Sono-physical and sono-chemical effects of ultrasound: Primary applications in extraction and freezing operations and influence on food components

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Sono-physical and sono-chemical effects of ultrasound: primary applications in extraction and freezing operations and influence on food components

Xizhe Fu[a], Tarun Belwal[a], Giancarlo Cravotto[b], Zisheng Luo[a*]

a. Zhejiang University, College of Biosystems Engineering and Food Science, Key Laboratory of Agro-Products Postharvest Handling of Ministry of Agriculture and Rural Affairs, Zhejiang Key Laboratory for Agri-Food Processing, Hangzhou 310058, People's Republic of China
b. Department of Drug Science and Technology, University of Turin, via P. Giuria 9, 10125 Turin, Italy and Sechenov First Moscow State Medical University, 8 Trubetskaya ul, Moscow, Russia.

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[*] Corresponding authors.
E-mail address: luozisheng@zju.edu.cn (Z. Luo).

Abstract

Ultrasound is an advanced non-thermal food-processing technology that has received increasing amounts of interest as an alternative to, or an adjuvant method for, conventional processing techniques. This review explores the sono-physical and sono-chemical effects of ultrasound on food processing as it reviews two typical food-processing applications that are predominantly driven by sono-physical effects, namely ultrasound-assisted extraction (UAE) and ultrasound-assisted freezing (UAF), and the components modifications to food matrices that can be triggered by sono-chemical effects. Efficiency enhancements and quality improvements in products (and extracts) are discussed in terms of mechanism and principles for a range of food-matrix categories, while efforts to improve existing ultrasound-assist patterns will also be seen. Furthermore, the progress of experimental ultrasound equipment for UAE and UAF as food-processing technologies, the core of the development in food-processing techniques, is considered. Moreover, sono-chemical reactions that are usually overlooked, such as degradation, oxidation and other particular chemical modifications that occur in common food components under specific conditions, and the influence of bioactivity, which can also affect food processing to varying degrees, are also summarised. Further trends as well as some challenges for, and limitations of, ultrasound technology for food processing, with UAE and UAF used as examples herein, are also taken into consideration and possible future recommendations were made.

Keywords

sono-physical effect; sono-chemical effect; ultrasound processing; freezing; extraction; ultrasonic equipment

1. Introduction

The aim of the modern food industry is to produce higher-quality and safer foods with lower costs, and ultrasonication is a versatile technique that can help to achieve these goals. The technology has thus received considerable amounts of attention thanks to its relatively high feasibility, lower processing costs and environmentally friendly nature [1]. Ultrasound (US), or ultrasonic waves, are acoustic waves, belonging to the category of mechanical waves, with frequencies that are higher than 20 kHz, which is beyond the audible frequency range of humans [2]. The piezoelectric effect has been widely utilised to produce ultrasonic waves since the 1880s, and more ceramic materials, such as quartz, barium titanate, lead zirconate and lead titanate, have been used to generate US with advancements in technology [3]. The US technique has gradually grown to be considered a promising and novel technology that can replace or assist traditional food-processing methods (Figure 1).

Transducers are generally the key parts of ultrasonic equipment as they are responsible for the conversion of mechanical or electrical energy into acoustic wave forms. Once acoustic vibration is produced by transducers, the sound wave travels throughout the vessel that is filled with the medium, and compression and rarefaction (high- and low-pressure regions) are created. Micro-bubbles, which are usually referred to as cavitation bubbles, are formed when the low pressure that is produced by rarefaction exceeds the threshold inter-molecular force of the liquid medium. If the acoustic pressure is not sufficient enough, the cavitation bubbles will enlarge and contract during rarefaction and compression cycles, respectively. However, if the bubbles cannot reach the critical size for collapse or implosion, the phenomenon is usually defined as stable cavitation, which weakens the cavitation effect. On the other hand, if the pressure is sufficient enough, the micro-bubbles will grow radically, and subsequently collapse or implode when the bubbles exceed their critical size. This is recognised as transient cavitation, during which high amounts of energy are released, subsequently resulting in instantaneous extreme temperature and pressure conditions (up to 5000 K and 1000 atm) at molecular level. This simultaneously produces high shear forces and turbulence around the cavitation bubbles [4]. The extreme conditions lead to the hydrogen-oxygen bonds in some water molecules being broken, subsequently producing hydrogen and hydroxyl radicals that can participate in or initiate chemical reactions [5]. In the meantime, microstreaming, which is derived from the implosion and the eddies caused by the nonlinear oscillations of bubbles, is also generated, causing violent turbulence that disrupts the surfaces of solid foodstuffs and facilitates the mixing of food materials and components in the medium. This also decreases diffusion boundary layers, thus accelerating mass and heat transfer [6].

Apart from the liquid whistle, which is excellent for mixing and homogenisation, there are two main types of US device: probe/horn reactors and bath reactor systems, which are both mostly based on piezoelectric transducers. Ultrasonic baths generally consist of a tank filled with water and at least one transducer, which is usually fixed at the bottom or in a side wall of the tank. The sound energy converted from the electric current by the transducers can be transferred into the medium within the bath. The significant difference in the probe/horn reactor systems is that a horn tip is attached to the transducer to amplify the intensity of irradiation, and the acoustic waves are directly transferred into the sample via the horn tip, whose end is submerged in the sample [7]. There are typically two categories of electric power or ultrasonic intensity employed by ultrasonic equipment: low-energy US and high-energy US. The former usually possess a higher frequency (higher than 100 kHz) and lower sound intensity (maximum at 1 W/cm^2) and is predominately employed for non-destructive purposes and for its weak- or non-cavitation effects that cause almost no physical or chemical changes in the treated materials [3, 8]. The latter, typically characterised by relatively lower frequency and higher power intensity (generally ranging from 1 to 1000 W/cm^2), produces fast-moving microbubble streams and bubble collapse, resulting in alternations in the physical, chemical and even biochemical properties of food. It thus finds applications in several food-processing fields, such as extraction, freezing, oxidation, filtration, dehydration, inactivation of microbes, nano-formulation, and the modification of food components.

Ultrasonic devices for food processing and modification are usually based upon high-intensity US, and special designs are applied for specific food materials and processing purposes. Taking UAE as an example, a basic ultrasonic bath equipped with several transducers, a timer and a heating unit is a widely studied prototype for UAE [9]. Several setups also include stirrer to make sure all the mixture is properly exposed to US irradiation [10, 11]. Similarly, a stirring effect with an ultrasonic horn has been introduced to enhance extraction efficacy [12].

With the development of advanced high-power ultrasonic generators, the applications of US technology in food processing have received increasing amounts of attention and existing food-industry unit operations have been upgraded [6], in a salient example of a “technology-push” [13]. Previous studies have revealed that ultrasonic technology can enhance the performance of unit operations, giving higher extraction yields, higher freezing rates, shorter oxidation periods, higher filtration and drying efficiency, better emulsification performance and assisting microbe inactivation to prolong shelf life. Furthermore, the ultrasonic process in other non-thermal novel food-processing techniques, such as ultra-high pressure, gamma-irradiation and pulse electric field, etc., is comparatively affordable, safe, environmentally-friendly, versatile, easy to manipulate and has lower energy consumption [3, 9]. US processes have also been found to protect qualitative characteristics, such as flavour, appearance, texture and biochemical activity. With a view to the increasing applications of the US technique in various food-processing unit operations [14], the present review specifically focuses on UAE and UAF techniques that mainly take advantage of the sono-physical and sono-chemical effects induced by US in food components. Moreover, the specific roles and significance of the US technologies involved in food processing are also discussed in detail.

2. Ultrasonic effect on physical properties of food products

Quality is one of the most critical food-product indices that guide consumer preference for food, and nearly all food-processing operations change food quality in some aspect. These quality aspects mainly consist of physical, chemical and nutritional indices. Of all the indices, the physical properties are perhaps of greatest significance as they play a decisive role in the initial judgment of product quality.

2.1. Liquid food products

US use during liquid food processing provides better mixing, smaller fat globule size, and better extraction performance, which all significantly influence physical properties, such as colour, viscosity, cloud phenomenon, stability, etc. Some researchers have used a bath type sonicator (working at 28 kHz, 420 W, for 30, 60 or 90 min) at a constant temperature of 20 ^{o}C to irradiate grapefruit juice, dramatically increasing its cloud value, and hence its quality [15]. Furthermore, ultrasonic treatment can also result in colour changes, in terms of variation of lightness (L^*), redness (a^*) and yellowness (b^*) indices [15]. There was a general decline in L^* and a^* value and a fluctuating variation in b^* value with longer ultrasonic treatment times. However, these changes were not easily seen by the naked eye [15]. The same research group then investigated the potential effect of ultrasonic treatment on the colouring pigment and viscosity of the grapefruit juice. A significant increase was observed for total carotenoids and lycopene content, which are the basic colouring pigments that maintain redness, whereas a decrease in viscosity was recorded after ultrasonication, suggesting the maintenance, or even an improvement, in grapefruit juice quality [16]. Campoli, Rojas, do Amaral, Canniatti-Brazaca and Augusto [17] have investigated a possible application of US in guava-juice processing to improve its quality. They found that even though ultrasonication decreased the total content of lycopene in guava juice, *in vitro* accessibility increased. Furthermore, the physical stability of the treated guava juice improved, due to a size reduction in the dispersed pulp particles, which prevented pulp sediment, while no effect on juice colour was noticed.

In the field of dairy products, pasteurised homogenised skim milk has been exposed to 20kHz US at 20W and 41W for up to 60min, leading to a reduction in turbidity and the size of casein micelles, fat globules and soluble particles, while the viscosity of milk was not significantly affected, as compared to the control group [18]. Another group has treated ayran (an acidic milk drink) with thermosonication (combination of ultrasonication and heating), which resulted in increased viscosity, and a positive effect on the consistency coefficient. Furthermore, a notabe decrease in the L^* value ($p<0.05$) was observed after thermal treatment alone, whilst thermosonication merely caused a slight change in the L^* value, suggesting that the latter could assuage the loss in product quality [19]. Yoghurt drinks were produced from the raw milk, which was exposed to ultrasonication at varying power levels (100, 125, 150 W) in combination with pre-heating treatment at 70 ^oC. This was found to improve major quality parameters, including delayed serum separation and increased apparent viscosity, as compared to conventional heating treatment (90 ^oC for 10 min), while the proximate composition and colour properties were not influenced significantly [20].

Increasing demand for high quality wine has led to advances in the development of alternative techniques for winemaking. Lowering biomass loss and improving the quality of the wine during winemaking is the major challenge, and US has been investigated as one of the most promising technologies for this challenge [1, 21]. During the winemaking process, US is mainly involved in the fruit/berry crushing and maceration processes. Owing to the significance of colour and aroma profile in wine quality evaluation, it is crucial to extract phenolic compounds (especially anthocyanins and tannins) and volatile compounds, which are mainly present inside the skin cell vacuoles. In the study performed by Ferraretto, Cacciola, Ferran Batlló and Celotti [22], results showed that the use of the ultrasonic technique in crushing and maceration was able to improve polyphenol extraction more quickly than classic maceration. Similarly, crushed grapes were treated with power US and were vinified over a range of maceration times (3, 6 and 8 days), resulting in higher concentrations of anthocyanins than in the control group. This change was closely associated with the organoleptic characteristics of the wine [23]. During the course of aging, ultrasonication has mainly been used to modify sensory properties. Zhang, Shen, Fan and García Martín [24] have tried to evaluate a number of physicochemical properties in red wine that was exposed to ultrasonication. The results illustrated that US, in general, was able to induce an increase in L^*, a^* and b^* values to some extent, indicating improvements in chromatic characteristics. An attempt to use sonication to accelerate the aging of steeped greengage wine has been made by Zheng, Zhang, Fang and Liu [25]. They found that sonication led to a smaller variation range of 1.18-1.25 (sum of absorbance at 420, 520, and 620 nm), suggesting that this process has no negative effect on wine colour. Moreover, improved sensory evaluation was observed after sonication at 45 kHz and 360 W for 30 min. Tchabo, Ma, Kwaw, Zhang, Li and Afoakwah [26] have reported that US exerted a positive effect on total phenol content and total flavonoid content, while it had a negative effect on total anthocyanin content, which might influence sensory characteristics. They also found that the organoleptic attributes responsible for the sensorial profile of wine processed by ultrasound-assisted aging was different to that of conventionally aged wines. Nevertheless, the evolution of colour properties of red wine that was exposed to ultrasonication throughout storage was investigated and similar patterns of changing tendencies in wine colour parameters and some typical phenolics were observed in both ultrasonically-treated and untreated wines. Furthermore, ultrasonically-treated wine undergoes a faster variation than untreated wine, in regard to the above-mentioned indices, indicating the potential of this technique to produce wine with a similar quality as conventionally-aged wine in a shorter time [27]. For wine spirit, a circulation system and ultrasonic transducer that encapsulated American oak chips was proposed, and the results revealed that ultrasonic treatment improved colour intensity, and TPI (total polyphenol index). Moreover, a sensorial assessment has shown that ultrasonically treated aged spirits had better ratings than initial distilled wine [28].

