

Review of ultrasound combinations with hybrid techniques for pectin extraction from agri-food by-products

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ABSTRACT

Pectin is a plant cell wall structural polysaccharide. It exhibits a variety of properties, including stabilizing, emulsifying, and thickening effects. Consequently, it is widely utilized in the food industry. Pectin extraction is commonly done by conventional method, which includes the use of mineral acids with heating, and obtains varying pectin yield and quality. Ultrasound technique is one of the novel methods for pectin recovery, that has improved its properties. Therefore, this paper presents a brief review about the pectin sources, structure, widespread applications, extraction possibility based on using ultrasound technique within and without the combination with other non-conventional techniques (enzymes, microwaves), and evaluation of the yield and physicochemical characteristics of the extracted pectin. As indicated by the cited literature, the application of hybridized methodologies generally exhibits higher extraction efficiency, considering the need for the associated variables to be optimized.

KEYWORDS

pectin, pectin recovery, ultrasound-assisted extraction, microwave extraction

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INTRODUCTION

Throughout the primary production and subsequent processing and distribution phases of plant based agri-food industry, various secondary products including peels, seeds, pulps, leaves, bran, kernels, and oil cake can be generated from numerous kinds of vegetables, fruit, legumes, root and tubers, cereal grains and oil-bearing plants (Difonzo et al., 2021; Jin et al., 2018; Pathania and Kaur, 2022). Fruit and vegetable by-products are rich in several healthful substances, including different vitamins, proteins, dietary fibers, carotenoids, alkaloids, and polyphenols, while cellulose, lignin, hemicellulose, and other polysaccharides (Nabi et al., 2024). Moreover, there is a promising opportunity to recover these valuable substances and reuse them in the food chain, thereby contributing in a greener and circular economy (Wedamulla et al., 2022).

Pectin is mostly found in fruits and vegetables, it is a heteropolysaccharide which consists of a D-galacturonic acid units' backbone with some side chains of neutral sugar, it is one of the essential components of the plant cell wall (Torres-León et al., 2018). Pectins have a wide range of applications in the pharmaceutical, cosmetics, and food industries such as emulsifiers, stabilizers, and gelling or thickening agents (Ma et al., 2020; Celus et al., 2018; Petkowicz et al., 2017). They are recognized as food additives in Europe under the codes E440a for amidated pectin and E440b for low- and high-methoxyl pectins (Williams, 2011). Moreover, pectin may provide several potential health benefits due to its ability to bind heavy metals in the digestive tract, reduce the cholesterol level, and heal wounds (Chandel et al., 2022). Its yield and characteristics might vary according to the source and processing technique; it can be separated from the plant tissue using a variety of techniques, such as enzymatic, chemical, physical, or a combination of treatments. Since each of these techniques may have a unique impact on the yield, purity, structure, and properties of pectin, they may differ in their basic concepts, features, and findings (Chandel et al., 2022; Bhat et al., 2024).

Acid-assisted extraction or conventional acid extraction (CAE) is the most used technique for pectin production from plant tissue on a commercial basis. This method is based on applying acids to fruit by-products under heating. Low pH values (1–3) accompanied with high-temperature heating hydrolyze and solubilize the pectin macromolecules from the cellulose network, releasing them into the extractive solvent (Duan et al., 2022; Gimba et al., 2022).

In industry, pectin extraction is commonly done by using sulfuric, hydrochloric, or citric acids at elevated temperatures between 60 and 90 °C for 1–6 h, then the alcoholic precipitation stage takes place (Sun et al., 2020; Freitas et al., 2020a). The conventional extraction method is inexpensive, it is easy to conduct and requires simple tools to operate. The process is also characterized by certain disadvantages, including low time efficiency, for example the conventional extraction time for pectin from grapefruit albedo is approximately 90 min, which is three times longer than the 25-min extraction time for ultrasound method (Bagherian et al., 2011). Furthermore, the quality of the obtained pectin is subject to variation due to its susceptibility to denaturation during the extraction process, which is time-consuming and requires significant amounts of solvent (Liew et al., 2019; Xiang et al., 2024). In addition to the presence of high quantities of precipitation agents and extensive energy demands of the driers, heating and mechanical grinding equipment are also necessary (Benassi et al., 2021).

Eco-friendly and unconventional methods such as ultrasounds, enzyme catalysis, subcritical fluids, microwaves, pulsed electric field, hydrostatic pressure and also the hybrid extraction involving the combination of two or more methods draw more attention because of their

positive findings including saving more energy and time, consuming less solvents, using lower temperature, and better quality and yield of the extracted pectin (Gentilini et al., 2014).

Ultrasonic extraction (UAE) uses high-frequency sound waves to create significant cavitation effects, which cause the breakdown of cell walls of the plant material as cavitation bubbles burst, a strong regional heat and pressure is generated and therefore pectin chains are released into the extraction medium (Ma et al., 2016; Zhang et al., 2021). When compared to CAE and other unconventional approaches, UAE has become an outstanding technology for pectin extraction, which improves the quantity, quality, and physicochemical attributes of pectin (Macias-Frotto et al., 2025).

This review provides information about pectin structure and its applications in food and focuses on the use of ultrasound for the extraction of pectin from agro-industrial by-products, the influence of ultrasound on yield and other properties, and the effect of the combination of ultrasound with other novel methods including microwave, enzyme and supercritical water.

Pectin structure

Pectin is a major constituent in plant primary cell wall and intercellular spaces. It is frequently linked to other polysaccharides such as cellulose, hemicellulose, and lignin, but with a more intricate structure (Scheller et al., 2007). Pectin structure differs according to its source, extraction parameters, other environmental factors, the processing method, and the maturity stage of the plant (Geerkens et al., 2015). Pectins are known to consist of three polysaccharide domains: homogalacturonan (HG), rhamnogalacturonan-I (RG-I), and rhamnogalacturonan-II (RG-II), which are linked together by covalent bonds throughout their backbones. Moreover, due to their similar backbones to HG, xylogalacturonan (XGA) and apiogalacturonan (AGA) are frequently regarded as pectins which could be found in a limited number of species or tissues (Zdunek et al., 2021). The linear homopolymer known as HG makes up roughly 65% of the pectin found in plant cell structures, HG (Fig. 1) is made up of 1,4-linked galacturonic acid units (GalA),

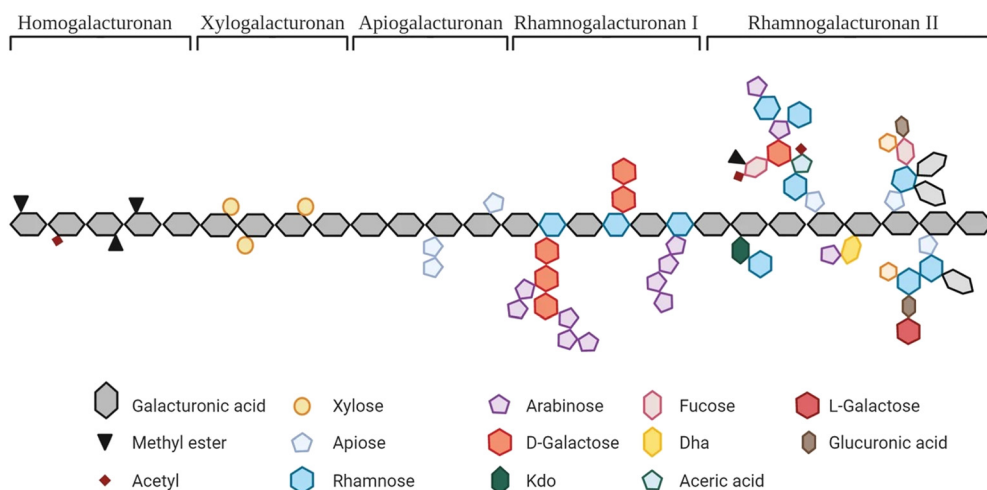


Fig. 1. The chemical structure of pectin (adapted from Beukema et al., 2020, under CC BY 4.0 License, without changes)

which form the polysaccharide's backbone, some of the carboxyl groups are methyl-esterified and it could be O-acetylated at C-2 or C-3 according to the plant source (Voragen et al., 2001). HG is regarded as the smooth region of pectin because of its lack to side chains (Ridley et al., 2001).

