

Review

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Review

Greener Pectin Extraction Techniques: Applications and Challenges

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Abstract: Pectin is a complex and versatile polysaccharide crucial for various industries. It functions as a thickener, gelling agent, emulsifier, and low-calorie food. Its anti-inflammatory and immunomodulatory properties have attracted biomedical interest, while its biodegradability and biocompatibility make it valuable for biomaterial applications. The effectiveness of these applications depends on the quality of pectin extraction procedures. While traditional extraction methods exist, green methodologies and alternative techniques have improved pectin's physicochemical properties—a significant advantage for industrial applications. Pectin can be extracted from various sources, with its molecular structure and functional groups analyzed through different characterization techniques. Modern green extraction methods include ultrasound-assisted extraction, pulsed ultrasound-assisted extraction, pulsed electric field, moderate electric field mediated extraction, microwave-assisted extraction, subcritical water extraction, enzyme-assisted extraction, ohmic heating-assisted extraction, ultrasound-assisted microwave extraction, ultrasound-assisted ohmic heating extraction, hydrothermal processing, high-pressure processing extraction, and dielectric barrier discharge extraction. This review examines these methods' advantages and disadvantages, along with their applications and future possibilities; it serves as a comprehensive guide for researchers exploring new pectin-rich sources and green extraction technologies for commercial applications.

Keywords: pectin; extraction; physical properties; characterization; greener techniques; applications

1. Introduction

Fruit processing industries generate substantial waste, primarily consisting of seeds and peels. In juice production facilities, approximately 50% of fruit becomes waste material, with peels accounting for 50–55% of this waste. This creates serious environmental challenges as microbial decomposition leads to the emission of greenhouse gases [1]. Consequently, it is now imperative that fruit waste be managed sustainably [2]. Food processing produces byproducts of bioactive compounds, including pectin. These pectins from the food processing industries are considered non-toxic heteropolysaccharides [3] that have also been actively used in the cosmetic and pharmaceutical industries [4]. Pectin has a variety of uses in the food, pharmaceutical, and healthcare industries, as well as in packaging regulations. In food processing, it acts as a thickener, emulsifier, and stabilizing agent. The pharmaceutical industry employs pectin to develop medications for lowering blood cholesterol, treating gastrointestinal disorders, and cancer therapy [5]. To support its continued use in the food industry, pectin must be recovered and extracted from food waste materials [6,7]. In plants, pectin naturally occurs in cell walls, intercellular spaces, and the central lamella, connected

through glycosidic linkages [8]. This compound is essential for mechanical strength and intercellular connections, providing plant tissue with its firmness and structure. Additionally, it contributes to plant cells' turgidity and resilience [9]. The term "pectin" encompasses various polymers that differ in molecular mass, chemical composition, and sugar concentration, as different plants produce pectin with distinct functional properties. Common sources of pectin include citrus peel, apple pomace, cocoa husk, and potato pulp [10]. Certain fruits such as apples, citrus fruits, blackberries, cranberries, gooseberries, grapes, and plums contain high levels of pectic components in their polysaccharides. Notably, mature banana peels contain higher pectin concentrations compared to other fruits [11]. Furthermore, the abundance of pectin in various fruits and vegetables demonstrates its role in maintaining cell wall strength, flexibility, and biological processes. Pectin content varies significantly by source: citrus peels contain 20-30% pectin, sugar beet yields 10-20%, while apple pomace contains less than 15% on a dry weight basis [12].