2.2. Non-Liquid food products

US is also employed in the processing of non-liquid food products for various purposes, including curing, meat tenderisation, to assist mixing, extraction, dehydration, freezing and many more. A study on US-assisted curing technology for porcine meat has been performed at a low-frequency (20 kHz) and relatively low-intensity (2-4 W/cm^2). The technique was found to soften the muscle fibres, which improved water-holding capacity and textural properties, as compared to the control group. Texture analysis showed a decrease in hardness and cohesiveness as well as higher springiness and lower chewiness after the US treatment, which all indicate improved tenderness. However, treatment for long periods or at increased power intensity (3.5 and 4.0 W/cm^2) caused protein denaturation, which leads to increased hardness and water-absorption limitations [29]. In another study, US has been applied to the bread-dough mixing process under a range of conditions, leading to a significant alteration in the physical properties of bread being observed [30]. A significant (19%) increase in bread volume, as well as reductions in bread mass and density, by 2.1% and 17% respectively, were recorded under high ultrasonic power and longer exposure times. Fish scale gelatin has been extracted using UAE (200 W, 40 kHz, 60 ^{o}C, 1 h). The resulting product presented a significantly higher storage modulus (5000 Pa), gelation point (22.94 ^{o}C), melting point (29.54 ^{o}C), and apparent viscosity than the analogue that was extracted using a conventional water bath [31]. US treatment gave better rheological behaviour, emulsifying properties, and higher gel strength. It should be noted that a reduction in triple-helix contents were recorded when the temperature was increased to 80 ^{o}C, which resulted in lower strength. In another study, the use of US to extract protein from peanut flour, at 100% amplitude for15 min, resulted in a decreased water-solubility index, nitrogen-solubility index, foam stability and emulsifying activity, whereas an increased water-absorption index and foam activity were found, as compared with the control treatment [32]. Although the protein microstructure was not apparently modified in this case, secondary structural changes may account for the alteration in the physico-chemical properties.

Furthermore, the mechanical and chemical effects caused by US have also caused the technique to gain considerable attention as a means to improve the quality and shelf-life of heat sensitive foods, such as fruits and vegetables. In one study, US exposure was applied to freshly harvested strawberries at 33 kHz and 60 W for different time durations (from 0 min to 60 min), and all the samples were tested over the storage period of 15 days at 4 ^{o}C [33]. The results showed that US treatment for 30-40 min caused the decrease in brightness to attenuate, while also leading to better redness preservation, smaller ΔE^* value variation and better fruit firmness, as compared to the untreated group. Nevertheless, a longer treatment time (60 min) showed detrimental effects on the parameters mentioned above. Freezing, which is an effective and well-known method for food preservation, can be dramatically favoured by ultrasonication. This is because of instantaneous nucleation, which can promote subsequent crystallisation. Xu, Zhang, Bhandari, Cheng and Sun [34] have stated that ultrasonic treatment can produce relatively small ice crystals, which markedly diminished the pore size of red radish and had a less destructive effect on microstructure, resulting in better preservation of firmness. In another study performed by Xin, Zhang and Adhikari [35], a $CaCl_2$ solution served as the freezing medium, and the UAF of broccoli was investigated. The results showed that, compared to normal freezing, texture properties and the colour index were better preserved with the involvement of US within an optimised intensity range of 0.250-0.412 W/cm^2. This may be explained by the cavitation phenomenon which generated several micro-bubbles that served as seeding sites for ice nucleation, while large dendritic ice crystals are crushed into smaller ice pieces during bubble collapse. This assists secondary ice nucleation and eventually forms fine and uniformly sized ice crystals, which greatly benefits the dough structure [36, 37]. Similarly, Hu, Liu, Li, Li and Hou [38] have investigated the influence of UAF on the quality and microstructure of frozen dough. They found that US pretreatment was better and should be introduced immediately when the temperature reaches the equilibrium freezing temperature. Furthermore, the quality (mainly elasticity) of the frozen dough that was exposed to US was significantly improved at 288, 360 and 418 W of ultrasonic power. An increased tendency in the whiteness index of mushroom (*L. edodes and P. eryngii*) has been recorded when UAF was applied at higher intensity (>0.27 W cm^{-2}) [39]. They reported the highest hardness value and chroma value with lower yellowness and browning indices in all mushroom varieties under ultrasonic treatment. Under a similar UAF treatment, porcine longissimus muscles have been reported to display a significantly smaller and more uniform ice-crystal size distribution, and markedly increased lightness compared to normal immersion freezing. Furthermore, no significant differences in $a^*$and b^*values were observed, suggesting that an improvement in frozen-meat quality can be achieved using UAF [40].

3. Ultrasound-assisted extraction and freezing – sono-physical effects

3.1. Ultrasound-assisted extraction (UAE)

3.1.1. Mechanism of UAE

Extraction is an important unit operation in the nutraceutical, pharmaceutical and cosmeceutical industries whether they aim to obtain a whole extract or separate specific compounds from a plant matrix [41, 42]. Apart from pharmaceutical manufacturing, extraction is a major step in the development of natural functional food, the valorisation of waste and by-products, and quality improvement during some manufacturing processes. The traditional extraction technology is solvent extraction, which demands a large amount of solvent and heating. Despite being a monumentally improved method, soxhlet extraction, which was established by Franz Ritter von Soxhlet in 1879, depends on diffusion through cell membrane, which limits extraction rates and yields. However, the cavitation and implosion caused by power US contributes to cell-wall rupture and increases the number of disrupted cells (Figure 2), thus weakening the limitation of cell structure on mass transfer from the solid matrix to the liquid phase [43]. Moreover, the reduction in the size of solid materials caused by power US also contributes to the increase in the contact surface area between the solid phase and the solvent (Figure 2). Many microscopic channels in tissue can be created, even when it is exposed to relatively low-power US in an ultrasonic bath, due to the sponge effect that is initiated by cycles of alternating compression and rarefaction, which facilitate the penetration of the solvent into the solid matrix. This moderate cell disruption is sufficient to increase mass transfer for extraction [44-46]. High temperature over the course of extraction, which is a critical factor in conventional extraction and one that facilitates diffusion and solvent permeation into the solid matrix, is not necessary with the assistance of US, and thus heat-sensitive compounds are protected. The implosion of cavitation bubbles and oscillation are able to cause fragmentation, erosion, sonoporation, capillary effect, shear forces, turbulence and mixing, which contribute to mass-transfer enhancement [47]. The use of US during extraction therefore allows the improved recovery of compounds with lower extraction times, temperatures, solvent volumes and energy consumption.

3.1.2. UAE instrumentation

The ultrasonic devices that are used in UAE can be roughly divided into 3 categories: US bath mode, sonotrode (US probe) mode and special-design mode, although the third is basically derived from the first two modes.

The US cleaning bath is the most popular and accessible approach for the first mode (Figure 3A). It is generally made up of at least one US transducer, a container, a timer knob and a heater. A mechanical stirrer is often required to make sure every matrix particle is irradiated by ultrasonic cavitation when the solid-material ratio to solvent is 1:5 or 1:10 [48]. In usual small and lab-scale UAE, a flask or tube serves as the extraction vessel and is half-submerged in water, which is the coupling liquid, and fixed at a specific position to obtain maximum cavitation. On larger scales, the US bath itself is considered to be the extraction vessel, and in this case, a mechanical stirrer is always necessary to stop the matrix from floating or sinking, and thus escape from the ultrasonic irradiation.

As for the ultrasonic probe mode, the system typically consists of a sonotrode, a container that can be fixed to the probe, a stirrer and sometimes a temperature-control system is also included (Figure 3B). For a commonly used lab-scale probe type extraction system, a double-wall vessel is usually selected to control temperature while extracting by cycling coolant or water inside the interlayer, and the tip of sonotrode is submerged in solvent to a specific height to obtain a relatively stable acoustic field. For the special-design mode for larger scale extraction with a probe type device (Figure 3C), a more powerful US probe is used together with sufficiently large bores with valves at suitable positions to allow the matrix and solvent to pass through. Some of the recently modified devices used for UAE that have been proposed in papers are listed in Table 1.

3.1.3. Effect on food components

A large number of recently published papers have investigated the advantages of UAE of food components (Table 2).

In a study performed by Al-Dhabi, Ponmurugan and Maran Jeganathan [49], US-assisted solid-liquid extraction was used to extract phenolic compounds (total phenolic content, total flavonoid content, chlorogenic acid, protocatechuic acid) from waste spent coffee grounds. The process was optimised using Box-Behnken statistical experimental design (BBD), and the yield under the optimum conditions (244 W ultrasonic power, 40^{o}C, 40 min, and 1:17 g/mL solid-liquid ratio) was confirmed. Another study has investigated araticum (*Annona crassiflora* Mart.), a type of popular fruit in the Brazilian Cerrado. It was found that high nominal ultrasonic power and short process times (less than 5 min) are effective for phenolic recovery from araticum peel when high-intensity US was employed, and the most abundant phenolic compounds recovered were epicatechin, rutin, chlorogenic acid, catechin and ferulic acid [50].

Backes, Pereira, Barros, Prieto, Genena, Barreiro and Ferreira [51] have compared the capabilities of three different extraction technologies, heat, US and microwaves, in the recovery of anthocyanin pigments from *Ficus carica* L. peel and they also optimised the extraction conditions. It was observed that US was the most effective technology, yielding 3.82 mg cyanidin 3-rutinoside/g residue, and the optimum condition was 21 min, 310 W of ultrasonic power and 100% of ethanol. A comparison between UAE and infusion extraction, in terms of the phenolic compounds derived from *Tilia cordata* fruit, was performed, and the total phenolic contents and total antioxidant obtained using UAE was significantly higher than those obtained via infusion extraction [52]. The influence of UAE conditions, including solvent type, solvent concentration, extraction time and temperature, on the extraction yield of olive-leaf-derived phenolic compounds, especially oleuropein and flavonoid contents, has been investigated, [53]. The authors stated that the optimum conditions included 50% acetone, 60^{o}C for 10 min extraction, and that they gave the highest yields of the major phenolic compounds, oleuropein and flavonoids from olive leaves. However, these were not the optimal conditions for other phenolics, such as hydroxytyrosol and phenolic acids. This fact clearly limits the simultaneous extractions of different types of phenolics. Unfortunately, the ultrasonic power, amplitude and the varying ultrasonic frequency were not reported. In another research project, conducted by Mehmood, Ishaq, Zhao, Yaqoob, Safdar, Nadeem, Munir and Wang [54], has made use of UAE in the extraction of bioactive compounds (mainly polyphenols) from blue butterfly pea flower (*Clitoria ternatea* L.). The technique was found to give higher yields than conventional extraction when optimised UAE conditions were adopted (20 kHz of ultrasonic frequency, 70% amplitude and 240 W, 150 min, 50 ^{o}C, solid-to-liquid ratio of 1 g: 15 mL, and 3 s ON and 3 s OFF for the ultrasonic mode). Besides the increasing trend in yield, the antioxidant activity of ultrasonically treated butterfly-pea-flower extract was also increased and its structure was only slightly affected.

Carotenoid, a type of natural colorant, which is characterised by its tetraterpene structure and that possesses health-promoting biological functions, has been extracted from cantaloupe waste under UAE, and a maximum yield of 124.6 μg/g under the optimal conditions (amplitude of 100%, 10 min, 80% hexane in hexane/acetone solvent, and solvent/solid ratio of 55 mL/g) was obtained [55]. Lutein, a commercially available and high-value product that belongs to the group of carotenoids, has been extracted from marine microalgae *Chlorella salina* with the assistance of US, and the extraction parameters were studied [56]. The results showed that the maximum yield (2.92±0.40 mg/g dry weight) was achieved at 40°C, 30 min extraction time with a 35 kHz frequency.

The yield of polysaccharides from *Verbascum thapsus* L., a common mullein flower, was notably enhanced by UAE (60 min, temperature 67.5 °C, ultrasonic power, and solvent/plant material ratio of 40 v/w) at relatively low temperature, and higher antioxidation activity was also observed [57]. It has been reported that higher polysaccharide yield (8.28%) and efficiency was obtained from *Volvariella volvacea* by UAE under the optimised conditions; 175 W of ultrasonic power, 57 °C, 33 min and a liquid-to-raw-material ratio of 25:1 were used [58]. A central composition design has been adopted, by Jiang, Yu, Li, Wang, Hu, Zhang and Zhou [59] , to optimise the UAE of polysaccharides from the roots of *Arctium lappa* L.. They stated that the highest extraction yield (8.22%) was attained under conditions of 158 W ultrasonic power, 50 °C, a water-to-raw-material ratio of 31 mL/g and a treatment duration of 83 min. Kia, Ganjloo and Bimakr [60] have optimised the UAE variables involved in the extraction of fenugreek-seed polysaccharides and this novel extraction method was compared with the conventional extraction. The results demonstrated that the maximum yield of fenugreek-seed polysaccharides (33.49%) was achieved under the optimal parameters (120 W of US power, 22 min, 30:1 mL/g of liquid-solid ratio). This yield was higher than that observed using conventional extraction for longer times at the same liquid-solid ratio. Apart from the higher extraction yield that can be acquired with US assistance, some properties are also influenced by this technique. A comparison of the *Ganoderma lucidum* polysaccharides that were extracted using a variety of different methods was performed, and the authors stated that lower molecular weight was obtained using UAE. Although the polysaccharides displayed the same monosaccharide compositions across the differing methods, the monosaccharide ratio was changed by US, and UAE decreased the reducing power of the polysaccharides [61].

As far as fatty acids, oils and lipids are concerned, an Iranian research group has recently investigated the effectiveness of the UAE treatment of *Aesculus hippocastanum* fruit oil, as optimised by response surface methodology using BBD, and compared it to the traditional Soxhlet method. The extraction yield was observed to increase by 21.82% (w/w) using UAE under the optimum conditions (60°C, 56.5 min, and 45:1 of solvent-to-plant-material weight ratio) [62]. In another paper, supercritical fluid extraction with CO_2 (40°C, pressure varied from 15 to 30 MPa) was compared with UAE in terms of *Spondias tuberosa* (umbu) oil extraction, and it was found that the highest yields were obtained by UAE with an ethanol/water mixture [63]. Not only were higher target-compound yields provided by UAE, the content of the high-value components within the target lipids can also be enhanced. Chanioti and Tzia [64] have compared the capabilities of UAE and traditional Soxhlet extraction (SE) to recover olive pomace oil, which is rich in sterols and squalene. It was revealed that the involvement of US resulted in oils with higher sterol and squalene contents than the SE equivalents.