The chemical structure of pectin (Fig. 1) shows the homogalacturonan (HG), xylogalacturonan (XG), apiogalacturonan (AG), rhamnogalacturonan II (RG-II), and rhamnogalacturonan I (RG-I) domains. The dominant structure of the whole pectin is the linear HG backbone which forms the smooth region, the highly branching RG I and RG II domains with smaller contributions from (XG) and (AG) make up the hairy regions.

Esterification can take place in pectins with methanol or acetic acid, in which methyl esterification is more prevalent than acetylation. The ratio of methylated esterified carboxylic groups of the GalA units of HG to the total carboxylic acid units is termed as the degree of esterification (DE), which could be higher than 50%, and in this case, they are known as high methoxyl (HM) pectins while those with less than a 50% ratio are known as low methoxyl (LM) pectins (Liew et al., 2018).

Low methoxyl (LM) pectins form gels in the presence of divalent cations, usually calcium, with pH values ranging from 2 to 6, and the addition of sugar is not necessary, they are used as a fat replacer in dairy products, emulsified meat, and dietetic products like low calorie carbonated beverages, whereas high methoxyl (HM) pectins can make gels at low pH (less than 3.5) with a high concentration of sugar (>55%) and they are commonly used in jellies, and desserts (Kamble et al., 2017; Marić et al., 2018). HM pectins can be converted to LM by applying a de-esterification process using pectin methyl-esterase enzyme or treatment with alkali (Cho et al., 2019). The percentage of galacturonosyl residues esterified with the acetyl group is known as the degree of acetylation (DA). Acetylation improves pectins' emulsifying and stabilizing properties while limiting their gel formation properties (Wandee et al., 2019).

The pectin extracted from sugar beet shows surfactant behavior that can enhance the stabilization of emulsions, due to its multiple acetyl groups (Phillips and Williams, 2009). High acetylation levels (up to 25%) in pectins are usually associated with poor gelling characteristics (Williams, 2011).

Some carboxyl groups of galacturonic acid can be replaced by amidated groups. In this case the percentage of the carboxylic groups in the amide form is known as the degree of amidation (DA). Amidated pectin tolerates more calcium changes that may occur while used and forms a thermoreversible gel with a better pectin solubility in water (Chen et al., 2020).

Xylogalacturonan and apiogalacturonan (Fig. 1) consist of homogalacturonan backbone substituted with xylose for xylogalacturonan, which is present in reproductive tissues like fruit and seeds, and one or two apiofuranosyl residues for apiogalacturonan, which is mainly identified in the walls of some aquatic monocotyledons (Ridley et al., 2001; Zandleven et al., 2006).

Rhamnogalacturonan-I (RG-I) (Fig. 1) accounts for approximately 20–35% of pectin, and consists of alternating quantities of rhamnose and galacturonic acid, of which 20–80% of the Rha (rhamnosyl) units are substituted at O-4 with neutral sugar side chains, mainly galactans and arabinogalactans (Coenen et al., 2007). Rhamnogalacturonan-II (RG-II) (Fig. 1) forms about 10% of the pectin structure, with a more complicated and conserved pattern compared to RG-I. It has a tiny backbone composed of 7–9 galacturonic acid residues, along with four side chains made up of arabinofuranose, arabinopyranose, glucopyranose, fucopyranose, apiofuranose, galactopyranose, and other rare monosaccharides like 3-deoxy-D-lyxo-2-heptulosaric acid

(Dha), ketodeoxyoctonic acid (Kdo), and aceric acid. Because of their non-ionic side chains, RG-I and RG-II are regarded as the hairy regions of pectin (Ridley et al., 2001; Dranca and Oroian, 2018; Noreen et al., 2017; Picot-Allain et al., 2020).

The structure of Rhamnogalacturonan-I (RG-I) is not conserved as Rhamnogalacturonan-II (RG-II) because it varies depending on the type of cell and developmental stage in terms of branching degree, side chain length, and monosaccharide composition (Peña and Carpita, 2004). The configuration of the three domains is still up for discussion. In the typical model, the HG, RG-I, and RG-II backbones make up block co-polymers by covalently crosslinking to each other. It is uncertain what the three major domains' exact positions are, one of the proposed structural models which is known as 'The smooth zone-hair zone model' the linear HGA residues alternately linked to branched RG-I residues (Mohnen, 2008), in contrast in the 'RG-I backbone model' HG -I is positioned as a side chain to RG-I (Coenen et al., 2007). In the 'living-thing-like' model, unsubstituted HGA attaches to the RG-I in a non-linear design, and the pectin backbone includes two linear HGA residues and one RG-I core alternately, where the HGA blocks can occupy both horizontal and vertical positions (Yapo, 2011).

Crosslinking between pectin molecules can take place via borate diester bonds between two RG-II regions, or Ca^{2+} bonds between demethyl-esterified homogalacturonan (HG) regions (Ridley et al., 2001). The side chains of RG-I region and the cellulose-hemicellulose network's fibers can physically interact and become entangled (Moore et al., 2008). Pectins can interact with extension and other hydroxyproline-rich proteins in the cell wall electrostatically or covalently primarily through glycosylation with RG-I arabinose and galactose (MacDougall et al., 2001).

Sources of food-waste-derived pectin and its applications

Juice production byproducts of some fruits such as apple, orange, lemon, grapefruit, and other citrus are typically used to obtain commercial pectin, as they contain significant amounts of pectic substances, ex. citrus peels, and apple pomace with 20–30% and 10–15% pectin on a dry weight basis respectively (Neckebroek et al., 2021; Gawkowska et al., 2018).

Sugar beet and sunflower by-products are also among the desirable sources of pectin, which encompass 10–20% pectin on a dry weight basis (Adetunji et al., 2017; Kalapathy and Proctor, 2001). Sunflower head residues pectin has high levels of galacturonic acid and a high molecular weight with fine gelling formation abilities (Iglesias and Lozano, 2004).

An estimated 931 million tons of food waste was produced globally in 2019 according to a report by UNEP. The fruit and vegetable industry contributes to the largest quantity of food waste in Europe (Osorio et al., 2021), these huge amounts of by-products may affect the environment negatively (Moorthy et al., 2015).

Numerous bioactive compounds can be extracted from these agro-industrial wastes, such as phenolic compounds, carotenoids, ascorbic acid, tocopherols, sterols, polypeptides and proteins, as well as oligo and polysaccharides (Gerschenson et al., 2021). In recent years, there has been a growing interest in the extraction of pectin from fruit and vegetable byproducts. This topic has been the focus of numerous studies, which aim to enhance the value of these raw materials while also mitigating environmental pollution.

New sources for pectin extraction are widely being reported in the literature such as the peel of passion fruit (Zhao et al., 2023), mango (Rojas et al., 2018), jackfruit (Freitas et al., 2020b),

kiwi (Karbuz and Tugrul, 2020), watermelon (Forouhar et al., 2023), pomegranate (Moorthy et al., 2015), papaya (Koubala et al., 2014), pumpkin (Lalnunthari et al., 2020), banana (Kham-sucharit et al., 2018), tomatoe waste (Sengar et al., 2020), potato pulp (Yang et al., 2019), coffee pulp (Reichembach and Petkowicz, 2020), okra pods (Mao et al., 2020), pistachios green hull (Chaharbaghi et al., 2017), cacao pod husks (Vriesmann and de Oliveira Petkowicz, 2017).

Pectin is a plant-based hydrocolloid approved as one of the safest and healthiest food additives globally and must comprise at least 65% of galacturonic acid as specified by the joint FAO/WHO Expert Committee and the European Commission (Müller-Maatsch et al., 2016). An estimated 60,000 tons of pectin were produced worldwide in 2015. Citrus peels provide around 85% of this pectin as shown (13% from oranges, 30% from limes, 56% from lemons), followed by apple pomace with 14%, and the remaining 1% from sugar beets (Ciriminna et al., 2016). Due to the increased consumer interest in the plant derived ingredients and natural food additives, the global pectin market is worth 1.07 billion USD in 2025, and it is expected to have grown to 1.43 billion USD in 2030 (Mordor intelligence, 2025).