Louis Nicolas Vauquelin isolated the molecule pectin from the fruit known as tamarind for the first time in 1790. Henri Braconnot first used pectin in 1825, derived from the Greek word "pektikos," which means solidifying or coagulating [13]. Modern nutritionists have shown particular interest in pectins as they serve as dietary fiber that increases transit time and glucose absorption in the digestive tract, leading to notable physiological effects [14]. The structure of pectin determines its physicochemical characteristics, making it essential to investigate the extracted pectin's structure. The source of the pectin, plant growth phases, and their extraction conditions significantly impact pectin structures [15]. The backbone of pectin is made up of galacturonic acids (GalpA) joined by (1,2)-linked β -L-rhamnose (Rhap). Galacturonic acid and its units are linked with additional substances found in the cell walls of plants, such as lignin, cellulose, or polyphenols [16]. Homogalacturonan (HG) and rhamnogalacturonan I (RG-I) are the most prevalent classes of these extremely complex polysaccharides that are covalently bonded. Rhamnogalacturonan II (RG-II), xylogalacturonan (XGA), and apiogalacturonan (AGA) are examples of small constituents of substituted galacturonans [17]. Given the intricate structure of the polysaccharides in pectin and the fact that plants retain the many genes needed to synthesise pectin, it is likely that pectin serves a variety of purposes in the growth and development of plants. During ripening, the pectin structure is hydrolyzed by enzymes such as pectinase and pectinesterase. The main job of the enzyme pectinase is to break down the pectin's whole structure by cleaving the primary pectin chain and its side branches, changing it into a common soluble polymer [18]. The chemical structure of pectin is very interesting as it consists of linear polysaccharides with a higher molecular weight varying between 75,000-125,000 g/mol [19]. The carboxyl groups can be found free or as salts with calcium, sodium, or other tiny counterion of residual uronic acid. They can also be found naturally esterified in certain situations, typically with methanol. The presence of free carboxyl groups contributes to pectin's acidic nature. The chemical structure of pectin is affected by its physicochemical properties, such as molar masses, extent of methylation, and esterification, which in turn is vital for functional characteristics like gelling, solubility, and viscosity [20]. The structure of pectin can be modified by non-sugar components like methanol, acetic acid, phenolic acids, and sometimes amide groups. In addition, these non-sugar components consist of polyesters, polyhydric alcohols, poly acids, reduced carbohydrates, certain polar carboxyl groups, and non-polar methyl groups [21].

The process of separating pectin from the source plant matter is the first step in using it. Historically, using acid for commercially extracting pectin has become the norm [22]. However, pectin extraction techniques remain an important challenge requiring further research. Optimizing the extraction process and improving the quality of the pectin need the use of an efficient extraction technique. Green extraction techniques have emerged as an alternative approach in recent years due to the growing awareness of environmental protection. Later on, several eco-friendly extraction techniques, such as enzyme-assisted extraction (EAE), microwave-assisted extraction (MAE), and ultrasound-assisted extraction (UAE), have been developed to improve pectin quality and efficiency [23–25]. This review will discuss many other green techniques for pectin extraction.

Pectin has many potential applications, from the industrial and pharmaceutical sectors to the primary food processing industries. Various applications of pectin have been demonstrated in Figure 1. The compound has gained significant importance in nutrition, food, and health sectors. Its molecular structure, comprising polar and nonpolar components, enables seamless integration into various food items [26]. Contemporary data suggests that people are more focused on healthy diets and are actively looking for feasible substitutes for petroleum-based plastic used in food packaging [27]. The edible polymers make excellent alternatives to these plastics, given their non-toxicity, environmental friendliness, and compatibility with most foods. Because of its capacity to gel and transport active substances like antimicrobials and antioxidants, pectin has justly found application in edible packaging [28,29]. Pectin is a valuable thickener [30], stabilizer [31–33], and emulsifier [34] in the food industry because of its multipurpose qualities. Pectin is frequently employed in jams, marmalades, and jellies because it can create a viscoelastic solution and a structural network. Pectin's origin and the way it is processed directs particular qualities, resulting in its varied uses. For instance, in comparison to pectin from other plant fruits, apple pectin is characteristically more viscous and provides dark shades. As a result, it works better with fillings and pastries. However, compared to apple pectin, citrus pectin is lighter and better fit as a texturing ingredient for jam and sweet jellies. So, it can be inferred that the structure of pectin can influence its use [35]. Additionally, pectin has also been shown to have biomedical and biomaterial applications. Although humans cannot digest or absorb pectin, it helps with good bacteria in the large intestine to provide prebiotic qualities [36]. Several researchers [37,38] have documented the health benefits of pectin use, which include preventing inflammatory and allergic illnesses, supporting cancer treatment, and reducing blood sugar and cholesterol levels. While numerous literature reviews cover pectin's extraction, structural chemistry, pharmaceutical applications, and its nutritional and functional properties in food packaging [39–43], comprehensive research on greener extraction techniques and their applications remains limited. This review examines green extraction methodologies of pectin, their physiochemical properties, structural characterization, and future multidisciplinary applications.