In order to explore and develop new natural protein sources, Ayim, Ma and Alenyorege [65] have attempted to obtain protein from tea (*Camellia sinensis* L.) residue via US-assisted sodium hydroxide extraction. A notably higher extraction yield of 134.8 mg/g (63.5%), compared with the use of NaOH alone and NaOH coupled with enzyme-assisted extraction, was observed under the optimum ultrasonic conditions (NaOH concentration of 0.13 M, 13 min, ultrasonic power of 377 W, solid-liquid ratio of 51.5 g/L). The UAE of protein from *Phaseolus vulgaris* L. var. *Ganxet* beans has been optimised, and the researchers described that the use of 0.4 M NaOH, followed by ultrasonic treatment for 60 min with 40 kHz and 250 W, gave the highest yield of 78.73% [66].

3.1.4. Green UAE and coupled methods

As the popularity of the new concept of green process and extraction grows together with the development of newer and advanced techniques, the engineering of UAE systems has gradually evolved from elementary systems and intermediary modifications to greener and food-grade solvent-based UAE systems and their integration with other techniques, thus notably improving overall process efficiency.

The combination of microwaves and US irradiation was developed to promote mass and heat transfer [67], high cell-rupture efficiency and the consequent release of constituents into the extraction solvent [68]. This is facilitated by the simultaneous microwave heating and the acoustic cavitation initiated by US [32]. Yu, Li, Duan, Liu, Duan and Shang [69] have reported that combining US and microwaves is a high-efficiency technology for the extraction of bioactive compounds from *Clinacanthus nutans*, especially for polyphenols, flavonoids, triterpenoids and vitamin C. A similar work on the US/microwave assisted extraction of natural dye from sorghum husk has also been performed, and the author stated that this combined technique with a blended extraction solvent was an efficient option for colorant extraction [70]. Supercritical fluid extraction, in which a substance is subjected to temperature and pressure above its critical point, is another efficient extraction method. This technique leaves no solvent contamination and allows low temperatures to be used, although it is limited by the low polarity of CO_2, which is a commonly used as supercritical fluid. This technique is thus particularly suitable for food components that possess similar polarities, such as oil and fats. A series of acoustically-assisted supercritical fluid extraction devices with different transducer geometries have recently been proposed for the recovery of phytochemicals from oregano and *Agave salmiana* bagasse. It was proven that extraction efficiency was enhanced by this novel mixed technique and that the multiplate transducer design was the preferable option [71, 72].

Enzyme-assisted extraction is a widely accepted substitute for traditional extraction due to its high efficiency, reduced solvent consumption, high selectivity and ease of operation. As the disruption of cell integrity enhances the release of components inside the cell, a combination of enzymatic characteristics and acoustic cavitation has been taken into consideration. In a study regarding tannin extraction from acorns, enzyme-based UAE was observed to be a high-efficiency approach to obtaining tannins, and optimal conditions were demonstrated [73].

In addition to the above-mentioned UAE-combined techniques, green extraction solvents are an emerging field that is likewise applied in the UAE process. Ionic liquids possess relatively low melting temperatures in liquid form, making them a preferable substitute for traditional organic solvents because of their traction benefits and eco-friendly attributes. Ionic liquids have been extensively employed in various extraction processes, such as liquid-phase micro-extractions, solid-phase extractions and so on. In a novel approach, ionic liquids have been incorporated into a UAE process as an extraction medium [74]. Jiang, Ning and Li [75] selected 1- butyl- 3-methylimidazolium bromide [(Bmim)Br] as their ionic liquid and reported that this ionic liquid-based UAE process was a simple, rapid, effective and sustainable method for the extraction of isochlorogenic acid C from *Chrysanthemum morifolium*. Furthermore, deep eutectic solvents, from the frontier trend, are another substitute for organic solvents. They are more competitive than ionic liquids, mainly due to their non-toxic nature and higher biodegradability. Deep eutectic solvents are generally made of two or three kinds of organic constituents in the form of hydrogen bond acceptors and donors, thus producing abundant hydrogen bonds, which grants them a definite type of polarity that is suitable for the extraction of various compounds. It is therefore reasonable to replace organic solvents with these quickly emerging eco-friendly solvents in UAE procedures. Deep-eutectic-solvent-based UAE has been widely employed in the extraction of organic samples from liquid samples, although their use with solid samples and for the extraction of inorganic components has also been proposed [76]. Chanioti and Tzia [77] have utilised choline chloride based natural deep eutectic solvent that contained citric acid, lactic acid, maltose and glycerol, as hydrogen bond donors, with 20% of water. US was then used in the extraction of phenolic compounds from olive pomace. According to the authors, the natural deep eutectic solvents containing citric acid and lactic acid showed the best extraction efficiency. This combined technology provides an excellent alternative to classic extraction applications. Multi-phase solvents are likewise considered to be a replacement for traditional solvents due to their selective partitioning of a target product between two or more phases. Basically, a two-phase system is formed by mixing two or more phase-forming substances in water, such as dissimilar and non-miscible polymers, different surfactants or polymers with salt [74]. Multi-phase partitioning, with ultrasonication as a pre-treatment, can enhance oil extraction, and the literature related to this theme has been reviewed by Panadare and Rathod [78]. Cloud point extraction, which is based on the clouding characteristics of surfactants at the cloud point (a temperature at which a solution turns into a turbid, cloud-like state), is another alternative solvent technology for separation and extraction. It involves two phases, one of which is a surfactant-rich phase that captures hydrophobic components and the other an aqueous phase that holds hydrophilic constituents when the temperature reaches the cloud point. A recent report has combined cloud-point extraction with UAE for the pre-concentration of Sb, Sn and Tl in food and water. This technique was chosen for its rapidity, effectiveness and eco-friendliness, and the optimal conditions were achieved utilizing fractional factorial design and response surface methodology [79].

3.2. Ultrasound-assisted freezing (UAF)

3.2.1. Mechanism of UAF

Freezing is an important method of preservation that can effectively extend the shelf life of food products. In the food industry, freezing is also one of the most important unit operations and is usually utilised to produce food products in a frozen state. There are two general phases during the freezing processes: initial ice nucleation and crystal growth towards the liquid phase. The quality of frozen food depends highly on the size distribution, location and even the shape of ice crystals [3, 6]. Generally, the speed of ice nucleation is slower than the growth of ice crystals in slower freezing conditions; ice crystals will grow to a comparatively large size that destroys the cell tissues of foodstuffs, rather than forming many small and uniform ice crystals within the cell, which can protect the microstructure of food [80]. Therefore, it is believed that accelerating the freezing process will decrease ice crystal size and preserve the original properties, which is usually related to producing food of high quality. In the current food freezing industry, commercial frozen foods are usually produced by conventional freezing techniques, such as air blast freezing, cryogenic freezing, immersion freezing, etc., which are rather slow due to the relatively low thermal conductivities of food, which range from 0.5 to 1.5 W/m^2K, subsequently resulting in larger, irregular and uneven ice crystals [80, 81]. US can enhance the rate of the freezing process by generating a mass of ice nuclei (cavitation bubbles) and, at the same time, increasing heat and mass transfer. This technique can be further utilized in freeze concentration and freeze drying, which can also improve the quality of products

In terms of the mechanism of UAF (Figure 4), it is generally believed that the freezing process can be accelerated by US due to the initiation of nucleation and increases in heat and mass transfer [6, 82]. The nucleation caused by US mainly consists of two phases; primary nucleation and secondary nucleation (Figure 4). The stage of primary nucleation is initiated when the temperature reaches the nucleation temperature, which is below the corresponding freezing temperature. A large amount of latent heat is released during this phase. It is widely believed that the extreme conditions (high pressure) created by US contribute to a decrease in the supercooling degree, driving the process of nucleation [6, 83]. Furthermore, the cavitation bubbles themselves serve as nuclei for ice nucleation when the size of the micro-bubbles reaches the critical threshold. The motion of the stable cavitation bubbles can result in microstreaming and eddies, subsequently enhancing heat and mass transfer and contributing to nucleation. During secondary nucleation, which is based on pre-existing ice crystals, US irradiation can break up the dendrites of this pre-existing ice into many smaller fragments, due to the collapse of cavitation bubbles and the shear force derived from the micro-flow, leading to more nucleation sites being produced. The collapse and motion of cavitation bubbles can change the fluid dynamics and increase the heat and mass transfer coefficient (Figure 4). Ice crystal growth can also be driven by the use of US largely from the enhancement of bulk-phase mass transfer. An enhanced heat-transfer coefficient is certainly beneficial to the rapid removal of sensible heat and latent heat that is released [81]. A theoretical model of inertial acoustic cavitation under the simulated conditions of ice nucleation has recently been proposed with the aim of accurately calculating the pressure and temperature at the bubble interface and in the liquid adjacent to the bubble surface before, and just after, collapse (parameters of simulation: initial bubble radius (R_0) was 5 μm, ambient temperature ($T_{l\infty}$) was -10 °C, acoustic pressure amplitude (P_{ac}) was 1.4 bar, acoustic frequency (f) was 29 kHz) [84]. The authors found that the gas pressure inside the bubble and the liquid pressure at the bubble surface were quite close at collapse. The rigorous model (model B, liquid acting as a normal heat-conducting medium) indicated that the gas inside the bubble was at 11300 K, and the pressure of the liquid at the surface of bubble was 23 GPa, while the interfacial temperature was relatively moderate (620 K), which was still lower than the critical value of water at 647 K. Given the endothermic dissociation of water vapour inside the cavitation bubble, bubble-core peak temperature and liquid temperature were decreased by 30 % and 5%, respectively. In the surrounding area of the bubble just after collapse, the pressure peak propagated within the liquid at an extremely high speed and attenuated as it departed from the bubble surface. The temperature profile exhibited a very steep, quasi-linear decreasing gradient in a rather narrow inner zone (100-200 nm) with a progressively declining gradient in a broader outer zone (1-2 μm). It should be noted that very high pressure (above 1 GPa) still existed after bubble collapse while the temperature was close to the ambient temperature in the same area, thus promoting sono-crystallisation processes [84, 85]. In the second part of the work, conducted by the same research group with identical simulation parameters, the nucleation rate and the nuclei number generated from a single collapsing bubble were theoretically estimated at a range of driving acoustic pressures, liquid ambient temperatures and bubble initial radius [86]. It can be observed that the collapse obviously induces huge local undercooling (up to -370 K, at 65 ps after collapse) just at the time of maximum pressure, and that nucleation might occur. However, this maximum undercooling is not exactly at the bubble surface, but a little bit away from the surface where the temperature is dramatically decreased, while the pressure is still extremely high. The volumetric nucleation rate was a function of liquid pressure and temperature in this research. In terms of the influence of liquid temperature on the nucleation rate, the decreasing temperature led to an increase in undercooling, thus causing a larger general nucleation rate. Meanwhile, the influence of liquid pressure on the nucleation rate is relatively complex and depended on two opposing effects: with the increasing pressure, the increased undercooling, together with solid density and melting enthalpy, contributed to an increased nucleation rate; yet, when the pressure was higher than 2.3 GPa, the surface tension was the predominant influence factor, causing a dramatic decrease in the nucleation rate. The integration of nucleation rate over time-after-collapse and the space surrounding the collapsed bubble determines the number of nuclei from a single collapse event. The number of nuclei increased with the enhancement of the undercooling level and acoustic pressure within the moderate pressure range, up to 220 kPa. However, when acoustic pressure was higher than 220 kPa with a given liquid supercooling level, the number of nuclei would not vary as the pressure increased. Furthermore, according to results obtained by Cogné, Labouret, Peczalski, Louisnard, Baillon and Espitalier [86], the ice nucleation temperature threshold, representing the conditions for the generation of at least one nucleus, could be described as a function of acoustic pressure and initial bubble radius. It was observed that a zone, located between an upper branch and a lower branch of each contour curve, possessed the possibility of nucleation, and a general optimal pressure range and bubble-size range were centred on 225 kPa and 8.5 μm, respectively. In a separate work, US theory, the penetration theory of mass transfer and energy conservation have been used as the basis for an investigation of heat and mass transfer characteristics to provide a deeper insight into US-assisted droplet freezing [87]. Firstly, cavitation bubble number and bubble radius were effectively controlled by means of adjusting the main parameters, namely US frequency and intensity. Higher US frequencies caused a larger number of bubbles with smaller radius, due to a more rapid change between positive pressure and negative pressure, thus leading to bubbles colliding and merging more drastically, while the increasing US intensity resulted in a decreasing number of bubbles of larger radius. Furthermore, the surface-renewal ratio for the droplets increased with intensity at specific frequencies, and decreased with ultrasonic frequency. The convective mass-transfer coefficient showed an increasing trend with higher ultrasonic intensity, while a declining trend with higher frequency. A similar pattern was observed in heat transfer. The reason was that higher frequencies led to smaller bubbles, which subsequently decrease the surface renewal ratio and caused mass and heat transfer to be weakened; even though there was a relatively smaller number of bubbles at higher US intensities, larger bubble radii might play a more important role in the surface renewal ratio, thus resulting in enhanced mass and heat transfer. These results provide new theoretical evidence for the UAF process.

3.2.2. UAF instrumentation

Several studies concerning UAF have described the experimental devices used, which roughly fall into three categories: namely full-immersion, half-immersion and non-immersion types (Figure 5) [81]. Generally speaking, the devices designed for UAF are basically made up of three parts: an ultrasonic system, coolant circulation coupled with a refrigeration-cycle system and temperature detection coupled with a data-recording system. Ultrasonic systems are generally made up of a number of transducers that are evenly attached at the bottom of a stainless bath and connected to the generator with variable power and frequency output. As for the coolant-circulation coupled with a refrigeration system, the coolant is pumped throughout the coil pipe located in the temperature-controlling section, and the coolant at the higher temperature will be cooled when it passes through the heat exchanger connected to the compressor to achieve cooling and temperature control. Temperature-detection coupled with the data-recording system typically includes several T/K-type thermocouples and data recorders connected to a computer. The full-immersion type devices (Figure 5A) have been widely applied in several freezing-property sample categories, including liquid samples, solid samples and semisolid samples [35, 39, 40, 83, 88-92]. The use of the other two types of device in freezing processes is relatively rare. Half-immersion type devices (Figure 5C) are characterised by a metal plate that is tightly bound to ultrasonic transducers, while samples inside the container (basically liquid and semisolid samples) are placed on the plate with the medium to improve US propagation between the plate and the container. Non-immersion type devices (Figure 5C) feature direct contact between samples and the UAF device without any coolant immersion. This avoids contamination by the coolant, but leads to ultrasonic-energy loss because of propagation via the air [93, 94]. Some of the modified experimental UAF devices are listed in Table 3.