Pectin has many applications in the food-processing industry such as thickeners, gelling agents, stabilizers, micro and nano encapsulating agent, film coating of fresh fruit and vegetables, and emulsifiers (Li et al., 2021; Sabater et al., 2021). Pectins exhibit notable gelling qualities. They form hydrogels and viscous and elastic solutions, and they are widely used in jellies, marmalades, fruit juices, and jams (Douglas et al., 2018). When compared to pectin derived from traditional sources, sugar beet pectin possesses a lower molecular weight, with a high concentration of acetyl groups, and neutral sugars. It also shows better emulsifying qualities but a weaker gelling ability which limits its use as a texturizing agent (Drusch, 2007; De Moura et al., 2020).

The characteristics of pectin vary according to its source, which affects its applications later, i.e. the color of apple pectin is darker and forms a more viscous solution in comparison with other sources of pectin making it more preferred for fillings and baked goods, while citrus pectin tends to have a lighter color and is used as a texturing agent in jams (Marić et al., 2018). Another use for pectin is a stabilizer due to its ability to increase the viscosity of the solutions, forming a strong network to keep particles suspended, and avoiding its precipitation, thus preventing the separation of a heterogeneous mixture into their components such as ice cream mix, fruit preparation with pieces, cocoa drinks, etc. (Belkheiri et al., 2021).

Pectin is also known as an emulsifying agent because of its structure characteristics including molecular weight, protein content, ferulic acid content, acetyl group, acetylation position, degree of esterification, and neutral sugar side chain, thus it is widely used in emulsified food such as low-fat mayonnaise and dressings, low-fat dairy products, emulsified meat products, and low-fat spreads (Yang et al., 2018a; Ngouémazong et al., 2015).

Recently, pectin is considered as one of the most promising encapsulating agents owing to its features as stabilizer, emulsifier, binder, safe food additive, and through the application of novel approaches including coacervation, nano spray drying, and nano complex creation, it can be utilized as a wall material for the encapsulation of several bioactive substances (Assadpour et al., 2017; Fathi et al., 2014). Pectin is used as a fat substitute in cake, and the resulting products showed similar mouthfeel and textural qualities to those made with fat (Lim et al., 2014).

Moreover, pectin can enhance the baking quality by improving the fermentation process of the dough and delaying the staling of the baked products, in addition to its effect on gluten elasticity (Correa et al., 2011).

Pectin is studied and used extensively in the creation of edible coatings and packaging materials due to its renewable, biodegradable, biocompatible and excellent mechanical qualities, inexpensive costs of production, and the potential to increase the food package's expiry date, with high viability to create single-layer films, films integrated with active ingredients, or even being a part in blends or bilayers with other polymers (Mendes et al., 2020).

It is also regarded as a soluble dietary fiber that may offer several health advantages because the intestinal fermentation of pectin produces acetate, propionate and butyrate, which are critical for preventing obesity, hypertension, Crohn's disease, and various forms of cancer (Gullón et al., 2013).

Pectin and pectin-derived compounds have antioxidant, antibacterial, antiglycation, anti-inflammatory qualities, ion binding and fat absorption ability (Koríem et al., 2014; Zhu et al., 2019; De Moura et al., 2020). This multipurpose polysaccharide is employed in the pharmaceutical and medical fields in drug encapsulation as a flexible drug delivery system and in tablet formulations as a binding material (Amini et al., 2009; Grassino et al., 2016).

Pectin extraction methods

Pectin extraction is a multifaceted process involving three primary steps (Cui et al., 2021). The first step is the pre-extraction treatment, which is followed by the extraction step, which involves the release of the pectin into the utilized solvent. The final step is pectin separation.

Pre-extraction treatment is a process that aims to enhance the stability of raw plant waste and protect pectin from degradation. This treatment involves a series of procedures, including cleaning, drying, grinding, and other methods, with the objective of eliminating enzymes and microorganisms (Dranca and Oroian, 2018).

Pectin can be extracted through a variety of methods, encompassing both conventional and innovative approaches. Acid extraction is a process that has been employed to convert insoluble pectin from apple pomace and citrus peels into a soluble form. This conversion is achieved by subjecting these waste materials to boiling in an acidic solution. A variety of novel methodologies are currently being employed to extract pectin from a variety of agro-industrial waste, including subcritical fluid extraction, ultrasound-assisted extraction, deep eutectic solvents, enzyme-assisted extraction, and microwave-based extraction, or a combination of one or more techniques (Adetunji et al., 2017).

Alcohol precipitation is the most widely used technique for pectin separation. Following the removal of the plant material from the extract, the solubilized pectin should be mixed with ethanol to precipitate it (Tsuru et al., 2021). The pectin can be precipitated at an ethanol concentration of 60%. Two methods are employed for the separation of the precipitated pectin: filtration and centrifugation.

In order to obtain pure pectin, it is necessary to implement additional washing processes that utilize varying concentrations of ethanol. These processes are intended to eliminate the impurities and acid ions that are utilized during the extraction stage. While ethanol is the optimal choice in terms of safety, methanol and isopropanol can also be employed for the precipitation process (Davitadze, 2023). Additional methods like ultrafiltration (Yapo et al., 2007), metal precipitation (Guo et al., 2015), have also been used for pectin separation.

Ultrasound-assisted extraction (UAE)

The concept of the UAE. The ultrasound-assisted extraction (UAE) combines the use of solvents and ultrasonic energy to separate specific compounds from various plant matrix (de Oliveira et al., 2016). Ultrasound is the mechanical waves that have a frequency higher than the human hearing threshold (>20 kHz). The waves are grouped according to the energy level at which they spread through the medium, as indicated by sound power (W), sound intensity (W m^{-2}), or sound energy density (W m^{-3}), and their frequency in two groups (Chemat et al., 2011):

- Low-intensity ultrasound, which operates at high frequency (100 kHz–1 MHz) and low power (usually $<1 \text{ W cm}^{-2}$). It is known for its non-invasive properties.
- High-intensity ultrasound operates at low frequency (16–100 kHz) and high power (typically $10\text{--}1,000 \text{ W cm}^{-2}$).

It is known for its destructive capabilities, by altering the mechanical and biochemical characteristics of the material through cavitation phenomena (Picó, 2013).

The transmission of ultrasound waves through the material initiates cycles of compression and rarefaction, thereby inducing molecular shifts and separation from their initial positions. The generation of cavitation bubbles is initiated by high-intensity sound waves, wherein the internal negative pressure during the rarefaction phase exceeds the attractive forces that bind molecules together, leading apart (Esclapez et al., 2011).

The cavitation bubbles enlarge in size until reaching the critical limit, then they are imploded by the effect of compression, which generates severe local circumstances as the temperature and pressure may be up to 5,000 K, 1,000 atm, and hot spots which can speed up the biochemical reactions in its surrounding area (Shen et al., 2023). Ultrasonic cavitation is the key mechanism behind the separation using ultrasound-assisted extraction (Tiwari, 2015). The collapsed cavitation bubbles produce shock waves and expedite inter-particle collision which leads to fragmentation in the cell wall (Giacometti et al., 2018).

Immediate cell fragmentation reduces the particle size, expands the surface area, and eases the release of cellular contents which induces the dissolution of the bioactive compounds in the extractive solvent (Roselló -Soto et al., 2015). Figure 2 depicts the cavitation effect of ultrasonic.

Recently several studies have suggested various ultrasonic mechanisms of UAE such as erosion, the sonoporation, local shear stress, and detexturation of plant structures in addition to fragmentation (Vinatoru et al., 2017; Khadhraoui et al., 2018).

Erosion induces the liberation of the components from the plant matrix into the solvent due to the implosion of the cavitation bubbles which causes regional destruction to the plant tissues. The eroded part will ease the penetration of solvents, which improves the yield of the extracted material (Chemat et al., 2017a; Petigny et al., 2013). Moreover, turbulence within the liquid media and shear force are also formed by the collapse of cavitation bubbles. This phenomenon leads to the rupture of the cell wall and the release of the inner compounds (Adetunji et al., 2017).

Sonoporation is defined as the process of pore formation on a cell membrane due to the activity of cavitation bubbles. This process enhances the membrane permeability, thereby facilitating the release of bioactive compounds from within the cell (Corbin et al., 2015; Azmir et al., 2013).