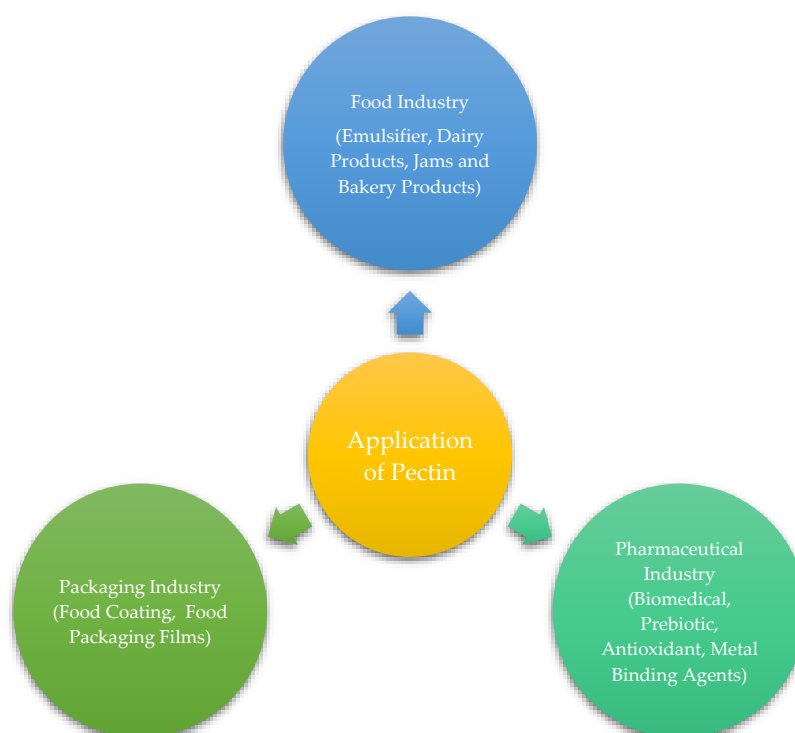


Figure 1. Various applications of pectin.

2. Physiochemical Properties

2.1. Percentage Yield

The extraction yield percentage varies based on several factors: the compound's nature, solvent type and polarity, temperature conditions, extraction method, presence of interfering substances, and the sample-to-solvent volume ratio. The pectin's yield was calculated using a conventional method and was determined using the following formula:

$$Y_{pec}(\%) = \frac{\text{Mass of dry pectin (g)} \times 100}{\text{Dry mass of sample used for extraction (g)}}$$

The pectin extraction depends on different factors and conditions. The temperature, pH and time for the utmost yield (23.64 %) for the isolation of pectin from citrus fruit is 94.13 °C, 1.45, and 114.7 min, respectively, according to Kamal et al. [44]. At a temperature of 80 °C, 1.5 pH and 60 min time with citric acid, the yield became 76 %, as concluded by Devi et al. [45]. The yield was maximum for the mango peel under temperature, pH, and time conditions, which were 90 °C, 1.5, and 120 min, respectively [46]. Meanwhile, the yield was 10.4 % to 59.3 % for the sundried peels under different conditions of temperature, pH, and time for the process [47]. Pectin obtained from cardamom lemon and China lemon is 8.08% and 12.73% using tartaric acid and ethanol [48,49]. Extraction of pectin from pomelo peel by the different methods of hot acid extraction (HAE), Microwave-assisted extraction (MAE), Ultrasound-assisted extraction (UAE) and Enzyme-assisted extraction (EAE) concludes that the outcome is highest in MAE and lowest in EAE. The instrumental data suggests no significant difference occurred among the different methods in the chemical structure. Still, the physicochemical properties are much different [50]. The demand for pectin is increasing across food, pharmaceutical, and fragrance industries, highlighting the need for a more efficient, simple, and environmentally friendly extraction process.

2.2. Color

Pectin powder, which is used across various industries, ranges from white to light brown in color. Its specific color varies by source due to the presence of additional compounds like essential oils and vitamins. Different citrus peels produce distinct colors: orange peel yields brown pectin, lime and white grapefruit peels produce golden yellow, lemon peel creates yellow, and red grapefruit peel results in golden yellow. These additional compounds help boost immunity, effectively turning waste materials into valuable resources [51–53].