3.2.3. Effect on food components

In previous research, UAF on apples was performed by Delgado, Zheng and Sun [90]. Apple cylinders were immersed in an ultrasonic bath that worked intermittently at a 40 kHz frequency and 131.3 W (0.23 W/cm^2). They found that the application of US (at

0ºC or -1ºC for 120 s with 30 s intervals) markedly improved the freezing rate, as compared with the untreated group, while the difference between the radial and tangential irradiated samples was not significant. Power US with a sonotrode, operating at 35 kHz, was applied in assisting the freezing of potato cubes, and the sonotrode worked when the temperature of the geometrical centre was within the range of -0.1 to -3.0ºC [95]. The authors reported that nucleation occurred significantly earlier when US was applied below -0.1ºC. Furthermore, the supercooling degree reached in the potato cubes was linearly correlated to ultrasonic temperature. However, only when US was applied at -2.0ºC was a significant reduction in freezing time observed. Subsequently, the UAF of potato spheres was assessed using experimental, numerical and analytical approaches to accurately predict temperature distribution, phase ratios and process time [89]. The results confirmed that US irradiation was able to quicken the characteristic freezing. Moreover, the author found that only within the range of 30-70% duty cycles could the shortest freezing time be achieved. This was due to the effect of the increased heat transfer coefficient and the thermal effect at the sample surface. Numerical simulations proposed by Kiani, Zhang and Sun [89] provided the temperature and water-fraction profiles in potatoes under different conditions. This can be used to support the study and control of several operation situations and provide a deeper understanding of freezing. More recently, a number of thermal conductivity models, several processing parameters and the geometric appearances of the sample (potato) have been studied using the same UAF device. The UAF of various shapes was accurately predicted, in comparison with experimental data, suggesting that an optimum condition of ultrasonic intensity and duty cycle is necessary to reduce freezing time [96]. Similarly, Cheng, Zhang, Adhikari, Islam and Xu [88] have used US to assist the immersion freezing of strawberries. They also reported that the achieved degree of supercooling in the US irradiation was linearly correlated to ultrasonic temperature. Moreover, the involvement of ultrasonication induced nucleation at a lower degree of supercooling at a range of temperatures. Unlike the result obtained by Comandini, Blanda, Soto-Caballero, Sala, Tylewicz, Mujica-Paz, Valdez Fragoso and Gallina Toschi [95] on the freezing of potato cubes, the characteristic freezing time was significantly decreased, as compared to the control group, at several temperatures, while, of all the operating temperatures, the minimum characteristic freezing time was achieved at -1.6ºC. Although higher US intensity general led to a lower supercooling degree, these two parameters had no linear correlation. Although organoleptic characteristics and corresponding parameters are very important indices for the assessment of any food processing technique, quality properties were unfortunately not evaluated in the above-mentioned investigations. However, a study performed by Xin, Zhang and Adhikari [35] , used US in the freezing of broccoli immersed in a $CaCl_2$ solution, and the results indicated that US decreased the freezing time, in the specific ultrasonic-intensity range of 0.250-0.412 W/cm^2, while also preserving the values of texture properties, colour and L-ascorbic acid content better than the normal freezing group. Furthermore, drip loss was also significantly diminished. Nevertheless, the quality of the frozen broccoli deteriorated when the ultrasonic intensity was out of the mentioned range. Another experiment concerning the UAF of red radish was conducted to explore the dynamic nucleation of ice and the delay from US onset to nucleation commencement in samples [97]. US irradiation (20 kHz) was exerted at varying parameters, such as duration time (0-15 s), onset temperature (ranging from -0.5 to -2^{o}C), and acoustic intensities (0.09-0.37 W/cm^2). The authors stated that nucleation was induced by US, that a linear equation was observed between the nucleation temperature of radish cylinders and US temperature, and that the optimal running condition was an US temperature of -0.5^{o}C, a 7s duration time and 0.26 W/cm^2 acoustic intensity, which implied that this technique was able to control the crystallisation process in freezing solid food. Microstructures and quality properties were subsequently investigated and the results showed that US treatment decreased the freezing time of red radish, resulting in smaller ice crystals, which caused less damage to cellular and tissue structures, leading to a reduction of drip loss and better preservation firmness [34]. Wrapping, as a physical barrier, has been introduced to the freezing process to effectively prevent solute uptake from the coolant ($CaCl_2$ solution) into the sample. This is a novel approach to controlling coolant uptake during the process. Moreover, the effect of power US on the physico-chemical properties of red radish during UAF was investigated in the same lab. The author illustrated that UAF granted a significant reduction in drip loss and the loss of phytonutrients, such as anthocyanins, vitamin C, and phenolics. Immersion freezing (IF) and UAF presented better texture preservation over the slow freezing (SF) mode, and radish tissue exhibited better cellular structure when the ultrasonic intensities were set at 0.17 and 0.26 W/cm^2, with less cell separation and disruption. Meanwhile, aromatic profiles have also been shown to be influenced by this process [98]. Moreover, US-assisted osmotic dehydration was taken into consideration as a pre-treatment for the freezing process. The application of US markedly reduced the times for dehydration and the subsequent freezing of the radish cylinders, which showed less freezable water content and better preservation in firmness and microstructure, as compared to the samples without US [99]. The effect of several freezing methods on the quality of lotus roots and their efficiency have been compared. The authors stated that ultrasound assisted-immersion freezing (30 kHz) had several advantages, in terms of freezing time, colour, firmness, tissue-microstructure preservation and drip loss, over the conventional air blast freezing and immersion freezing, while the drawback of this method is its relatively high vitamin C loss throughout the course of the process [100]. This finding is clearly in contrast with the conclusion of research on red-radish freezing conducted by Xu, Zhang, Bhandari, Cheng and Islam [98]. This may be due to the different fruit/vegetable materials, which present varying microstructures, and the different ultrasonic devices and experimental conditions used in the investigations.

In addition to fruits and vegetables, this technique has also been adopted for other food materials. One attempt conducted by Islam, Zhang, Adhikari, Chen and Xu [39] focused on the immersion freezing of mushrooms. The authors reported that ultrasonication worked at 0.39 W/cm^2 and 20 kHz, which notably reduced the nucleation time by 24~53%, depending on the species treated (*Lentinula edodes, Agaricus bisporus and Pleurotus eryngii*). Less drip loss (around 10%) and a higher whiteness index (in *L. edodes and P. eryngii*) were attributed to US application when acoustic intensity was above 0.27 W/cm^2, whereas the highest chroma value, with lower yellowness and brown indices, and the highest textural hardness values were achieved at 0.39 W/cm^2 acoustic intensity. The activities of polyphenol oxidase and peroxidase decreased with increasing ultrasonic intensity, suggesting that there is a benefit to quality preservation. The US equipment was later improved to direct contact mode and US was applied during freezing and the frozen storage course of mushrooms, by the same research group, in order to investigate crystal morphology [93]. They found that US irradiation initiated the nucleation of ice and that the ice crystals were mainly within the range of 0-80 micros when US was utilized. A larger size range of 50-180 microns was observed in the control group, implying that improved frozen-product quality can be obtained compared to the control. In the dough-freezing field, an ultrasonic bath has been used at a 25 kHz frequency to investigate the quality and microstructure influence induced by US [38]. The authors found that the total freezing time was significantly shortened, by more than 11% at power levels of 288 and 360 W, and that heat transfer was more efficiently enhanced in the latter two stages (phase-transition stage and solid-state temperature decrease stage) than in the first stage (liquid-state temperature). The formation of a large number of tiny ice crystals inside the frozen dough led to crystal nucleation also being enhanced by US, which granted an increased maximum penetration force to the frozen dough. Although this novel freezing technique was able to simultaneously accelerate the freezing process and form tiny and uniform ice crystals that protected the dough-network structure [36], the literature on this topic is comparatively limited. As far as US-assisted meat freezing is concerned, the effect of this technique on the freezing rate and quality of porcine longissimus muscles at several ultrasonic power settings has been evaluated, and the researchers stated that the freezing process was effectively accelerated at a certain ultrasonic power level (180 W), while no significant difference was observed in terms of colour values, pH values and cooking loss compared with the normal immersion freezing group and control group [40]. Moreover, they pointed out that US treatment at 180 W was able to substantially diminish ice-crystal size and make the distribution more uniform. The effect of UAF on muscle quality and the physicochemical properties of common carp (*Cyprinus carpio*) during freezing storage has recently been investigated [101]. The results showed that UAF samples exhibited smaller ice crystals, and thus caused less damage to muscle tissue, lower thawing and cooking losses, higher shear force over the 90-180 days storage, higher protein thermal stability, as well as lower thiobarbituric acid reactive substances and total volatile basic nitrogen values during storage than those in the normal immersion freezing (IF) and air freezing (AF) groups. This result implies that frozen fish maintain higher quality during freezing storage. Moreover, UAF processing was found to lead to the reduction of mobility and loss of immobilised and free water, which is in accordance with the results obtained by Zhang, Niu, Chen, Xia and Kong [40].

3.2.4. UAF coupled methods

In order to produce frozen food products of higher quality in a shorter time and at a lower cost, researchers make great efforts to improve the UAF technique. Previously, Hu, Sun, Gao, Zhang, Zeng and Han [102] tried to inject bubbles into the liquid before US-assisted freezing for 3-5 s at 0.21 W/cm^2. They found that the nucleation of the liquid with bubbles occurred at temperatures close to the irradiation temperature with a shorter delay, compared to the samples without pre-existing bubbles, and that some of the pre-existing bubbles may be similar to those generated by US. This result suggests that this method could be utilized to initiate and control the nucleation process in liquid samples. Subsequently, Xu, Zhang, Bhandari, Sun and Gao [103] investigated the influence of CO_2 infusion on a model solid food system, and the results showed that infused CO_2 significantly reduced the freezing time and ice crystal size. Compared with samples that are only exposed to US, this combined treatment led to lower water loss, higher gel strength and better texture properties, while water mobility and the distribution of freeze-thawed samples were also influenced. Therefore, the existence of bubbles (CO_2) promoted UAF in this solid food model, indicating that there may be promising applications in real food in the solid state. Micro-nano bubbles (MNBs) have recently been tentatively introduced into sucrose and maltodextrin solutions during a UAF process by Zhu, Sun, Zhang, Li and Cheng [92]. They found that existing MNBs were able to effectively accelerate ice nucleation and crystal growth for both the sucrose and maltodextrin solutions because of the significant reduction in the supercooling degree and freezing time, compared to conventional immersion freezing. However, any existing air, mainly voids, in plant tissues could attenuate US propagation and weaken the effectiveness of UAF. Freezing time and several quality parameters have been assessed in a variety of fruits and vegetables of varying structure, and the results indicated that a higher percentage of voids in plant tissues led to lower US effectiveness. Furthermore, the total freezing time reduction fitted a power function of the volume of voids [104]. The authors stated that UAF was more effective in freezing fruits or vegetables that possessed a highly dense structure. Soluble soybean polysaccharide (SSPS) has been applied as a novel cryoprotectant in conjunction with UAF during the freezing process of grass carp surimi [91]. The results exhibited that UAF clearly accelerated the freezing process, within the power range 300-540 W, and the optimal US treatment conditions were found to be 300 W for 10 s with an interval of 40 s, and repeat 5 times. Compared with the samples without SSPS, it was found that its addition was able to alleviate the quality property deterioration of grass carp surimi, including the decreases in Ca^{2+}-ATPase activity, total sulphydryl content, active sulphydryl content, salt extractable protein content and water-holding capacity. The addition of 3% SSPS was the optimal ratio to effectively mitigate protein denaturation in this sample during frozen storage at -18^{o}C, indicating that a promising advance in UAF may be conjunction with other beneficial food-derived agents.

4. Chemical changes in food components: sono - chemical effects

4.1. Polyphenolics

The use of US applications for food processing purposes is generally carefully considered in terms of the preservation of bioactive compounds (namely, phenolic compounds, carotenoids, anthocyanins, etc.) in food materials because of their various beneficial functional properties, such as anti-oxidation, anti-cancer and anti-inflammation activity etc. The US treatment conditions selected during food processing procedures are therefore important for the preservation of these components, both because of their organoleptic characteristics and their health-benefits.