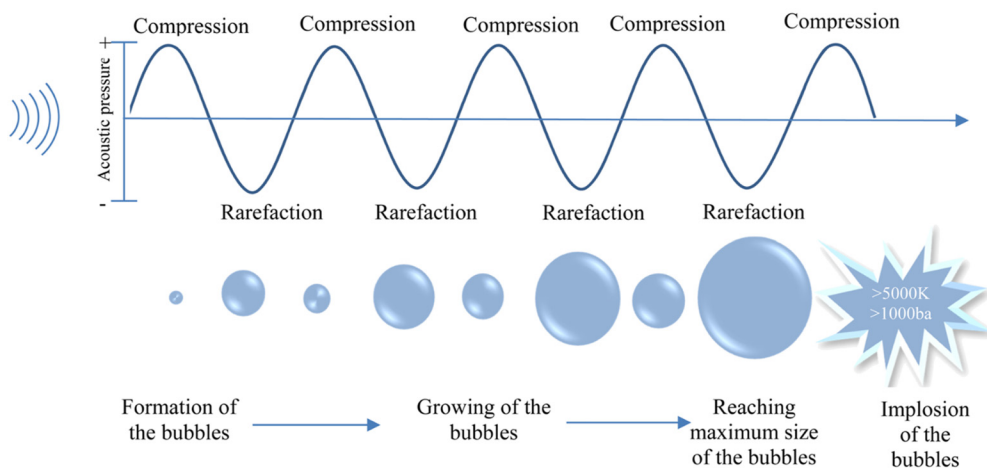


Fig. 2. The formation of cavitation bubbles in UAE (Bui et al., 2020) (Reproduced with permission)

Finally, detexturation is defined as the disruption of cell walls by the ultrasound effect. This process facilitates the permeation of the solvent into the material, thereby enhancing its interaction with the target molecule (Mason et al., 1996). During UAE extraction several mechanisms most probably take place, which may explain the improved yield of UAE (Vilkhu et al., 2011).

Ultrasonic system

High-power ultrasound has been applied using an ultrasonic bath and ultrasonic probe. It is focused entirely on using an energy generator and a transducer which alter electrical energy into the required acoustic energy (Yao et al., 2020). The most popular kind of ultrasonic device is the ultrasonic bath (Fig. 3A and B), which primarily includes one or more ultrasonic transducers that are attached to the four walls or the bottom of the stainless-steel tank. The sample is distributed in a suitable within the tank solvent (Chemat et al., 2017b). The ultrasonic baths are affordable, easy to use with the ability to handle several samples at the same time. However, their efficiency is compromised by the decline in intensity experienced as the sound waves pass through liquid contained in the bath and sample vessel before reaching the sample (Tiwari, 2015; Kumar et al., 2021). The ultrasonic probe (Fig. 3C) is made up of a probe or horn connected to a transducer. Through the probe, the ultrasonic energy is emitted into the sample. Probes with lower tip diameters produce a greater cavitation effect, but the effect is limited to a smaller field. Although the cavitation impact is less in probes with bigger tip diameters, the energy is dispersed over a greater region (Kumar et al., 2021). This method is frequently employed in the case of low volume samples. The probes immersion in the sample container facilitates the transmission of ultrasound to particular zones of the sample, thereby enhancing the cavitation mechanism and reducing energy dissipation (Adetunji et al., 2017). However, the ultrasonic probe is prone to corrosion and damage due to direct contact and the potential chemical reactions between the analyzed material and the probe (Shen et al., 2023). Figure 3 presents a schematic illustration of ultrasonication equipment, ultrasonication bath (A and B) and probe (C).

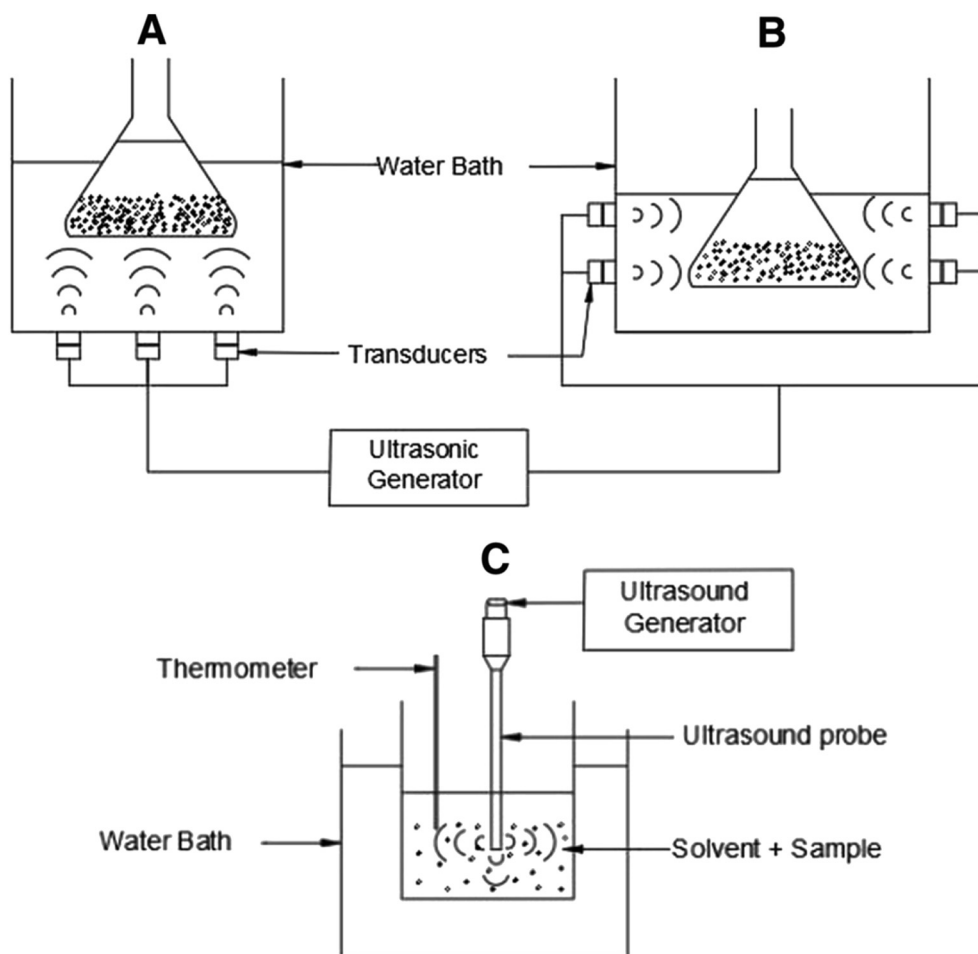


Fig. 3. Schematic illustration of ultrasonication equipment. Ultrasonication bath (A and B) and probe (C) (Thilakarathna et al., 2023) (Reproduced with permission)

The advantages and disadvantages of UAE

The UAE method has been shown to offer several benefits, including its simplicity in execution (Lavilla and Bendicho, 2017). Furthermore, it has been demonstrated to be a successful extraction technique for a wide range of compounds, irrespective of their polarity or molecular weight (Chemat et al., 2017b). This method is also characterized by its environmental sustainability, which produces pure outcomes rapidly (Maran et al., 2017; Kumar et al., 2021).

Compared to conventional techniques, ultrasound can be used at lower temperatures, which protect heat-sensitive substances from degradation – the ideal temperature range of UAE is usually from 40 to 60 °C (Christou et al., 2021).

Concurrently, the UAE is regarded as an energy-saving strategy that reduces energy consumption during extraction due to the utilization of lower extraction temperatures (Alupului et al., 2009).

The UAE procedure has been observed to exhibit several vulnerabilities. A salient issue is the considerable expense associated with UAE equipment and maintenance (Zhou et al., 2021). Furthermore, during the operation of the ultrasonic equipment, noise levels of approximately 65 dB are produced. The incorporation of a sound shield constitutes a viable solution to this challenge; however, it does result in an increase in the equipment's cost (Shen et al., 2023).

Another issue is the need for UAE parameters to be optimized. Numerous variables can affect the outcome of UAE, as the extraction parameters of different bioactive substances related to different substrates are varied, so a lot of experiments must be carried out to improve the extraction process (Shen et al., 2023).

Ultrasound-assisted extraction for pectin

Several fields have successfully used ultrasonic technology in many applications, including non-destructive testing, medical testing, welding, distance measurement, cleaning, and food technology (Yao et al., 2020). The physical and/or chemical effects of ultrasound on procedures such as inactivation of microorganisms and enzymes, modulation of living cell growth, reduction of particle sizes, homogenization, mixing, crystallization, dehydration, filtration, fermentation, degassing, and extraction have led to growing interest on its application in the food industry (Kiani et al., 2011).

