explore the sequencing process and its potential impact on the extraction of phenolics from agro-byproducts.

**Keywords:** Combined extraction methods, Green extraction, Polyphenols, Waste valorization, Ultrasound extraction

## 1. Introduction

Food processing creates millions of tons of byproducts (peels, spent grains, cobs, brans, etc.) annually, leaving a substantial financial burden on food processors and causing adverse ecological impacts. Nevertheless, several studies have proven that these byproducts have high phytoconstituents,

indicating that extracting them from agro-byproducts is paramount [1]. One notable group of bioactive compounds is polyphenols, which have gotten prominence because of their antioxidant, allelopathic, antibacterial, and ultraviolet (UV)-protective properties. Phenolics range from 200 to 3500 kDa and are generally produced from the malonic or shikimic acid pathways, representing

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8–10 k molecules globally [2]. Since phenolics counteract abiotic and biotic stresses, chelate oxidant metals, scavenge reactive nitrogen and oxygen species, and replace antioxidants, they provide pleiotropic shields against degenerative illnesses and nitrooxidative damage [3]. Hence, polyphenols are becoming increasingly popular for food fortification as nutraceuticals and additives in various sectors (food processing, pharmaceuticals, cosmetics, etc.) [3,4].

Extracting polyphenols is a crucial phase before assessing the prospective utilization of a plant sample for therapeutic uses. Heating under reflux, Soxhlet, maceration, percolation, etc. [5] are conventional extraction (CE) methods to extract polyphenols from byproducts. However, CE have numerous drawbacks (polymerization of HO\*, recurrent conjugation, etc.) [1]. Consequently, Avant-garde extraction escalation technologies have been used to enhance extraction while retaining strict green chemistry and reaching extraction objectives quickly [4,6]. Avant-garde extraction escalation technologies (emerging extraction technologies), such as cold plasma (CP), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), pulsed electric fields (PEF), etc., have been underlined by scientists [4,7]. The extraction processes employed in this study are characterized by their advanced nature, as they utilize many stimuli that induce cell lysis via internal physicochemical instability or extracellular activity [8,9]. Therefore, the efficacy of their application is contingent upon their application technique and the prevailing physicochemical circumstances [1]. Likewise, it is also worth noting that merging Avant-garde extraction escalation technology approaches is becoming a hot research topic [3,10,11]. Nonetheless, the combined Avant-garde extraction technologies (non-thermal/thermal extraction matrix) for extracting polyphenols from agro-byproducts is limited.

Hence, this review delved into the principles and success stories of Avant-garde extraction methods and their combinative effects of these technologies for extracting polyphenols from byproducts (Fig. 1). The methodology of this review follows a comprehensive literature review approach to provide an overview of the Avant-garde extraction to maximize agro-byproducts' polyphenols (Fig. 2). The data for this study were obtained from online databases, including Web of Science and [lens.org](https://www.lens.org). The search terms used were “extraction of polyphenols from waste ([lens.org](https://www.lens.org)),” “extraction of polyphenols from waste using HHPE, PEF, PLE, UAE, HVED, SWE, SCFE, MAE, and their combinative effects

#### Abbreviations

CE	Conventional extraction
HHPE	High hydrostatic pressure
PEF	Pulsed-electric field
HVED	High voltage electrical discharges
SCF	Supercritical fluid
SWE	Subcritical water extraction
MAE	Microwave-assisted extraction
UV	Ultraviolet
CP	Cold plasma
UAE	Ultrasound-assisted extraction
TPC	Total phenolic content
GAE	Gallic acid equivalent
GRAS	Generally recognized as safe
TFC	Total flavonoid content
RSM	Response surface methodology
HPLE	Hot-pressurize liquid extraction
SA	Solvent aerosolization
SCFE	Supercritical fluid extraction
CBS	Cocoa bean hulls
Mw	Molecular weight
EF	Electric fields
CCC	Countercurrent chromatography
PLC	Preparative liquid chromatography
DPPH	2,2-Diphenyl-1-picrylhydrazyl
TAC	Total anthocyanin content

(SCFE-PLE, SCCO<sub>2</sub>-SWE, SCCO<sub>2</sub>-MAE, HHPE + UAE, PEF + UAE) (Web of science)” to capture all relevant studies on the topic (Fig. 2). The data were painstakingly examined, and relevant papers were filtered based on relevance and quality. The inclusion criteria for this review were research papers published in English until September 2023 that reported on the use of these technologies to extract polyphenols from agro-byproducts.

## 2. Key factors influencing the biorefinery approach for agro-byproduct phenolic compounds' extraction, and their mechanisms

### 2.1. Solvents' polarity

Determining the distribution coefficient and extraction of polyphenol in the agro-product relies on the interplay amongst the solubilization of the solvent and the phenolics' solubility. The ability of solvents to establish hydrogen bonds plays a crucial role in this context, specifically in facilitating solvation and liberating species bound inside a matrix. Consequently, an enhanced diffusivity of the extractant inside the matrix leads to a corresponding decrease in the stability of the hydrogen bond network present in the matrix's structure, resulting in a higher degree of solvation for the analyte of interest [12].

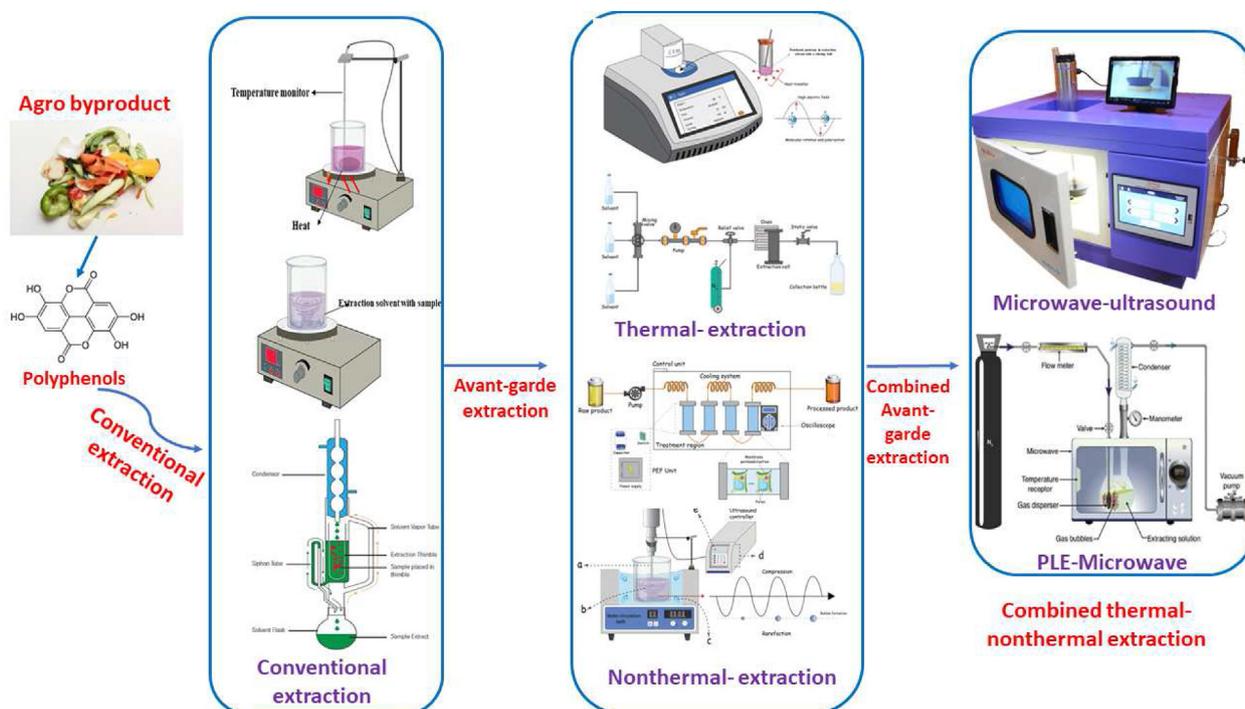


Fig. 1. Schematic diagram to describe the overall scope of the review article.

The polarity of an extractant plays a crucial role in determining the selectivity of the partition system, hence facilitating the distribution of different polyphenols within the extract. Polar protic solvents tend to yield more favorable extraction results due to the typically polar nature of polyphenol derivatives. These derivatives exhibit varying degrees of hydrophilicity or lipophilicity per the number and conjugation of phenolic groups. Thus, the use of aliphatic alcohols (for instance, ethanol and methanol) and organic extractants (polar) (e.g., ethyl acetate and acetone) are the common methods to extract polyphenols from agro-byproducts [13,14]. In extracting polyphenols from citrus peels, extractants such as ethyl acetate, ethanol, methanol, and acetone are frequently utilized. By decocting grapefruit, mandarin, and orange byproducts in acidified water and methanol (1:1 v/v, pH of 2) and washing with H<sub>2</sub>O and acetone (30:70), extracts high in narirutin, naringin, and hesperidin flavanones were obtained [15].

On the other hand, polar phenolics such as cinnamic and benzoic acids may not be completely soluble in organic extractants. As a result, most applications use mixtures with different levels of water. Thus, a simple decoction in water and methanol (30:70) at room temperature was effective for extracting kaempferol, coumaric acid, and caffeic acid derivatives from seven European *Vicia faba*

cultivars with greater antioxidant activities (3.1–4.73 µg Trolox equivalent (TE)/g dry weight (dw)) and was strongly associated with total phenolic content (TPC) [16]. Moreover, a water and acetone (1:1) mixture extracted maximum phenolics from the hazelnut involucre (370.42 mg of GAE/g) and walnut septum (67.03 mg of GAE/g) [17]. Both formulations showed promise in treating skin hyperpigmentation, wrinkle formation, cancer, obesity, and diabetes [17]. Besides, a fraction containing 10 phenolics (comprising rutin, epicatechin, chlorogenic acid, epigallocatechin, isoquercetin, ferulic acid, catechin, kaempferol, caffeic acid, and quercetin) with positive impacts on gut microbiota, fatty acid metabolism, and lipid homeostasis of rats, was extracted in methanol (10% v/v) [18]. In addition, using several extractants in consecutive cycles is an alternative approach to enhance the accumulation of extracted polyphenolic compounds. For instance, notable quantities have been obtained by utilizing mixtures of acidified methanol and water in a 1:1 ratio, as well as water and acetone in a 30:70 ratio, on ground peel derived from three different shades of pears (*Opuntia ficus indica*). These pears exhibited a TPC of ~9.64 to ~12.28 mg GAE/g. Additionally, the analysis revealed the presence of 68 extractable and 15 hydrolyzable polyphenols [19].

Even though non-generally recognized as safe (non-GRAS) extractants are used extensively in

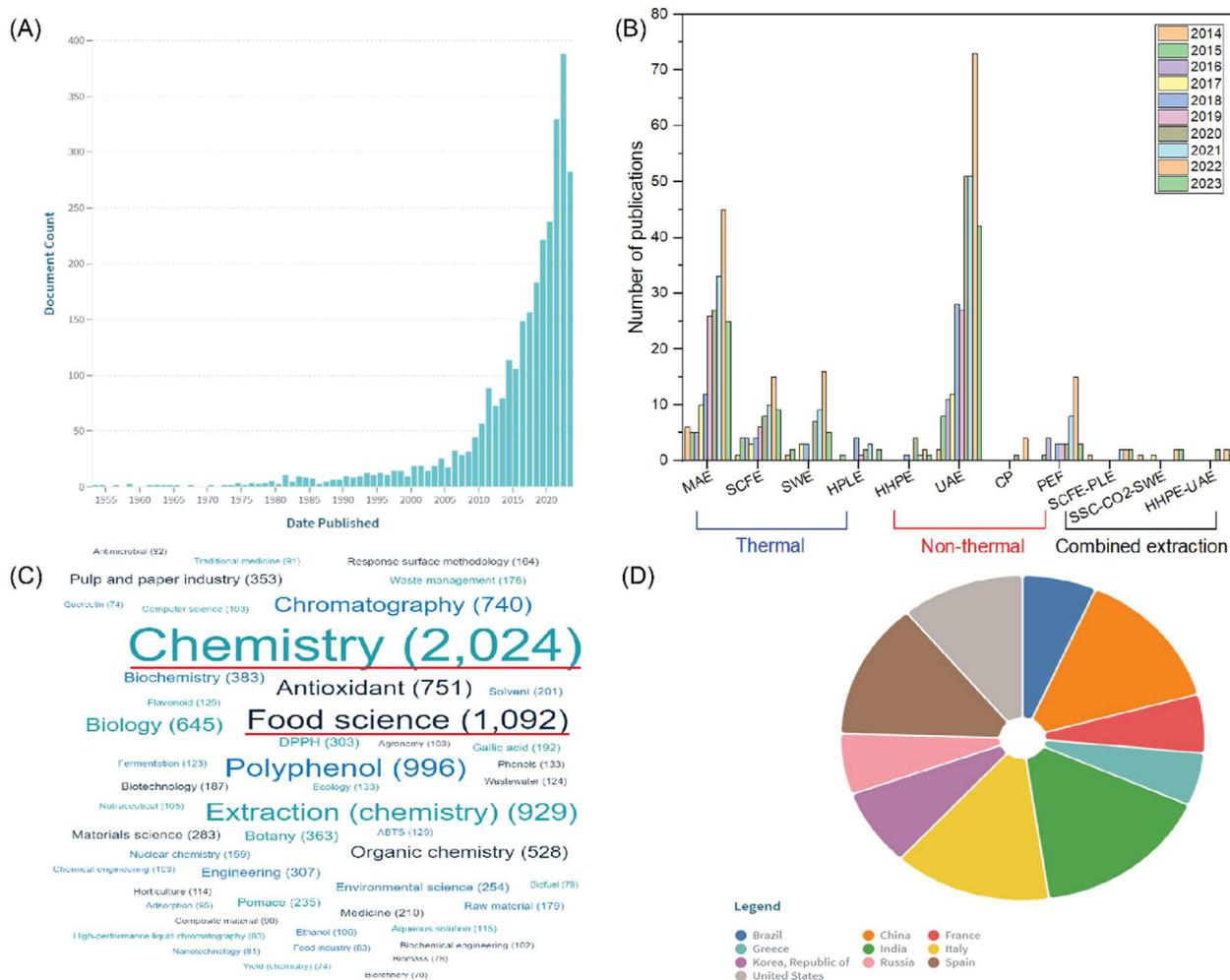


Fig. 2. (A) Scholarly works (only peer-reviewed journal articles) on the extraction of polyphenols over time (Data were taken from [Lens.org](https://lens.org) on 15th September 2023) (B) extraction of polyphenols from waste using thermal, nonthermal and their combinations (Data were taken from web of science on 15th September 2023). (C) Fields of study and (D) most active countries that have published extraction of polyphenols from waste (Data were taken from [Lens.org](https://lens.org) on 15th September 2023).

extraction procedures, they are potentially toxic and biologically aggressive, making their use in the food, cosmetics, and pharmaceutical sectors highly controversial. For example, methanol's high toxicity makes it unsuitable for human contact or consumption applications. Instead, ethanol is non-toxic and environmentally benign [20], yields higher polyphenols, and is amenable to large-scale processing capabilities. Consequently, 3.32 and 0.95 mg/g extracts of TPC and TFC, correspondingly, were obtained from melon peels (extracted at 30 °C, 24 h in ethanol (95% v/v)), with 0.29 mg/g of apigenin-7-glycoside and 0.33 mg/g of hydroxybenzoic acids being the extremely copious phenolic compounds [21]. Similarly, polyphenols were extracted from avocado peels [22] and fragrant *Thymus serpyllum* herbal dust (0.06 mg/g GAE) [23] using 50% ethanol.

Conversely, grape pomace has been tested in comprehensive valorization conditions for pomace residues using HPLC in polar media (hydroalcoholic) or a non-polar organic solvent-water mixture [24]. Ethanol and H<sub>2</sub>O mixture have been efficiently studied in extracting polyphenols from peanut skin [25], peels of citrus [26], pequi's peel [27], papery onion skin [28], grape's seeds [29], and seeds of blackberry [30], where ethanol/water systems have been applied. The necessity of polar extractants for efficient extraction can be ascribed to glycosides in certain polyphenols, such as anthocyanins. Glucosides exhibit greater solubility in water, whereas aglycones demonstrate enhanced solubility in alcohols. Conversely, flavonols and anthocyanins exhibit improved solubility in polar extractants [4,7]. Anthocyanins possess solubility in polar extractants because of hydroxyl groups and sugars within their

chemical structure [31]. Consequently, hydroalcoholic extractants are frequently employed to extract anthocyanins. H<sub>2</sub>O has a polarity value of 10, while methanol and ethanol have polarity indices of 5.1 and 4.3, respectively [32]. Consequently, the enhanced efficiency of extraction can be ascribed to a reduction in the polarity of the extractant used for extraction, achieved by increasing the ethanol content [32]. This finding aligns with multiple research that conducted experiments with mixing water and pure solvents and concluded that the combined solvent exhibited more efficacy in extracting phenolics than the single extraction solvent [3,13,31,32].

## 2.2. Acid and pH

Acids are commonly employed as extractants for phenolics because of their ability to facilitate the disruption of cell membranes and serve as a solvent for water-soluble polyphenols [33]. Lao et al. [33] demonstrated that acidified organic solvents were the most efficient in extracting anthocyanins from corn. Excessive acid utilization might result in the impairment of the intermolecular bonds responsible for binding the anthocyanin molecule to co-pigments or metals, as well as the partial hydrolysis of the glycosidic link, hence diminishing the overall extraction yield [32,34]. Besides, too much acid can obstruct extraction by altering the polyphenols because of (acyl) glycoside hydrolysis [35]. Hence, a proper ratio is needed when applied in the extraction of phenolics. For instance, membranes and analytes can be dissolved in acidified ethanol or methanol. Furthermore, excessive acids can also hydrolyze sugar and acyl labile residues simultaneously [3,4]. Given the complexities of extraction setups, *a priori* modeling of chemo-thermodynamic principles of various solvents, like the Hansen solubility parameters theoretical technique or non-random two-liquid segment activity coefficient technique, can help determine their solubilization and suitability [36]. Given this perspective, using modeling tools to address the numerous factors impacting yield to get an optimal solution is highly captivating [37,38]. For example, chemometric models that employ the response surface methodology (RSM) aim to distinguish response factors from various underlying components and develop predictive models to guide efficient approaches [39,40].

The ionic strength of the solvent, which influences the solubility of the polyphenols and their interactions with the byproduct matrix, can also impact the extraction performance. Several experiments have determined the best pH to extract

flavonoids from byproducts. In Mai et al. [41] study, the effect of the extractant's pH on the extraction of flavonoids' extra from *Euonymus alatus* was examined. The results revealed that recovery rates rose in acidic pH ranges (2.5–3.5) and fell at higher pH levels. Another study that looked at polyphenols in this situation found that the solvent's pH impacted how well polyphenols were extracted from pomegranate peel, with an acidic medium producing the most remarkable results [42]. The lower extraction yields were observed for pH > 7. The most significant outcomes for extracting phenolics from the bark of *Citrus reticulata* were achieved using slightly acidic electrolyzed H<sub>2</sub>O (pH 6.20). Nevertheless, an acid-electrolyzed water pH of 3.24 showed higher flavonoid production [43]. A higher TFC was found at pH 2 when polyphenols were extracted from *Satsuma mandarin* leaves, providing yet another illustration of the impact of pH as the acidic media produced the highest TPC and TFC [44].

According to observations in the literature, an acidic medium typically produces higher flavonoid yields. This polyphenol pattern can be clarified by the acidic pH encouraging phenolics attached to proteins and carbohydrate polymers to break down. As a result, phenols become protonated at low pH levels, changing them from hydrophobic molecules to ones that interact more firmly with the hydrophobic micellar surfactant and more easily permeate the micelles. As protons are more active at higher pHs, phenols become deprotonated and exhibit stronger ionic properties, which reduces the hydrophobic phenolics' solubility in micelles. As a result, as the pH decreases, more phenols are extracted [7,45].

## 2.3. Temperature

Temperature is another crucial variable in polyphenols' extraction from agro-byproduct. The extractants' surface tension and viscosity are reduced as the temperature rises, boosting the diffusion coefficient and, thus, the rate of mass transfer and efficiency of extraction [38,46,47]. Nonetheless, higher temperatures can accelerate the decomposition of labile phenolics such as anthocyanins [48]. The significant deterioration may arise from the fracture of covalent bonds or the accelerated oxidation resulting from thermal extraction. This process can lead to the breakdown of anthocyanins into chalcone structures upon heating, followed by the conversion of the latter into coumarin glucoside derivatives, resulting in the B-ring loss [49]. Furthermore, to minimize chemical

deterioration, anthocyanin extractions' temperature of  $\leq 60$  °C (mostly at 20–50 °C) is required [50].

Three theories were presented by Maillard & Berset [51] to illustrate how polyphenols behave at high temperatures. First, when the lignin that is bonded to phenolic acids are broken, the insoluble polyphenols may be released. It has been demonstrated that the amount of bound phenolic acids is twice as large as the amount of free phenolic compounds (measured after plant tissue has been hydrolyzed). Second, high temperatures may cause lignin to break down, producing more phenolic acids. This may account for the phenolic yield in PLE extractions rising with the temperature. Finally, the polyphenols' heat breakdown may occur at high temperatures. The decrease in polyphenolic yield during high-temperature extractions is most frequently attributed to thermal degradation [52]. However, research using CE methods has revealed that heat degradation occurs at 80 °C as opposed to 150–200 °C in hot-pressurize liquid extraction (HPLE), which means that thermal degradation alone cannot explain the behavior of polyphenols. The inconsistent findings in the literature could be related to the other two mechanisms outlined above. Therefore, the lignin-phenolic acid bonds cleavage or the breakdown of the lignin itself producing additional phenolic acid may cause the increase in TPC found at high temperatures in PLE extractions.