However, US treatment can still influence the content and activities of the compounds existing in a food matrix. In a previous research performed in 2008, the authors identified the potential of the sono-chemical hydroxylation of phenolic compounds, in which it was indicated that phenolic compounds in food materials can be modified by US [105]. In a later work, a model extraction solution was constructed and several UAE factors, including ultrasonic power, frequency, sonication time, temperature and other extraction factors, were considered. Notably, the degradation of gallic acid, the target component, was observed during the UAE procedure, but the degradation products and mechanism were not clearly revealed [106]. Similarly, catechin, a common phenolic compound mainly found in tea leaves, degraded under specific ultrasonic irradiation, and the degradation ratio increased, increasing ultrasonic frequency and input power within a specific range. In addition, the authors established a mathematical model to predict the degradation level of catechin under different ultrasonic conditions [107]. In another study, 14 flavonoids (eriocitrin, narirutin, neohesperidin, quercitrin, eridictyol, didymin, naringenin, luteolin, sinensetin, nobiletin, tangeretin, naringin, hesperidin and quercetin) that are commonly found in citrus have been exposed to ultrasonication to investigate the sono-chemical effects on these flavonoids. The authors stated that the first thirteen flavonoids were relatively stable with only slight degradation being observed, whereas quercetin was degraded significantly by US treatment, especially in 80% ethanol aqueous solution, at lower temperatures and longer exposure times. Furthermore, four types of reactions were initiated by US irradiation, oxidation, addition, polymerisation, and decomposition, which led to the generation of dimer, alcohol addition, oxidation and decomposition products [108]. Phenolic acids were also tested to evaluate the sono-chemical effects and stability. In a study performed by Qiao, Ye, Sun, Ying, Shen and Chen [109], seven free phenolic acids were prepared in model systems and exposed to ultrasonic irradiation. The researchers found that five phenolic acids (protocatechuic acid, *p*-hydroxybenzoic acid, vanillic acid, *p*-coumaric acid, and ferulic acid) were stable, while two phenolic acids (caffeic acid and sinapic acid) were degraded under US treatment. Furthermore, they indicated that lower temperatures assisted the degradation, while decomposition and polymerisation reactions occurred simultaneously, thus generating corresponding decarboxylation products and dimers [109]. Later, the similar degradation of caffeic and sinapic acids under flat sweep frequency and pulsed ultrasound (FSFP) were observed, and it was noted that FSFP ultrasound had a stronger sonochemical effect on sinapic acid than caffeic acid [110]. In a model wine system, the production of the xanthylium cation, an oxidized dimer of catechin, from catechin was proven to be accelerated to some extent by long-term US treatment with an interval [111].

The degradation of β-carotene was investigated under high-power US and the main degradation products were three Z-isomers of β-carotene and seven β-apo-carotenals/ones [112]. The stability of all-trans lutein, one of the carotenoids under ultrasonication in the paper, has also been studied, and the results showed that US induced isomerisation to its isomers, including 13-*cis* lutein, 13'-*cis* lutein, 9-*cis* lutein and 9'-*cis* lutein, which increased with higher ultrasonic frequency and power [113]. Moreover, the authors noted that lower temperatures favoured the instability of all-trans lutein, which runs counter to the Arrhenius law, but is in accordance with previous results obtained by Qiao, Sun, Chen, Fu, Zhang, Li, Chen, Shen and Ye [108] and Qiao, Ye, Sun, Ying, Shen and Chen [109]. Furthermore, all-*trans* lutein epoxidation nearly occurred as the ultrasonic reaction time prolonged.

The US-induced degradation of cyanidin-3-glucoside, an anthocyanin commonly found in plant materials, has been reported by [114]. Unfortunately, however, the evaluation of degradation was performed only according to changes in the UV-Vis spectrum and a lack of HPLC and LC-MS data poorly support the degradation reaction and the mechanism. In another study concerning sono-chemical effects on cyanidin-3-O-glucoside, results showed that the formation of methylpyranocyanidin-3-O-glucoside from cyanidin-3-O-glucoside was accelerated in a simulation system, and the yield was increased by 32.5% by US irradiation at 100 W for 40 min [115].

More recently, a very interesting sono-transformation of amorphous tannic acid into regularly shaped crystalline ellagic acid particles has been reported, and the size, morphology and bio-activity of ellagic acid micro-nanocrystals was finely tuned by adjusting the appropriate US parameters [116].

As for the specific food matrix, a significant reduction in the amount of anthocyanins in mulberry juice has been obtained after US exposure [117]. However, other authors have claimed that ultrasonication at mild temperatures (43-45°C for 10-30 min) can enhance most of the bioactive compounds in orange juice as well as favouring radical scavenging activity, as compared to the control group without US [118]. Similarly, US (28 kHz, 60 W, 15 min) has been observed to cause a significant upsurge in the phenolic and antioxidant properties of lactic-acid-fermented mulberry juice [119]. In a study on the UAE of bioactive compounds from butterfly pea flower, it was noted that an increasing trend in the antioxidation activities of US-treated samples was observed, as compared to conventional extraction [54]. The use of US in the pre-drying treatment of an apple juice has been found to lower procyanidin polymerisation and the degradation of catechin dimers. Furthermore, the varying performance of US in different liquid media was revealed by the authors. In the case of distilled water, ultrasonication has been reported to caused almost an uncontrolled increase in polyphenol leakage, whereas in sucrose solution, the superficial impregnated layer was able to efficiently hamper polymer leakage [120]. Wang, Wang, Ye, Vanga and Raghavan [121] have demonstrated that 12-min of US treatment can enhance the antioxidant activity of strawberry juice. A research group has previously reported that sonication can reduce ascorbic acid content by 11% [122]. The influence of US on ascorbic acid in passion-fruit juice has also been studied during its storage. An immediate decrease in ascorbic-acid content, which was equal to that of untreated samples after the second day of storage, was induced by US treatment [123]. However, the stability of ascorbic acid under sonication at different temperatures has recently been investigated in a model juice system, and it was proven to be stable under all sonication conditions [124].

4.2. Starch

Starch is one of the most common and versatile biopolymers and it is in ample supply at relatively cheap prices. Starch exists as semi-crystalline granules in its native form, which limits its applications. It is therefore usually modified by genetic, chemical, physical or enzymatic methods to produce new desired properties. On the one hand, the widespread existence of starch in plant matrices means that ultrasonic processing would influence or even modify the starch component, leading to property alterations. On the other hand, the limitations on genetic techniques applied by legislation and the extensive public boycott on the use of genetic and chemical modification to create new features in food mean that physical methods are regarded as one of the most promising ways to modify starch in food. Ultrasonication, has received much attention in the starch-modification field. The influence of US on starch composition, the physical structure of starch granules, their physicochemical properties, some modifications and usage have been reviewed by Zhu [125]. Herein, we shall only focus on the sonochemical effect on starch and recent relevant research.

Corn starch pastes with varying amylose contents were exposed to ultrasonication, and the results have shown that the molecular scission that was initiated by US occurred at the C-O-C bond of the α-1,6 glycosidic linkage, and higher amylose content led to a lower breakage ratio, which was attributed to the aggregation of high-amylose starch pastes [126]. As for the graft polymerisation of starch and butyl acrylate, this reaction was strongly enhanced, in terms of graft ratio, graft efficiency and monomer conversion, by US irradiation [127]. Moreover, it has been found that the acetylation of dioscorea starch was always promoted by US [128]. In a study regarding the synthesis of water-soluble octenyl succinates of carboxymethyl starch, the authors reported that the esterification time was shortened to several minutes, from 24 h, using US irradiation and the dimethyl sulphoxide/*p*-toluenesulphonic acid system. Excellent emulsifying efficiency and surfactant performance properties were acquired [129]. US and pullulanase have been simultaneously applied to pea starch and a synergistic debranching effect was observed, producing 73.5% linear glucans, 18% slowly digestible starch and 26% resistant starch in the resulting product after 6 h of debranching [130].

Starches with different granule sizes have been exposed to US irradiation (25 kHz), and it was revealed that the US caused profound cavities and fractures on the granule surface, and starch with larger granule sizes was affected more intensely by ultrasonication. Moreover, peak viscosity increased with US, while swelling power and solubility decreased after ultrasonic irradiation [131]. However, in a study concerning sweet potato starch, US treatment (dual frequency) caused a lower peak-viscosity value, increased starch solubility and transmittance [132]. In the relatively high frequency range of US (0.5~1 MHz), the maximum number of pits per granules has been found to be proportional to the US frequency, with values of approximately 7, 10 and 11 at 0.50, 0.85, and 1 MHz, respectively. Furthermore, there was an optimum granule size for which a maximum pit number was obtained, while larger or smaller granule sizes resulted in fewer pits per granule [133]. The influence of US on the granule porosity of starch that is derived from a range of different plant materials has also been investigated by Sujka [134]. They noted a significant increase in the specific surface area of all studied starches after ultrasonication. The sono-effect on the average diameter of mesopores differed with the varying conditions [134]. The physical and structural properties of native starch have been observed to improve under the effect of an ultrasonic field in an acidic environment, and the double helix structure was modified. The particle size of starch changed from 1596 nm to 80 and 42 nm after ultrasonication and acid hydrolysation [135]. Apart from modifications to starch properties, ultrasonication has also been used to improve the hydration process without modifying any starch properties, which was mainly attributed to inertial flow and the sponge effect [136].

4.3. Bioactive polysaccharides

Polysaccharides are another class of common and important component that are usually used to modify the physical and flavour properties of food. The structure of polysaccharides is generally presented as linear, while random coil structure and helices exist in certain solvent conditions. The different conditions and structural properties of polysaccharides can lead to ultrasonication having a range of different effects. It has been proven that high-intensity US can trigger a large number of sono-chemical reactions in polysaccharides, such as glycosylation, acetalisation, oxidation, C-D, C-heteroatom, and C-C bond formations [7, 137]. It has also been shown to assist a variety of representative carbohydrate syntheses, including hydroxy-group manipulation (acylation, protection/deprotection, acyl group migration), thioglycoside synthesis, azidoglycoside synthesis, 1,3-dipolar cycloaddition and the reductive cleavage of benzylidene, and series of glycosylation reactions that employ thioglycosides, glycosyl trichloroacetimidate, glycosyl bromide and glycosyl acetate as the glycosyl donors [138]. Most of the available references are mainly related to the UAE of polysaccharides from different species and/or different tissues. Solubility, basic reactivity, thickening, gelation and digestibility have been carefully reviewed by Hao Feng, Gustavo V. Barbosa-Cánovas and Weiss [7]. Herein, we shall only focus on recent reports of the influence of US on bioactive polysaccharides and its tendency to modify these species.

The polysaccharides extracted from leaves of *Rhododendron aganniphum* have been found to possess higher positive radical-scavenging activity than the control group for hydroxyl, superoxide and 1,1-diphenyl-2-picrylhydrazyl (DPPH) radicals when exposed to ultrasonic irradiation [139]. A similar phenomenon has been observed by Hou, Wu, Kan, Li, Xie and Ouyang [140] in chestnut polysaccharides (*Castanea mollissima* Blume). They found that the transition temperature, enthalpy value, and characteristic FT-IR spectrum was changed by US irradiation. Moreover, antioxidation activity, in terms of reducing power, DPPH-, ABTS- and hydroxyl radical-scavenging activity, were significantly enhanced by ultrasonication. Polysaccharides from *Carex meyeriana* Kunth have been investigated after serval processes, and it was proven that higher antioxidation activities, as evaluated by DPPH and ABTS radical scavenging activity, were obtained after ultrasonic treatment [141]. A dextran sodium sulphate colitis mouse model has been used to prove that the intestinal anti-inflammation activity of polysaccharides from seeds of *Plantago asiatica* L. was enhanced by ultrasonic irradiation. This effect was demonstrated by a decrease in macrophage inflammatory protein-2 secretion, and levels of inflammatory cytokines in inflamed colons [142]. In addition to US's ability to enhance partial radical-scavenging activity (hydroxyl radical and ferrous ion), ultrasonic modification has been shown to cause polysaccharides, derived from the seeds of *Ziziphus jujuba* Mill var. *spinose*, to possess a notably stronger effect on inducing Cyclooxygenase-2 (COX-2) expression in RAW264.7 macrophages, as compared with hot-water extraction treatment [143]. The influence of US on the bioactivities of polysaccharides derived from blackcurrant fruits have also been investigated, and the authors stated that US irradiation clearly enhanced the antioxidant, α-amylase and α-glucosidase inhibition activities of the polysaccharides. In terms of antioxidation activities, lipid peroxidation inhibition, and DNA-damage protection activities were also included, in addition to partial radical scavenging ability [144]. The polysaccharide-protein complexes that were derived from a number of mushrooms [*G. frondosa* (maitake), *C. versicolor* (Yunzhi) and *L. edodes* (shiitake)] have been processed by US and showed generally higher antioxidation activities than those treated using conventional hot-water extraction [145]. However, there are also some published articles that have reported opposing results. Kang, Chen, Li, Wang, Liu, Hao and Lu [61] have reported that polysaccharides from *Ganoderma lucidum* showed lower antioxidant activity after ultrasonication than after a hot-water extraction process. The activity was evaluated using the reducing power, DPPH radical scavenging ability and cellular protective effect from ultraviolet radiation damage in yeast cells.

Although it has been proven that a large number of sono-chemical reactions of polysaccharides can be initiated by high-intensity US [137, 138], previous research has mainly been performed under specific organic chemical reaction conditions, which are not perfectly capable of accounting for the sono-chemical reactions in current complex food systems. A greater number of investigations into sono-chemical reactions in food and delicate food model systems therefore need to be performed. Furthermore, although the influence of US on bioactivities has been extensively investigated, the mechanisms and structural modifications, especially in functional groups, that are related to these bioactivity changes are still to be clarified. Additionally, in polysaccharides research from mulberry leaves, the authors have stated that antioxidant activity decreased with increasing polysaccharide purity, and highly concentrated polysaccharides were proved to have very little antioxidant activity. However, antioxidant activities significantly improved after the addition of quercetin [146]. This phenomenon may be attributed to the specific polysaccharide source, and it is still necessary to enlarge the range of polysaccharide sources to evaluate the bioactivity of polysaccharides from different plant and animal tissues. Moreover, in most conditions, polysaccharides are investigated as crude polysaccharides or as a mixture containing other components without further purification. Bioactivity, under some conditions, may therefore include contributions from other components and not only from the polysaccharides themselves. The influence of US on polysaccharides and other components related to the polysaccharides, and the synergistic or antagonistic effects between them, are therefore expected to be clearly elucidated in the near future.