Many factors may affect the efficiency of the US extraction process including ultrasonic equipment (bath or probe, power, frequency, etc.), extractive solvent properties (such as viscosity, surface tension, concentration, and purity of the used solvent, etc.), and the parameters of the environment (such as temperature, pressure, etc.), besides other factors (Hernandez-Hernandez et al., 2024).

There are two ways to apply UAE: pulsed ultrasound-assisted extraction (PUAE) and continuous ultrasound-assisted extraction (CUAE) (Wani and Uppaluri, 2024). Both modes depend on the formation of cavitation bubbles through the liquid phase, as well as the contemporaneous development of extreme pressure and temperature conditions (5,000 °C and 2,000 atm), which cause the examined sample to break down rapidly (Aung et al., 2022). PUAE uses intermittent ultrasonic pulses, to alternate between rest and sound application intervals which permit the release of a small amount of heat and therefore the isolation of heat-sensitive molecules including pectin with less degradation. This method combines the reduction of potential heat-induced degradation and boosting the extraction effectiveness (Haque et al., 2025).

Mineral acids like nitric, sulfuric, and hydrochloric acids are often used in the pectin conventional extraction process for commercial purposes regardless of their toxicity and not being environmentally friendly (Sun et al., 2020). Organic acids, such as citric acid, are considered safer alternatives to mineral acids because they are non-toxic and environmentally friendly (Xu et al., 2018). Moreover, pectin extracted with organic acids had higher molecular weights, viscosities, and degrees of esterification compared to inorganic acids, due to their lower dissociation constants, which may lessen their hydrolyzing abilities and therefore depolymerization capacity (Cho et al., 2019). The ultrasound technique has been widely used to extract bioactive

constituents such as polyphenols, carotenoids, and aroma from plant matrix and oil from oilseeds (Akhtar et al., 2023; Jalili et al., 2018).

Several studies have revealed the benefits of using ultrasonication extract particular compounds, such as pectin, by lowering both treatment time and energy usage when compared to the conventional extraction methods. The cavitation effect of ultrasonic waves enhances solvent penetration and mass transfer and consequently leads to a better pectin extraction process as it is a soluble fibre (Sengar et al., 2020; de Oliveira et al., 2016).

Rai et al. (2025) revealed that the energy consumption for ultrasound-assisted extraction of sweet lime peel pectin was almost 4 times lower than the conventional method, considering the operating condition for UAE (Temperature = Room Temp, Time = 30 min) and CA (Temperature = 90 °C, Time = 90 min). Pectin's functional qualities can be preserved by increasing its solubilization as well as limiting its degradation through using optimized ultrasonic conditions (Yi et al., 2024).

UAE showed promising results in pectin extraction from different sources including, banana peel (Phaiphan, 2022), pomegranate peel (Firat et al., 2023), pineapple peel (Shivamathi et al., 2022), citrus limetta peels (Panwar et al., 2023), carrot pomace (De Laet et al., 2024), pomelo peel (Wani and Uppaluri, 2024), custard apple peel (Shivamathi et al., 2019), eggplant peel (Kazemi et al., 2019), gac pulp (Tran et al., 2021), sugar beet flakes (Dranca and Mironeasa, 2024). Table 1 summarizes the optimum UAE conditions for pectin extraction from various plant peel, pomace, and rind. Panwar et al. (2023) revealed that the UAE improved the yield of *Citrus limetta* peels pectin (28.73%) as compared to the conventional extraction process (22.03%), which was conducted in their former study at a higher temperature (90 °C) and longer extraction time (95 min) (Panwar et al., 2022). the extracted pectin also showed better antioxidant properties and thermal stability than the commercial pectin and the same functional properties (water/oil holding capacities and emulsifying properties). De Laet et al. (2024) also reported that the UAE gives a higher pectin yield from Carrot pomace (57.2%) when compared to the conventional method (37.4%) while using similar optimized parameters. These results could be attributed to the ultrasonication effect only, considering the use of a thermostatic water bath during UAE, which prevents the associated overheating and provides stable temperature. Their findings also highlighted the pH as the most significant parameter on pectin yield and molecular structure, while the amplitude affected the pectin yield only. Moreover, Tran et al. (2021) demonstrated that Gac pulp pectin extracted by ultrasound-assisted extraction (UAE) and acidic hot water extraction (AHWE) techniques showed good antioxidant properties, solubility, and comparable structure but with enhanced viscosity and yield by almost 10% in the case of UAE. A recent study by Wani and Uppaluri (2024) revealed that using ultrasound-assisted extraction (UAE pulsed mode) enhanced pomelo peel pectin yield (40.41%) and degree of esterification (59.95%) in comparison to the acidic hot water extraction (ACHWE) (14.8%) and (51.5%), respectively It has been documented that this technique can also be utilized in continuous or pulsed modes to optimize results, noting that the pulsed mode could be more recommended due to the reduction of the heat release and the improved solvent penetration and cell wall disruption driven by agitation and induced particle collisions. Their results also pointed out that the pH was the most influential parameter on the pectin yield and degree of esterification, and citric acid was the best extracting agent compared to other acids (HNO₃, HCl, and CH₃COOH) used in this study.

Table 1. The operating and optimized conditions of ultrasound assisted pectin extraction of different types of agricultural waste

Matrix	Probe/ Bath	US parameters	Extraction conditions	Solid/liquid ratio (W/V) g mL ⁻¹	Method optimization (Yes/No) Optimized conditions	Yield %	References
Banana peel	Bath	P = 185 W f = 40 kHz A: not informed	pH = (1, 2, 3) T = (35, 40 45) °C t = (10, 15, 20) min hydrochloric acid	(1:12)	Yes/central composite design pH = 3.68 T = 33.12 °C t = 17.12 min	2.62	Phaiphan (2022)
Pomegranate peel	Bath	P = 172 W f = 40 kHz A = (30–45–60) %	pH = (1.5, 2.25, 3). T = (60, 70, 80) °C t = (10, 20, 30) min hydrochloric acid	(1:10)	Yes/Box Benhken Design pH = 1.5, T = 69 °C t = 29 min, A = 32%	11.56	Firat et al. (2023)
Pineapple peel	Probe	P: not informed f= 20 kHz A = 75% pulse mode	pH = (1–2) T = (50–80) °C t = (15–30) min	(1:10–20)	Yes/central composite design pH = 1.0 T = 70.83 °C t = 21.88 min SL = 1:15.20	16.24	Shivamathi et al. (2022)
<i>Citrus limetta</i> peels (Sweet lemon)	Probe	P = 500 W f = 20 kHz A = (10–50) % pulse mode	pH = (1–3) T = (30–50) °C t = (10–30) min citric acid	(1:30)	Yes/Box–Behnken design (BBD) A = 37% pH = 1.9 T = 40 °C t = 24 min	28.73	Panwar et al. (2023)
Carrot pomace	Probe	P: not informed f = 20 kHz A = (40,70,100) % continuous mode	T = (60,70,80) °C t = (5,15,25) min citric acid/pH = (2,2.5,3) citric acid/ pH = (1,2,3)	(1:40) (1:60) (1:80)	Yes/Orthogonal Minimally Aliased Response Surface (OMARS) design A = 100% pH = 1.2 T = 80 °C t = 11.2 min SL = 1:80	57.2	De Laet et al. (2024)
Pomelo peel	Bath	P: not informed f = 37 kHz A: not informed pulse mode	pH = 1.5–3.5 T = 65 °C t = 5–30 min citric acid	(1:15)	Yes/face-centered design (FCD) pH = 1.7 t = 16.11	40.41	Wani and Uppaluri (2024)

(continued)