#### 2.4. Extraction time

The duration of the extraction has a crucial role in extracting polyphenols from byproducts. The occurrence of incomplete extraction is well acknowledged when the time of the extraction process is insufficient. As extraction time increase, the polysaccharides and oligosaccharides probably formed strong associations with a significant amount of phenolic compounds. Consequently, these phenolics were released from their bound state and transformed into free polyphenols [38]. This phenomenon occurred due to the extended exposure of the sample to the extractant, allowing sufficient time for the desired phenolics to move into the extractant [13,53]. However, an extended duration of extraction would lead to the inefficient allocation of resources and financial loss. Besides, extraction time is an important parameter since excessive light or oxygen exposure can degrade phenolics, reducing their ability to scavenge radical nitrogen and oxygen species. On the other hand, other polyphenols are prone to volatility or oxidation, necessitating quick methods or circumstances to shield the solubilized portions from oxygen or

light degradation and avoid releasing polyphenols into the gaseous phase [35]. Considering these factors, the time for specific phenolics to reach partition equilibrium must be doubled. However, antioxidants, such as butylated hydroxytoluene or ascorbate, can be added to the extraction cocktail to strengthen chemical protection and post-extraction shielding methods, such as encapsulation, prior to manufacturing the end-formulations [54,55].

#### 2.5. Pretreatment prior to extraction

Pretreatments, including homogenization, milling, grinding, and drying, are frequently employed to decrease particle size before extraction, which improves the yield of targeted phenolics because of an increase in the surface area [4,13]. Solvent aerosolization (SA) has also successfully extracted polyphenols by maximizing the extraction surface. For example, SA was environmentally friendly in determining the TPC in 42 olive oil samples and saved time and cost [56]. However, protocolation and pretreatment significantly influence the extract's phytoconstituents. Therefore, water activity ( $a_w$ ) ought to be reduced through drying or lyophilization [57]. As established in the loss of polyphenols because of the instability of native flavan-3-ols and 5-O-caffeoylquinic acid (from apple pomace), drying impacted the preservation of polyphenols' native structures [58,59]. Because drying might induce unexpected changes in the phenolic, sample preparations ought to be thoroughly investigated and extrapolated carefully [59,60]. In spite of the lack of a comprehensive investigation, lyophilization has proven to be more successful than thermal drying in retaining phenolics and keeping the functional capability of extracts like those reported in olive pomace [61].

To summarize, plant phenolic extractability from agro-byproduct is influenced by several physico-chemical parameters that require a meticulous balance amongst natural structure partition and protection to maintain the functionality of the extract (phenolics). In addition, biological variables such as the target polyphenols' microheterogeneity and their pairing with matrix compounds also influences the extraction yield.

### 3. Conventional biorefinery methods for byproducts' polyphenols

#### 3.1. Percolation

The most common method for making liquid extracts is percolation (Fig. 3A). The definition of

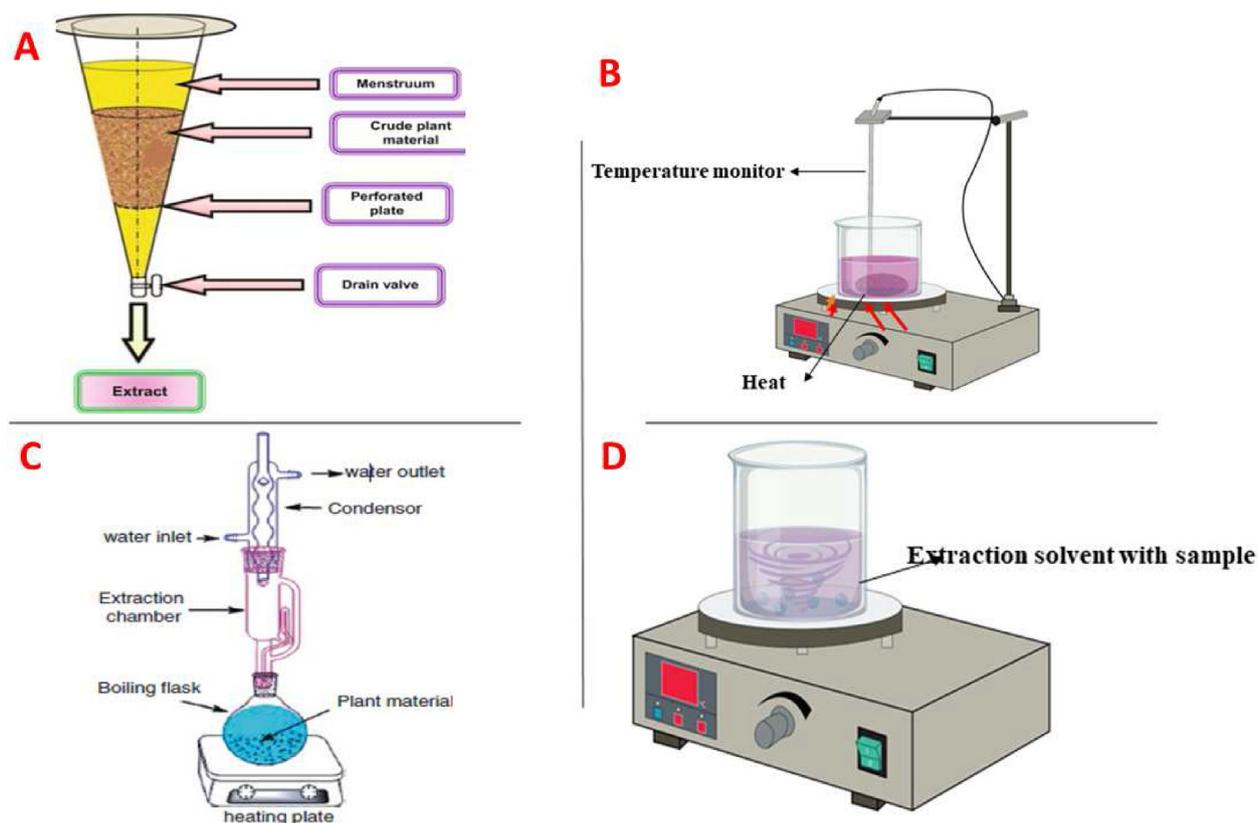


Fig. 3. Conventional extraction methods (A) percolation [12], (B) decoction, (C) Soxhlet extraction, and (D) maceration.

percolation is to pass a liquid through a solid material drop by drop. In the process of percolation, a fresh solvent is supplied from the top, and the solvent—typically ethyl alcohol—is gradually forced down while slowly passing via the plant material and slowly packing itself with polyphenols [62]. The plant material needs to be carefully shredded, taking care not to shred the particles too fine, before adding it to the percolator as it will be more difficult to separate the tiny particles from the solvent if the particles are too fine. As a result, there would be residue at the bottom of the percolator and a hazy extract. To facilitate the smooth diffusion of polyphenols into the extractant, it is suitable to wet the plant matrix with the extractant to allow the plant cell to lengthen [63]. The solvent is poured into the percolator from the top once the plant material has been placed within (Fig. 3A). The extraction solvent percolates via the plant material at a rate specific to the agro-byproduct being extracted. To provide the solvent enough time to enter agro-byproduct cells and extract the polyphenols, the flow rate of the solvent should be manageable. Nonetheless, the rate at which the solvent percolates should not be too slow, as this would require more solvent to

achieve full extraction. Generally, the extractant flow rate is 5 mL per min for 1 kg of plant material [62].

The chemistry of the phenolics to be extracted determine which extraction solvent is best for the percolation. Water-alcohol extractant mixture is frequently used, producing an exceptionally efficient extraction [64]. For instance, 70% ethanol was used to extract phenols, especially epicatechin, while petroleum ether was utilized to extract flavonoids and other antioxidants [65]. Interestingly, alkaloids from wild fruits were extracted by percolation using an inorganic aqueous hydrochloric acid solution, aside from alcohol [62]. But because alcohol is a preservative, it also has the benefit of keeping the extract intact. After the procedure is complete, the byproduct pressed to the extract the residually absorbed solvent, and the residual solution is added to the extract. The extraction process concludes when a colorless liquid free of polyphenols elutes from the percolator and concentrated [62]. Several polyphenolics have been isolated using this method from matrices of food byproducts [64]. The percolation approach has the same time-consuming, large solvent volume issues

as the maceration method but also has sample size, extraction length, and polyphenol solubility issues.

### 3.2. Decoction

Using the decoction technique (Fig. 3B), plant samples are either boiled for a shorter time (15–60 min) or are covered with boiled water and left to stand for a predetermined amount of time. This technique works best with water-soluble and heat-stable polyphenols in crude byproducts [62]. The type of agro-byproduct and the extracted polyphenols will determine how long the boiling process takes. For example, decoction and infusion methods have been employed to extract flavonoids and phenols from rhizomes and leaves at 100 °C [66]. According to reports, the *Syzygium cumini* bark extract obtained by decoction as an extractive method showed notable antioxidant and anti-glycation potential [67]. Alternatively, you can boil the byproduct for 1 h, including tree bark and branches. After boiling, the mixture is allowed to cool before being filtered and supplemented with cold water to get the desired solution concentration. Once the decoction process is finished, the mixture is filtered to acquire the liquid extract. However, the decoction procedure will likely yield an extract with a high concentration of unwanted phenolic compounds. Besides, there are better approaches for polyphenols that are thermolabile than this one.

### 3.3. Soxhlet extraction

Soxhlet extraction (Fig. 3C) is one of the crucial approaches when it comes to eco-friendly methods. The Soxhlet method involves adding the sample into a thimble and replacing the solvent in the flask with a circular bottom. The new solvent is run through the thimble throughout the extraction procedure. Once the liquid reaches the top, the thimble holder allows the siphon to return the solvent to the flask with a circular bottom (Fig. 3C). Until the process reaches saturation, this procedure is repeated. Over half of the extractant is utilized for phenolic extraction studies (24–50 h) [64].

The Soxhlet extraction process is still considered because of its greater simplicity. These methods do have certain limitations, though. This method involves using a lot of solvents (300–500 mL per sample), 10–30 g of sample (byproduct), extended extraction time (18–24 h per the sample), and very high heat energy loss. However, it is a better extraction method because maceration requires lower temperatures, takes less time, and yields a larger yield of polyphenolic content. For example,

the flavonoid and phenolic content of the *S. cumini* seed kernel was investigated [68]. According to Mahindrakar and Rathod [68], the TPC of the soxhlet extraction was ~30 mg GAE/g for 6 h and 100 °C, but the TPC of the batch extraction (maceration) was 79.87 mg GAE/g for 105 min at 50 °C. Using process intensification, a study on curcumin extraction was examined using maceration and Soxhlet extraction. Juxtaposed to Soxhlet, which required virtually the same yield at higher temperatures, batch extraction (maceration) had a higher extraction yield (~7.89 mg/g) at 30 °C [69]. Therefore, maceration is a more financially viable and promising method.

### 3.4. Maceration

A straightforward extraction technique called maceration immerses the coarsely ground or powdered agrobyproduct in a suitable solvent for a long time at room temperature while stirring occasionally (Fig. 3D). The mixture is strained via a net or sieves with microscopic holes once the extraction process is finished. The marc is then crushed, and the liquid extract can stand before decantation or filtration. Doing maceration in a stoppered container is preferable to reduce solvent loss by evaporation [62]. The solvent should not evaporate and yield a concentrated extract throughout the extraction process. Vacuum evaporation is commonly used to concentrate the product [8]. Choosing the right solvent for the maceration is essential since it will identify the classes of polyphenols that can be recovered from the samples. The extraction of thermolabile polyphenols may also be made possible by the solvent. Besides, the crucial variables to consider are time and the agitation speed. If the magnetic stirrer's speed changes, turbulence may result from creating a vortex [64]. These factors also make an increase in the mass transfer rate feasible. Therefore, the stirrer's speed should be between 180 and 240 rpm [64]. Increased speed results in significant fluctuations in the equilibrium concentration and, consequently, the diffusion coefficient. Consequently, the entire investigation is carried out until all polyphenolic components are extracted, and the procedure achieves equilibrium [70].

The primary drawback of the process is its lengthy extraction time and low efficiency [62]. However, given that chokeberries yielded higher concentration of anthocyanins, optimal conditions may attribute significant efficiency to this procedure [71]. However, a study that used microwave, reflux, ultrasonic, and maceration extractive methods to

extract flavonoids from *Cajanus cajan* leaves found that the maceration procedures produced the lowest yield [72]. Similarly, Boateng et al. [8] stated that extraction yield for MAE' was ~38% higher than maceration and MAE took ~30 min to extract polyphenols, whereas maceration took ~510 min.

## 4. Thermal extraction methods

### 4.1. Supercritical fluid extraction (SCFE)

The SCFE combines both liquid and gas inside a single phase, as depicted in Fig. 4A. The presence of low surface tensions offers protection to more unstable phenolic constituents, reduces the viscosity of organic liquids, enhances the diffusion of phenolics, and facilitates the partitioning of polyphenols in the supercritical zone (when pressures and temperatures exceed their critical thermodynamic points) due to diffusivity properties similar to gases [73]. Due to their increased solubility and solvating capabilities under high pressure, and temperatures, dual liquid–gas SCFs exhibit remarkable versatility as extractants for many products, both in laboratory and industrial settings [3]. In addition, the extraction process of SCF has the potential to be non-reactive towards food elements and pose no harm to human health. In addition, it is noteworthy that SCF extraction does not

adversely impact the environment due to the complete avoidance of toxic solvents while also reducing the high energy requirements associated with alternative extraction methods [74].

Numerous literary studies have provided evidence that using SCFE positively impacts the polyphenols' extraction from byproducts (Table 1). Using SCFE has demonstrated its efficacy in preserving the integrity and functionality of labile phenolic compounds, enhancing its applicability across a wide range of plant matrices. This advancement in SCFE technology has extended its application beyond its initial utilization in decaffeinated tea and coffee. This aligned with the extraction of mangosteen [75], resveratrol [76], and numerous polyphenols of *Vitis vinifera* [77]. Analogous findings have been documented by Mazzutti et al. [78] about the effects of polyphenols derived from cocoa bean hulls (CBS) and by Campone et al. [79], who extracted onion peel's flavonoids (Table 1). The enhanced extraction of phenolics from agro-byproducts via SCFE may be attributed to the better solvating and solubility capabilities at high pressure and temperature, as well as the dual liquid–gas state of the supercritical fluid, which renders it very versatile as an extractant for a diverse array of byproducts. Previous studies have documented comparable findings of polyphenols' extraction from

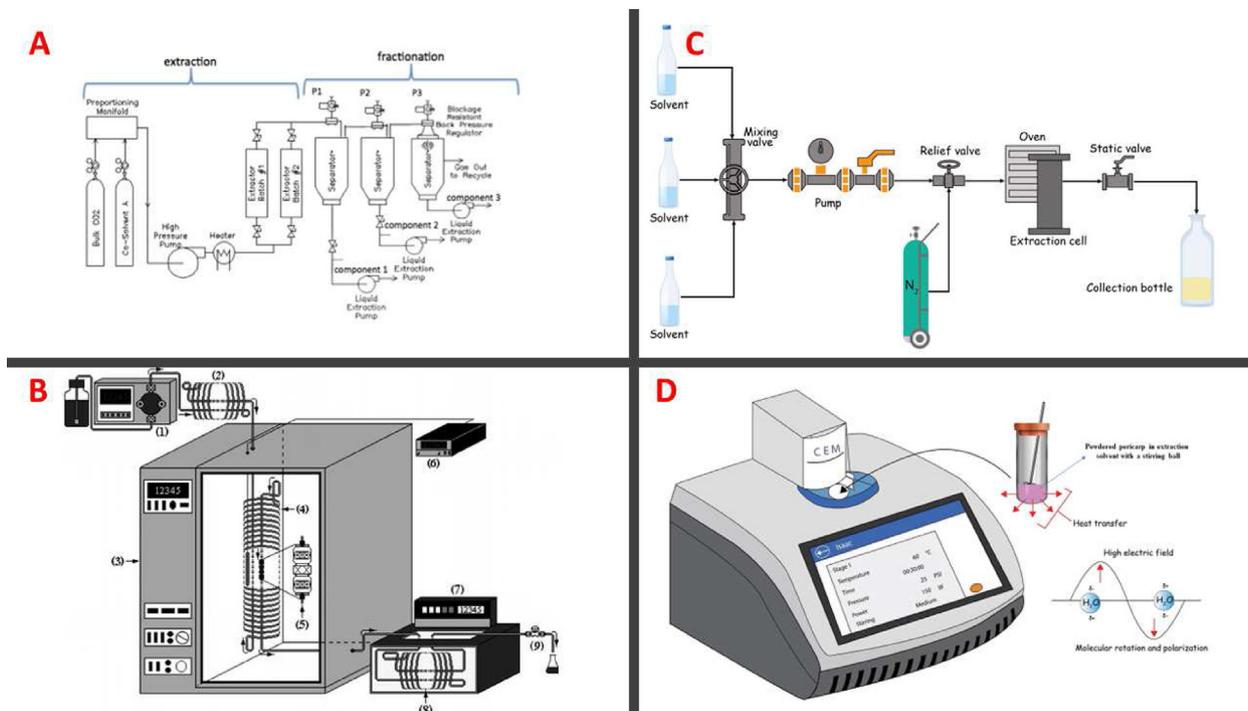


Fig. 4. Experimental setup for thermal polyphenol extraction form agro-byproduct utilizing (A) Subcritical fluid, (B) Subcritical water [9], (C) Hot pressurized liquid, and (D) Microwave extraction technologies [8,9].

Table 1. Agro-byproducts' phenolic extraction using thermal Avant-garde technologies.

Extraction	Byproduct	Optimum conditions	Results	Reference
SCF	Cocoa bean hulls	120 min, 20 MPa, and 40 °C	TPC = 35–51 mg/g	[78]
	Onion skin	120 min, EtOH (85%, v/v) with CO <sub>2</sub> , 10 MPa, 10 mL/min of CO <sub>2</sub> flowrate, and 40 °C.	Protocatechuic acid = 0.14 mg/g and quercetin = 0.53 mg/g	[79]
	Spent coffee grounds	EtOH/H <sub>2</sub> O (2:1 v/v), 80 °C, 20 MPa for 25 min	High TPC (294.47–392.96 mg/100 g), similar PLE.	[88]
	Oat bran	35 MPa, 50 °C, 15 g/min and 300 min.	Higher ferulic acid in SCF (~35 µg/g) than CE (~30 µg/g).	[86]
	Rice bran	Extractant: CO <sub>2</sub> + EtOH (5–10%), 40 MPa, 120 min and 40 °C.	SCF had higher gallic acid (~30.5 mg/g) than CE (~29.7 mg/g).	[210]
	Orange pomace	15–35 MPa, 40–60 °C, and EtOH (6%, v/v).	TPC = 0.36–0.57 mg/g	[82]
	<i>Peganum harmala</i> seeds	55 °C, and 30 MPa	TPC = 79.04 mg/g and TFC = 7.10 mg/g	[81]
	Spent black tea	EtOH/H <sub>2</sub> O (1:1), 0.3 MPa and 12 °C.	Higher TPC in SCF (80.82 mg/g) for SCFE than HWE.	[84]
	Mango peel	35 MPa, 55 °C, and ethanol (20%, v/v)	SCF had higher gallic acid (30.5 mg/g) than CE (29.7 mg/g)	[210]
	SWE	Defatted coffee cake and powder.	Defatted cake (200 °C), coffee powder (175 °C), 22.5 MPa, and 9 min	(Coffee powder) TPC = 26.6 mg/g (Defatted cake) TPC = 55.7 mg/g
Sorghum bran		S/L ratio (35 mL/g), ~145 °C, and 21 min.	TPC = ~47.3 mg/g.	[91]
Yarrow by-products		198 °C, 3 MPa, and 16.5 min.	TPC = ~52.4 to ~128 mg/g.	[102]
Hayward kiwifruit pomace		170–225 °C, 3 MPa, S/L of 1:100 g/mL 30 min.	TPC = ~26.16–86.26 mg/g	[104]
Onion skin		170–230 °C, 30 min and 5 MP	TPC = 200 mg/g and TFC = 90 mg of QE/g.	[103]
Papaya seeds		70–150 °C, 5–40 min, 4 mL/min flow rate, and 10 MPa.	Ferulic acid: 22.7 µg/g, mandelic acid: 122.7 µg/g, vanillic acid: 108 µg/g	[105]
Spent coffee		180–240 °C, 6 g, and 20–6 MPa	TPC and TFC of 33.1–51.2 and 15.13–25.51 mg/g, respectively.	[107]
Green coffee byproduct		22–30 MPa, 150–250 °C, 10 mL/min and 36 min.	TPC = ~27 mg/g	[101]
Chestnut shells		30 min and 220 °C.	TPC = 315.2–496.8 mg/g	[108]
Distillery stillage		30-min with S/L of 1:15 at ~140 °C (TPC) or (~200 °C) for TFC.	TFC = 1.24 mg/g and TPC = 4.88 mg/g	[96]
HPLE	Coffee spent ground	7 MPa, 30 min, and 150–220 °C.	TPC = 70.3 mg/g	[100]
	Grape pomace	150 °C, EtOH: H <sub>2</sub> O 32.5:67.5, and 1 MPa.	HPLE's TPC was ~19× higher than CE (0.23 mg of GAE/g)	[121]
	<i>Vitis vinifera</i> L. pomace	H <sub>2</sub> O: EtOH (40:60, v/v), 160 °C, 5 min, 1 MPa.	HPLE's TPC was ~1.5 to 2.5× more than CE.	[118]
	Grape marc	Acidified H <sub>2</sub> O: EtOH (pH 2) 1:1, 40 °C, 10 MPa and 40 min	TPC = 28.66 mg/g	[120]
	Avocado peel	200 °C, EtOH: H <sub>2</sub> O 1:1, 40 min, and 11 MPa.	TPC was 34 mg/g	[22]
	Orange peel	EtOH: H <sub>2</sub> O 3:1 (v/v), 65 °C, 40 min and 10 MPa	The extraction yield was ~35%, and TPC was ~15 mg/g	[116]
	Pomegranate peel	200 °C, EtOH: H <sub>2</sub> O (1:1), 20 min and 10.34 MPa	TPC was ~149 mg/g	[117]
	Winery byproducts and olive oil mill	100 °C, EtOH: H <sub>2</sub> O 1:1, 5 min, 1 cycle, and 10.3 MPa	(Olive pomace) TPC was ~9.5 mg of GAE/g, winery byproduct: TPC was 3.58 mg of GAE/g)	[122]
	<i>Tetrapleura tetraptera</i>	43 min, 220 °C, and L/S of 60 mL/g	TPC of 8.92 mg/g	[46]
	Spent coffee grounds	195 °C	TPC = 19–26 mg/g, Caffeine was 3–9 µg/g.	[112]
Olive Pomace	52.3% of EtOH and 136.5 °C.	HPLE had a higher TPC (1.66 g/kg) than CE (0.28 g GAE/kg)	[113]	
Beetroot byproduct	7.5–12.5 MPa, 40 °C, and 3 mL/min	HPLE had a higher TPC (~0.61 mg/g) for HPLE than CE (0.13 mg/g)	[114]	
<i>Eucalyptus intertexta</i> leaves	26.6% EtOH, 179 °C and 36 min.	TPC = 180.7 mg/g.	[115]	
Maqui leaves	200 °C, EtOH (23% v/v), and 3 extraction cycles	HPLE's TPC was ~3× more than maceration	[119]	