4.4. Proteins

Proteins, are an essential nutrient for humans. Not only can they serve as a source of exogenous amino acids for the human diet, they are critical functional ingredients of food to grant them special flavours, and some physical properties, such as the stabilisation of foams and emulsions, gelation and viscosity. High-intensity US can lead to the modification of proteins via sono-chemical effects, thus causing functionality changes in proteins. The basic effects of high-energy US on functional properties, such as solubility, water-retention capacity, gelling properties, foaming properties and emulsifying properties has been carefully reviewed by Higuera-Barraza, Del Toro-Sanchez, Ruiz-Cruz and Márquez-Ríos [147] . More recently, further cases of foaming-property modification have been appropriately summarised by Gharbi and Labbafi [148]. Herein, we shall only update the readership on advances in functional-property modification by US, as well as providing some insights into its influence on structural modification.

In terms of functional-property modification by US, a recent study concerning the exposure of whey protein to ultrasonication has demonstrated that US treatment can provide a narrow and smaller particle size distribution (0.683 ± 0.225 μm) upon the treatment of a broad bimodal particle size distribution (2.453 ± 0.717 μm). Significantly higher solubility and a considerably higher storage modulus (G') were thus granted. The product was also more elastic and had significantly higher heat stability, as compared with the untreated group [149]. The SDS-PAGE profile showed marked decreases in the band density of low molecular weight molecules (β-lactoglobulin and α-lactalbumin), as compared to untreated whey protein. Structural analyses revealed that a smaller, regular and more homogenous structure was acquired after ultrasonication. Amiri, Sharifian and Soltanizadeh [150] have reported that US irradiation led to better water-holding capacity, gel strength, emulsifying properties, elasticity, stiffness and reducing viscosity in beef myofibrillar protein, whereas the opposite trend was observed after 30 min of treatment. However, Zhang, Regenstein, Zhou and Yang [151] have indicated that the water-holding capacity of myofibrillar protein gel only improved, due to a denser and uniform gel microstructure, in the moderate intensity range (no more than 600 W) of US. Furthermore, they observed an increase in solubility, surface hydrophobicity and decreased turbidity after high intensity US treatment. High-intensity US has also been used to modify whey proteins, and several functional properties were changed, including decreased particle sizes, increased surface hydrophobicity, solubility, emulsion activity and stability [152].

As far as structural modification is concerned, De Leo, Catucci, Di Mauro, Agostiano, Giotta, Trotta and Milano [153] have revealed the structural changes to membrane proteins that are responsible for the photosynthetic reaction triggered by US, causing the denaturation of the protein and the loss of its ability to absorb and convert light energy into a charge-separated state. Multi-frequency power US has been employed to unfold the structure of *zein* and *glutelin*, and different ultrasonic modes led to varying changes in the α-helices and β-sheets of *zein* and *glutelin*. This process could be utilised for protein proteolysis [154]. It has been proven that the use of power US treatment during a beef-brining procedure increased beef-protein aggregation, via disulfide crosslinking, augmented β-sheet content, at the cost of decreasing α-helix content, and free sulfhydryl residues as well as protein surface hydrophobicity [155]. However, the treatment of chicken myofibrillar protein with pulsed US has led to different structural changes. A decrease in α-helices and β-sheets, and an increase in the β-turns of chicken myofibrillar protein were observed after pulsed ultrasonication. Furthermore, the reactive sulphur (SH) content and disulfide bonds (S-S) of samples increased while the total SH contents decreased in the first 6 min of treatment [156]. A different result still has been observed by Yang, Li, Li, Oladejo, Ruan, Wang, Huang and Ma [157] in rice protein after ultrasonication. They reported a decrease in α-helix and β-turn content together with increases in β-sheet and random coil content after a variety of ultrasonic treatments. Furthermore, ultrasonic treatment also caused protein deformation and unfolding leading to hydrophobic groups being exposed. Yang, Li, Li, Oladejo, Wang, Huang, Zhou, Wang, Mao, Zhang, Ma and Ye [158] have confirmed that ultrasonication is able to unfold defatted wheat-germ protein, loosen protein structure and expose more hydrophobic amino-acid residues for access by alcalase. It can therefore be summarised that US generally led to decreases in α-helix content, several changes in β-turn and β-sheet content and an increase in random coils, suggesting that the protein structure unfolded and was deformed to range of degrees as a result of the varying acoustic-field conditions. This treatment generally led to the exposure of inner hydrophobic amino-acid residues. Jambrak, Mason, Lelas, Paniwnyk and Herceg [159] have indicated that both the high-intensity US types (probe and bath) caused a decrease in the particle size and molecular weight of whey proteins. Although enzymes are generally considered to be inactivated by the structural changes that US causes [160], the active effect of US on some α-amylases (heat-stable (HSA) and liquozyme (LQA) from *Bacillus licheniformis*) has recently been suggested as providing beneficial structural modifications. This highlights the potential of US to modulate the activity of α-amylase [161].

It becomes clear that the structural changes in proteins that are caused by ultrasonication basically depend on the nature of the protein, the ultrasonic wave-energy intensity, sonication time and medium conditions, including pH, temperature, ionic strength, solvent polarity, etc. Although extensive research has been carried out on the structural and functional changes that result from ultrasonication, there is still a lack of detailed information on precise descriptions for the structural and conformational changes at the “structural biology” level, which could clearly shed light upon sonication-driven functional modification. Furthermore, the underlying mechanisms and the procedure for the ultrasonic modification of proteins still require exploration. Changes in sulfhydryl groups indicate that free radicals, produced during cavitation, may react with amino-acid residues. This suggests that sono-chemical effects may carry more weight on protein modification than previously thought. It may therefore be of the upmost importance that more attention be paid to the chemical modification of protein under high intensity US, as it may be able to influence the flavour of food materials and enzyme activity.

5. Trends, challenges and recommendations

Trends

The advantages of UAE are clear and, for this reason, the food industry is particularly interested in its adoption. There are therefore several novel strategies for this technique's development in order to improve extraction efficiency and meet the requirements of "green chemistry". Combinations of the US technique with other novel or conventional technologies are very much a hot topic. These combinations mainly include microwaves, supercritical fluids and enzyme-assisted extractions being coupled with US in order to utilize the synergistic effects of these techniques. In order to meet the demands of green extraction, UAE can replace conventional extraction solvents with novel solvents, such as ionic liquids, deep eutectic solvents, multi-phase solvents, cloud point techniques etc. Moreover, new developments in the design of ultrasonic instruments to maximise US's contact with a sample matrix in a continuous flow system are in demand. In fact, "ultrasonic circulation extraction" is a typical configuration that has been proposed by F. Chen, Q. Zhang, J. Liu, H. Gu, and L. Yang (2017). It includes a specially designed U shape vessel, an ultrasonic probe and stirrer, which drives the circulation flow of the solvent. In this case, the concentration of the ultrasonic power into a smaller part of the vessel and even exposure of the food matrix to US energy converge to provide higher yield ratios and rates. Some studies into the scaling of UAE from the lab to pilot and industrial scales have been proposed. An industrial scale ultrasonic reactor working in batch mode was initially created for natural-product extraction in 1998 [162]. Later, more large-scale UAE devices, such as a multi-horn flow system, a multi-transducer sonication plate, etc., were built [163]. Large-scale UAE equipment has been applied for polyphenol extraction from apple pomace by Pingret, Fabiano-Tixier, Bourvellec, Renard and Chemat [164], and they reported that a 30% higher yield of total phenolic content and higher antioxidant activity were obtained using UAE, as compared to the conventional method. A scale-up investigation of green-tea-polyphonic compound extraction has been performed in continuous flow mode with US assistance, and it was proven that markedly higher extraction yields were obtained with UAE [165]. More recently, it has been reported that a high-power, industrial-scale US device (possessing 2 tons/h processing capacity) provided significant enhancements to the extraction yield (22.7%) of virgin olive oil, and the resulting oil possessed higher phenol content than the control oil [166]. As reported by Cravotto *et al*., efficient big scale production requires a process intensification with flow US reactors [167].

Regarding UAF, research has mainly focused on improving freezing efficiency and the quality of the frozen products. Similarly to UAE, there are many novel physical techniques, apart from US, that have been evaluated and adopted to assist freezing. These include microwaves, static electric fields, high-pressure, etc. However, coupled applications that make the most of synergistic effects are still rare, and thus require further investigation. Pre-existing gas bubbles, especially micro-nano bubbles, have been proven to promote nucleation, accelerate the freezing process and even decrease ice crystal size. With respect to quality improvement in frozen products under UAF, novel cryoprotectants, such as soluble soybean polysaccharide, have been introduced to protect the quality of frozen products. More cryoprotective compounds, such as Konjac Glucomannan Hydrolysates, trehalose, alginate oligosaccharides, etc., should also be taken into consideration in the near future as means to take advantage of synergistic protective effects that are similar to the soluble soybean polysaccharide case [91]. Some research into the use of plastic wrapping to prevent direct contact between the coolant and the food matrix in UAF processes has been performed (W. Gao, Hou, & Zeng, 2019). However, the development of nontoxic or harmless coolants for most immersion freezing or half-immersion freezing conditions that are assisted by US could protect food materialand is thus another trend for UAF. Although UAF is a useful technology for the food-freezing industry, there is still a lack of large/pilot scale equipment for the scale-up of this technique from the lab scale. Although the funding that is needed for the development of large-scale UAF equipment is considerable, it is necessary if the industry is to take advantage of UAF. Furthermore, operating conditions and parameters for large-scale use still require further optimisation in future work.

Challenges

- Despite the comparatively low cost of ultrasonic equipment, scale-up for industrial production, or even pilot-scale, use is slow because the need of *ad hoc* tailored plants, limiting the investigation and optimisation of large-scale practices.
- The impact on the stability of components in food matrices under US treatment for excessive time periods or at extremely high-power intensities could lead to notable compound degradation or oxidation.
- Contamination by coolants can affect frozen-product quality under UAF.

Recommendations

- Besides specifying general process parameters, some good practice issues that are often neglected also need to be highlighted in papers. These include the volume, material, geometrical features, and the position of the extraction vessel. Furthermore, the US power that is exerted on an extraction matrix should be determined and clearly mentioned.
- The aluminium foil test needs to be conducted to determine the effective position of the extraction vessel in an ultrasonic bath. The use of hydrophones or better of cavitometers can provide important information on the intensity and distribution of cavitation. The actual ultrasonic power entering the vessel also needs to be determined using calorimetric measurements.
- For scale-up experiments, ultrasonic intensity must be taken into consideration along with the geometric design, position and the ultrasonic power entering the extraction vessel.
- UAE is not a linear process, meaning that simply increasing the size of ultrasonic equipment is impractical, as has been shown by results obtained from the laboratory-scale experiment. Other process parameters must therefore be taken into consideration during scale-up. These include, kinetic studies, US intensity, power, geometrical design, etc.
- Lower frequency and power intensity should be preferred for US treatments in order to avoid the detrimental physicochemical effects that can be triggered by US. In addition, some strong reducing agents, such as ethanol and ascorbic acid should be added to scavenge the free radicals that are generated by cavitation, thus

protecting food components.

- It is imperative that researchers and the manufacturers of US devices more closely cooperate to meet the challenges of ultrasonic instrument for better ultrasonic-device efficiency for industrial uses.
- Conventional extraction solvents, which are usually harmful and toxic, can be replaced with less toxic or even non-toxic and environmentally friendly solvents.
- More research is needed to prevent direct contact between coolant and food matrices under UAF.

6. **Conclusions**

US applications have demonstrated the possibilities and promising trends of enhancing processing efficiency and food-product quality in many application aspects under mild processing conditions, whether US is used alone or in hybrid reactors. The physicochemical effects initiated by US, such as those provided by the UAE and UAF procedures, play critical roles in process-rate enhancements as well as in nutritional and sensory quality improvements in target food materials and food-derived compounds. The assistance that US provides in UAE and UAF processes is basically driven by sono-physical effects; UAE is mainly based on cell disruption, the formation of microjets and microchannels, and mass transfer enhancements caused by acoustic cavitation, while UAF basically depends on assisting nucleation (primary nucleation), the mechanical breaking of existing ice crystal into new tiny ice cores (secondary nucleation), and mass and heat transfer enhancements. However, sono-chemical effects will cause notable degradation and oxidation as well as inducing some specific chemical reactions under a variety of conditions while food materials are exposed to US. These sono-chemical reactions are generally complicated because of the complexity of food matrices and varying reaction and ultrasonic conditions. Nevertheless, sono-chemical effects on food systems during US treatment are usually overlooked and still unclear. The chemical reactions and mechanisms initiated by US under specific food and treatment conditions therefore need to be systematically investigated, which would make it possible to take advantage of sono-chemical effects while eliminating the US's detrimental impact in specific application scenarios, especially in relation to functional properties and bioactivity modification. Moreover, investigations into US processing, in particular UAE and UAF, are mainly on the laboratory-scale, meaning that the scale-up to pilot-scale, or even industrial-scale, applications will require specific attention and optimisation, which will be a major step in its commercial exploitation and will ensure that the best use of this emerging, sustainable and green technique will be made.