Table 1. Continued

Matrix	Probe/ Bath	US parameters	Extraction conditions	Solid/liquid ratio (W/V) g mL ⁻¹	Method optimization (Yes/No) Optimized conditions	Yield %	References
Custard apple peel	Probe	P: not informed f = 20 kHz A = 70% pulse mode	pH = (1–3) T = (50–80) °C t = (10–30) min hydrochloric acid	(1: 10 – 1:25)	Yes/central composite design pH = 2.36 T = 63.22 °C t = 18.04 min SL = 1/23.52	8.93	Shivamathi et al. (2019)
Gac (<i>Momordica cochinchinensis</i>) pulp	Bath	P = 150–250 W f: not informed A = 60–100% continuous mode	pH = (1.5–3.5) T = 60 °C t = (15–35 min) citric acid	(1:50)	Yes/Box–Behnken design (BBD) P = 200 W pH = 1.5 t = 35 min	56.45	Tran et al. (2021)
Eggplant peel.	Probe	P = 50–150 W f: not informed A: not informed continuous mode	pH = 1.5–3 T: not informed t = (10–30) min acid used: not informed	(1: 15–40)	Yes/Box–Behnken design (BBD) P = 50 W pH = 1.5 t = 30 min	33.64	Kazemi et al. (2019)
Sugar beet flakes	Bath	P = 200 W f = 25 kHz, A = 60, 80, 100% pulsed mode	pH = (1, 1.5 or 2) T = 60 °C t = 20, 40 or 60 min citric acid	(1:10) (1:15) (1:20)	Yes/Box–Behnken design A = 80% pH = 1.60 t = 47 min. (SL) = 1:16	3.40	Dranca and Mironeasa (2024)

P: Power, f: Frequency, A: Amplitude, T: temperature; t: time; SL: solid to liquid.

Contrary to the above mentioned-studies, [Firat et al. \(2023\)](#) found that pectin yield from pomegranate peel using classical solvent extraction (17.22%) was higher than that registered by the UAE (11.56%), which may be explained by the adverse effect of the ultrasound on the separation of liquid and solid phases as it fragments the plant matrix, but with good stability of its emulsions during 5 days of storage at room temperature compared to other pectin included in the study. Similarly, [Dranca and Mironeasa \(2024\)](#) also reported a reduction in the pectin yield of the Sugar beet flakes obtained by the PUAE (3.40%) but with a notably higher GalA content (90.34 g/100 g) in comparison with the conventional method as yield and GalA content were 20.80%, 76.12 g/100 g respectively under the optimal extraction conditions. [Phaiphan \(2022\)](#) and [Kazemi et al. \(2019\)](#) suggested that the extracted pectin from banana peel and eggplant, respectively, under the optimized ultrasonic parameters could be promising sources of pectin production, due to the similar features of the banana peel pectin to the commercial citrus pectin since both can be categorized as high methoxyl pectins that are suitable for use as rapid-set pectin types. In addition to the advantageous functional properties (emulsifying and foaming properties, water and oil holding capacity, the high degree of crystallinity and the 33.64% extraction yield), it is abundant in galacturonic acid (66.08 mg/100 mg) and total phenolic content (96.81 mg GAE/g pectin), while exhibiting low ash and protein contents of the eggplant pectin.

[Shivamathi et al. \(2019, 2022\)](#) carried out the characterization of pectin extracted from pineapple peel and custard apple peel, respectively. Their finding in both cases confirmed the crystalline nature, and the absence of anti-nutritional factors (oxalate, phytate, aflatoxin, saponin and tannin). The pectin also exhibited good antioxidant properties at concentrations of 15 and 20 mg mL⁻¹ for the pectin of pineapple and custard apple peels, respectively.

The combination of ultrasound with other technologies

There is a growing trend toward the synergistic application of two or more novel techniques to improve the efficiency of the pectin extraction process. These techniques include microwave-ultrasound, and ultrasound enzymes ([Liew et al., 2016](#); [Chen et al., 2015](#); [Nadar and Rathod, 2017](#)).

The primary goal of integrating two or more unconventional technologies is to bring the advantages of each method, which is related to time and resource savings ([Soquetta et al., 2018](#)). Studies have shown that integrating green technologies can have positive, synergistic effects, such as the reduction of the used solvents and the extraction time, improving the yield and the quality of the extracted material from various plant materials and their byproducts ([Li et al., 2017](#); [Estrada-Gil et al., 2022](#)).

Ultrasound-assisted microwave extraction (UAME)

In UAME, ultrasound extraction and microwave extraction are combined. Microwaves (MWs) are generated at a frequency range of approximately 2.45 GHz (12 cm) for extraction purposes. This leads to dielectric heating mainly through the absorption of energy by water and other polar compounds present in wet biomass or a specific sample, which is transformed into molecular motion, then the temperature rise of the intracellular liquids, due to which the water evaporates and puts pressure on the cell walls, damaging it ([Hahn et al., 2012](#)). Furthermore, MWs cause hydrogen bonds to break and the migration of dissolved ions starts, which increases the solvent's penetration into the sample ([Kadam et al., 2013](#)).

Microwave-assisted extraction (MAE) has several advantages, including low energy input, simple controllability, quick processing times, and minimal solvent requirements (Al Hattab, 2015).

The method has been described as practical, with applications in both laboratory and industrial settings. It does not necessitate a complex series of steps and is not restricted to a particular category of materials. Instead, it is versatile, encompassing a diverse array of sample types, including natural products, food items, and plant materials (Chew et al., 2023; Bhadange et al., 2024).

However, it should be noted that microwave-assisted extraction (MAE) is not without drawbacks, as the purity of the bioactive compounds that are extracted may be adversely affected by the release of interfering chemicals. This phenomenon is attributable to microwave interaction with matrix components (Azhar et al., 2023).

The extent to which microwave energy can penetrate a sample is constrained. In the context of complex matrices, this approach can potentially lead to imbalanced heating, which can hinder the extraction of bioactive chemicals. Moreover, improper usage of microwaves can result in the localized warming of specific regions within the sample, potentially leading to the degradation or alteration of heat-sensitive substances (Parappa et al., 2024).

De Los Santos et al. (2024) utilized microwave-assisted extraction to optimize pectin extraction from pineapple peels. Their findings demonstrated that the microwave could serve as a viable method for pectin extraction in a relatively short time, within two-minute duration. Nonetheless, under optimal conditions, the pectin yield was 1.01%, which is lower than the findings of earlier research on pineapple pectin extraction using the conventional method. Therefore, more optimization with different parameters should be considered.

Recently, the combination of ultrasound extraction and microwave extraction has shown better results in the extraction of the bioactive compound from various plant matrices compared to the application of each technology alone (Wen et al., 2020). According to Wang et al. (2020) the yield of the extracted flavonoid compounds from *Eucommia ulmoides* leaves by UMAE (2.454%) was higher than UAE (2.041), MAE (1.731%), and solvent extraction (1.568%).

The physical effects of ultrasound cause a solvent to penetrate the membrane walls of the cell more deeply, aiding the release of the cell's contents and enhancing mass transfer as the cell wall is disrupted, while microwave rapidly heats the sample, and the consequent evaporation of moisture can burst the cell wall and it improves the release of the target compound into the solvent (Chemat et al., 2017a; Vinatoru et al., 2017).

It has been suggested that the combination of these two methods can result in several synergistic benefits, such as a reduction in extraction time, rapid damage to the cell wall's outer layer by ultrasonic energy, increased energy efficiency, and lower costs (Sun et al., 2019; Moorthy et al., 2017).

Hence, the successful application of ultrasound-assisted microwave extraction (UAME) has been documented for several active ingredients, including polysaccharides from plants (Chen et al., 2010), vegetable oil (Cravotto et al., 2008), lycopene (Lianfu and Zelong, 2008), and certain types of pectin (Yang et al., 2019).

UAME showed promising results in pectin extraction from different sources including jackfruit peel (Xu et al., 2018), sugar beet pulp (Peng et al., 2015), watermelon rind (Forouhar et al., 2023), brapefruit albedo (Bagherian et al., 2011), pomelo peels (Liew et al., 2019), fig skin (Gharibzahedi et al., 2019a, 2019b). Table 2 provides an overview of the operating and optimized conditions used to extract pectin by ultrasound-assisted microwave extraction.