(continued on next page)

Table 1. (continued)

Extraction	Byproduct	Optimum conditions	Results	Reference
MAE	Hibiscus calyx	8 min, 700 W & subsequent extraction (acidic aqueous solution) for 6 h	The anthocyanin was 1.63 mg/g, and TPC was 29.62 mg/g.	[123]
	Kiwiberly leaf	72–94 °C, 300 W, and 1–5 min	TFC = 136.8 mg/g and TPC = 629.5 mg/g	[124]
	Pomegranate Fruits Peels	61.48 & 79 °C, 10 & 12.2 min, ~3797 & 3576 W and L.S ratio of ~40 and ~38%	TPC = 5.542 mg of GAE/g	[127]
	Orange juice waste	120 min and ~6 kW	TPC of ~37.7 mg of GAE/g	[126]
	Black carrot pomace	9.8 min, 348 W, L/S (19.3 mL/g) and EtOH (19.8%)	TPC = 0.26 mg GAE/mL	[125]
	Apple fruit dust	EtOH (40%), 15.2 min, 400 W	TPC = 36.99 mg of GAE/g	[129]
	Chardonnay grape marc	48% EtOH, ~1.8 g of sample, and 10 min	TPC = 1.21 mg of GAE/mL	[128]
	Black rice husk	175 °C and 31.11 s	TFC (3.04 mg/100 g) and TAC (3.39 mg/100 g)	[130]
	<i>Thymus serpyllum</i> byproduct	48% EtOH, 40.3 mg/mL S/L ratio, 86 s.	TPC = ~57 mg GAE/g	[132]
	Chestnut waste	L/S ratio = 50 mL/g, 107 °C and 5 min.	TPC = 344 mg/g, CT = 129.6 mg/g and extraction yield = 25%	[131]

**Note**

TPC-Total phenolic acid, GAE-Gallic acid equivalent, TFC-Total flavonoid acid, PLE, pressurized liquid extraction, SWE, subcritical water extraction; SCFE, supercritical fluid extraction, HPLE, hot-pressurized liquid extraction, MAE, microwave-assisted extraction, SWE, subcritical water extraction, CE, conventional extraction, EtOH, ethanol, L/S, liquid to solid ratio, TAC, total anthocyanin content.

sugar beet leaves [80] and *Peganum harmala* seeds [81].

Furthermore, a study by Espinosa-Pardo et al. [82] showed that the orange pomace' TPC significantly increased after undergoing biotransformation with SC-CO<sub>2</sub> (25 MPa and 60 °C) proceeded by fermentation. The TPC (~21.2 mg/g) was twice as high as the control group. The extract derived from the pulp of black chokeberry, which was extracted using SC-CO<sub>2</sub> (10% ethanol, 24.9 MPa, and 68 °C), exhibited an inhibitory influence on the proliferation of breast cancer cells, as reported by Wenzel et al. [83]. The fitness of semi-continuous SCFE for the pilot-scale polyphenol extraction from wasted black tea was evaluated by Rajapaksha and Shimizu [84]. The occurrence of SCFE increased the diffusivity and solubility of phenolics. In contrast to hot water extraction, the TPC exhibited a higher level (80.82 mg GAE/g), as seen in Table 1. The findings can be ascribed to the emergence of a moderately polar environment and the enhanced ability of ethanol to create intermolecular interactions with phenolics [85]. Comparable findings have been stated for apple pomace and oats bran [86]. Chai et al. [87] extracted flavonoids (quercetin and quercetin derivatives) and phenolic acids (protocatechuic acid, ferulic acid, gallic acid, vanillic acid, and p-coumaric acid derivatives) from Lees produced by the pisco-making method at 20 MPa and 39.85 °C. Analogous results have been documented for spent coffee grounds [88] and wild vegetables [89].

#### 4.2. Subcritical water extraction (SWE)

SWE (Fig. 4B), applies pressures >21.8 MPa and H<sub>2</sub>O (200 °C ≥ temperature <374 °C) to maintain the liquid state stable. Mostly, 5 MPa and 100–250 °C are adequate to retain liquid H<sub>2</sub>O [90]. SWE can be done in either continuous or batch mode. Consequently, it can be shown that pure H<sub>2</sub>O possesses remarkable temperature-reliant dynamic abilities that enhance mass transfer efficiency and enable the selective extraction of diverse phenolics [91,92]. The viscosity of an aqueous solution is decreased within the subcritical region, leading to an improvement in the diffusivity and mass transfer rates. In addition, the dielectric constant exhibits a reduction upon the disruption of hydrogen bonds, resulting in a reduced polarity of H<sub>2</sub>O. Consequently, H<sub>2</sub>O demonstrates comparable behavior to conventional organic solvents like ethanol or methanol, exhibiting analogous solvation characteristics. Controlling temperature under medium pressures can impact H<sub>2</sub>O ion product, density, and dielectric constant,

enhancing its versatility in extracting phenolic compounds with lower polarity [92].

Multiple findings have proved that using SWE enhances the efficiency of polyphenol extraction from different byproducts (Table 1). Ibrahim et al. [93] showed that the SWE method at 200 °C for 30 min showed an affinity for extracting polyphenols from apple pomace (Table 1). In research on peels by Cai et al. [94], it was shown that the use of SWE (2 cycles, 90 °C, 80% ethanol, and 15 min) resulted in an improved extraction yield (~2.44 mg/g) compared to UAE (~2.29 mg/g) and CE (~2.189 mg/g) for purple sweet potato peels. In contrast to anthocyanins, the tannins extracted from red and white grape pomace exhibited an extraction yield of 68 mg/g (2.5 MPa and 200 °C), according to Yammine et al. [95]. This extraction yield surpassed that obtained for SWE using a mixture of H<sub>2</sub>O and ethanol (1:1 v/v) at 25 °C. In the study by Mikucka et al. [96], on phenolic extraction from distillery stillage (Table 1), the highest TPC of ~4.88 mg GAE/g and TFC of 1.24 mg QE/g were attained at 140 °C and 200 °C, respectively. These optimal conditions were achieved by a 30 min SWE using a solvent-to-liquid ratio of 15:1 (v:w). The TPC and TFC exhibited a statistically significant reduction as the extraction duration (90 min), and temperature (260 °C) increased, with TPC decreasing 4.4× and TFC decreasing 3.9×. The polyphenols in coriander seeds were recovered through SWE, as conducted by Zeković et al. [97]. The maximum TPC of 10 mg/g was attained (8.76 MPa, 100 °C, and 10 min). This TPC was more than that obtained by CE. Pavlić et al. [98] reported comparable findings, indicating that the highest TPC (~79.8 mg GAE/g) was obtained by SWE (202 °C and 15.8 min) for sage herbal dust. Furthermore, Naffati et al. [99] revealed that using SWE had a higher efficiency in recovering phenolics from *Arctostaphylos uva-ursi* compared to CE. Pedras et al. [100] employed a semi-continuous SWE technique to extract polyphenols from a coffee-waste grind. A higher yield of polyphenols was obtained at temperatures of 200 and 220 °C compared to the hydro-alcoholic extraction. Pedras et al. [100] postulated that the liberation of polyphenols inside the matrix of spent coffee grounds and the potential conversion of lignin into polyphenols could explain this observed enhancement. Previous studies have documented comparable investigations on the SWE of phenolics from sorghum bran [91], defatted cake and coffee powder [101], yarrow byproducts [102], onion skin [103], Hayward kiwifruit pomace [104], papaya seeds [105], grape seeds [106], spent coffee grounds [107] and chestnut shells [108].

#### 4.3. Hot-pressurized liquid extraction (HPLE)

Pressured liquid extraction (Fig. 4C), or advanced HPLE, involves the dissolution of solid matrices in extractants at >100 °C while maintaining a liquid state [109]. Secondary intermolecular forces disrupt when ethanol or H<sub>2</sub>O is employed as extracting agents under elevated temperatures (50–250 °C) and pressures (4–20 MPa). This process accelerates the release and dissolution of polyphenols bound inside the matrix [110]. The encapsulation and extraction of the solvent and sample can be performed either in a static state, where short intervals are employed, or in a dynamic state, where the solvent is continuously delivered into the chamber [20]. Consequently, employing HPLE exhibits notable advantages such as enhanced extraction efficiency, reduced extractant consumption, elimination of filtration requirements, and compatibility with extractants suitable for food-grade applications. These attributes contribute to the development of ecologically conscientious strategies [20].

Works of literature have provided evidence that HPLE substantially affects phenolics' extraction from agro-byproducts (Table 1). The comparative analysis conducted by Ref. [111] revealed that the HPLE method (20 min, 176 °C, and 10.3 MPa) exhibited a higher extraction efficiency than SCFE (60 min, 30 MPa, 15% ethanol, and 40 °C). Shang et al. [112] investigated the effects of different operating parameters (pressure, temperature, and time) on extracting polyphenols from coffee grounds using HPLE. The findings indicated that the extraction process had positive effects, potentially resulting in a TPC of ~23 mg GAE/g. Additionally, it was demonstrated that HPLE is an efficient approach to optimize the extraction parameters of caffeine and TPC from used coffee grounds within a relatively short duration, owing to its high mass transfer capabilities. Similarly, a study conducted by Cea Pavez et al. [113], used HPLE to extract olive pomace' phenolics (Table 1). The TPC was much higher when using the HPLE (1.65 g/kg) than CE (0.28 g/kg). The optimized HPLE extract exhibited a much higher concentration of secoiridoids and flavonoids, which was 3–4× that of CE. Similar outcomes have been documented in phenolic extraction from beetroot waste [114], *Eucalyptus intertexta* leaves [115], orange peel [116], olive pomace [113], *Tetrapleura tetraptera* L. [46], and pomegranate peel [117].

In Allcca-Alca et al. [118] research, *V. vinifera* L. pomace' skin and seeds' polyphenols were recovered utilizing HPLE (Table 1). The authors reported that HPLE was able to extract 1.5× and 2.5× of

polyphenols from the seeds and skins of grape pomace, correspondingly, in comparison to CE, which utilized 60% acetone at 1 atm and 30 °C. The observed result can potentially be elucidated by using ethanol as a co-extractant in HPLE, which reduces the solvents' polarity. Consequently, this reduction in polarity facilitates polyphenols' solubility by establishing molecular interactions involving London and dipole dispersion forces. Rivera-tovar et al. [119] applied HPLE to extract phenolics from maqui. The findings (Table 1) showed that under the optimum conditions, the TPC (~205 mg GAE/g) extracted using HPLE was 3× higher than the maceration method. Therefore, it was recommended that HPLE is an efficient method to extract catechin, quercetin, and protocatechuic acid. Previous studies have shown comparable results for the extraction of polyphenols from grape marc' [120], grape pomace [121], olive oil mill and winery waste [122], and avocado peel [22].

#### 4.4. Microwave-assisted extraction (MAE)

The MAE has been used to efficiently extract particularly polyphenols. The phenomenon of microwave radiation is shown to engage in interactions with chemically linked water molecules present within the confines of the microwave oven, leading to the generation of elevated pressure and temperature levels (Fig. 4D). The microwaves interact with the agro-byproduct matrix's cell walls and tissues. The absorption of photonic energy from electromagnetic waves during this interaction results in heating moisture confined within the plant's matrix. Consequently, the byproduct's matrix experiences moisture loss because of the influence of electromagnetic radiation. Subsequently, the agro-byproducts' cell walls undergo substantial pressure at subcellular and cellular levels, leading to swelling during mechanical alloying and extrusion. Thus, the rupture of cells and subsequent swelling lead to structural alterations in the byproduct matrix, promoting an increased solutes transfer. Consequently, this process facilitates the migration of polyphenols from the cellular matrix of the agro-byproduct into the extractant during MAE. Microwaves effectively interact with dipolar molecules by inducing ion conduction or dipole rotation, resulting in efficient agitation, rapid heating, and disruption of hydrogen bonding within the extraction system [123]. The enhancement of matrix desorption and mass transfer of extractable polyphenols to the solvent occurs when the accessibility of cell components to the extractant increases. This leads to reduced

extraction durations and decreased volumes of extractants required.

Research has shown that MAE substantially impacts agro-byproducts' phenolic extraction (Table 1). MAE-based techniques have been investigated to extract polyphenols, such as anthocyanins, phenolic acids, and flavonoids, from olive pomace [122] and grape juice byproducts [124] in equivolumetric water: ethanol (Table 1). Kumar et al. [125] stated that MAE outperformed UAE and CE in extracting phenolics from black carrot pomace. The TPC for MAE was 2.64 mg GAE/mL and was performed within 9.8 min (348 W, ethanol (19.8% v/v), and time (9.8 min)). Previous studies have reported comparable results regarding the extraction of phenolics from various sources, including orange juice byproduct [126], pomegranate fruit peels [127], chardonnay grape marc [128], apple fruit dust [129], black rice husk [130], chestnut waste [131] and *T. serpyllum* L. product [132].

The utilization of MAE to extract phenolics (for instance, phenolic acids, anthocyanins, flavonoids, etc.) from olive pomace byproducts has been investigated by Tapia-Quirós et al. [122]. Similarly, using MAE to extract polyphenols from grape juice byproducts has also been explored by Silva et al. [124]. Arboleda Meija et al. [133] stated the utilization of an interconnected MAE (pH 2, ethanol (75% v/v), and 350 W) in conjunction with a filter membrane for extracting polyphenols from red grape lees. The study identified syringic acid, gallic acid, (+)-catechin, galocatechin derivative, and catechin derivative in the extracted samples. The experimental conditions achieved in a sealed chamber, where extractants are subjected to high pressure and temperature to optimize extraction, exhibited similarities to MAE and HPLE. According to Kumar et al. [125], MAE demonstrated comparable or even better performance than UAE and CE in extracting phenolics from black carrot pomace (TPC of 2.64 mg GAE/mL). Comparable outcomes have been reported on phenolic extraction from pomegranate fruits peels [127], black rice husk [130], chardonnay grape marc [128], *T. serpyllum* L. byproduct [132], apple fruit dust [129], chestnut waste [131], purple corn pericarp [8], Chicory byproducts [134], and Fennel byproducts [134].

## 5. Non-thermal extraction methods

### 5.1. High-hydrostatic pressure extraction (HHPE)

According to Scepankova et al. [135], the primary effects of HHPE (Fig. 5A) are observed in the non-covalent interactions, specifically hydrophobic

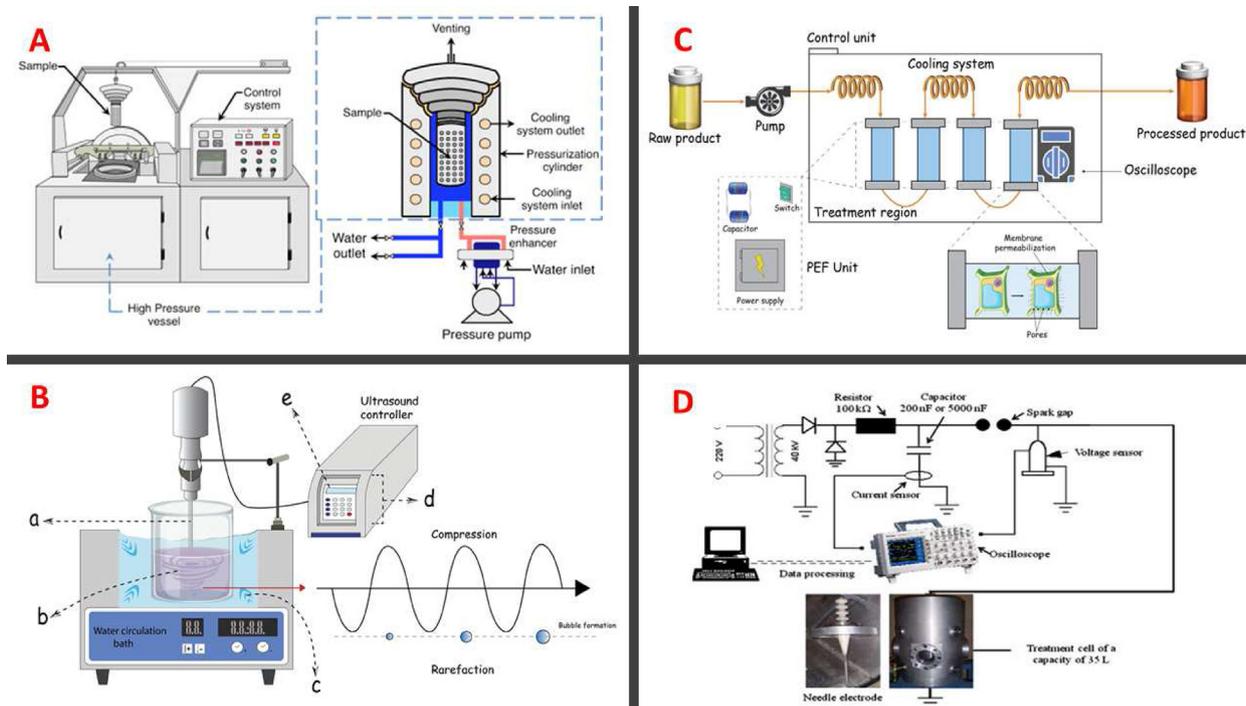


Fig. 5. Experimental setup for non-thermal polyphenol extraction from agro-byproduct using (A) High-hydrostatic pressure [109], (B) Ultrasound [31], (C) Pulsed electric field, and (D) High voltage electric discharge system [172].

bonds, van der Waals forces, and hydrogen electrostatic bonds. Consequently, it can be deduced that compounds with low molecular weight (Mw), such as flavonoids and pigments, are less influenced by HHPE. Consequently, the HHPE exhibits enhanced preservation of bioactive compounds. The correlation between pressure and the solubility of polyphenols establishes pressure as a pivotal element in the extraction process. Increasing the pressure enhanced the equilibrium concentration within the extract, consequently leading to an elevation in solubility. Furthermore, the scientists documented in a subsequent study that pressure directly influenced organelles' rupture and cellular contents' intermingling [136]. A considerable proportion of the target constituents are discharged into the solvent due to the rupture of the cellular membrane, resulting in the liberation of the cytoplasm upon the reduction of pressure. The high efficiency of the HHPE technique can be primarily ascribed to two variables, as stated by Fernandes et al. [136]. In phenolic extraction, HHPE is influenced by temperature, extractant-to-solid ratio, pressure, concentration, type of extractant, and duration [137].

Studies have been conducted on HHPE of polyphenols from agricultural byproducts (Table 1). Fernandes et al. [136] applied HHPE to extract polyphenols from pansies (*Viola wittrockiana*) using

RSM (Table 2). According to Fernandes et al. [136], it was observed that TPC, tannins, and anthocyanins were 65.1 mg/g, 42.8 mg/g, and 6.15 mg/g, correspondingly, which were 1.8 and 1.4× higher than the control. The study conducted by Alexandre et al. [138] employed HHPE to extract byproducts from figs. Compared to extractions at 0.1 MPa, HHPE led to an 8–13% increase in antioxidant capacity and augmentation in TPC, TFC, and condensed tannins (8–11%). Combining high pressure with extended extraction time and increased ethanol concentration enhanced the yield of various polyphenols obtained from fig byproduct processing. According to Pascal's hypothesis, applying HHP treatment results in immediate and uniform pressure distribution throughout the material, facilitating a rapid, straightforward, and effective extraction method [139]. Moreover, HHPE consistently produced better extraction, up to 35% for TFC at 40% ethanol, 600 MPa, and 30 min. Alexandre et al. [140] demonstrated that using HHPE (30 min, 470 MPa, 55% ethanol) for extracting phenolics from pomegranate peel significantly enhanced extraction yields. The highest flavonoids and tannins were at 492 and 600 MPa, respectively. In contrast, the extraction of anthocyanins reached its maximum at 395 MPa. Xi and Yan [141] observed a comparable outcome when extracting flavonoids from white Flos

Table 2. Agro-byproducts' phenolic extraction using non-thermal Avant-garde technologies.