2.3. Moisture and Ash Content

Pectin obtained from any source is a hygroscopic compound. The following formula is used to get any sample's moisture content. Usually, 5gm of dried sample in a Petri dish is placed in a hot air oven at 130°C for 2 hours, cold and kept in a desiccator, and later, the weight is taken to calculate the moisture content.

$$\text{Percentage of moisture} = \frac{W_1 - W_2}{\text{Weight of Sample}} \times 100$$

W_1 = Weight of the Petri dish with sample (g); W_2 = Weight of the petri dish with dried sample (g)

The pectin extracted from lemon has a moisture content of 8.59–8.62 %, less than other citrus fruits. Low moisture content prevents the microorganisms from growing and is easier to store for a long time [54]. However, the orange peel powder showed higher MC values of 10.12% and 9.6% compared to sweet lime peel (9.4% and 9.0 %) and papaya peel (8.92% and 8.8%) extracted through similar treatment with HCl and citric acid [55]. The formation of gels from pectin is better in quality if the ash content is less. Hence, determining ash content is one of the main requirements for usage in different industries. The below equation calculates the percentage of ash. In this process, 1.2 g. of sample is ignited and heated for 3-4 hours at 600°C, cooled and weighted accurately until a constant reading is obtained.

$$\text{Percentage of ash} = \frac{W_2 - W_1}{\text{Weight of Sample}} \times 100$$

Where W_1 = Weight of the empty Petri dish (g) W_2 = Weight of the Petri dish with ash (g)

The papaya peel powder contained higher amounts of ash content (3%) than the orange peel (2.6%) and sweet lime peel (2.4%) extracted with HCl. However, different scenarios were observed with citric acid, where sweet lime showed higher ash amounts (3.28%) than papaya peel (2.9%) and orange peel (2.1%). The ash content in the pectin obtained from lemon peel is much lower than that of other fruits, so it is widely used in making gels [56].

2.4. Galacturonic Acid Content

In plants, pectin mainly contains galacturonic acid units. The amount of pectin in plants decreases from the primary cell wall to the plasma membrane. Galacturonic acid is also noted as a pectin carbohydrate. Plants have three pectin domains: homogalacturonan, rhamnogalacturonan I, and rhamnogalacturonan II. Xylogalacturonan and homogalacturonan are also terms of pectin because of the presence of homogalacturonan as a backbone. This is known as substituted galacturonan, and the backbone comprises d-galacturonic acid units [57]. Gelling ability depends on the degree of methyl esterification. The names higher and lower methoxy pectin are based on the degrees of methyl esterification, which will be higher and lower than 50%, respectively [58]. The pectin content is higher in citrus and apple pomace than others, so these fruits are used for commercial extraction of pectin. The pectin extracted contains more than 65% galacturonic acid for the gel formation [31,59]. *Pectin commonly represses a natural sugar, rhamnogalacturonan*, that affects braking and knots in the galacturonic acid chain [12,60]. Galacturonic acid content was estimated using the formula:

$$\text{Galacturonic Acid Content (\%)} = \frac{176 \times \text{Methoxyl Content} \times 100}{31 \times \text{DM}}$$

Where, 176 and 31 are the molecular weights of galacturonic acid and methoxyl groups, respectively.