Table 2. The operating and optimized conditions of ultrasound assisted microwave extraction of different types of plant byproducts

Matrix	Mode of application	Operating conditions				Method optimization (Yes/No) Optimized conditions	Yield (%)	References
		Ultrasound power	Microwave power	Time/ Temperature/ pH/ Extracting agent	Solid/Solvent Ratio (w/v)			
Jackfruit peel	ultrasonic-microwave apparatus (UAE followed by MAE)	P = 50 W f = 40 KHz	P = 500 W, f = 2450 MHz	t = (20–40) min T = (70–90) °C pH = 2 citric acid	SL = (1:20–1:60)	Yes/response surface methodology (RSM) T = 86 °C, t = 29 min, SL = 1:48	21.5%	Xu et al. (2018)
Sugar beet pulp	ultrasonic-microwave apparatus (UAE followed by MAE)	P = 50 W f = 40 KHz	P = (10–800) W f = 2450 MHz	t = (30,35, 45) min T = (85,90, 95)°C hydrochloric acid	SL = (1:25,1:30,1:35)	Yes/Box Behnken design (BBD) T = 92 °C, t = 37 min, SL = 1:30	26.16%	Peng et al. (2015)
Grapefruit	UAE as a pretreatment for MAE	probe P: not informed f: not informed t = 30 min pulse mode	P = 0.45 kW f: not informed t = 10 min	pH = 1.5 hydrochloric acid	SL = (1:30)	No	31.88%	Bagherian et al. (2011)
Fig skin	UAE as a pretreatment for MAE	probe P = 70 W f = 24 kHz T = 70 °C t = (10–30) min	P = (300–600) W f: not informed t = 5–15 min	pH = 1.4 citric acid	SL = (1:10–1:30)	Yes/central composite rotatable design (CCRD) SL = 1:24.66 Ultrasound	13.97%	Gharibzahedi et al. (2019a)

(continued)

Table 2. Continued

Matrix	Mode of application	Operating conditions				Method optimization (Yes/No) Optimized conditions	Yield (%)	References
		Ultrasound power	Microwave power	Time/ Temperature/ pH/ Extracting agent	Solid/Solvent Ratio (w/v)			
Fig skin	UAE as a pretreatment for MAE	probe P = 70 W f = 24 kHz T = 70 °C t = 25 min	P = 600 W, f: not informed t = 3.5 min	pH = 1.4 citric acid	1/20 g mL ⁻¹	pretreatment: t = 11.67 min microwave-assisted extraction P = 580.9 W, t = 21.35 min No	11.71%	Gharibzahedi et al. (2019b)
Pomelo peels	UAE as a pretreatment for MAE	bath P: not informed f = 40 kHz t = (12–28) min	P = (350–650) W f: not informed t = (4–12) min	pH = 1.7 – 2.3 citric acid	1/29 g mL ⁻¹	Yes/Box–Behnken design pH = 1.8; ultrasound pretreatment: t = 27.52 min; microwave-assisted extraction t = 6.40 min; P = 643.44 W	36.33 %	Liew et al. (2016)

T: temperature; t: time; SL: solid to liquid, P: Power, f: Frequency.

The synergetic effect of applying both technologies was confirmed by [Forouhar et al. \(2023\)](#) as the yield of watermelon rind pectin increased from 14.4 to 19.08% by using MAE and UAME respectively. It was also noted that the pectin extracted by UAME showed a darker colour, a lessened degree of esterification, and galacturonic acid content along with the pretreatment power and time increase. [Peng et al. \(2015\)](#) revealed that the yield, molecular weight, uronic acid content, and the degree of esterification of sugar beet pulp pectin extracted by UMAE was 26%, 636 kDa, 75%, and 82%, respectively, while the traditional method yielded lower values of 20%, 296 kDa, 72%, and 63%, with weaker emulsification capacity, emulsion stability, and thickening capacity.

Moreover, [Xu et al. \(2018\)](#) extracted pectin from jackfruit peel using UMAE as well as conventional method by citric acid and revealed that galactose, rhamnose, arabinose, glucose, and galacturonic acid were present in significant amounts in the pectins extracted using both techniques, with a high degree of esterification, while UMAE enhanced pectin yield with stronger antioxidant properties when compared to the conventional method. [Bagherian et al. \(2011\)](#) reported that UAME produced the best extraction yield of pectin from grapefruit albedo (31.88%) in comparison with other methods including MAE (27.81%), UAE (17.92%) and CAE (19.26%). They also emphasized that during US pretreatment, the intermittent sonication was more efficient and produced higher yields than continuous sonication. This could be attributed to the ability of the intermittent sonication to produce pulsation in the ultrasonic waves, which lowers the mass boundary layer and facilitates the release of pectin. [Liew et al. \(2016\)](#) reported that UMAE is the ideal approach for pectin extraction from pomelo peels, producing the best yield (36.33%), compared to other methods UAE (14.25%), MAE (27.65%), and MUAE (30.50%) at the same optimized conditions. It was also noted that the microwave power had the biggest effect on the degree of esterification, while pH was the dominant in the pectin yield. In addition, [Liew et al. \(2019\)](#) revealed that (UMAE) at the same optimized conditions from their previous study showed the least energy usage and the best extraction efficiency technique for pectin from pomelo peel compared to the conventional and dynamic subcritical water extractive technique. UMAE was proven to be the superior extraction technique for HM pectin while SWE may be used to extract LM pectin. They also studied the effect of using acid in the extraction process by applying UMAE and CAE with and without using acid. It was noted that pectin yields were higher when citric acid was used as an extraction agent due to the important role of acid in promoting the hydrolysis of protopectin to produce pectin. [Gharibzahedi et al. \(2019a\)](#) compare the pectin extraction yield from fig skin using different techniques, including CAE (6.05%), UAE (8.74%), MAE (9.26%), and UAME (11.71%), their findings demonstrate that UAME obtained the highest GalA content (76.85%), molecular weight (6.91×103 kDa), emulsifying activity (61.2–61.3%), emulsion stability (94.3–95.2%), and yield. Moreover, all extracted pectins in this study can be classified as LMP and none of the applied extraction technologies result in a significant alteration of the chemical structure or monosaccharide composition of pectin which was proved by Fourier-transform infrared spectroscopy (FTIR) and high-performance liquid chromatography (HPLC).

[Gharibzahedi et al. \(2019b\)](#) also optimized the extraction process of fig skin pectin by using UAME and noted that under the optimal conditions, the extracted pectin showed significant antioxidant and cytotoxic inhibitory properties against HepG2 and A549 cells, with high level of neutral monosaccharides (glucose, fucose, arabinose, galactose, rhamnose, and mannose) and uronic acids.

Ultrasound-assisted enzymatic extraction (UAEE)

In UAEE, enzymatic and ultrasound extraction are combined. Enzyme-assisted extraction (EAE) is a biological extraction technique that depends on particular enzymes to selectively convert primary glycosides into secondary glycosides or aglycones, or degrade cell wall, that help stimulate the release of bioactive constituents and improve the extraction yield.

Regarding pectin extraction from plant food materials, wastes, and by-products, EAE offers significant advantages over conventional methods since it conducts at lower temperatures, uses less energy and chemicals, and increases the pectin's yield (Liew et al., 2016). On the other hand, this extraction technique is costly due to the price of enzymes, which are rather costly for large quantities of raw material (Roman-Benn et al., 2023). Furthermore, the extraction process of pectin is time consuming and challenging due to the inability of existing enzymes to fully hydrolyze plant cell walls, and the effect of several factors such as temperature, dissolved oxygen levels, and nutrient availability on the enzyme response (Saha et al., 2018).

Enzymes are utilized to enhance the extraction process by disrupting the plant cell wall matrix and thereby increasing its permeability (Roselló-Soto et al., 2016).

It has been demonstrated that enzymes alone are incapable of facilitating the mass migration of solvents and target particles within or beyond the medium. The UAE process has been shown to enhance the efficiency of the enzymatic extraction process through the use of ultrasonic cavitation, which disrupts the structural integrity of the substrate, facilitating the subsequent discharge of targeted components. The employment of various physical techniques, such as shaking and UAE, has been demonstrated to promote mass transfer, offering a promising approach for enhancing the exchange of materials both within and on the surface of the substrate (Chemat et al., 2011).

A study by De Carvalho et al. (2020) has demonstrated that low-frequency ultrasound was efficient in advancing the cellulase activity under specific circumstances (time = 170 s, US intensity = 180.8 W cm^{-2} , and temperature = 25°C), which was the result of optimization process for the parameters sonication time (10–330 s), US intensity ($120.6\text{--}263.7 \text{ W cm}^{-2}$), and temperature ($25\text{--}50^\circ\text{C}$) using central composite rotatable design.