Extraction	Byproduct	Optimum conditions	Results	Reference
HHPE	Grape Pomace	30 °C, ~268 MPa, and ~3.3 min	The extraction yield was ~56%. Malvidins, which made up 55.77% of the TAC	[142]
	Pansies ( <i>Viola wittrockiana</i> )	15 min and EtOH (35% v/v) and 384 MPa	TPC = 65.1 mg GAE, tannins = 42.8 mg TAE and anthocyanins = 6.15 mg C3G/g	[136]
	Fig byproducts	600 MPa, 18 & 29 min, and EtOH (<15% v/v) except for flavonoids (48% v/v)	An increase of 8–11% of TPC, TFC, and CT than extracts at 0.1 MPa	[138]
	Olive leaves	500 MPa and 5.5 min	Higher TPC (73.6 mg/g) than CE (71.1 mg/g).	[139]
	Flos Sophorae flower	400–534 MPa for 5 min at 25 °C, EtOH (53–80% v/v), and L:S ratio (13–47 mL/g)	TFC was 200 mg QE/g. The content of rutin, kaempferol, quercetin, and genistein was ~368.7, 104.2, 89.2, and 32.3 mg/g, correspondingly.	[141]
UAE	Pomegranate peel	300 and 600 MPa for 5, 17.5, and 30 min at 20 °C with EtOH (0–80% v/v)	TFC increased to 24% at 600 MPa, EtOH (40% v/v), and 17.5 min.	[140]
	Residues of berry press	96% EtOH with 0.5% of TFA, L/S 100:1, 20 min and <30 °C	The yield was ~1.2x more than microwave or Soxhlet. TPC was 15 mg of GAE/g and anthocyanin: 1.4 mg/g.	[152]
	Potato peels	80% Methanol (80%) and L/S of 10:1	TPC was ~4.2 mg of GAE/g	[158]
	Olive leaves	EtOH: H <sub>2</sub> O (1:1 v/v), and 25 °C, 60 min, and L/S 20:1	TPC was ~0.72 mg of GAE/g and TFC was ~0.39 mg of QE/g	[155]
	Vine cans	H <sub>2</sub> O: EtOH 1:1, 20 °C, L/S 50:1 and 60 min	TPC was ~32.6 mg of GAE/g and TFC was ~9.5 mg of EC/g	[149]
	Mandarin peel	80% Acetone, T = 48 °C, and t = 40 min	The extraction yield was ~1.7× more than CE, and TPC was ~160 mg/g	[148]
	Jujube byproduct	EtOH (50.16% v/v), 29 °C, 15.94 min and L:S ratio 34:1 mL/g.	TPC, TFC, and CT were 23.83 mg GAE/g, 4.86 mg QE/g, and 157.10 mg CE/g, respectively	[147]
	Mango peel	EtOH (25% v/v), amplitude 75%, and 30 min.	Ellagic acid augmented up to 2.5×	[145]
	Mango seed	60 μm amplitude and 20 min.	Ellagic acid augmented up to 4.4×	[145]
	Olive pomace	490s, pH: 5.6 at 46 μm amplitude	The maximum extraction yield was 3.6 mg GAE/mL.	[146]
PEF	Coffee silver skin	E = 1.3–4.4 kV/cm = 5–20 μs, N = 500–1000, and f = 50 Hz	Increase in extraction yield of TPC ~1.03–1.21×	[167,205,206,208–211]
	Lemon peel	30 pulses of 30 μs and EF of 7 kV/cm	Hesperidin: 0.84 mg/g, Eriocitrin: 1.76 mg/g	[161]
	Tepals of <i>Crocus sativus</i>	E = 1.2 kV/cm	TPC increased by 44.36%	[168]
	Cocoa bean shell	E = 1.5–3 kV/cm, t <sub>t</sub> = 5–20 μs, and N = 500–1000, and f = 50 Hz	Increase in extraction yield of TPC (~1.01–1.19×),	[167]
	Potato Peels	EtOH (52%), 50 °C and 230 min, EF 25 kV, pulse widths (τ = 3–25 μs), f = 1–450 Hz	PEF samples showed higher TPC (10%)	[162]
	Olive pomace	~1 to 6.5 kV/cm, ~15 μs pulse width, and ~0.9 to 51.1 kJ/kg	Increase in TPC by 91.6% with reduced extraction time	[163]
	Lemon Residues	E = 1–9 kV/cm, U = 0–7.6 kJ/kg, N = 0–100, and t = 3 μs	Increase in extraction yield of TPC (up to 300%)	[161]
	<i>Sideritis raiseri</i> .	Maximum voltage 25 kV	Increase by up to 146% the yield of TPC	[169]
	<i>Sideritis scardica</i>	E = 0.2 kV/cm	TPC increased by 35.25%	[168]
	Pomegranate peels	E = 10 kV/cm	Compared to the US and HVED, PEF is more useful in extraction.	[165]
	Thinned peach byproducts	E = 5 kV/cm	TPC of 0.4 mg GAE/g	[166]
	Olive leaves	25% v/v EtOH:H <sub>2</sub> O, pulse time 2 μs, 0.85 kV/cm, period of 100 μs, and 15 min.	38% increase in TPC extractability with a 117% increase for specific metabolites	[164]
	Apple pomace	U = 17, 100 kJ/kg, E = 2–3 kV/cm, and N = 9, 115	TPC of 181.4–223.5 μg GAE/g	[170]

HVED	Banana peels	Cocoa bean shell	Orange peels	Pomegranate peels	Sage extracts	Gac leaves	Red onion peel	Olive leaves	Tobacco waste	Mandarin Peel	Orange pomace
	t = 70 μs, E = 1.3–6.45 kV/cm, and V = 6.2–40 kV	100 Hz, 30 min, and L/S ratio 47 mL/g	40 kV, L/S ratio of 8:1 mL/g, 50 °C, 0.5 Hz, 222 kJ/kg, and 5 mm electrode distance.	25 °C, H <sub>2</sub> O, 9 kV, 12 mL/min, L/S ratio of 35 mL/g, and 30 min	25 kV, 9 min, and ethanol (33%)	Electric field strength of 4000 kV/m min	L/S ratio of 40 and energy input of 49.37 J/m	Argon, 9 min, 20 kV, and 50% EtOH	47.16 Hz, and 15 min	97.56 Hz and 5.1 min	10 min and 18 Kv
	Increase in TPC (60%) and increase in antioxidant capacity (38%) [171]	Catechin (284.33 mg/kg), gallic acid (219.17 mg/kg) and epicatechin (358.90 mg/kg) [177]	The extraction yield was 0.7% [179]	TPC was 196.7 mg of GAE/g [174]	TPC was 57.21 mg of GAE/g [178]	Higher TPC (48.46 mg/g) than CE (39.90 mg/g) [182]	A steep rise in TPC [175]	The TPC in HVED was 3.2× than CE. [176]	Chlorogenic acid was 3.91 mg/mL [180]	Hesperidin content (366.19 mL/g) [181]	TPC, and extraction yield of 617.76 mg/L, and 64%, correspondingly. [183]

Note: GAE-Gallic acid equivalent, TFC-Total flavonoid acid, TPC-Total phenolic acid, GAE-Gallic acid equivalent, E = Electric field intensity, t<sub>p</sub> = Pulse repetition time, t = Pulse duration, N = Pulse number, t<sub>n</sub> = Inter-train pause, U = Specific energy input, f = Frequency, n = Train number, T = Temperature, V = Voltage, L:S = liquid to solid ratio, HVED = High-voltage electrical discharges, UAE = ultrasound-assisted extraction, HHPE, high hydrostatic pressure extraction, PEF, pulsed electric field, CE, conventional extraction, TAC, total anthocyanin content, EtOH, ethanol, C3G, cyanidin-3-glucoside.

Sophorae buds. The researchers showed that using ethanol (75% v/v), 460 MPa and an extractant-to-sample ratio of 32 mL/g led to the highest TPC (203 mg/g). Comparable results have been stated by Dobrincić et al. [139] on the HHPE of olive leaves' polyphenols and Putnik et al. [142] on grape skin pomace. Due to its capacity to deprotonate charged groups, rupture hydrophobic interactions, and salt bridges in cell membranes, which may result in a higher permeability, HHPE enhanced the extraction yields on grape pomace.

### 5.2. Ultrasound-assisted extraction (UAE)

Ultrasounds are characterized by acoustic wave properties, with frequencies between 20 and 2000 kHz) [47]. Fig. 5B shows a schematic representation of the UAE [31]. The transmission of mechanical energy in an isotropic manner occurs through rapid cycles of compression and rarefaction (Fig. 5B). Nanobubbles are generated at several nucleation sites along the propagation channels due to rapid oscillations in either low or high pressure. The bubbles accumulate energy through a series of compression-rarefaction oscillations until they surpass the resistance threshold and undergo a catastrophic collapse. Therefore, the simultaneous collapse of large vacuum bubbles generates powerful forces of shearing that propagate as microjets and shock waves. This process, known as cavitation in sonochemistry, has been extensively [143,144]. Applying mechanical energy by sonication induces notable chemical and physical alterations in the plant matrix [4]. Collisions disrupt the structural integrity of cellular components such as cell walls and membranes, reducing the size of particles within a given sample. Furthermore, the enhanced surface area resulting from mechanical impacts facilitates the acceleration of solvent entry and the dispersion and mass transfer of polyphenols [4].

Table 2 illustrates the significant works in UAE for byproduct recovery. In Ojeda et al. [145] study, UAE was employed to extract mango peels and seeds' polyphenols. The results showed that the UAE augmented TPC by 33%. Remarkably, mangiferin rose by 53% in the peel; ellagic acid extraction increased by up to 2.5× and 4.4× in the peel and seed extracts, respectively. Comparable findings have been documented regarding the phenolics from olive pomace [146], jujube byproduct [147], mandarin peel [148], vine cans [149], and grape skin [150]. Low-powered UAE on orange peels had 1.8× more polyphenols than maceration and had 64.35 mg/g of hesperidin [148]. Compared to traditional extraction, UAE recovered 1.5× more

flavonoids (nobiletin and tangeretin) from orange peels [151]. In American cranberry residues, the UAE had a greater phenolic extraction than Soxhlet and MAE [152]. The most effective way for extracting phenolics from lime peel was in the UAE, which surpassed MAE by 33% [153]. Comparable findings have been documented on citrus peel [154], olive leaves [155], carob pods [156], pomace of black carrot [157], and potato peels [158].

### 5.3. Pulsed electric field (PEF)

In PEF electrotechnology (Fig. 5C), pulses ranging from 0.14 to 1 kV or even >25 kV are discharged from a few  $\mu$ s to numerous 100s into a closed container with a sample. Electric potentials stress the cell membrane and form irreversible or reversible holes based on time, energy, and pulsation. Once the cell membrane has been decreased, PEF increases electrical permeability, conductivity, and solute diffusion, which speeds up the extraction of polyphenols [159]. For the mechanism of action, electroporation is the predominant action. The complicated cell membrane electroporation procedure produced by PEF considers the cells' size, electro-physical characteristics, the surrounding media's pH, the osmotic agents, and spatial orientation [160]. As a result of the applied PEF, a transmembrane potential differential could lead to membrane rupture across the cell membrane or local structural changes. A physiological and biochemical shift occurs when the transmembrane potential threshold is exceeded, causing the cell membrane to permeabilize irreversibly or temporarily. The effects of electrically induced instabilities and modifications in the energy balance brought on by PEF on cell membranes in terms of membrane cleavage are explained by non-pore and pore models [160].

Table 2 summarizes significant works on PEF application for byproducts recovery. Byproducts, such as lemon peels [161], potato peels [162], olive pomace [163], etc., have all shown improvement in solvent usage and extraction yield and time (Table 2) when PEF was applied. In research by Pappas et al. [164], olive leaves waste phenolics were extracted using PEF, and reported that PEF increased the TPC by 38% compared to the control. PEF increases diffusivity by causing changes in cell permeabilization, thereby causing the migration of polyphenols to an extractant. Similar findings have been reported for PEF extraction of pomegranate peels [165] and thinned peach byproducts [166]. Using PEF to extract phenolics from coffee silver skin and cocoa bean shells (CBS) was researched in

a study by Barbosa-Pereira et al. [167]. The authors stated that using PEF increased TPC in extracts from CBS and coffee silver skin by 1.8–19.5% and 3–21%, correspondingly, compared with CE. Comparable findings have been reported on PEF extraction of lemon peel's polyphenols [161], tepals of *Crocus sativus* [168], olive pomace [163], *Sideritis raiseri* [169], apple pomace [170], banana peels [171] and potato peels [162].

### 5.4. High voltage electrical discharges (HVED)

The reasoning, strategy, and technology used in HVED (Fig. 5D) are comparable to those used in PEF, except that electric discharges happen at a tiny location, making it a viable choice for CE [172]. The two electrodes' high electric fields (EF) (20–40 kV) transmit via the extractant toward the sample (agro byproduct) and through the discharging chamber, electroporating the cell membrane and wall above a certain potential threshold [173]. The genesis of this physical process is the disintegration of H<sub>2</sub>O molecules on an anode-to-cathode electric streamer, which is caused by extreme EF, and it is connected with high cavitation or turbulent shock, Joule heating, and ultraviolet radiation. As a result, this electro-technology is a more efficient means of cell disintegration. Moreover, the process will be accelerated by air bubbles initially found in the H<sub>2</sub>O or created due to local heating.

Table 2 summarizes the significant works on the application of HVED for byproduct recovery. To extract phenolics from pomegranate peel, Xi et al. [174] reported using a continuous HVED extraction system of the “converged electric field type (CEFT).” They compared CEFT to maceration. The TPC of continuous HVED (196.7 mg/g) was significantly higher than maceration (158.9 mg/g). Besides, there was a 23.78% increase in the yield for HVED, which was attributed to improved mass transfer resulting from disruptions of sample cells under electrical breakdown [175]. Comparable findings have been reported on HVED of polyphenols from red onion peel [175], olive leaves [176], cocoa bean shells [177], sage extracts [178], and orange peels [179]. Besides, the effectiveness of the HVED and PEF extractions of phenolics from pomegranate peels has been researched by Rajha et al. [165]. According to the study, HVED was 1.3 and 3× more successful than UAE and PEF techniques for recovering polyphenols. They also demonstrated that, in contrast to UAE, HVED, and CE, the PEF preferentially extracted and improved the extraction of ellagic acid (740 mg/kg), while HVED (345 mg/kg) increased gallic acid extraction. Comparable findings have

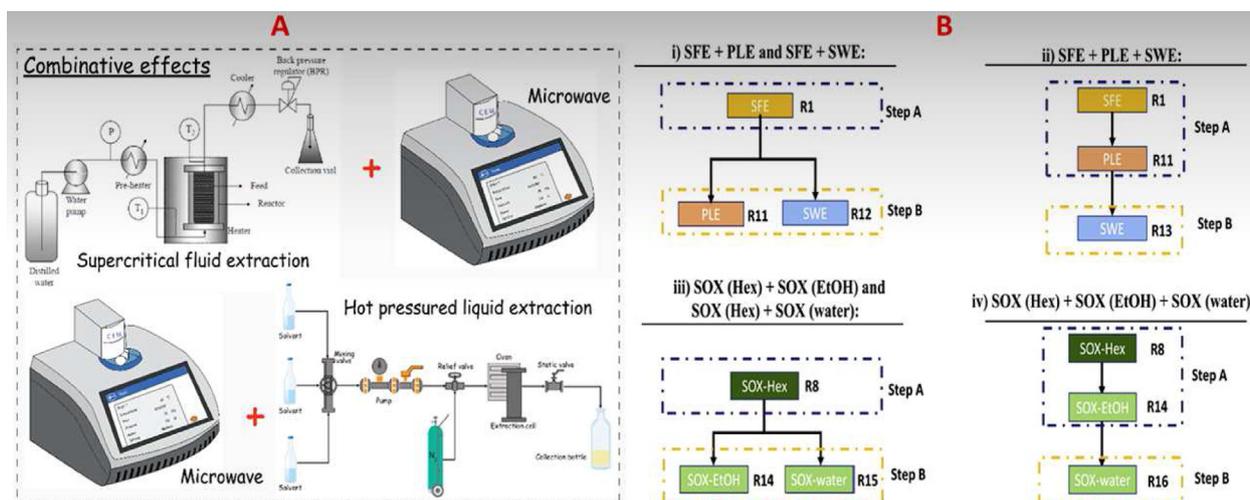


Fig. 6. (A). Combined thermal-thermal extraction of polyphenols (B) consecutive extraction for pink pepper fruits [184].

been reported for tobacco waste [180], mandarin peel [181], Gac leaves [182], and orange pomace [183].

## 6. Integrations of Avant-garde extraction technologies

Due to the growing tendency toward environmentally friendly extractions, the food industry primarily focuses on lowering manufacturing costs by either speeding up the process or improving polyphenolic yield. Nonetheless, there are ways to balance solvent use, product quality, and manufacturing costs, even if no perfect extraction technique depends entirely on the technology applied. Therefore, even if each extraction method described above is efficient, combining many

methods can result in better results [184]. Additionally, this method (Figs. 6–7) enables the purification of priceless phenolic components from various agro-byproducts and the progressive extraction of distinct polyphenols [185]. Therefore, this section presents the potential of combined Avant-garde extraction technologies in phenolic extraction from agro-byproducts.

### 6.1. Thermal-thermal Avant-garde extraction technologies

#### 6.1.1. SCFE-PLE

The SCFE-PLE potential for future applications is extensive, particularly in sequential or in situ extraction processes. Marlou et al. [186] utilized SCFE-PLE to extract polyphenols from the leaves of

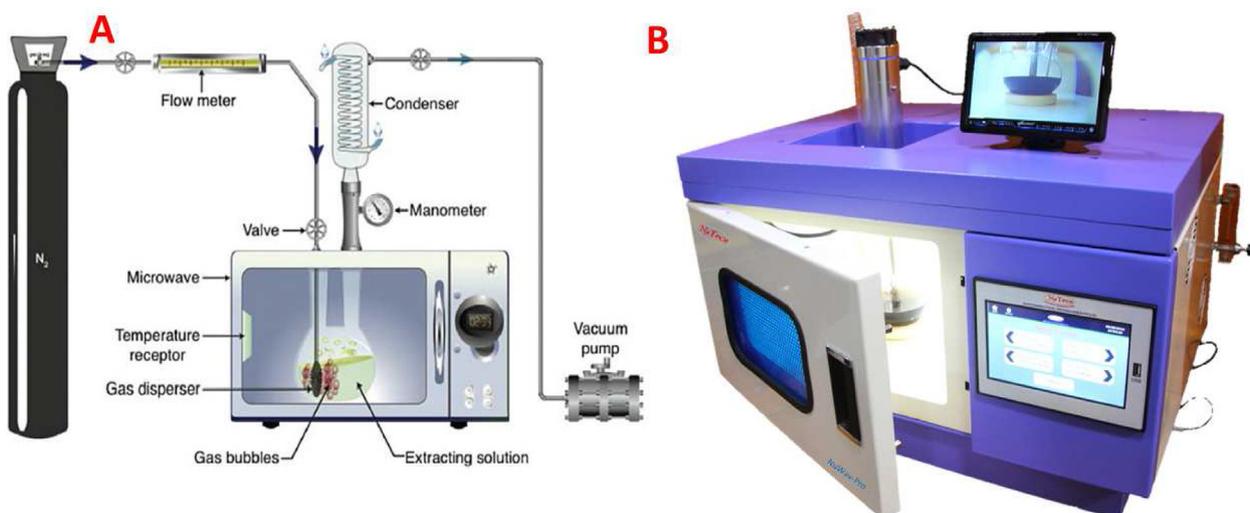


Fig. 7. Combined (A) therma-thermal (PLE + MAE) [185] (B) thermal-non-thermal (MAE-UAE) extraction of polyphenols [203].

*Sida rhombifolia* (Table 3). The maximum TPC of 91 mg GAE/g was accomplished by the PLE method using the SCFE residue and employing a rapid depressurization rate. In addition, 31 polyphenols were found by LC-MS/MS. Compared to extraction methods that use a single phase, the integrated approach, which involves assays in two processes, has shown that process integration leads to improved utilization of raw materials. This is achieved by boosting the efficiency of extraction and recovery of valuable phenolic fractions, such as flavonoids and phenolic acids. The observed results seem to be influenced by two primary factors: the initial stage involving the elimination of lipid and wax components and the subsequent stage characterized by enhanced mass transfer during PLE as a result of cell wall disruption caused by either SCFE depressurization or the utilization of the Soxhlet technique, which impacts the structure of the particles. The present study proposes a viable approach for extracting essential phenolics from the initial product by employing a process integration strategy. The subsequent fraction consists of a second product abundant in polar molecules possessing antioxidant properties. This product is obtained after the initial fraction, and non-polar compounds were extracted using either SCFE or Soxhlet extraction (with hexane) as a subsequent stage. Analogous findings have been reported by Carolina et al. [187] on *Capsicum chinense*, Vardanega et al. [188] on granadilla waste (*Passiflora ligularis*, Juss.), and Teixeira et al. [189] on *Pachira aquatica* seeds.

Mazzutti et al. [78] used SCFE-PLE to extract cocoa bean hull (CBH) phenolics (Table 3). The SCFE-PLE had a higher TPC (44 mg GAE/g) than PLE (9.6 mg GAE/g) and SCFE (4 mg GAE/g). Based on the results obtained, Mazzutti et al. [78] concluded that using SCFE-PLE demonstrates potential as a viable technique for generating two significant fractions derived from CBH. Specifically, this method produces a lipid-enriched extract during the SCFE phase and a phenolic-rich product during the PLE phase. Furthermore, the extract obtained from the second segment had the highest caffeine and theobromine, two compounds of significant bioactivity. Zhang et al. [190] developed HPLE merged with countercurrent chromatography (CCC) and preparative liquid chromatography (PLC) to extract and isolate *Cicer arietinum* seeds' phenolics. The HPLE extraction was conducted using an aqueous ethanol solution with a volume-to-volume ratio of 60%. The extraction process occurred at 80 °C and 5 min. Subsequently, the PLC and CCC sample loops were filled with the HPLE extract. When optimizing the CCC and PLC

separations, the polarity of the phenolics in the HPLE extract was considered. A total of 11 flavonoid-type compounds were potentially extracted using the synergistic use of CCC (countercurrent chromatography) and PLC. Therefore, the novel continuous extraction and online isolation technique can extract phenolics from byproducts.