Figures

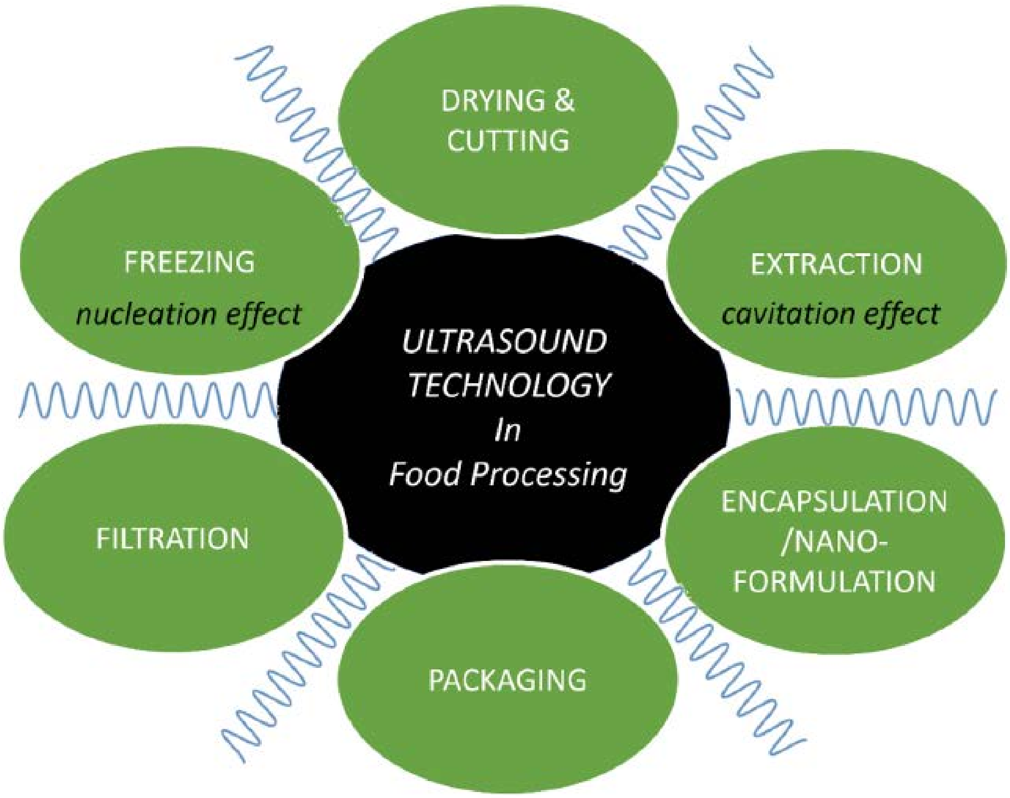


Figure 1: Use of ultrasound technology in food processing

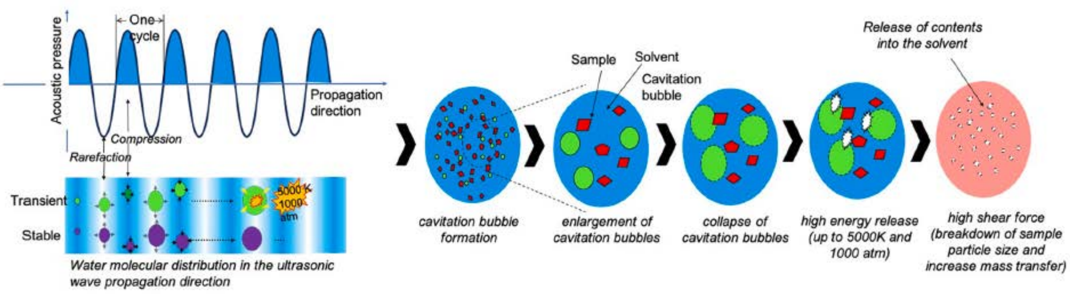


Figure 2: Possible mechanism of Ultrasonic-assisted extraction (UAE) during food processing

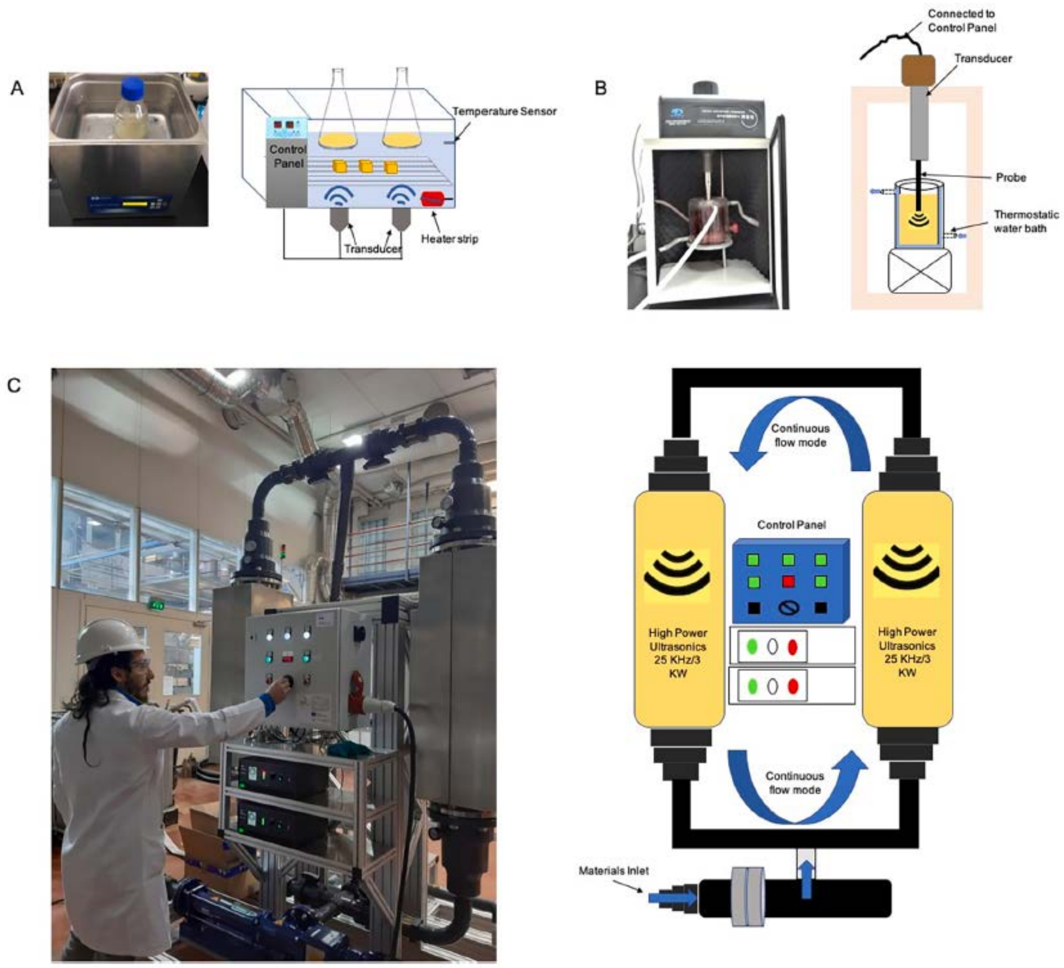


Figure 3: Different types of UAE instrumentations used during food components extraction

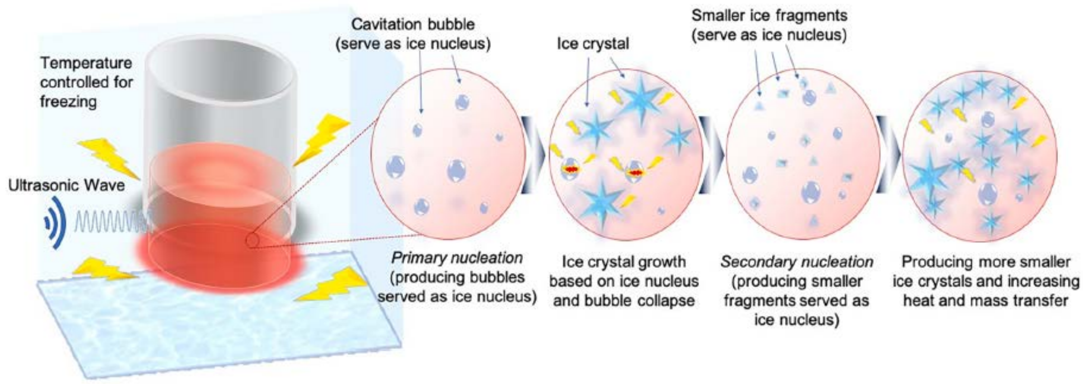


Figure 4: Possible mechanism of Ultrasonic-assisted freezing (UAF) during food processing

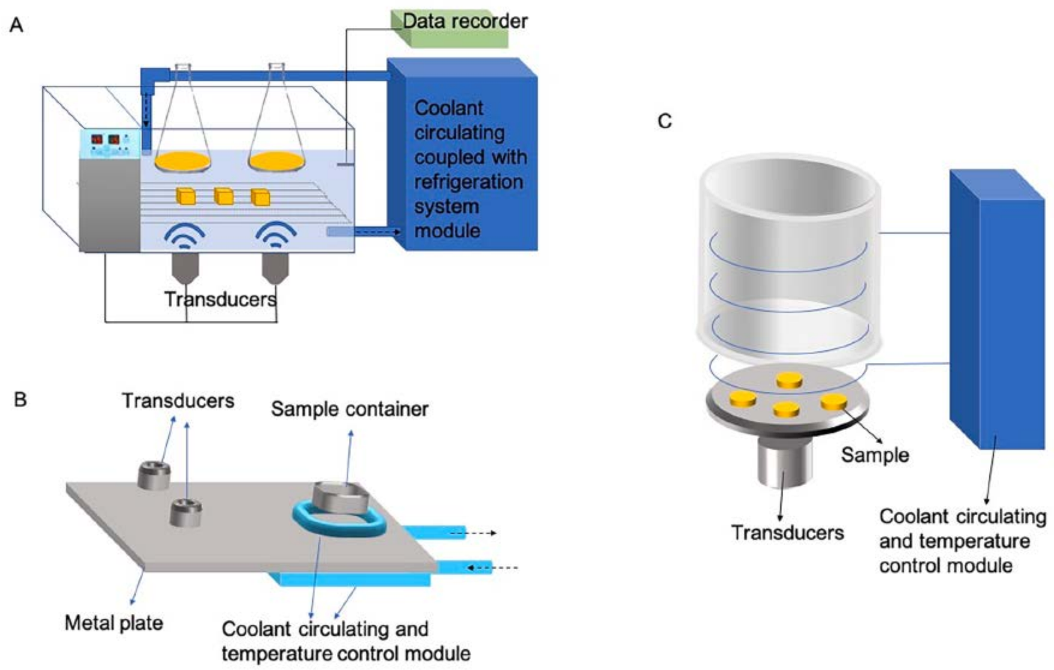
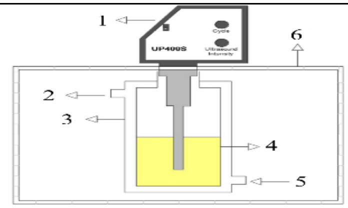
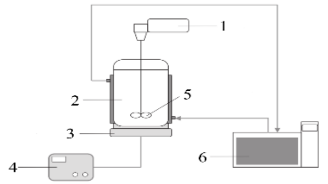
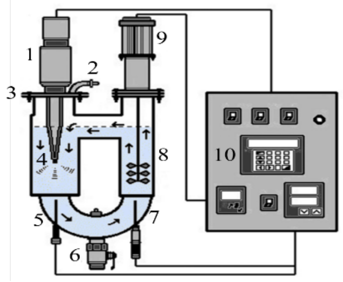


Figure 5: Different types of UAF instrumentations used during food processing. (A) Full immersion, (B) Half-immersion and (C) Non-immersion

Table 1: Modified UAE instruments used in food-component extraction over the last 3 years (2016 onwards)

Matrix	Target component	Diagram note	Device diagram	Reference
Mortierella isabellina	lipid	1. High-intensity ultrasound processor; 2. Water; 3. Jacketed; 4. Biomass+solvent; 5. Water; 6. Soundproof box;		[168]
Cichorium intybus L. var. *sativum* (chicory) grounds	polyphenols	1. Motor; 2. Double-wall glass extractor; 3. Transducer; 4. Ultrasound generator; 5. Double pale agitator; 6. Thermostat;		[169]
Trollius chinensis flowers	orientin and vitexi	Ultrasonic circulating extraction: 1.Ultrasonic generator, 2. vent, 3. inlet, 4. transducer, 5. heater, 6. outlet, 7. temperature sensor, 8. agitator, 9. reflux-circulating controller, 10. control system;		[170]

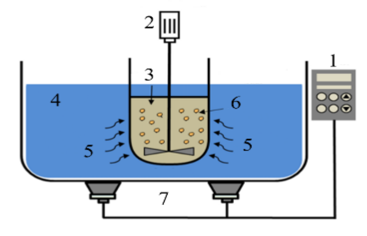
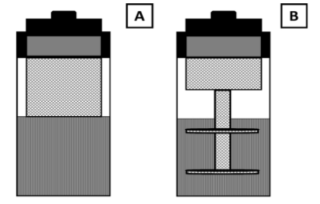
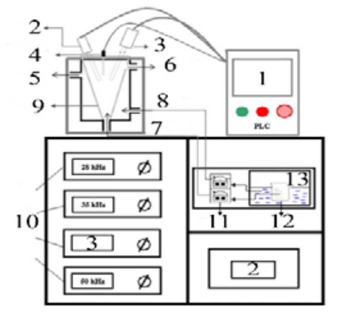
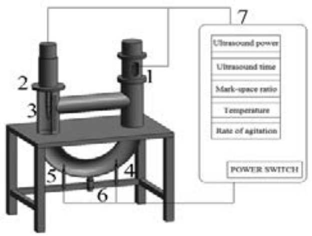
Taxus chinensis	paclitaxel	1. Ultrasound generator; 2. Mechanical stirrer; 3. Methanol; 4. Water; 5. Ultrasound; 6. Biomass; 7. Ultrasound transducer;		[171]
Agave salmiana bagasse	antioxidants and saponins	Ultrasound transducers combined with supercritical fluid extraction: (A) cylindrical head-mass transducer, (B) multiplate head-mass transducer;		[72]
Helianthus annuus L. (Sunflower) meal	Protein	Dual frequencies ultrasound equipment: 1.Touch panel, 2.20 kHz, 3.40 kHz, 4.Temperature sensor, 5.Water outlet to water bath, 6.Sample outlet to sample vessel, 7. Sample inlet, 8.Water inlet, 9.Process chamber, 10.Ultrasonic generators with varying frequencies, 11. Two pumps, 12. Sample, 13. Water bath;		[172]
Idesia polycarpa	oils	ultrasonic homogenate-circulating extraction equipment: (1) agitator, (2) heater, (3) temperature sensor, (4) inlet, (5) outlet, (6) ultrasonic generator, (7) control panel (including power, time, temperature, mark-space ratio, rate of agitation control and power switch).		[173]