2.5. Equivalent Weight and Molecular Weight

It is necessary to determine the equivalent weight, which is vital in knowing the pectin's quality. The molecular chain of pectin, which contains the free galacturonic acid, is the equivalent weight of the pectin. The physical properties, like viscosity, water binding, etc., are due to free galacturonic acid [49]. The titration method can be used to determine the equivalent weight in this process 0.5 g of pectin sample was mixed with 5 ml of ethanol and 1.0 g of sodium chloride along with 100 ml of distilled water so that all the sample was dissolved well which will be titrated with 0.1 M sodium hydroxide solution using the phenol red indicator. Pectin obtained from different citrus fruits, including citrus sinensis (10,000 g/mol) and citrus maxima (1250 g/mol), have higher and lower equivalent weights. It concludes that citrus sinensis has more viscosity and water-binding properties than others, leading to its usage in food and cosmetics [61–63]. The equivalent weight fluctuates based on the solvent used and pH in the extraction of pectin [64]. At lower pH, pectin polymerisation occurs, reducing free acid content and creating pectin with a higher equivalent mass. In contrast, higher pH produces pectin with a lower equivalent mass. Pectin is a large molecule typically found with high molecular weight, making it difficult to digest in its natural form. Human digestive enzymes cannot break down pectin effectively, so its structure needs modification to enhance its biological activity. The molecular mass of black mulberry is also higher, which indicates higher antioxidant properties [60,65]. The low-molecular-weight pectin extracted from okra gained interest because of its improved physicochemical properties, which could be helpful in industry for different applications. The low molecular weight is better than the higher one in the food industry. It could be used as an effective gelling agent and emulsifier, which is increasingly applied for its prebiotic values in functional foods.

2.6. Methoxyl Content (MeO)

Methoxyl content in the pectin greatly influences its gel capacity. Usually, commercial pectin contains 8–11% methoxyl content and causes high sugar gels exceeding 65%. Pectin has a higher

methoxyl content when dissolved in water than pectin, which has a lower methoxyl content [66]. Yu et al. [67] reported that microwave-assisted extracted pectin (MAE) has 8.35% methoxyl content and exhibited higher apparent viscosity, thermal and emulsion properties. The pectin obtained via ultrasound-assisted extraction (UAE), enzyme-assisted extraction (EAE) and hot acid extraction methods (HAE) does not show significant differences in methoxyl content. In MAE, elevated microwave radiation levels decrease pH, and prolonged irradiation can reduce value [68]. Pectin extracted from apple pomace using an organic acid mixture (ORGS) showed a methyl content of 10.85%. This might be because ORGS has lower hydrolyzing capabilities and a lower dissociation constant [69]. Details of the methoxyl content of the pectin obtained from various sources and methods are listed in Table 1 [67,69–89].

Table 1. Details of methoxyl content (MeO) and Degree of esterification (DE) for pectin extracted from various sources using different methods.

Pectin Source	Extraction method	MeO (%)	DE (%)	Ref.
Pomelo peel	Hot acid extraction	7.43	55.67	[67]
	Microwave-assisted extraction	8.35	55.34	
	Ultrasound-assisted extraction	7.08	51.42	
	Enzyme-assisted extraction	6.62	47.71	
Apple pomace	Hot acid extraction	8.13	63.80	[69]
	Extraction using citric acid	9.69	63.42	
	Organic acid mixture extraction	10.85	64.55	
	Microwave-assisted extraction	9.30	64.80	
Citrus sinensis- Poncirus trifoliata	Ultrasound-assisted extraction	8.79	64.18	[70]
	Hot acid extraction	10.20	62.50	
	Electromagnetic induction extraction	09.9	61.00	
	Extraction using HCl	-	59.37	
Orange Peel Waste	Extraction using citric acid	-	57.75	[71]
Sea buckthorn peel	Extraction using citric acid	3.04	84.4	[72]
	Microwave-assisted extraction	4.77	73.8	
	Ultrasound extraction with and without heat treatment	4.22	77	
	Enzyme-assisted extraction-ultrasound treatment	3.9	47.6	
Sweet lime	Hydrothermal extraction	8.8	71.2	[74]
	Conventional solvent extraction	6.8	61.1	
	Extraction using HCl	9.3	82.7	
	Microwave-enzyme-assisted extraction	-	68.43	
Lemon peels	Enzyme-assisted extraction	-	67.89	[76]
	Acid extraction using H ₂ SO ₄	2.86	75.98	
	Microwave-assisted extraction	09.30	78.95	
	Microwave-assisted extraction	4.91	31.36	
Feijoa (Acca sellowiana) fruit	Extraction using HCl	-	27.63	[80]
	Extraction using citric acid	-	50.27	
	Maleic acid extraction	-	44.88	

Pumpkin peels	Extraction using HNO ₃	6.20	66.53	[81]
	Extraction using citric acid	7.23	66.57	
Eggplant Peels	Extraction using citric acid	5.76	61.33	[82]
Orange peel	Hot water extraction	13.81	96.58	[83]
Breadfruit's peel	Acidic extraction using HCl	15.78	96.70	[84]
Papaya's peel		5.5	33.67	