#### 6.1.2. SCCO<sub>2</sub>-SWE

Integrated SCCO<sub>2</sub> and SWE have been applied to extract phenolics from agro-byproducts. The implementation of sequential SCCO<sub>2</sub>-SWE extraction could yield beneficial outcomes, and it enables the retrieval of the complete range of polyphenols exhibiting diverse polarity. In a study by Banožić et al. [191], tobacco waste was extracted using a combination of SC-CO<sub>2</sub> and SWE. The optimum parameters (Table 3) were employed in the SC-CO<sub>2</sub> extraction process to recover the lipophilic compounds from the tobacco byproduct. The residues underwent a subsequent extraction process using SWE-SC-CO<sub>2</sub> extraction. The hydrophilic fractions of the SWE extracts contained significant amounts of phenolics (chlorogenic acid, 3,4-dihydroxybenzoic acid, neochlorogenic acid, and rutin). The study revealed that the tobacco waste exhibited significant DPPH scavenging activity, ranging from ~77.9 to 93.4%, and possessed TPC ranging from around 8.0%–10.1%. A two-stage extraction process (SC-CO<sub>2</sub> preceded by SWE) can increase the extraction rate because removing fats during SC-CO<sub>2</sub> allows for better dissolution of the other polyphenols in subcritical water. In addition, the polyphenols were significantly higher than those without SC-CO<sub>2</sub>. Anthocyanins extraction from *Hibiscus sabdariffa* L. was conducted utilizing a dual technique approach in a study by Rizkiyah et al. [192]. The initial step involved the utilization of SC-CO<sub>2</sub> to eliminate the outer layer or particle surface of the roselle plant, focusing on recovering polyphenols. This was subsequently followed by the implementation of SWE in the second stage, aimed at extracting anthocyanins. The experimental setup involved the utilization of SC-CO<sub>2</sub> (Table 3), resulting in an extraction yield of 18.20% and a TPC of 80.34 mg/100 g. Furthermore, the optimal conditions for SWE produced a total anthocyanin content (TAC) of 1224.61 mg/100 g (Table 3). To enhance the diffusivity and density of the extractant to extract anthocyanins, an integrated approach using SCCO<sub>2</sub> and SWE was employed, utilizing a solvent with greater pressure. Furthermore, integrated SC-CO<sub>2</sub> and SWE have proven effective in achieving a significant increase in anthocyanin than the previous extraction method.

Table 3. Agro-byproducts' phenolic extraction using combined Avant-garde technologies.

Combined	Extraction	Byproduct	Optimum conditions	Yield/Quantity	Reference
Thermal-thermal	SCFE-PLE	<i>Sida rhombifolia</i> leaves	SFE at 30 MPa and 59.85 °C, proceeded by PLE with EtOH at 79.85 °C and 10 MP and SWE at 129.85 °C and 10 MPa.	PLE had higher TPC (~91 mg/g) from the SFE. 31 phenolics were obtained.	[186]
		Cocoa bean hulls	SCFE (40 °C and 20 MPa) and PLE (70 °C and 10 MPa).	SCFE-PLE had higher TPC (44 mg/g) than SFE (4 mg/g) and PLE (96 mg/g).	[78]
		Biquinho pepper	SCFE- (50 °C and 15 MPa) followed by PLE (65 °C and 10 MPa)	SFE <sub>PLE</sub> led to a lower COM (5316.41 US\$/kg).	[212]
	SCFE-SWE	Granadilla waste	SCFE 40 °C and 20 MPa. PLE 75% EtOH, 70 °C and 10 MPa	SCFE-PLE had a higher TPC (66 mg/g) than UAE (23 mg/g)	[188]
		<i>Pachira aquatica</i> seeds	SCFE 60 °C, 120 min and 30 MPa PLE EtOH (99.5%) at 10 MPa and 60 °C	TPC = 53.66–350.29 mg GAE/100 g	[189]
	SCCO <sub>2</sub> -PLE	Calafate pomace	SC-CO <sub>2</sub> 60 °C, 144.6 min and 35.9 MPa. PLE, 1500 psi, 25 °C, and 5 min.	TPC = 33 mg/g and anthocyanins = 8 mg/g	[213]
		Cranberry pomace	SCFE at ~42 MPa, 53 °C, and 158 min, ~55.9% (PLE-EtOH at 83 °C, using 3 cycles, 15 min each) of EtOH-soluble extract and 6.50% (PLE-H <sub>2</sub> O at 130 °C, 3 cycles, 10 min each) of H <sub>2</sub> O-soluble extracts.	Proanthocyanidins and anthocyanins were ~532.2 and 0.42 mg/g, correspondingly.	[214]
	SCCO <sub>2</sub> -SWE	Tobacco waste	SC-CO <sub>2</sub> , 61.2 °C and 30 MPa SWE, 150 °C, 23 min and L/S ratio: 28 mL/g.	neochlorogenic acid (0.24–0.53%), Chlorogenic acid (0.46–0.74%), and rutin (0–1.025%),	[191]
		<i>Hibiscus sabdariffa</i>	SC-CO <sub>2</sub> (1st stage) was 19.13 MPa, 60 °C, and 4.31 mL/min. SWE (2nd stage) was 137 °C, 6.14 mL/min and 9.48 MPa.	This technique yielded an anthocyanin content of 1224.61 mg/100 g.	[192]
	SCCO <sub>2</sub> -PLE-SWE	Pink pepper fruit	SCFE 59.9 °C and at 30 MPa PLE with EtOH at 59.85 °C and 10 MPa. SWE at 10 MPa and 129.85 °C.	91.97 mg/g produced by SWE in a single phase to 163.13 mg/g for SFE + PLE + SWE.	[184]
	MAE-SCCO <sub>2</sub>	Roselle seed	SCFE 1.64 MPa and 158 °C MAE 300 W	Low TFC (6.4524 mg QE/g) and Higher TPC (18.2244 mg GAE/g).	[194]
		Mango peel	50 g/mL, 90 s, and 800 W	The TPC (52.08 mg/g) and antioxidant activities (2.75 mmol TE./g)	[195]
	MAE-SWE	Black rice straw and bran	S/L ratio (5% (w/v)), 90 °C and 5 s	The anthocyanin content was 62.8 mg/100 g	[196]
Non-thermal-non-thermal	HHPE + UAE	Pomegranate peel	UAE, 70 °C, 480 W and 3 cycles and 10 MPa	TPC for HHPE + UAE 61.72 mg GAE/g was higher than the control.	[10]
		Tomato waste	HHPE; nitric acid (0.1 mol/L), 80 °C, 300 MPa and 10–45 min UAE: 400 W, 30 kHz, and amplitude of 95%.	Higher TPC than the conventional method	[199]
		Pomegranate peel	UAE 400 and 600 W merged an initial expansion gas pressure (0.5 and 1 MPa)	An increase in extraction yield by more than 20% with respect to the control.	[198]
		Defatted passion fruit bagasse	PLE; EtOH: H <sub>2</sub> O (75% v/v/v), 10.0 MPa. UAE; 240, 440, and 640 W at 65 °C.	PLE + UAE had 60% more TPC and piceatannol than the control.	[200]

(continued on next page)

Table 3. (continued)

Combined	Extraction	Byproduct	Optimum conditions	Yield/Quantity	Reference
Thermal-non-thermal	PEF + UAE	Gac leaves	HHPE; 300 MPa, 25 °C. for 3 min UAE; 20 min, 150 W and 25 °C	The TPC for HHPE + UAE (52.81 mg/g) was higher than the control (39.90 mg/g).	[182]
		Passion fruit rinds	60 °C, 360 W/cm <sup>2</sup> 10 MPa, with a flow rate of 10 g/min	100% of TPC was extracted	[215]
		Acorns	Cellulase, 3.44 g/L of the enzyme at 38 °C for 2.51 h, with a power of 97.92 W	Increase in extraction yield of 63.16%	[216]
		Grape Stems	PEF: The pulse was 1 ms and 1 Hz UAE; 35 kHz and 320 W	PEF-UAE increased the TPC (35%) than CE.	[202]
		Rosemary byproduct	Pulse at 1.1 kV/cm, pulses of 30 µs, and 12.48 min. 0.1% NaCl (1: 1.4 w/v for rosemary).	UAE + PEF (2.97 mg GAE/g) was 1.5× higher than UAE alone	[201]
	HVED + UAE	Gac leaves	HVED: E = 4000 kV/m min. UAE; 20 min, 150 W and 25 °C	Highest TPC (52 mg/g), TFC (6.2 mg QE/g),	[182]
	MAE-UAE	<i>Satureja macrostema</i>	MAE 40 °C and 500 W	The TPC in MAE-UAE (134 mg/g) was higher than the reflux extraction	[207]
		<i>Larix decidua</i> bark	Ultrasound 40 kHz and 50 W	The TPC in MAE-UAE (~596 mg GAE/g) was higher than CE (~567 mg GAE/g)	[206]
		<i>Clinacanthus nutans</i>	Time (~120 s), MW power (~300 W), and ultrasonic amplitude (99.68%).	TPC and TFC were ~8.8 mg GAE/g and ~25 9 mg QE/g, respectively.	[209]
		Jackfruit peels	Distilled H <sub>2</sub> O, S/L of 1: 55 g/mL, 90 W, and extraction cycle with 75 S.	Higher TPC (8.14 mg/g) and chlorogenic acid (2.53 mg/g).	[205]
Sorghum husk		EtOH (63% v/v), L/S ratio 34 mL/g, 160 W and 20 min	MAE-UAE extraction yield was 3.6× higher than CE. MAE-UAE had higher luteolinidin and apigeninidin.	[208]	
HPLE-UAE	<i>Osmanthus fragrans</i> flowers	20 min, and 55 °C using ultrasonic power (360 W) and MW power (14–33 W). Pulsation was 2s:2s pulsed-on: off time.	TFC = 7.86 mg QE/g	[217]	
	Passion fruit bagasse	EtOH (48.15% v/v), MW time (6.43 min), US time (10 min), and power (370.9 W). HPLE = 65–75 °C UAE = 240–260 W	HPLE-UAE led to a 60% increase in the thermolabile polyphenols	[200].	

## Note

TPC-Total phenolic acid, GAE-Gallic acid equivalent, TFC-Total flavonoid acid, SCFE, supercritical fluid extraction, HPLE, hot pressurized liquid extraction, SCCO<sub>2</sub>, supercritical CO<sub>2</sub> extraction, MAE, microwave-assisted extraction, SWE, subcritical water extraction, UAE, ultrasound-assisted extraction, EtOH, ethanol.

In Rebelatto et al. [184] study, the authors employed an integrated SCFE, HPLE, and SWE to extract polyphenols from pink pepper fruits (Fig. 6B). Following a rapid decrease in pressure, the findings indicated that the HPLE achieved the highest TPC (~91 mg/g) from the SCFE residue. In addition, 31 polyphenols were detected. The present research indicates that integrating Avant-garde extraction technologies is a highly advantageous approach for acquiring two separate fractions (recovered extracts) with specific characteristics: a lipid-rich fraction and a phenolic-rich fraction. The observed outcome may be attributed to the structural alterations imposed on the agro-byproducts during the first extraction process, facilitating the retrieval of the targeted polyphenols. Analogous outcomes have been stated by Vandepoosele et al. [193] that the coupling of supercritical CO<sub>2</sub> and H<sub>2</sub>O had a caffeine extraction of ~60%.

### 6.1.3. MAE with other Avant-garde extraction technologies

Yusoff and Leo [194] studied the phenolic extraction from roselle seeds under MAE using subcritical water as the solvent. Without temperature control, the solvent had subcritical conditions (1.64 MPa and 158 °C), leading to higher polyphenols' (~18.2 mg GAE/g) after 300 W and 10 min. In Sánchez-Camargo et al. [195] research, SCCO<sub>2</sub>-MAE was employed to extract mango peels' phenolics (Table 3). The maximum TPC (52.08 mg GAE/g) was reported. The utilization of this sequential strategy led to the production of extracts at a faster rate and with higher specificity for the extraction of crucial compounds compared to CE. Furthermore, this technique achieved these outcomes while employing reduced solvent. Furthermore, the application of RSM effectively assessed the MAE utilizing depleted biomass derived from SCFE. The study revealed that microwave power and liquid content significantly influence the antioxidant capacity and TPC in relation to the solid-to-liquid ratio. A similar finding was reported by Moirangthem et al. [196] that integrated MAE-SWE from the straw had extraction efficiency for anthocyanins at 85.8%. Furthermore, SCCO<sub>2</sub>-MAE had a higher antioxidant activity than CE. Despite the findings above, research on the application of MAE-SCFE/SWE in separation science is still in its early stages, indicating its potential for future advancements in this field.

## 6.2. Non-thermal-non-thermal Avant-garde extraction technologies

### 6.2.1. Integrated HHPE + UAE

Low-frequency UAE can be integrated into HHPE in various ways, including 1. Pretreatment in

ultrasound devices separate from high-pressure extraction apparatus, 2. Ultrasonically pretreatment inside the extractor by linking the extraction vessel to the ultrasound, and sonication in pulses or while the HHPE equipment is running. Various methods for coupling the UAE device to the extraction vessel include configurations that place the extraction vessel into the ultrasound bath and connect the ultrasonic probe to the extraction vessel [197].

Santos et al. [198] used expansion gas in high-pressure H<sub>2</sub>O to optimize cavitation using UAE (Table 3) and stated an increase in extraction yield by more than 20%. Comparable results have been stated by Ninčević Grassino et al. [199] on tomato waste, as shown in Table 3. According to the authors, sequential extraction of tomato peel' phenolics can be accomplished by combining HHPE and UAE with more established ones (Soxhlet). Furthermore, reducing the extraction time with HHPE + UAE makes it possible to effectively substitute CE and produce a higher concentration of polyphenols. The extraction approach suggested in this work may offer two crucial advantages: producers may discover a way to reduce waste disposal costs, and consumers may seize the chance to reintroduce separated components into food. Analogous results have been documented by Nguyen et al. [182] in which they reported that TPC in HHPE-UAE (52.81 mg/g) was higher than UAE (48.97 mg/g), HHPE (45.69 mg/g), and CE (39.90 mg/g).

Viganó et al. [200] employed the dynamic pressurized liquid extraction (PLE) approach to examine how process factors affected the extraction of phenolics from passion fruit bagasse. The research was done in an extraction unit that included an extraction cell connected to an ultrasonic probe and an HPLC pump that continuously pumped a pressured solvent during the extraction. The authors assessed the effect of temperature and ultrasonic power on extraction yields (Table 3). The findings revealed that PLE assisted by ultrasound augmented the polyphenolic yields, leading to 60% more TPC and piceatannol. The polyphenolic yields imply that the primary mechanism driving PLE assisted by the ultrasound from defatted passion fruit bagasse was the increase in temperature instigated by the ultrasonic waves. This demonstrates that PLE with ultrasound assistance has a high prospect for enhancing byproducts polyphenols extraction. Nevertheless, additional cost analysis is needed to prove the economic sustainability of the proposed techniques.

### 6.2.2. PEF + UAE or HVED + UAE

Görgüç et al. [201] applied PEF + UAE to extract polyphenols from the Rosemary byproduct (Table 3)

and reported that UAE + PEF (2.97 mg GAE/g) was 1.5× higher than UAE alone. Likewise, Ntourtoglou et al. [202] extracted polyphenols from grape stems (Table 3) and reported that PEF-UAE augmented the TPC extraction (35%) compared to the control (PEF alone). This could be explained by the combinative effect of the electroporation by (PEF) and the cavitation effect by ultrasound that cleaves the cell membrane and allows polyphenols to be released, thus increasing the TPC yield. Nguyen et al. [182] extracted polyphenols from Gac leaves using HVED + UAE and observed that the highest TPC (52 mg GAE/g), TFC (6.2 mg QE/g) was found in the HVED + UAE compared to UAE, conventional extraction, and HVED. The combinative effect of ultrasound's disruption of cell walls in UAE and the rearrangement of the electric ion in HVED increased polyphenols' extraction.

### 6.3. Thermal and non-thermal combination

It is important to acknowledge that studies have combined thermal and non-thermal Avant-garde extraction technologies (Fig. 7). For instance, a combination of MAE with UAE has been applied to enhance the extraction of polyphenols [203]. Utilizing MAE-UAE has demonstrated a synergistic impact on enhancing extraction yields while reducing expenses (Table 3). Trujillo-Mayol et al. [204] documented the extraction of avocado peels using UAE-MAE. The combination technique yielded the highest TPC of 281 mg GAE/g. Additionally, this technique demonstrated high efficacy (TPC of 281 mg GAE/g), with MAE and UAE achieving a TPC of 275 mg GAE/g and 270 mg GAE/g, respectively. According to the study conducted by Trujillo-Mayol et al. [204], it was observed that MAE's economic viability is enhanced when energy prices are elevated. Simultaneously, the combined effect of UAE-MAE was more efficient for byproducts with higher costs. In a study conducted by Jha et al. [130], it was found that the combination of UAE for 10 min and MAE for 31 s increased the TFC of milled black rice husk (30 µg/g). Additionally, the TPC increased to ~1.7 mg GAE/g, and the concentration of anthocyanins reached ~34 µg/g.

The efficient polyphenols extraction from jackfruit peels was achieved via a sequential MAE-UAE extraction process, as demonstrated by Jiang et al. [205]. The researchers observed an increase in the phenolic purity of the extract from 13.59% to 49.07%, indicating its potential as a valuable source of powerful antioxidant activity. According to the findings of this research, it was recommended that the utilization of jackfruit peel be considered to scale

up a batch adsorption–desorption unit on a large scale. This would facilitate the production of antioxidant phenolics from the peels. Applying the HPLC technique with UAE (65–75 °C and 240–260 W) resulted in a notable 60% increase in the thermolabile polyphenols extraction from passion fruit bagasse [200]. Analogous findings have been documented on phenolic extraction from *Larix decidua* bark [206], *Satureja macrostema* [207], sorghum husk [208], and *Clinacanthus nutans* [209].

## 7. Conclusion and future studies

Avant-garde extraction methods exhibit enhanced efficiency, potential cost-effectiveness, and scalability. Despite the progress made in this field, achieving the ideal practicality, specificity, and efficiency levels in plant byproduct extraction remains a significant challenge that has yet to be overcome. Based on the information above, it is preferable to achieve a harmonious equilibrium between complete restoration and minor alteration of the polyphenolic native structure throughout the recovery process. Even with notable technological advancements, the most effective methodology for extracting specific polyphenols continues to be impacted by the characteristics of the agro-byproduct, the desired phenolic compounds, and the inherent structural associations. Hence, further investigation is required before the widespread recognition and appreciation of byproducts.

Moreover, using innovative extraction techniques, specifically the combinative effects approach, can enhance extraction yields within a reduced timeframe, enhance the quality of the resulting product, and mitigate environmental concerns. There is an increasing trend of integrating novel extraction approaches in contemporary research. In contrast to CE, Avant-garde extractions yield higher polyphenolic yield. The incorporation of advanced technology has the potential to improve the progress of biologically active extraction processes. Numerous advantages are associated with this phenomenon, encompassing swiftness, convenience, and security. However, it is important to establish a pilot program for comprehensive industrial extraction first. Subsequently, utilizing advanced technologies requires laboratory-scale research to demonstrate integrated efficiency. This, in turn, calls for collaboration among academia, government, and business partners in a multidisciplinary approach to leverage their respective competencies. Besides, acknowledging that implementing an integrated process and the sequential application of diverse Avant-

garde extraction technologies hold significant significance is crucial. The differential use of the integrated process, including MAE and UAE, to extract phenolic compounds from agro-byproducts should be distinct from the sequential execution of UAE followed by MAE for the same objective. Therefore, further investigation is required on sequencing and its potential impact on extracting phenolic chemicals from agricultural wastes.

## Conflicts of interest

No conflict of interest.