Utilizing an enzymatic and ultrasonic extraction approach in combination could be a useful strategy for increasing yields and reducing target compound degradation to produce pectin of higher quality with lower costs and shorter extraction time (Yang et al., 2018b; Marathe et al., 2019). The commonly involved enzymes in the pectin extraction process are cellulase, protease, hemicellulase, alcalase, xylase, polygalacturonase, α -amylase, neuraminidase, β -glucosidase, endo polygalacturonase and pectinesterase (Jeong et al., 2014; Yuliarti, et al., 2015). Even though each enzyme has a distinct function, combining different enzymes can increase pectin yield. Effective pectin extraction can be achieved with commercial multi-enzyme mixes (Wikiera et al., 2015). Depending on the enzyme formula, raw materials, and the terms of extraction, there are significant variations in the quality and quantity of pectin extracted through enzyme-assisted extraction (Zhang et al., 2020). In this context, UAEE was utilized to extract pectin from sisal waste (Yang et al., 2018b), orange peel (Bosch and Malgas, 2023), red beet pomace (Abou-Elseoud et al., 2021), artichoke by products (Sabater et al., 2020), carrot (Encalada et al., 2019).

Table 3 shows the operating conditions of the UAEE for pectin extraction from different agro-industrial by-products.

Table 3. The operating and optimized conditions of ultrasound assisted enzymatic extraction of different types of plant byproducts

Matrix	Ultrasound operating conditions (pretreatment)			Enzyme treatment				Yield%	References
	Probe/ Bath	Experimental conditions	Time/ temperature/ Extraction solvent	Enzyme type	Enzyme concentration	Solid/ liquid ratio medium	Incubation parameters		
Orange peel	ultrasonic bath	P = 300 W f = 40 kHz	t = 30 min T = 80 °C pH = 5 sodium citrate	celluclast	64.7 mg mL ⁻¹	1/19 (g mL ⁻¹)	T = 50 °C, under shaking at 70 rpm for 4h	26.9	Bosch and Malgas (2023)
Red beet pomace	probe	P: not informed f: not informed A = 75%	t = (15–30– 45) min sodium citrate buffer (pH 4.8)	xylanase/ cellulase enzymes mixture	1:1.5 xylanase to cellulase (200 U/g xylanase and 300 U/g cellulase)	1:15 (g mL ⁻¹)	T = 50°C, pH = 4.8 under shaking at 150 rpm for (1–2–4) h.	33.5 using 45 min of ultrasonication time with 4 h enzymatic treatment	Abou-Elseoud et al. (2021)
Artichoke	probe	P = 400 W f = 20 kHz pulse mode	t = 1–2–4–6 h/T = 50 °C (6.5% w/v) sodium acetate buffer	celluclast	10.1 U/g	(6.5% w/v)	T = 50 °C pH = 5 under shaking at 200 rpm for 6h	13.9% (for six hours sonication time)	Sabater et al. (2020)
Sisal waste	probe	P = 450 W f = 20 kHz	t = 60 min 0.6% ammonium oxalate	celluclast	88 U/g	1/15 (g mL ⁻¹)	T = 50 °C pH = 4.0 (HCl), under constant shaking (200 rpm) for 20 h	14.6%	Yang et al. (2018b)

(continued)

Table 3. Continued

Matrix	Ultrasound operating conditions (pretreatment)			Enzyme treatment				Yield%	References
	Probe/ Bath	Experimental conditions	Time/ temperature/ Extraction solvent	Enzyme type	Enzyme concentration	Solid/ liquid ratio medium	Incubation parameters		
Discarded carrot	probe	F = 20 kHz A = 80% pulse mode	$t = 20$ min sodium citrate buffer (pH 5.20)	hemicellulase or cellulase	hemicellulase (0.125 g g^{-1} carrot powder) <u>or</u> cellulase (0.025 g g^{-1} carrot powder)	1/100 (g mL^{-1})	$t = 5$ h at 40 °C	27.1% (hemicellulase)	Encalada et al. (2019)

T: temperature; t : time; SL: solid to liquid, P: Power, f: Frequency, A: Amplitude.

A recent study by [Bosch and Malgas \(2023\)](#) compared UAEE with traditional acid extraction for pectin extraction from orange peel. The study found that Pectin yields were almost similar for the UAE (26.9%) and acid extraction methods (22.1%). However, the pectin’s molecular weight (70.22) kDa and degree of esterification (8%) were significantly lower than those of its counterpart (197.78 kDa, 55%) respectively, because the Celluclast enzyme cocktail degraded the polysaccharide during the extraction process. Similarly, [Sabater et al. \(2020\)](#) revealed that UAEE was the most effective method for pectin extraction from artichoke by-products as it obtained the highest yield (13.9%) in comparison with six hours of enzymatic extraction (11.3%), ultra-sound extraction (7.1%), and acid extraction using nitric acid (5.8%), while the Pectin extracted by acids extraction contained the highest GalA contents (90.2%)’, followed by enzymatic extrac-tion (72.33%), ultrasound extraction (61.58%) and UAEE (59.28%). [Encalada et al. \(2019\)](#) reported that UAEE improves the pectin yield from carrot leftovers either using cellulase or hemicellulase in comparison with other methods including ultrasound and enzyme-assisted extraction. It was noted that the highest yield (27.1%) and the optimal rheological properties with the greater structural integrity was a result of US-pretreatment. Significantly, [Abou-Elseoud et al. \(2021\)](#) emphasized that the combination of ultrasonic pretreatment accompanied with enzymatic treatment enhanced the pectin yield of red beet pomace during a shorter extraction time. The study revealed that 15–45 min ultrasonic pretreatment accompanied with the enzy-matic treatment for an hour led to 84–92% increase in pectin yield, while the enzymatic treatment for 4 h with the same ultrasonic pretreatment caused only 2–16% rise in pectin yield. Their findings also indicated that the utilization of a combination of mixed enzymes yielded a substantially higher yield in comparison to the application of individual enzymes. In addition, [Yang et al. \(2018b\)](#) compared the effect of reversing the order of the sequential extraction methods by applying the ultrasonic and enzymatic extractions in two distinct sequences with the same conditions, which is summarized in [Table 3](#). They revealed that the pectin yield from sisal waste when enzyme was followed by ultrasonic treatment was 31.1%, which was higher by

Table 4. The advantages and disadvantages of UAME and UAEE

Techniques	Advantages	Disadvantages
UAME	• Time saving and environmentally friendly method due to less solvent needs (Haque et al., 2025) • Improved pectin purity and yield (Roman-Benn et al., 2023).	• It is recommended that heating be conducted under strictly controlled conditions to prevent degradation. • Specialized equipment requires a large initial expense. • The difficulty of optimizing many parameters, including microwave in-tensity, processing duration, and sonication duration related to both methods (Dixit et al., 2025)
UAEE	• Improve extraction efficiency (Shen et al., 2023) • Save of the active ingredients from degradation (Yang et al., 2018b)	Optimizing UAEE parameters (power, sonication time, enzyme concentration, etc.) for pectin extraction from different substrates can be complex and time-consuming.

16% than the yield when ultrasonic was followed by enzyme treatment, which may be explained by the insufficiency of the synergistic action in this case.

The advantages and disadvantages of UAME and UAEE

The integration of UAE with other technologies such as microwave and enzyme has shown a synergistic effect, by the utilization of diverse mechanisms, resulting in improved yield and quality of the extracted compound. However, it can show some drawbacks. Table 4 summarizes the advantages and disadvantages of UAME and UAEE.

CONCLUSION

Pectin is a heteropolysaccharide, which is found in the cell walls of numerous plant species, it can be extracted from fruit and vegetable by-products by different methods, with a wide range of uses in food processing and pharmaceutical sectors, because of its properties as swelling, thickening, gelling, emulsifying agents. Ultrasound is a novel method of pectin extraction that can be applied alone or in conjunction with other approaches like enzymatic and microwave, in order to offer more effective and eco-friendly alternatives to the conventional pectin extraction method and minimize undesired changes with higher yield in most cases.

It was noted that critical factors like the power, frequency, amplitude, size, and dimensions of the ultrasound bath, as well as the probe tip utilized, are frequently left unspecified when it comes to the usage of ultrasound, it is difficult to compare different treatments and get clear idea about the effect of ultrasound if these statistics are not included, so it is important to conduct more detailed studies to improve the use of this technology in pectin extraction.

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