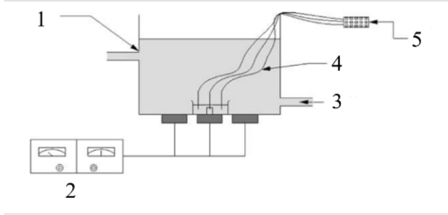
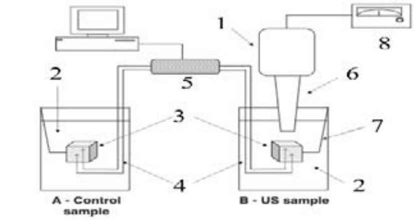
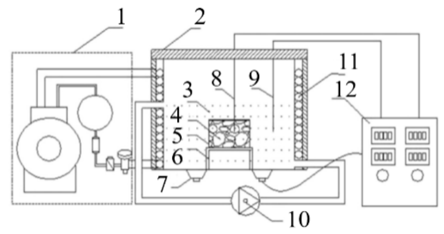
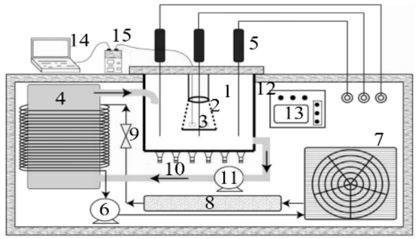
Table 2.Extraction of various food components as assisted by US over the past 3 years (2016 onwards)

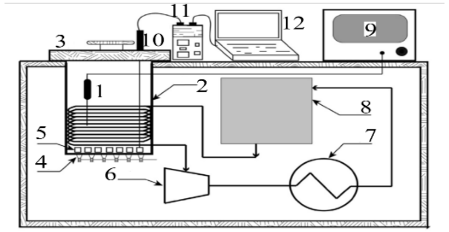
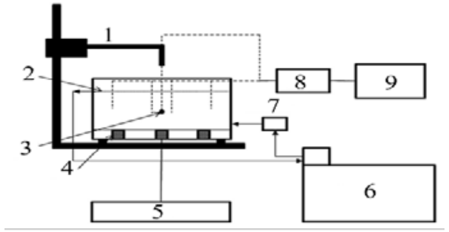
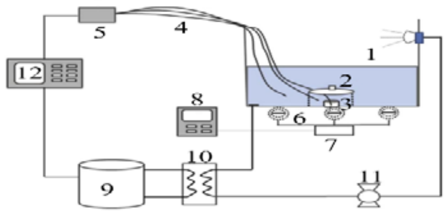
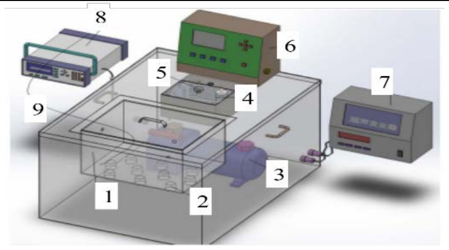
Plant material	Target component	Solvent	Final operating condition	Major results	references
Zanthoxylum bungeanum leaves	Flavonoids	60% (v/v) aqueous ethanol	Leave size of 40 mesh, temperature of 50 °C, ultrasonic power of 400 W, 15 min, the ratio of leaves weight (g) to ethanol solution volume (mL) was 1:25 (w/v)	Maximum four flavonoids yield of 120.84 mg/g	[174]
Citrus aurantium peel	Phenolic compounds	50% (v/v) aqueous ethanol	Fresh peel, 4 g of dry ground peels or 9 g of fresh peels immersed in 200 mL solvent, 20 kHz, power 130 W, 12.5 min at 80% radiation; probe-type sonicator	Maximum phenolic content yield of 40.95 mg/g	[175]
Solanum tuberosum L. (potato) by-product from industry	Phenoliccompounds	55% (v/v) aqueous ethanol	ultrasound bath during 35 min at 35 °C and 1:10 for the sample/solvent ratio	Maximum yield of phenolic compounds in five potato species	[176]
Camellia sinensis (green tea) leaves	polyphenols and flavonoids	water	Temperature of 77°C, tea to water ratio of 73 g/L, amplitude of 77%, power of 500 W, frequency of 20 kHz and 30 min	Maximum yield for total polyphenolsconcentration of 12318 mg_{EAG}/L and total flavonoids concentration of 3774 mg_{R}/L	[177]
Citrus reticulata Blancocv. Sainampueng (mandarin) peel	Phenolic compounds and hesperidin	80% (v/v) aqueous acetone	48 °C of temperature, 56.71W of ultrasonic power for 40 min	Maximum yield (26.52%), total phenolic(15,263.32 mg Eq gallic/100 g DW) and hesperidin (6435.53 mg/100 g DW); UAE showed greater extraction efficiency than microwave-assisted extraction for the same extraction temperature and time	[178]
Avena sativa L. (defatted oat)	Phenolic compounds	80% (v/v) aqueous ethanol	600 W of ultrasonic power, frequency of 40 kHz, 70 °C of temperature, 25 min	Maximum phenolic content yield	[179]
defatted soybean flour (genotype is “Laura”)	Phenolic acid	methanol and 10% HCl ina ratio of 85:15 (v/v)	20 kHz of ultrasonic frequency, 25 °C of temperature, 30 % amplitude of maximum power, 10 min	Higher yield of phenolic acid	[180]

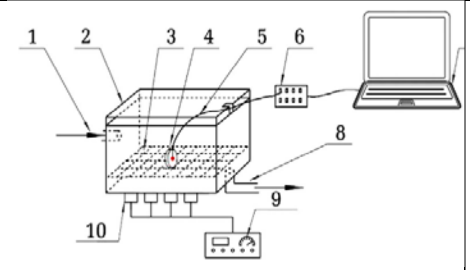
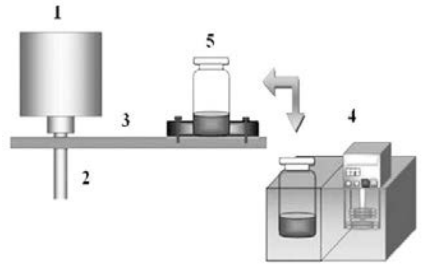
Olive (olive tree pruning biomass (OTP) and olive millleaves (OML))	Antioxidant (manly phenolic compounds and flavonoid)	ethanol/water ratio 54.5% and 51.3% for OTP and OML, respectively	70% amplitude, 15 min, power of 400 W, 24 kHz of frequency, continuous mode with no pulse without temperature control	Maximum yield of total phenolic content and total flavonoid content were obtained;	[181]
Hibiscus sabdariffa calyces	anthocyanins	39.1% (v/v) aqueous ethanol	26.1 min for extraction time, 296.6W of power, 30 g/L of solid/liquid ratio, 30-35 ºC, frequency of 20 kHz	UAE was the most efficient method yielding 51.76 mg delphinidin-3-O-sambubioside + cyanidin-3-O-sambubioside/ g residue.	[182]
Zingiber officinale Roscoe (ginger)	Gingerols and polysaccharides	$[C_4mim]BF_4$ concentration of 1.5 mol/L (ionic liquid),	ultrasonic power of 200 W, 25 ºC, solid/liquid ratio of 1:20 (g/ml) and extraction time of 10 min	significantly improved yield of total gingerols (12.21 mg/g) and ginger polysaccharides compared with traditional extraction methods	[183]
Helianthus annuus L. (Sunflower) meal	Protein	Water (pH was adjusted to 8.0 by 1 mol/L NaOH)	Dual frequencies at 20/40 kHz, power density (220 W/L), temperature (45 ºC) and extraction time (15 min)	Maximum protein content yield of 54.26% and 0.13 kW hr for energy consumption	[172]
oil peony seed (cakes)	Monoterpene Glycoside	67% (v/v) aqueous ethanol	liquid-to-solid ratio of 27 mL/g, 120 W of ultrasonic power, ultrasonic extraction time of 16 min, ultrasonic extraction temperature of 26 ºC	Maximum monoterpene Glycoside yield of 10.24%	[184]
citrus aurantium L. (sour orange) peel	pectin	Acidified distilled water (with citric acid)	ultrasound power of 150W, irradiation time of 10 min and pH of 1.5, 20 kHz of frequency, 20 v/w of the liquid/solid ratio,	Maximum extraction yield was 28.07±0.67%	[185]
Juglans regia L. (walnut) kernel	oil	n-hexane solvent	ultrasonic time of 47 min, 2.0% cellulase concentration and 111 min incubation time, ratio of 1:4 solid/solvent, 45±5 ºC of temperature,	Maximum yield of oil; similar color parameters, higher total phenolic content, iodine value, higher unsaturated fatty acids, and lower peroxide value compared with conventional method;	[186]
Idesia polycarpa	oil	Water with enzyme	liquid-solid ratio of 7.13, the incubation time of 2.94 h, and the incubation temperature of 48.74 ºC, enzyme (hemicellulose:	maximum fruit oil recovery of 79.36% withan error of 0.17%	[173]

			cellulose: pectinase=1:1:1, w/w/w), other factors are not significant variables according to authors.		
Moringa oleifera seeds	oil	petroleum ether	ultrasonic power of 200W, frequency of 40 kHz, temperature of 30 ºC, ultrasonic time for 5 min and stirred for 15 min at 325 r/min	Maximum oil yield of 35.77 % under this condition	[187]
Inonotus hispidus (mushroom)	melanin	1.5 mol/L NaOH aqueous	NaOH concentration of 0.56 mol/L, solid-liquid ratio of 1:50, ultrasonic power of 300 W, extraction temperature of70 ºC, and ultrasonic time of 70 min	Extraction rate increased by 37.33% compared with the non-ultrasound control group.	[188]
Cassia singueana Del. (Fabaceae) root	anthraquinones	methanol	extraction time 25.00 min, temperature 50 ºC, and solvent to sample ratio of 10 mL/g.	Maximum extraction yield of 1.65±0.07% for crude anthraquinones	[189]
Cymbopogon martinii	Geraniol	65 mL volume of 1 mol/L aqueoussolution of sodium cumene sulfonate	65% ultrasound amplitude, 60 W ultrasound power, 70% cycle time, and 16 min of sonication time, 26 kHz of frequency and 200 W of power	Improved geraniol yield of 1.9012% (w/w)	[190]
olive pomace	hydroxytyrosol, maslinic acid and oleanolic acid	ethanol concentration of 90% in water	extraction temperature of 50 ºC, extraction time of 5 min, liquid to solid ratio of 30 mL/g, ultrasound intensity of 135.6 W/cm^2, and ultrasound frequency of 60 kHz	Maximum yield of target components, and UAE is effective and greener technique with the lowest E factor, energy consumption and carbon emission compared with microwave-assisted extraction and solvent extraction	[191]

Table 3.Modified UAF instruments used in preserving food over last 10 years (2009 onwards)

Matrix	Category	Diagram note	Device diagram	Reference
Apple	full-immersion	1. Ultrasonic bath; 2. Generator; 3. Refrigerated circulator; 4. Thermocouples; 5. Data logger		[90]
Potato	full-immersion	1. Transducer; 2. Cooling solution (-6 ºC); 3. Potato cubes; 4. Thermocouples; 5. Data logger; 6. Sonotrode; 7. Retaining needle; 8 Ultrasound generator		[95]
Strawberry	full-immersion	1. Refrigerating system; 2. Ultrasonic tank; 3. Coolant; 4. Samples; 5. Perforated cage; 6. Samples holder; 7. Ultrasonic transducer; 8 and 9. Thermometer probe; 10. Refrigerated circulator; 11. Evaporator; 12. Control panel		[88]
Mushroom	full-immersion	1. Freezer cavity; 2. Sample holder; 3. Samples; 4. Coolant reservoir; 5. Thermocouple; 6. Compressor; 7. Condenser; 8. Reservoir for refrigerant; 9. Valve; 10. Ultrasound transducer; 11. Immersion fluid circulation pump; 12. Control panel; 13. Digital screen; 14. Computer; 15. Digital thermometer		[39]

Mushroom	non-immersion	1. Built in thermocouple; 2. Freezer cavity; 3. Cover; 4. Ultrasound transducer; 5. Samples; 6. Compressor; 7. Condenser; 8. Reservoir for refrigerant; 9. Control panel; 10. Thermocouple probe; 11. Digital thermometer; 12. Computer		[93]
Potato	full-immersion	1. Stand; 2. Fluid level; 3. Potato/copper sphere; 4. Transducer; 5. Ultrasonic generator; 6. Refrigerated circulator; 7. Flow meter; 8. Data logger; 9. PC		[89]
Radish	full-immersion	1. Immersion freezing tank; 2. Sample cage; 3. Sample; 4. Type K thermocouples; 5. Temperature controller; 6. Ultrasound transducers; 7. Ultrasound generator; 8. Control system of ultrasound; 9. Refrigeration units; 10. Refrigeration cycle system; 11. Pump; 12. Control panel		[97]
Porcine longissimus muscles	full-immersion	1. Immersion freezing tank; 2. Sample cage; 3. Sample; 4. Type K thermocouples; 5. Temperature controller; 6. Ultrasound transducers; 7. Ultrasound generator; 8. Control system of ultrasound; 9. Refrigeration units; 10. Refrigeration cycle system; 11. Pump; 12. Control panel		[40]

Grass carp surimi	full-immersion	1. Circulating freezing liquid inlet; 2. Ultrasonic processor; 3. Special rack bar; 4. Experimental sample placement; 5. T-type thermocouple; 6. Multichannel data recorder; 7. Computer; 8. Circulating cryogenic liquid outlet; 9. Ultrasonic control panel; 10. Ultrasonic transducer		[91]
Mannitol's aqueous solution	half-immersion	1. Ultrasonic transducer; 2. Coupling element; 3. Vibrating plate; 4. Cooling bath; 5. 4ml glass vial fixed to the plate and filled with mannitol solution		[94]

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