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## References

- [1] Alexandre EMC, Moreira SA, Castro LMG, Pintado M, Saraiva JA. Emerging technologies to extract high added value compounds from fruit residues: sub/supercritical, ultrasound-, and enzyme-assisted extractions. *Food Rev Int* 2018;34:581–612. <https://doi.org/10.1080/87559129.2017.1359842>.
- [2] Martillanes S, Rocha-Pimienta J, Delgado- Adámez J. Agrifood by-products as a source of phytochemical compounds. *Descr Food Sci* 2018. <https://doi.org/10.5772/intechopen.79434>.
- [3] Gil-Martín E, Forbes-Hernández T, Romero A, Cianciosi D, Giampieri F, Battino M. Influence of the extraction method on the recovery of bioactive phenolic compounds from food industry by-products. *Food Chem* 2022;378. <https://doi.org/10.1016/j.foodchem.2021.131918>.
- [4] Dzah CS, Duan Y, Zhang H, Wen C, Zhang J, Chen G, et al. The effects of ultrasound assisted extraction on yield, antioxidant, anticancer and antimicrobial activity of polyphenol extracts: a review. *Food Biosci* 2020;35:100547. <https://doi.org/10.1016/j.fbio.2020.100547>.
- [5] Barba FJ, Zhu Z, Koubaa M, Sant'Ana AS, Orlie V. Green alternative methods for the extraction of antioxidant bioactive compounds from winery wastes and by-products: a review. *Trends Food Sci Technol* 2016;49:96–109. <https://doi.org/10.1016/j.tifs.2016.01.006>.
- [6] Carciochi RA, D'Alessandro LG, Vauchel P, Rodriguez MM, Nolasco SM, Dimitrov K. Valorization of agrifood by-products by extracting valuable bioactive compounds using green processes. Elsevier Inc.; 2017. <https://doi.org/10.1016/b978-0-12-811521-3.00004-1>.
- [7] Dzah CS, Duan Y, Zhang H, Authur DA, Ma H. 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* 2020;137:109598. <https://doi.org/10.1016/j.foodres.2020.109598>.
- [8] Boateng ID, Mustapha A, Daubert CR, Kuehnel L, Kumar R, Flint-Garcia S, et al. Novel two-pot microwave extraction of purple corn pericarp's phenolics and evaluation of the polyphenol-rich extract's product quality, bioactivities, and structural properties. *Food Bioprocess Technol* 2023. <https://doi.org/10.1007/s11947-023-03072-7>.
- [9] Moreno E, Reza J, Trejo A. Extraction of polycyclic aromatic hydrocarbons from soil using water under subcritical conditions. *Polycycl Aromat Comp* 2007;27:239–60. <https://doi.org/10.1080/10406630701462916>.
- [10] Sumere BR, de Souza MC, dos Santos MP, Bezerra RMN, da Cunha DT, Martinez J, et al. Combining pressurized liquids with ultrasound to improve the extraction of phenolic compounds from pomegranate peel (*Punica granatum L.*). *Ultrason Sonochem* 2018;48:151–62. <https://doi.org/10.1016/j.ultsonch.2018.05.028>.
- [11] Santos-Zea L, Gutiérrez-Urbe JA, Benedito J. Effect of ultrasound intensification on the supercritical fluid extraction of phytochemicals from Agave salmiana bagasse. *J Supercrit Fluids* 2019;144:98–107. <https://doi.org/10.1016/j.supflu.2018.10.013>.
- [12] Alara O, Abdurahman NH, Ukaegbu CI. Extraction of phenolic compounds: a review. *Curr Res Food Sci* 2021;4:200–14. <https://doi.org/10.1016/j.crf.2021.03.011>.
- [13] Dzah CS, Duan Y, Zhang H, Serwah Boateng NA, Ma H. Latest developments in polyphenol recovery and purification from plant by-products: a review. *Trends Food Sci Technol* 2020;99:375–88. <https://doi.org/10.1016/j.tifs.2020.03.003>.
- [14] Ji D, Wang Q, Lu T, Ma H, Chen X. The effects of ultrasonication on the phytochemicals, antioxidant, and polyphenol oxidase and peroxidase activities in coffee leaves. *Food Chem* 2022;373:131480. <https://doi.org/10.1016/j.foodchem.2021.131480>.
- [15] Reynoso-Camacho R, Rodríguez-Villanueva LD, Sotelo-González AM, Ramos-Gómez M, Pérez-Ramírez IF. Citrus decoction by-product represents a rich source of carotenoid, phytosterol, extractable and non-extractable polyphenols. *Food Chem* 2021:350. <https://doi.org/10.1016/j.foodchem.2021.129239>.
- [16] Valente IM, Maia MRG, Malushi N, Oliveira HM, Papa L, Rodrigues JA, et al. Profiling of phenolic compounds and antioxidant properties of European varieties and cultivars of Vicia faba L. pods. *Phytochemistry* 2018;152:223–9. <https://doi.org/10.1016/j.phytochem.2018.05.011>.
- [17] Rusu ME, Fizeşan I, Pop A, Gheldiu AM, Mocan A, Crişan G, et al. Enhanced recovery of antioxidant compounds from hazelnut (*Corylus avellana L.*) involucre based on extraction optimization: phytochemical profile and biological activities. *Antioxidants* 2019;8:1–27. <https://doi.org/10.3390/antiox8100460>.
- [18] Alim A, Li T, Nisar T, Ren D, Liu Y, Yang X. Consumption of two whole kiwifruit (*Actinide chinensis*) per day improves lipid homeostasis, fatty acid metabolism and gut microbiota in healthy rats. *Int J Biol Macromol* 2020;156:186–95. <https://doi.org/10.1016/j.ijbiomac.2020.04.028>.
- [19] Amaya-Cruz DM, Pérez-Ramírez IF, Delgado-García J, Mondragón-Jacobo C, Dector-Espinoza A, Reynoso-Camacho R. An integral profile of bioactive compounds and functional properties of prickly pear (*Opuntia ficus indica L.*) peel with different tonalities. *Food Chem* 2019;278:568–78. <https://doi.org/10.1016/j.foodchem.2018.11.031>.
- [20] Chaves JO, de Souza MC, da Silva LC, Lachos-Perez D, Torres-Mayanga PC, Machado AP da F, et al. Extraction of flavonoids from natural sources using modern techniques. *Front Chem* 2020;8. <https://doi.org/10.3389/fchem.2020.507887>.
- [21] Mallek-Ayadi S, Bahloul N, Kechaou N. Characterization, phenolic compounds and functional properties of Cucumis melo L. peels. *Food Chem* 2017;221:1691–7. <https://doi.org/10.1016/j.foodchem.2016.10.117>.
- [22] Figueroa JG, Borrás-Linares I, Lozano-Sánchez J, Segura-Carretero A. Comprehensive identification of bioactive compounds of avocado peel by liquid chromatography coupled to ultra-high-definition accurate-mass Q-TOF. *Food Chem* 2018;245:707–16. <https://doi.org/10.1016/j.foodchem.2017.12.011>.
- [23] Ž Mrkonjić, Rakić D, Kaplan M, Teslić N, Zeković Z, Pavlić B. Pressurized-liquid extraction as an efficient method for valorization of thymus serpyllum herbal dust

towards sustainable production of antioxidants. *Molecules* 2021;26. <https://doi.org/10.3390/molecules26092548>.

- [24] Ferri M, Vannini M, Ehrnell M, Eliasson L, Xanthakis E, Monari S, et al. From winery waste to bioactive compounds and new polymeric biocomposites: a contribution to the circular economy concept. *J Adv Res* 2020;24:1–11. <https://doi.org/10.1016/j.jare.2020.02.015>.
- [25] Franco D, Rodríguez-Amado I, Agregán R, Munekata PES, Vázquez JA, Barba FJ, et al. Optimization of antioxidants extraction from peanut skin to prevent oxidative processes during soybean oil storage. *LWT–Food Sci Technol* 2018;88:1–8. <https://doi.org/10.1016/j.lwt.2017.09.027>.
- [26] Gómez-Mejía E, Rosales-Conrado N, León-González ME, Madrid Y. Citrus peels waste as a source of value-added compounds: extraction and quantification of bioactive polyphenols. *Food Chem* 2019;295:289–99. <https://doi.org/10.1016/j.foodchem.2019.05.136>.
- [27] Caldeira ASP, Mbiakop UC, Pádua RM, van de Venter M, Matsabisa MG, Campana PRV, et al. Bioguided chemical characterization of pequi (*Caryocar brasiliense*) fruit peels towards an anti-diabetic activity. *Food Chem* 2021;345:1–10. <https://doi.org/10.1016/j.foodchem.2020.128734>.
- [28] Saptarini NM, Wardati Y. Effect of extraction methods on antioxidant activity of papery skin extracts and fractions of maja cipanas onion (*Allium cepa* L. Var. *ascalonicum*). *Sci World J* 2020;2020. <https://doi.org/10.1155/2020/3280534>.
- [29] Kato-Schwartz CG, Corrêa RCG, de Souza Lima D, de Sá-Nakanishi AB, de Almeida Gonçalves G, Seixas FAV, et al. Potential anti-diabetic properties of Merlot grape pomace extract: an in vitro, in silico and in vivo study of  $\alpha$ -amylase and  $\alpha$ -glucosidase inhibition. *Food Res Int* 2020;137:109462. <https://doi.org/10.1016/j.foodres.2020.109462>.
- [30] Wajs-Bonikowska A, Stobiecka A, Bonikowski R, Krajewska A, Sikora M, Kula J. A comparative study on composition and antioxidant activities of supercritical carbon dioxide, hexane and ethanol extracts from blackberry (*Rubus fruticosus*) growing in Poland. *J Sci Food Agric* 2017;97:3576–83. <https://doi.org/10.1002/jsfa.8216>.
- [31] Boateng ID, Kumar R, Daubert CR, Flint-Garcia S, Mustapha A, Kuehnel L, et al. Sonoprocessing improves phenolics profile, antioxidant capacity, structure, and product qualities of purple corn pericarp extract. *Ultrason Sonochem* 2023;95:106418. <https://doi.org/10.1016/j.ultrasonch.2023.106418>.
- [32] Boateng ID, Mustapha A, Kuehnel L, Daubert CR, Kumar R, Agliata J, et al. From purple corn waste (pericarp) to polyphenol-rich extract with higher bioactive contents and superior product qualities using two-step optimization techniques. *Ind Crop Prod* 2023;200:116871. <https://doi.org/10.1016/j.indcrop.2023.116871>.
- [33] Lao F, Giusti MM. Extraction of purple corn (*Zea mays* L.) cob pigments and phenolic compounds using food-friendly solvents. *J Cereal Sci* 2018;80:87–93. <https://doi.org/10.1016/j.jcs.2018.01.001>.
- [34] Ongkowitzo P, Luna-Vital DA, Gonzalez de Mejia E. Extraction techniques and analysis of anthocyanins from food sources by mass spectrometry: an update. *Food Chem* 2018;250:113–26. <https://doi.org/10.1016/j.foodchem.2018.01.055>.
- [35] Dzah CS, Duan Y, Zhang H, Golly MK, Ma H. Enhanced screening of key ultrasonication parameters: total phenol content and antioxidant activity assessment of Tartary buckwheat (*Fagopyrum tataricum*) water extract. *Separ Sci Technol* 2019;00:1–10. <https://doi.org/10.1080/01496395.2019.1675704>.
- [36] Ballesteros-Vivas D, Álvarez-Rivera G, Morantes SJ, Sánchez-Camargo A del P, Ibáñez E, Parada-Alfonso F, et al. An integrated approach for the valorization of mango seed kernel: efficient extraction solvent selection, phytochemical profiling and antiproliferative activity assessment. *Food Res Int* 2019;126:108616. <https://doi.org/10.1016/j.foodres.2019.108616>.
- [37] Akpabli-Tsigbe NDK, Ma Y, Ekumah JN, Osabutey J, Hu J, Xu M, et al. Two-step optimization of solid-state fermentation conditions of heilong48 soybean variety for maximum chlorogenic acid extraction yield with improved antioxidant activity. *Ind Crops Prod* 2021;168:113565. <https://doi.org/10.1016/j.indcrop.2021.113565>.
- [38] Dzah CS. Optimized ultrasound-assisted recovery, HPLC/LC-MS identification and biological activities of *Tetrapleura tetraptera* L. dry fruit polyphenols. *Food Chem Adv* 2022:100093. <https://doi.org/10.1016/j.focha.2022.100093>.
- [39] Boateng ID, Yang XM. Process optimization of intermediate-wave infrared drying: screening by Plackett–Burman; comparison of Box–Behnken and central composite design and evaluation: a case study. *Ind Crops Prod* 2021;162:113287. <https://doi.org/10.1016/j.indcrop.2021.113287>.
- [40] Fakayode OA, Aboagarib EAA, Yan D, Li M, Wahia H, Mustapha AT, et al. Novel two-pot approach ultrasonication and deep eutectic solvent pretreatments for watermelon rind delignification: parametric screening and optimization via response surface methodology. *Energy* 2020;203. <https://doi.org/10.1016/j.energy.2020.117872>.
- [41] Mai X, Liu Y, Tang X, Wang L, Lin Y, Zeng H, et al. Sequential extraction and enrichment of flavonoids from *Euonymus alatus* by ultrasonic-assisted polyethylene glycol-based extraction coupled to temperature-induced cloud point extraction. *Ultrason Sonochem* 2020;66:105073. <https://doi.org/10.1016/j.ultrasonch.2020.105073>.
- [42] Motikar PD, More PR, Arya SS. A novel, green environment-friendly cloud point extraction of polyphenols from pomegranate peels: a comparative assessment with ultrasound and microwave-assisted extraction. *Separ Sci Technol* 2021;56:1014–25. <https://doi.org/10.1080/01496395.2020.1746969>.
- [43] Soquetta MB, Tonato D, Quadros MM, Boeira CP, Cichoski AJ, de Marsillac Terra L, et al. Ultrasound extraction of bioactive compounds from Citrus reticulata peel using electrolyzed water. *J Food Process Preserv* 2019;43:1–9. <https://doi.org/10.1111/jfpp.14236>.
- [44] Çiğeroğlu Z, Körbaşlar İ, Şahin S, Köprücü G. Optimization and kinetic studies of ultrasound-assisted extraction on polyphenols from Satsuma Mandarin (*Citrus Unshiu* Marc.) leaves. *Iran J Chem Chem Eng* 2017;36:163–71.
- [45] Dzah CS, Duan Y, Zhang H, Ma H. Effects of pretreatment and type of hydrolysis on the composition, antioxidant potential and HepG2 cytotoxicity of bound polyphenols from Tartary buckwheat (*Fagopyrum tataricum* L. Gaerth) hulls. *Food Res Int* 2021;142:110187. <https://doi.org/10.1016/j.foodres.2021.110187>.
- [46] Dzah CS. Optimized pressurized hot water extraction, HPLC/LC-MS characterization, and bioactivity of *Tetrapleura tetraptera* L. dry fruit polyphenols. *J Food Sci* 2023;88:175–92. <https://doi.org/10.1111/1750-3841.16422>.
- [47] Chen X, Ding J, Ji D, He S, Ma H. Optimization of ultrasonic-assisted extraction conditions for bioactive components from coffee leaves using the Taguchi design and response surface methodology. *J Food Sci* 2020;85:1742–51. <https://doi.org/10.1111/1750-3841.15111>.
- [48] Herrman DA, Brantsen JF, Ravisankar S, Lee KM, Awika JM. Stability of 3-deoxyanthocyanin pigment structure relative to anthocyanins from grains under microwave assisted extraction. *Food Chem* 2020;333:127494. <https://doi.org/10.1016/j.foodchem.2020.127494>.
- [49] Jafari SM, Mahdavee Khazaei K, Assadpour E. Production of a natural color through microwave-assisted extraction of saffron tepal's anthocyanins. *Food Sci Nutr* 2019;7:1438–45. <https://doi.org/10.1002/fsn3.978>.
- [50] Oancea S. A review of the current knowledge of thermal stability of anthocyanins and approaches to their stabilization to heat. *Antioxidants* 2021;10. <https://doi.org/10.3390/antiox10091337>.
- [51] Maillard MN, Berset C. Evolution of antioxidant activity during kilning: role of insoluble bound phenolic acids of

- barley and malt. *J Agric Food Chem* 1995;43:1789–93. <https://doi.org/10.1021/jf00055a008>.
- [52] Weremfo A, Adulley F, Adarkwah-Yiadom M. Simultaneous optimization of microwave-assisted extraction of phenolic compounds and antioxidant activity of avocado (*persea americana* mill.) seeds using response surface methodology. *J Anal Methods Chem* 2020;2020. <https://doi.org/10.1155/2020/7541927>.
- [53] Boateng ID, Yang XM, Li YY. Optimization of infrared-drying parameters for Ginkgo biloba L. seed and evaluation of product quality and bioactivity. *Ind Crops Prod* 2020;160:113108. <https://doi.org/10.1016/j.indcrop.2020.113108>.
- [54] Paulo F, Santos L. Deriving valorization of phenolic compounds from olive oil by-products for food applications through microencapsulation approaches: a comprehensive review. *Crit Rev Food Sci Nutr* 2021;61:920–45. <https://doi.org/10.1080/10408398.2020.1748563>.
- [55] Nani M, Krishnaswamy K. Circular economy for food industry waste: development and characterization of spray-dried acid whey encapsulated in millet matrix. *ACS Food Sci Technol* 2023;3:123–33. <https://doi.org/10.1021/acscfoodscitech.2c00326>.
- [56] Mirón C, Sánchez R, Prats S, Todolí JL. Total polyphenol content and metals determination in Spanish virgin olive oils by means of a dispersive liquid-liquid aerosol phase extraction method and ICP-MS. *Anal Chim Acta* 2020;1094:34–46. <https://doi.org/10.1016/j.aca.2019.10.009>.
- [57] Boateng ID, Zhang W, Li Y, Saalia FK, Yang X. Non-thermal pretreatment affects Ginkgo biloba L. seed's product qualities, sensory, and physicochemical properties. *J Food Sci* 2022;1–18. <https://doi.org/10.1111/1750-3841.15999>.
- [58] Birtic S, Régis S, Le Bourvellec C, Renard CMGC. Impact of air-drying on polyphenol extractability from apple pomace. *Food Chem* 2019;296:142–9. <https://doi.org/10.1016/j.foodchem.2019.05.131>.
- [59] Boateng ID, Yang X. Thermal and non-thermal processing affect Maillard reaction products, flavor, and phytochemical profiles of Ginkgo biloba seed. *Food Biosci* 2021;41:101044. <https://doi.org/10.1016/j.fbio.2021.101044>.
- [60] Chen XM, Ma Z, Kitts DD. Effects of processing method and age of leaves on phytochemical profiles and bioactivity of coffee leaves. *Food Chem* 2018;249:143–53. <https://doi.org/10.1016/j.foodchem.2017.12.073>.
- [61] Difonzo G, Aresta A, Cotugno P, Ragni R, Squeo G, Summo C, et al. Supercritical CO<sub>2</sub> extraction of phytochemicals from olive pomace subjected to different drying methods. *Molecules* 2021;26. <https://doi.org/10.3390/molecules26030598>.
- [62] Bitwell C, Indra S Sen, Luke C, Kakoma MK. A review of modern and conventional extraction techniques and their applications for extracting phytochemicals from plants. *Sci African* 2023;19:e01585. <https://doi.org/10.1016/j.sciaf.2023.e01585>.
- [63] Cittan M, Altuntaş E, Çelik A. Evaluation of antioxidant capacities and phenolic profiles in *Tilia cordata* fruit extracts: a comparative study to determine the efficiency of traditional hot water infusion method. *Ind Crops Prod* 2018;122:553–8. <https://doi.org/10.1016/j.indcrop.2018.06.044>.
- [64] Sridhar A, Ponnuchamy M, Kumar PS, Kapoor A, Vo DVN, Prabhakar S. Techniques and modeling of polyphenol extraction from food: a review, vol. 19. Springer International Publishing; 2021. <https://doi.org/10.1007/s10311-021-01217-8>.
- [65] Paz JEW, Contreras CR, Munguía AR, Aguilar CN, Inungaray MLC. Phenolic content and antibacterial activity of extracts of *Hamelia patens* obtained by different extraction methods. *Braz J Microbiol* 2018;49:656–61. <https://doi.org/10.1016/j.bjm.2017.03.018>.
- [66] Mahmudati N, Wahyono P, Djunaedi D. Antioxidant activity and total phenolic content of three varieties of Ginger (*Zingiber officinale*) in decoction and infusion extraction method. *J Phys Conf Ser* 2020;1567:4–10. <https://doi.org/10.1088/1742-6596/1567/2/022028>.
- [67] Perera PRD, Ekanayake S, Ranaweera KKDS. Antidiabetic compounds in *Syzygium cumini* decoction and ready to serve herbal drink. *Evidence-Based Complement Altern Med* 2017;2017. <https://doi.org/10.1155/2017/1083589>.
- [68] Mahindrakar KV, Rathod VK. Ultrasonic assisted aqueous extraction of catechin and gallic acid from *Syzygium cumini* seed kernel and evaluation of total phenolic, flavonoid contents and antioxidant activity. *Chem Eng Process - Process Intensif* 2020;149:107841. <https://doi.org/10.1016/j.cep.2020.107841>.
- [69] Shirsath SR, Sable SS, Gaikwad SG, Sonawane SH, Saini DR, Gogate PR. Intensification of extraction of curcumin from *Curcuma amada* using ultrasound assisted approach: effect of different operating parameters. *Ultrason Sonochem* 2017;38:437–45. <https://doi.org/10.1016/j.ulsonch.2017.03.040>.
- [70] Shewale S, Rathod VK. Extraction of total phenolic content from *Azadirachta indica* or (neem) leaves: kinetics study. *Prep Biochem Biotechnol* 2018;48:312–20. <https://doi.org/10.1080/10826068.2018.1431784>.
- [71] Čujić N, Šavikin K, Janković T, Pljevljakušić D, Zdunić G, Ibrić S. Optimization of polyphenols extraction from dried chokeberry using maceration as traditional technique. *Food Chem* 2016;194:135–42. <https://doi.org/10.1016/j.foodchem.2015.08.008>.
- [72] Shi J. Microwave-assisted extraction of flavonoids from *Cajanus cajan* leaves. *Chin Tradit Herb Drugs* 2011;24:2235–9.
- [73] Dassoff ES, Li YO. Mechanisms and effects of ultrasound-assisted supercritical CO<sub>2</sub> extraction. *Trends Food Sci Technol* 2019;86:492–501. <https://doi.org/10.1016/j.tifs.2019.03.001>.
- [74] Wrona O, Rafińska K, Możeński C, Buszewski B. Supercritical fluid extraction of bioactive compounds from plant materials. *J AOAC Int* 2017;100:1624–35. <https://doi.org/10.5740/jaoacint.17-0232>.
- [75] Pimentel-Moral S, Borrás-Linares I, Lozano-Sánchez J, Arráez-Román D, Martínez-Férez A, Segura-Carretero A. Supercritical CO<sub>2</sub> extraction of bioactive compounds from *Hibiscus sabdariffa*. *J Supercrit Fluids* 2019;147:213–21. <https://doi.org/10.1016/j.supflu.2018.11.005>.
- [76] Becze A, Babalau-Fuss VL, Varaticeanu C, Roman C. Optimization of high-pressure extraction process of antioxidant compounds from *feteasca regala* leaves using response surface methodology. *Molecules* 2020;25:1–14. <https://doi.org/10.3390/molecules25184209>.
- [77] Da Porto C, Natolino A. Supercritical fluid extraction of polyphenols from grape seed (*Vitis vinifera*): study on process variables and kinetics. *J Supercrit Fluids* 2017;130:239–45. <https://doi.org/10.1016/j.supflu.2017.02.013>.
- [78] Mazzutti S, Rodrigues LGG, Mezzomo N, Venturi V, Ferreira SRS. Integrated green-based processes using supercritical CO<sub>2</sub> and pressurized ethanol applied to recover antioxidant compounds from cocoa (*Theobroma cacao*) bean hulls. *J Supercrit Fluids* 2018;135:52–9. <https://doi.org/10.1016/j.supflu.2017.12.039>.
- [79] Campone L, Celano R, Piccinelli AL, Pagano I, Carabetta S, Sanzo R Di, et al. Response surface methodology to optimize supercritical carbon dioxide/co-solvent extraction of brown onion skin by-product as source of nutraceutical compounds. *Food Chem* 2018;269:495–502. <https://doi.org/10.1016/j.foodchem.2018.07.042>.
- [80] Maravić N, Teslić N, Nikolić D, Dimić I, Šereš Z, Pavlić B. From agricultural waste to antioxidant-rich extracts: green techniques in extraction of polyphenols from sugar beet leaves. *Sustain Chem Pharm* 2022;28:100728. <https://doi.org/10.1016/J.SCP.2022.100728>.
- [81] Melloul S, Zehioua R, Meniai AH. Supercritical CO<sub>2</sub> extraction of bioactive compounds from local *Peganum Harmala* plant seeds and optimization of the extraction yield and the antioxidant activities. *Sustain Chem Pharm* 2022;28:100729. <https://doi.org/10.1016/J.SCP.2022.100729>.

- [82] Espinosa-Pardo FA, Nakajima VM, Macedo GA, Macedo JA, Martínez J. Extraction of phenolic compounds from dry and fermented orange pomace using supercritical CO<sub>2</sub> and cosolvents. *Food Bioprod Process* 2017;101:1–10. <https://doi.org/10.1016/j.fbp.2016.10.002>.
- [83] Wenzel J, Wang L, Horcasitas S, Warburton A, Constine S, Kjellson A, et al. Influence of supercritical fluid extraction parameters in preparation of black chokeberry extracts on total phenolic content and cellular viability. *Food Sci Nutr* 2020;8:3626–37. <https://doi.org/10.1002/fsn3.1645>.
- [84] Rajapaksha S, Shimizu N. Pilot-scale extraction of polyphenols from spent black tea by semi-continuous subcritical solvent extraction. *Food Chem X* 2022;13:100200. <https://doi.org/10.1016/j.fochx.2021.100200>.
- [85] Rajapaksha DSW, Shimizu N. Valorization of spent black tea by recovery of antioxidant polyphenolic compounds: subcritical solvent extraction and microencapsulation. *Food Sci Nutr* 2020;8:4297–307. <https://doi.org/10.1002/fsn3.1726>.
- [86] Walters M, Lima Ribeiro AP, Hosseinian F, Tsopmo A. Phenolic acids, avenanthramides, and antioxidant activity of oats defatted with hexane or supercritical fluid. *J Cereal Sci* 2018;79:21–6. <https://doi.org/10.1016/j.jcs.2017.09.010>.
- [87] Chai YH, Yusup S, Kadir WNA, Wong CY, Rosli SS, Ruslan MSH, et al. Valorization of tropical biomass waste by supercritical fluid extraction technology. *Sustain Times* 2021;13:1–24. <https://doi.org/10.3390/su13010233>.
- [88] Araújo MN, Azevedo AQPL, Hamerski F, Voll FAP, Corazza ML. Enhanced extraction of spent coffee grounds oil using high-pressure CO<sub>2</sub> plus ethanol solvents. *Ind Crops Prod* 2019;141:111723. <https://doi.org/10.1016/j.indcrop.2019.111723>.
- [89] Aryal S, Baniya MK, Danekhu K, Kunwar P, Gurung R, Koirala N. Total Phenolic content, Flavonoid content and antioxidant potential of wild vegetables from western Nepal. *Plants* 2019;vol. 8. <https://doi.org/10.3390/plants8040096>.
- [90] Essien SO, Young B, Baroutian S. Recent advances in subcritical water and supercritical carbon dioxide extraction of bioactive compounds from plant materials. *Trends Food Sci Technol* 2020;97:156–69. <https://doi.org/10.1016/j.tifs.2020.01.014>.
- [91] Luo X, Cui J, Zhang H, Duan Y. Subcritical water extraction of polyphenolic compounds from sorghum (*Sorghum bicolor* L.) bran and their biological activities. *Food Chem* 2018;262:14–20. <https://doi.org/10.1016/j.foodchem.2018.04.073>.
- [92] Zhang J, Wen C, Zhang H, Duan Y, Ma H. Recent advances in the extraction of bioactive compounds with subcritical water: a review. *Trends Food Sci Technol* 2020;95:183–95. <https://doi.org/10.1016/j.tifs.2019.11.018>.
- [93] Ibrahim S, Santos R, Bowra S. Optimization of subcritical water mediated extraction of apple pomace polyphenolics and their antioxidant activity. *J Chromatogr Separ Tech* 2018;9. <https://doi.org/10.4172/2157-7064.1000410>.
- [94] Cai Z, Qu Z, Lan Y, Zhao S, Ma X, Wan Q, et al. Conventional, ultrasound-assisted, and accelerated-solvent extractions of anthocyanins from purple sweet potatoes. *Food Chem* 2016;197:266–72. <https://doi.org/10.1016/j.foodchem.2015.10.110>.
- [95] Yammine S, Delsart C, Vitrac X, Peuchot MM, Ghidossi R. Characterisation of polyphenols and antioxidant potential of red and white pomace by-product extracts using subcritical water extraction. *Oeno One* 2020;54:263–78. <https://doi.org/10.20870/oeno-one.2020.54.2.2346>.
- [96] Mikucka W, Zielinska M, Bulkowska K, Witonska I. Subcritical water extraction of bioactive phenolic compounds from distillery stillage. *J Environ Manag* 2022;318:115548. <https://doi.org/10.1016/j.jenvman.2022.115548>.
- [97] Zeković Z, Kaplan M, Pavlič B, Olgun EO, Vladić J, Canli O, et al. Chemical characterization of polyphenols and volatile fraction of coriander (*Coriandrum sativum* L.) extracts obtained by subcritical water extraction. *Ind Crops Prod* 2017;87:54–63. <https://doi.org/10.1016/j.indcrop.2016.04.024>.
- [98] Pavlič B, Vidović S, Vladić J, Radosavljević R, Cindrić M, Zeković Z. Subcritical water extraction of sage (*Salvia officinalis* L.) by-products - process optimization by response surface methodology. *J Supercrit Fluids* 2017;116:36–45. <https://doi.org/10.1016/j.supflu.2016.04.005>.
- [99] Naffati A, Vladić J, Pavlic B, Radosavljevic R, Gavarić A, Vidović S. Recycling of filter tea industry by-products: application of subcritical water extraction for recovery of bioactive compounds from *A. uva-ursi* herbal dust. *J Supercrit Fluids* 2017;121. <https://doi.org/10.1016/j.supflu.2016.11.010>.
- [100] Pedras BM, Nascimento M, Sá-Nogueira I, Simões P, Paiva A, Barreiros S. Semi-continuous extraction/hydrolysis of spent coffee grounds with subcritical water. *J Ind Eng Chem* 2019;72:453–6. <https://doi.org/10.1016/j.jiec.2019.01.001>.
- [101] Mayanga-Torres PC, Lachos-Perez D, Rezende CA, Prado JM, Ma Z, Tompsett GT, et al. Valorization of coffee industry residues by subcritical water hydrolysis: recovery of sugars and phenolic compounds. *J Supercrit Fluids* 2017;120:75–85. <https://doi.org/10.1016/j.supflu.2016.10.015>.
- [102] Vladić J, Jakovljević M, Molnar M, Vidović S, Tomić M, Drinić Z, et al. Valorization of yarrow (*Achillea millefolium* L.) by-product through application of subcritical water extraction. *Molecules* 2020;25. <https://doi.org/10.3390/molecules25081878>.
- [103] Munir MT, Kheirkhah H, Baroutian S, Quek SY, Young BR. Subcritical water extraction of bioactive compounds from waste onion skin. *J Clean Prod* 2018;183:487–94. <https://doi.org/10.1016/j.jclepro.2018.02.166>.
- [104] Kheirkhah H, Baroutian S, Quek SY. Evaluation of bioactive compounds extracted from Hayward kiwifruit pomace by subcritical water extraction. *Food Bioprod Process* 2019;115:143–53. <https://doi.org/10.1016/j.fbp.2019.03.007>.
- [105] Gonçalves Rodrigues LG, Mazzutti S, Vitali L, Micke GA, Ferreira SRS. Recovery of bioactive phenolic compounds from papaya seeds agroindustrial residue using subcritical water extraction. *Biocatal Agric Biotechnol* 2019;22:101367. <https://doi.org/10.1016/j.cbac.2019.101367>.
- [106] Barriga-Sánchez M, Rosales-Hartshorn M. Effects of subcritical water extraction and cultivar geographical location on the phenolic compounds and antioxidant capacity of Quebranta (*Vitis vinifera*) grape seeds from the Peruvian pisco industry by-product. *Food Sci Technol* 2022;42. <https://doi.org/10.1590/FST.107321>.
- [107] Getachew AT, Chun BS. Influence of pretreatment and modifiers on subcritical water liquefaction of spent coffee grounds: a green waste valorization approach. *J Clean Prod* 2017;142:3719–27. <https://doi.org/10.1016/j.jclepro.2016.10.096>.
- [108] Pinto D, Vieira EF, Peixoto AF, Freire C, Freitas V, Costa P, et al. Optimizing the extraction of phenolic antioxidants from chestnut shells by subcritical water extraction using response surface methodology. *Food Chem* 2021;334:127521. <https://doi.org/10.1016/j.foodchem.2020.127521>.
- [109] Uwineza PA, Waśkiewicz A. Recent advances in supercritical fluid extraction of natural bioactive compounds from natural plant materials. *Molecules* 2020;25. <https://doi.org/10.3390/molecules25173847>.
- [110] Wijngaard H, Hossain MB, Rai DK, Brunton N. Techniques to extract bioactive compounds from food by-products of plant origin. *Food Res Int* 2012;46:505–13. <https://doi.org/10.1016/j.foodres.2011.09.027>.
- [111] Nastić N, Lozano-Sánchez J, Borrás-Linares I, Švarc-Gajić J, Segura-Carretero A. New technological approaches for recovering bioactive food constituents from sweet cherry (*Prunus avium* L.) stems. *Phytochem Anal* 2020;31:119–30. <https://doi.org/10.1002/pca.2872>.
- [112] Shang YF, Xu JL, Lee WJ, Um BH. Antioxidative polyphenolics obtained from spent coffee grounds by pressurized liquid extraction. *South African J Bot* 2017;109:75–80. <https://doi.org/10.1016/j.sajb.2016.12.011>.

- [113] Cea Pavez I, Lozano-Sánchez J, Borrás-Linares I, Nuñez H, Robert P, Segura-Carretero A. Obtaining an extract rich in phenolic compounds from olive pomace by pressurized liquid extraction. *Molecules* 2019;24:1–17.
- [114] Battistella Lasta HF, Lentz L, Gonçalves Rodrigues LG, Mezzomo N, Vitali L, Salvador Ferreira SR. Pressurized liquid extraction applied for the recovery of phenolic compounds from beetroot waste. *Biocatal Agric Biotechnol* 2019;21. <https://doi.org/10.1016/j.bcab.2019.101353>.
- [115] Chamali S, Bendaoud H, Bouajila J, Camy S, Saadaoui E, Condoret J-S, et al. Optimization of accelerated solvent extraction of bioactive compounds from Eucalyptus intertexta using response surface methodology and evaluation of its phenolic composition and biological activities. *J Appl Res Med Aromat Plants* 2023;100464. <https://doi.org/10.1016/j.JARMAP.2023.100464>.
- [116] Barrales FM, Silveira P, Barbosa P de PM, Ruviano AR, Paulino BN, Pastore GM, et al. Recovery of phenolic compounds from citrus by-products using pressurized liquids — an application to orange peel. *Food Bioprod Process* 2018;112:9–21. <https://doi.org/10.1016/j.fbp.2018.08.006>.
- [117] García P, Fredes C, Cea I, Lozano-Sánchez J, Leyva-Jiménez FJ, Robert P, et al. Recovery of bioactive compounds from pomegranate (*punica granatum L.*) peel using pressurized liquid extraction. *Foods* 2021;9. <https://doi.org/10.3390/iecbm2020-08582>.
- [118] Allcca-Alca EE, León-Calvo NC, Luque-Vilca OM, Martínez-Cifuentes M, Pérez-Correa JR, Mariotti-Celis MS, et al. Hot pressurized liquid extraction of polyphenols from the skin and seeds of *vitis vinifera L.* Cv. negra criolla pomace a peruvian native pisco industry waste. *Agronomy* 2021;11:0–9. <https://doi.org/10.3390/agronomy11050866>.
- [119] Rivera-tovar PR, Dolores M, Camilo C, Salom M, Domínguez H, Ricardo P. Multi-response optimal hot pressurized liquid recovery of extractable polyphenols from leaves of maqui (*Aristotelia chilensis* [Mol] Stuntz). *Food Chem* 2021;357. <https://doi.org/10.1016/j.foodchem.2021.129729>.
- [120] Pereira DTV, Tarone AG, Cazarin CBB, Barbero GF, Martínez J. Pressurized liquid extraction of bioactive compounds from grape marc. *J Food Eng* 2019;240:105–13. <https://doi.org/10.1016/j.jfoodeng.2018.07.019>.
- [121] Huamán-Castilla NL, Campos D, García-Ríos D, Parada J, Martínez-Cifuentes M, Mariotti-Celis MS, et al. Chemical properties of *vitis vinifera* carménère pomace extracts obtained by hot pressurized liquid extraction, and their inhibitory effect on type 2 diabetes mellitus related enzymes. *Antioxidants* 2021;10:1–14. <https://doi.org/10.3390/antiox10030472>.
- [122] Tapia-Quirós P, Montenegro-Landívar MF, Reig M, Vecino X, Alvarino T, Cortina JL, et al. Olive mill and winery wastes as viable sources of bioactive compounds: a study on polyphenols recovery. *Antioxidants* 2020;9:1–15. <https://doi.org/10.3390/antiox9111074>.
- [123] Cassol L, Rodrigues E, Zapata Noreña CP. Extracting phenolic compounds from *Hibiscus sabdariffa L.* calyx using microwave assisted extraction. *Ind Crops Prod* 2019;133:168–77. <https://doi.org/10.1016/j.indcrop.2019.03.023>.
- [124] Silva AM, Pinto D, Fernandes I, de Freitas V, Cádiz-Gurrea M de la L, Costa P, et al. An insight into kiwiberry leaf valorization: phenolic composition, bioactivity and health benefits. *Molecules* 2021;26:1–15. <https://doi.org/10.3390/molecules26082314>.
- [125] Kumar M, Dahuja A, Sachdev A, Kaur C, Varghese E, Saha S, et al. Valorisation of black carrot pomace: microwave assisted extraction of bioactive phytochemicals and antioxidant activity using Box–Behnken design. *J Food Sci Technol* 2019;56:995–1007. <https://doi.org/10.1007/s13197-018-03566-9>.
- [126] Petrotos K, Giavasis I, Gerasopoulos K, Mitsagga C, Papaioannou C, Gkoutosidis P. Optimization of the vacuum microwave assisted extraction of the natural polyphenols and flavonoids from the raw solid waste of the pomegranate juice producing industry at industrial scale. *Molecules* 2021;26. <https://doi.org/10.3390/molecules26041033>.
- [127] Skenderidis P, Leontopoulos S, Petrotos K, Giavasis I. Optimization of vacuum microwave-assisted extraction of pomegranate fruits peels by the evaluation of extracts' phenolic content and antioxidant activity. *Foods* 2020;9. <https://doi.org/10.3390/foods9111655>.
- [128] Garrido T, Gizdavic-Nikolaidis M, Leceta I, Urdanpilleta M, Guerrero P, de la Caba K, et al. Optimizing the extraction process of natural antioxidants from chardonnay grape marc using microwave-assisted extraction. *Waste Manag* 2019;88:110–7. <https://doi.org/10.1016/j.wasman.2019.03.031>.
- [129] Pavlič B, Naffati A, Hojan T, Vradić J, Zeković Z, Vidović S. Microwave-assisted extraction of wild apple fruit dust—production of polyphenol-rich extracts from filter tea factory by-products. *J Food Process Eng* 2017;40:1–11. <https://doi.org/10.1111/jfpe.12508>.
- [130] Jha P, Das AJ, Deka SC. Optimization of ultrasound and microwave assisted extractions of polyphenols from black rice (*Oryza sativa* cv. Poiréton) husk. *J Food Sci Technol* 2017;54:3847–58. <https://doi.org/10.1007/s13197-017-2832-0>.
- [131] Tomasi IT, Santos SCR, Boaventura RAR, Botelho MS. Optimization of microwave-assisted extraction of phenolic compounds from chestnut processing waste using response surface methodology. *J Clean Prod* 2023;395. <https://doi.org/10.1016/j.jclepro.2023.136452>.
- [132] Jovanovi AA, Vaji UV, Brankovi S. Polyphenol extraction in microwave reactor using by-product of *Thymus serpyllum L.* and biological potential of the extract. *J Appl Res Med Aromat Plants* 2022;31. <https://doi.org/10.1016/j.jarmp.2022.100417>.
- [133] Arboleda Meija JA, Parpinello GP, Versari A, Conidi C, Cassano A. Microwave-assisted extraction and membrane-based separation of biophenols from red wine lees. *Food Bioprod Process* 2019;117:74–83. <https://doi.org/10.1016/j.fbp.2019.06.020>.
- [134] Baiano A, Romaniello R, Giametta F, Fiore A. Optimization of process variables for the sustainable extraction of phenolic compounds from chicory and Fennel by-products. *Appl Sci* 2023;13. <https://doi.org/10.3390/app13074191>.
- [135] Scepankova H, Martins M, Estevinho L, Delgado I, Saraiva JA. Enhancement of bioactivity of natural extracts by non-thermal high hydrostatic pressure extraction. *Plant Foods Hum Nutr* 2018;73:253–67. <https://doi.org/10.1007/s11130-018-0687-9>.
- [136] Fernandes L, Casal SIP, Pereira JA, Ramalhosa E, Saraiva JA. Optimization of high pressure bioactive compounds extraction from pansies (*Viola × wittrockiana*) by response surface methodology. *High Press Res* 2017;37:415–29. <https://doi.org/10.1080/08957959.2017.1347925>.
- [137] Khan SA, Aslam R, Makroo HA. High pressure extraction and its application in the extraction of bio-active compounds: a review. *J Food Process Eng* 2019;42:1–15. <https://doi.org/10.1111/jfpe.12896>.
- [138] Alexandre EMC, Araújo P, Duarte MF, de Freitas V, Pintado M, Saraiva JA. High-pressure assisted extraction of bioactive compounds from industrial fermented fig by-product. *J Food Sci Technol* 2017;54:2519–31. <https://doi.org/10.1007/s13197-017-2697-2>.
- [139] Dobrinčić A, Repajic M, Elez Garofulić I, Tuden L, Dragović-Uzelac V, Levaj B. Comparison of different extraction methods for the recovery of olive leaves polyphenols. *Processes* 2020;8:1008.
- [140] Alexandre EMC, Araújo P, Duarte MF, de Freitas V, Pintado M, Saraiva JA. Experimental design, modeling, and optimization of high-pressure-assisted extraction of bioactive compounds from pomegranate peel. *Food Bioprocess Technol* 2017;10:886–900. <https://doi.org/10.1007/s11947-017-1867-6>.
- [141] Xi J, Yan L. Optimization of pressure-enhanced solid-liquid extraction of flavonoids from *Flos Sophorae* and evaluation

- of their antioxidant activity. *Sep Purif Technol* 2017;175:170–6. <https://doi.org/10.1016/j.seppur.2016.10.013>.
- [142] Putnik P, Bursać Kovacević D, Ježek D, Sustić I, Zorić Z, Dragović-Uzelac V. High-pressure recovery of anthocyanins from grape skin pomace (*Vitis vinifera* cv. Teran) at moderate temperature. *J Food Process Preserv* 2018;42:1–11. <https://doi.org/10.1111/jfpp.13342>.
- [143] Alolga RN, Osae R, Essilfie G, Saalia FK, Akaba S, Chikari F. Sonication, osmosonication and vacuum-assisted osmosonication pretreatment of Ghanaian garlic slices: effect on physicochemical properties and quality characteristics. *Food Chem* 2021;343:128535. <https://doi.org/10.1016/j.foodchem.2020.128535>.
- [144] Zhang W, Boateng ID, Zhang W, Jia S, Wang T, Huang L. Effect of ultrasound-assisted ionic liquid pretreatment on the structure and interfacial properties of soy protein isolate. *Process Biochem* 2022;115:160–8. <https://doi.org/10.1016/j.procbio.2022.02.015>.
- [145] Ojeda GA, Sgroppo SC, Sánchez Moreno C, de Ancos Sigüero B. Mango ‘criollo’ by-products as a source of polyphenols with antioxidant capacity. Ultrasound assisted extraction evaluated by response surface methodology and HPLC-ESI-QTOF-MS/MS characterization. *Food Chem* 2022;396. <https://doi.org/10.1016/j.foodchem.2022.133738>.
- [146] Ó Rodríguez, Bona S, Stäbler A, Rodríguez-Turiénzo L. Ultrasound-assisted extraction of polyphenols from olive pomace: scale up from laboratory to pilot scenario. *Processes* 2022;10. <https://doi.org/10.3390/pr10122481>.
- [147] Berkani F, Serralheiro ML, Dahmoune F, Ressaissi A, Kadri N, Remini H. Ultrasound assisted extraction of phenolic compounds from a jujube by-product with valuable bioactivities. *Processes* 2020;8:1–17. <https://doi.org/10.3390/pr8111441>.
- [148] Nipornram S, Tochampa W, Rattanatruiwong P, Singanusong R. Optimization of low power ultrasound-assisted extraction of phenolic compounds from Mandarin (*Citrus reticulata* Blanco cv. Sainampung) peel. *Food Chem* 2018. <https://doi.org/10.1016/j.foodchem.2017.08.114>.
- [149] Dorosh O, Moreira MM, Rodrigues F, Peixoto AF, Freire C, Morais S, et al. Vine-canes valorisation: ultrasound-assisted extraction from lab to pilot scale. *Molecules* 2020;25. <https://doi.org/10.3390/molecules25071739>.
- [150] Gerardi C, D’amico L, Migoni D, Santino A, Salomone A, Carluccio MA, et al. Strategies for reuse of skins separated from grape pomace as ingredient of functional beverages. *Front Bioeng Biotechnol* 2020;8:1–13. <https://doi.org/10.3389/fbioe.2020.00645>.
- [151] Wang Q, Qin X, Liang Z, Li S, Cai J, Zhu Z, et al. HPLC–DAD–ESI–MS2 analysis of phytochemicals from Sichuan red orange peel using ultrasound-assisted extraction. *Food Biosci* 2018;25:15–20. <https://doi.org/10.1016/j.fbio.2018.07.001>.
- [152] Klavins L, Kviessis J, Nakurte I, Klavins M. Berry press residues as a valuable source of polyphenolics: extraction optimisation and analysis. *Lwt* 2018;93:583–91. <https://doi.org/10.1016/j.lwt.2018.04.021>.
- [153] Rodsamran P, Sothornvit R. Extraction of phenolic compounds from lime peel waste using ultrasonic-assisted and microwave-assisted extractions. *Food Biosci* 2019;28:66–73. <https://doi.org/10.1016/j.fbio.2019.01.017>.
- [154] Papoutsis K, Pristijono P, Golding JB, Stathopoulos CE, Bowyer MC, Scarlett CJ, et al. Screening the effect of four ultrasound-assisted extraction parameters on hesperidin and phenolic acid content of aqueous citrus pomace extracts. Elsevier Ltd; 2018. <https://doi.org/10.1016/j.fbio.2017.11.001>.
- [155] Cedola A, Palermo C, Centonze D, Nobile MA Del, Conte A. Characterization and bio-accessibility evaluation of olive leaf extract-enriched “Taralli”. *Foods* 2020;9. <https://doi.org/10.3390/foods9091268>.
- [156] Clodoveo ML, Crupi P, Muraglia M, Corbo F. Ultrasound assisted extraction of polyphenols from ripe carob pods (*Ceratonia siliqua* L.): combined designs for screening and optimizing the processing parameters. *Foods* 2022;11. <https://doi.org/10.3390/foods11030284>.
- [157] Agcam E, Akyıldız A, Balasubramaniam VM. Optimization of anthocyanins extraction from black carrot pomace with thermosonication. *Food Chem* 2017;237:461–70. <https://doi.org/10.1016/j.foodchem.2017.05.098>.
- [158] Kumari B, Tiwari BK, Hossain MB, Rai DK, Brunton NP. Ultrasound-assisted extraction of polyphenols from potato peels: profiling and kinetic modelling. *Int J Food Sci Technol* 2017;52:1432–9. <https://doi.org/10.1111/ijfs.13404>.
- [159] Rybak K, Wiktor A, Witrowa-Rajchert D, Parniakov O, Nowacka M. The effect of traditional and non-thermal treatments on the bioactive compounds and sugars content of red bell pepper. *Molecules* 2020;25:1–17. <https://doi.org/10.3390/molecules25184287>.
- [160] Ranjha MMAN, Kanwal R, Shafique B, Arshad RN, Irfan S, Kieliszek M, et al. A critical review on pulsed electric field: a novel technology for the extraction of phytoconstituents. *Molecules* 2021;26:1–23. <https://doi.org/10.3390/molecules26164893>.
- [161] Peiró S, Luengo E, Segovia F, Raso J, Almajano MP. Improving polyphenol extraction from lemon residues by pulsed electric fields. *Waste and Biomass Valorization* 2019;10:889–97. <https://doi.org/10.1007/s12649-017-0116-6>.
- [162] Frontuto D, Carullo D, Harrison SM, Brunton NP, Ferrari G, Lyng JG, et al. Optimization of pulsed electric fields-assisted extraction of polyphenols from potato peels using response surface methodology. *Food Bioprocess Technol* 2019;12:1708–20. <https://doi.org/10.1007/s11947-019-02320-z>.
- [163] Andreou V, Psarianos M, Dimopoulos G, Tsimogiannis D, Taoukis P. Effect of pulsed electric fields and high pressure on improved recovery of high-added-value compounds from olive pomace. *J Food Sci* 2020;85:1500–12. <https://doi.org/10.1111/1750-3841.15122>.
- [164] Pappas VM, Lakka A, Palaiogiannis D, Athanasiadis V, Bozinou E, Ntourtoglou G, et al. Optimization of pulsed electric field as standalone “green” extraction procedure for the recovery of high value-added compounds from fresh olive leaves. *Antioxidants* 2021;10. <https://doi.org/10.3390/antiox10101554>.
- [165] Rajha HN, Abi-Khattar AM, El Kantar S, Boussetta N, Lebovka N, Maroun RG, et al. Comparison of aqueous extraction efficiency and biological activities of polyphenols from pomegranate peels assisted by infrared, ultrasound, pulsed electric fields and high-voltage electrical discharges. *Innovat Food Sci Emerg Technol* 2019;58:102212. <https://doi.org/10.1016/j.ifset.2019.102212>.
- [166] Redondo D, Venturini ME, Luengo E, Raso J, Arias E. Pulsed electric fields as a green technology for the extraction of bioactive compounds from thinned peach by-products. *Innovat Food Sci Emerg Technol* 2018;45:335–43. <https://doi.org/10.1016/j.ifset.2017.12.004>.
- [167] Barbosa-Pereira L, Guglielmetti A, Zeppa G. Pulsed electric field assisted extraction of bioactive compounds from cocoa bean shell and coffee silverskin. *Food Bioprocess Technol* 2018;11:818–35. <https://doi.org/10.1007/s11947-017-2045-6>.
- [168] Lakka A, Bozinou E, Makris DP, Lalas SI. Evaluation of pulsed electric field polyphenol extraction from *Vitis vinifera*, *sideritis scardica* and *Crocus sativus*. *Chem Eng* 2021;5:25. <https://doi.org/10.3390/chemengineering5020025>.
- [169] Athanasiadis V, Pappas VM, Palaiogiannis D, Chatzimitakos T, Bozinou E, Makris DP, et al. Pulsed electric field-based extraction of total polyphenols from *sideritis raiseri* using hydroethanolic mixtures. *Oxygen* 2022;2:91–8. <https://doi.org/10.3390/oxygen2020008>.
- [170] Pollini L, Cossignani L, Juan C, Mañes J. Extraction of phenolic compounds from fresh apple pomace by different non-conventional techniques. *Molecules* 2021;26:1–10. <https://doi.org/10.3390/molecules26144272>.
- [171] Hendrawan Y, Larasati R, Wibisono Y, Umam C, Sutan SM, Choviya Hawa L. Extraction of phenol and antioxidant compounds from kepok banana skin with PEF pre-

- treatment. *IOP Conf Ser Earth Environ Sci* 2019;305. <https://doi.org/10.1088/1755-1315/305/1/012065>.
- [172] Boussetta N, Vorobiev E, Reess T, De Ferron A, Pecastaing L, Ruscassié R, et al. Scale-up of high voltage electrical discharges for polyphenols extraction from grape pomace: effect of the dynamic shock waves. *Innovat Food Sci Emerg Technol* 2012;16:129–36. <https://doi.org/10.1016/j.ifset.2012.05.004>.
- [173] Li Z, Fan Y, Xi J. Recent advances in high voltage electric discharge extraction of bioactive ingredients from plant materials. *Food Chem* 2019;277:246–60. <https://doi.org/10.1016/j.foodchem.2018.10.119>.
- [174] Xi J, He L, Gong Yan L. Continuous extraction of phenolic compounds from pomegranate peel using high voltage electrical discharge. *Food Chem* 2017;230:354–61. <https://doi.org/10.1016/j.foodchem.2017.03.072>.
- [175] Hejazi S, Siahpoush V, Ostadrahimi A, Kafil Gazi Jahani B, Ghasempour Z. High-voltage electric discharge as pre-treatment for efficient extraction of bioactive compounds from red onion peel. *Innovat Food Sci Emerg Technol* 2022;81:103153. <https://doi.org/10.1016/j.ifset.2022.103153>.
- [176] Žuntar I, Putnik P, Bursać Kovačević D, Nutrizio M, Šupljika F, Poljanec A, et al. Phenolic and antioxidant analysis of olive leaves extracts (*Olea europaea* L.) obtained by high voltage electrical discharges (HVED). *Foods* 2019;8:248.
- [177] Jokić S, Pavlović N, Jozinović A, Aćkar, Babić J, Šubarić D. High-voltage electric discharge extraction of bioactive compounds from the cocoa bean shell. *Chem Biochem Eng Q* 2019;33:271–80. <https://doi.org/10.15255/CABEQ.2018.1525>.
- [178] Nutrizio M, Gajdoš Kljusurić J, Badanjak Sabolović M, Bursać Kovačević D, Šupljika F, Putnik P, et al. Valorization of sage extracts (*Salvia officinalis* L.) obtained by high voltage electrical discharges: process control and antioxidant properties. *Innovat Food Sci Emerg Technol* 2020;60. <https://doi.org/10.1016/j.ifset.2019.102284>.
- [179] El Kantar S, Boussetta N, Rajha HN, Maroun RG, Louka N, Vorobiev E. High voltage electrical discharges combined with enzymatic hydrolysis for extraction of polyphenols and fermentable sugars from orange peels. *Food Res Int* 2018;107:755–62. <https://doi.org/10.1016/j.foodres.2018.01.070>.
- [180] Banožić M, Jozinović A, Grgić J, Miličević B, Jokić S. High voltage electric discharge for recovery of chlorogenic acid from tobacco waste. *Sustain Times* 2021;13. <https://doi.org/10.3390/su13084481>.
- [181] Banožić M, Šafranko S, Bogadi D, Aladić K, Jokić S. Intensification of polyphenols extraction from Mandarin peel by high voltage electric discharge: effect of process parameters and optimisation. *Kem u Ind* 2022;71:699–709. <https://doi.org/10.15255/kui.2022.023>.
- [182] Nguyen TMC, Gavahian M, Tsai PJ. Effects of ultrasound-assisted extraction (UAE), high voltage electric field (HVEF), high pressure processing (HPP), and combined methods (HVEF+UAE and HPP+UAE) on Gac leaves extraction. *Lwt* 2021;143:111131. <https://doi.org/10.1016/j.lwt.2021.111131>.
- [183] Shahram H, Taghian Dinani S. Influences of electro-hydrodynamic time and voltage on extraction of phenolic compounds from orange pomace. *Lwt* 2019;111:23–30. <https://doi.org/10.1016/j.lwt.2019.05.002>.
- [184] Rebelatto EA, Rodrigues LGG, Rudke AR, Andrade KS, Ferreira SRS. Sequential green-based extraction processes applied to recover antioxidant extracts from pink pepper fruits. *J Supercrit Fluids* 2020;166. <https://doi.org/10.1016/j.supflu.2020.105034>.
- [185] Ekezie FGC, Sun DW, Cheng JH. Acceleration of microwave-assisted extraction processes of food components by integrating technologies and applying emerging solvents: a review of latest developments. *Trends Food Sci Technol* 2017;67:160–72. <https://doi.org/10.1016/j.tifs.2017.06.006>.
- [186] Márton D, Mazzutti S, Vitali L, Maria C, Müller O, Regina S, et al. Integrated extraction approach to increase the recovery of antioxidant compounds from *Sida rhombifolia* leaves. *J Supercrit Fluids* 2019;149:10–9. <https://doi.org/10.1016/j.supflu.2019.03.013>.
- [187] Carolina A, Aguiar D, Paula A, Fernando C, Angolini F, Rodrigues D, et al. Sequential high-pressure extraction to obtain capsinoids and phenolic compounds from biquinho pepper (*Capsicum chinense*). *J Supercrit Fluids* 2019;150:112–21. <https://doi.org/10.1016/j.supflu.2019.04.016>.
- [188] Vardanega R, Fuentes FS, Palma J, Bugueño-Muñoz W, Cerezal-Mezquita P, Ruiz-Domínguez MC. Valorization of granadilla waste (*Passiflora ligularis*, Juss.) by sequential green extraction processes based on pressurized fluids to obtain bioactive compounds. *J Supercrit Fluids* 2023;194:1–10. <https://doi.org/10.1016/j.supflu.2022.105833>.
- [189] Teixeira GL, Maciel LG, Mazzutti S, Barbi RCT, Ribani RH, Ferreira SRS, et al. Sequential green extractions based on supercritical carbon dioxide and pressurized ethanol for the recovery of lipids and phenolics from *Pachira aquatica* seeds. *J Clean Prod* 2021;306. <https://doi.org/10.1016/j.jclepro.2021.127223>.
- [190] Zhang Y, He Y, Liu C, Liu C, Li S. In vitro screening and isolation of human aromatase inhibitors from *Cicer arietinum* by a novel continuous online method combining chromatographic techniques. *J Separ Sci* 2018;41:483–92. <https://doi.org/10.1002/jssc.201700990>.
- [191] Banožić M, Gagić T, Čolnik M, Ž Knez, Škerget M, Jerković I, et al. Sequence of supercritical CO<sub>2</sub> extraction and subcritical H<sub>2</sub>O extraction for the separation of tobacco waste into lipophilic and hydrophilic fractions. *Chem Eng Res Des* 2021;169:103–15. <https://doi.org/10.1016/j.cherd.2021.03.005>.
- [192] Rizkiyah DN, Putra NR, Idham Z, Hazim A, Aziz A, Azizi M, et al. Recovery of anthocyanins from *Hibiscus sabdariffa* L. Using a combination of supercritical carbon dioxide extraction and subcritical water extraction. 2023.
- [193] Vandepoosele A, Bernard D, Draye M, Piot C, Chatel G. Innovative coupling of supercritical carbon dioxide and water for the recovery of lipids and polyphenols from spent coffee grounds. 2021.
- [194] Yusoff NI, Leo CP. Microwave assisted extraction of defatted roselle (*Hibiscus sabdariffa* L.) seed at subcritical conditions with statistical analysis. *J Food Qual* 2017;2017. <https://doi.org/10.1155/2017/5232458>.
- [195] Sánchez-Camargo A del P, Ballesteros-Vivas D, Buelvas-Puello LM, Martínez-Correa HA, Parada-Alfonso F, Cifuentes A, et al. Microwave-assisted extraction of phenolic compounds with antioxidant and anti-proliferative activities from supercritical CO<sub>2</sub> pre-extracted mango peel as valorization strategy. *Lwt* 2021;137. <https://doi.org/10.1016/j.lwt.2020.110414>.
- [196] Moirangthem K, Ramakrishna P, Amer MH, Tucker GA. Bioactivity and anthocyanin content of microwave-assisted subcritical water extracts of Manipur black rice (*Chakkhao*) bran and straw. *Futur Foods* 2021;3:100030. <https://doi.org/10.1016/j.fufo.2021.100030>.
- [197] Zabol GL, Viganó J, Silva EK. Low-frequency ultrasound coupled with high-pressure technologies: impact of hybridized techniques on the recovery of phytochemical compounds. *Molecules* 2021;26. <https://doi.org/10.3390/molecules26175117>.
- [198] Santos MP, Souza MC, Sumere BR, da Silva LC, Cunha DT, Bezerra RMN, et al. Extraction of bioactive compounds from pomegranate peel (*Punica granatum* L.) with pressurized liquids assisted by ultrasound combined with an expansion gas. *Ultrason Sonochem* 2019;54:11–7. <https://doi.org/10.1016/j.ultsonch.2019.02.021>.
- [199] Ninčević Grassino A, Ostojić J, Miletić V, Djaković S, Bosiljkov T, Zorić Z, et al. Application of high hydrostatic pressure and ultrasound-assisted extractions as a novel approach for pectin and polyphenols recovery from tomato peel waste. *Innovat Food Sci Emerg Technol* 2020;64. <https://doi.org/10.1016/j.ifset.2020.102424>.
- [200] Viganó J, Assis BF de P, Náthia-Neves G, dos Santos P, Meireles MAA, Veggi PC, et al. Extraction of bioactive compounds from defatted passion fruit bagasse (*Passiflora edulis* sp.) applying pressurized liquids assisted by

ultrasound. *Ultrason Sonochem* 2020;64:104999. <https://doi.org/10.1016/j.ultsonch.2020.104999>.

- [201] Görgüç A, Bircan C, Yölmaz FM. Sesame bran as an unexploited by-product: effect of enzyme and ultrasound-assisted extraction on the recovery of protein and antioxidant compounds. *Food Chem* 2019;283:637–45. <https://doi.org/10.1016/j.foodchem.2019.01.077>.
- [202] Ntourtoglou G, Drosou F, Chatzimitakos T, Athanasiadis V, Bozinou E, Dourtoglou VG, et al. Combination of pulsed electric field and ultrasound in the extraction of polyphenols and volatile compounds from grape stems. *Appl Sci* 2022;12. <https://doi.org/10.3390/app12126219>.
- [203] Biophlox. Microwave-ultrasonic-ultraviolet synthesis/extraction/reactor system n.d. <https://www.biophlox.com/lab-equipment/general-lab-equipment/lab-others/Microwave-Ultrasonic-Ultraviolet-Synthesis-Extraction-Reactor-System-NuWav-Pro>.
- [204] Trujillo-Mayol I, Céspedes-Acuña C, Silva FL, Alarcón-Enos J. Improvement of the polyphenol extraction from avocado peel by assisted ultrasound and microwaves. *J Food Process Eng* 2019;42:1–11. <https://doi.org/10.1111/jfpe.13197>.
- [205] Jiang Z, Shi R, Chen H, Wang Y. Ultrasonic microwave-assisted extraction coupled with macroporous resin chromatography for the purification of antioxidant phenolics from waste jackfruit (*Artocarpus heterophyllus* Lam.) peels. *J Food Sci Technol* 2019;56:3877–86. <https://doi.org/10.1007/s13197-019-03858-8>.
- [206] Sillero L, Prado R, Labidi J. Simultaneous microwave-ultrasound assisted extraction of bioactive compounds from bark. *Chem Eng Process - Process Intensif* 2020;156:108100. <https://doi.org/10.1016/j.cep.2020.108100>.
- [207] Alonso-Carrillo N, Aguilar-Santamaría M de los Á, Vernon-Carter EJ, Jiménez-Alvarado R, Cruz-Sosa F, Román-Guerrero A. Extraction of phenolic compounds from *Satureja macrostema* using microwave-ultrasound assisted and reflux methods and evaluation of their antioxidant activity and cytotoxicity. *Ind Crops Prod* 2017;103:213–21. <https://doi.org/10.1016/j.indcrop.2017.04.002>.
- [208] Wizi J, Wang L, Hou X, Tao Y, Ma B, Yang Y. Ultrasound-microwave assisted extraction of natural colorants from sorghum husk with different solvents. *Ind Crops Prod* 2018;120:203–13. <https://doi.org/10.1016/j.indcrop.2018.04.068>.
- [209] Yu Q, Li C, Duan Z, Liu B, Duan W, Shang F. Ultrasonic microwave-assisted extraction of polyphenols, flavonoids, triterpenoids, and Vitamin C from *Clinacanthus nutans*. *Czech J Food Sci* 2017;35:89–94. <https://doi.org/10.17221/82/2016-CJFS>.
- [210] Villacís-Chiriboga J, Voorspoels S, Uyttendaele M, Ruales J, Camp J Van, Vera E, et al. Supercritical CO<sub>2</sub> extraction of bioactive compounds from mango (*Mangifera indica* L.) peel and pulp. *Foods* 2021;10:1–22. <https://doi.org/10.3390/foods10092201>.
- [211] Boussetta N, Lesaint O, Vorobiev E. A study of mechanisms involved during the extraction of polyphenols from grape seeds by pulsed electrical discharges. *Innovat Food Sci Emerg Technol* 2013;19:124–32. <https://doi.org/10.1016/j.ifset.2013.03.007>.
- [212] de Aguiar AC, Osorio-tobón JF, Viganó J, Martínez J. Economic evaluation of supercritical fluid and pressurized liquid extraction to obtain phytonutrients from biquinho pepper : analysis of single and sequential-stage processes. *J Supercrit Fluids* 2020;165. <https://doi.org/10.1016/j.supflu.2020.104935>.
- [213] Ortiz-Viedma J, Bastias-Montes JM, Char C, Vega C, Quintriqueo A, Gallón-Bedoya M, et al. Sequential bio-refining of bioactive compounds of high functional value from calafate pomace (*berberis microphylla*) using supercritical CO<sub>2</sub> and pressurized liquids. *Antioxidants* 2023;12. <https://doi.org/10.3390/antiox12020323>.
- [214] Tamkutė L, Liepuoniūtė R, Pukalskienė M, Venskutonis PR. Recovery of valuable lipophilic and polyphenolic fractions from cranberry pomace by consecutive supercritical CO<sub>2</sub> and pressurized liquid extraction. *J Supercrit Fluids* 2020;159. <https://doi.org/10.1016/j.supflu.2020.104755>.
- [215] Pereira DTV, Zabot GL, Reyes FGR, Iglesias AH, Martínez J. Integration of pressurized liquids and ultrasound in the extraction of bioactive compounds from passion fruit rinds: impact on phenolic yield, extraction kinetics and technical-economic evaluation. *Innovat Food Sci Emerg Technol* 2021;67. <https://doi.org/10.1016/j.ifset.2020.102549>.
- [216] Luo X, Bai R, Zhen D, Yang Z, Huang D, Mao H, et al. Response surface optimization of the enzyme-based ultrasound-assisted extraction of acorn tannins and their corrosion inhibition properties. *Ind Crops Prod* 2019;129:405–13. <https://doi.org/10.1016/j.indcrop.2018.12.029>.
- [217] Yu J, Lou Q, Zheng X, Cui Z, Fu J. Sequential combination of microwave- and ultrasound-assisted extraction of total flavonoids from *osmanthus fragrans* lour. Flowers. *Molecules* 2017;22. <https://doi.org/10.3390/molecules22122216>.