2.7. Degree of Esterification (DE)

The DE value shows the number of esterified carboxylic acids in pectin, comparing the total number of carboxylic acid groups. In industry, this parameter indicates the gel-forming capacity of pectin. When DE exceeds 50% pectin, it is categorized as high methoxyl pectin (HM); lower than 50% is classified as low methoxyl content (LM). HM pectin quickly forms a gel at high temperatures and low pH. In contrast, LM pectin forms rigid gels by cross-linkage with calcium or multivalent cations [90]. Details of the degree of esterification of pectin obtained from various sources and methods are listed in Tables 1 and 2. A significant change in the DE value was reported for pectin obtained from Pomelo peel [67]. The harsh conditions, followed in MAE and UAE, caused higher de-esterification of polygalacturonic chains, which reduced DE value. Citric acid, ultrasonic, and traditional heat extraction did not appear to change the degree of esterification of apple pectin [69], while the sonication time exhibited a negative effect on the DE for pectin obtained from dragon fruit peel [91]. An increase in the sonication time from 15 to 35 minutes reduced the DE value from 37.84 to 31.48%. Under prolonged exposure to sonication, an increase in de-esterification of polygalacturonic chains occurs, causing the decrement in DE value.

Table 2. Details Degree of esterification (DE) and Degree of acylation (DA) for pectin extracted from various sources using different methods.

Pectin Source	Extraction method	DE (%)	DA (%)	Ref.
Jack fruit	Extraction using citric acid	75.82	0.478	[88]
Jack fruit	Extraction using HNO ₃	74.82	0.418	[88]
Potato pulp	Extraction using HCl		11.92	[86]
Mosambi peels	Extraction using HCl	51.81	0.51	[87]
Sugar Beet Flakes	Extraction using citric acid		21.90	[85]
Kinnow peels	Extraction using HCl	51.86	0.47	[87]
Sugar Beet Flakes	Microwave-assisted extraction		21.90	[85]
Potato pulp	Extraction using H ₂ SO ₄		10.51	[86]
Orange peels	Extraction using HCl	53.55	0.48	[87]
Jack fruit	Extraction using H ₂ SO ₄	72.82	0.567	[88]
Sugar Beet Flakes	Pulsed ultrasound-assisted extraction		22.58	[85]
Chempedak	Extraction using citric acid	69.01	0.060	[88]
Potato pulp	Extraction using HNO ₃		10.51	[86]
Chempedak	Extraction using HNO ₃	69.1	0.328	[88]
Potato pulp	Extraction using citric acid		9.21	[86]
Potato pulp	Extraction using acetic acid		15.38	[86]
Chempedak	Extraction using H ₂ SO ₄	66.34	0.328	[88]
Coffee Arabica pulp	Extraction using H ₂ SO ₄	78.5	1.10	[89]

In the citric acid extraction method (CE), a decrease in pH value resulted in a reduction in DE value (65 to 57%) as reported for pectin extracted from Saveh pomegranate peel [92], while a decrease

in DE value (44.7% to 24.3%) during the increase in the citric acid concentration by Hundie et al. [93]. A similar trend was seen in the pectin extracted from Pulp in Pods of Riang by Apirattananusorn et al. [94].

2.8. Polydispersity Index (PDI)

The polydispersity index (PDI) indicates the range of molecular weight distribution. It is the ratio between the average molecular weight (M_w) and the number of average molecular weights (M_n). A larger PDI indicates a wider M_w distribution. It can be seen that the values of PDI are quite divergent from each other for all samples. This behaviour is acceptable since pectin from the exact origin has a wide range on the polydispersity index value (Table 3) [74,95–98]. High temperatures and longer extraction times used in hydrothermal (HT) and conventional solvent methods break C-O bonds, causing the PDI value [74]. PDI of HT-extracted pectin was near the commercial pectin value, indicating a lower range of molecular mass distribution. A higher PDI value was reported for pectin extracted from dragon fruit using the enzyme-assisted method by Du et al. [95]. It was higher when comparing the PDI value of pectin extracted using ultrasonic-assisted hot acid extraction to cold or hot water extraction for pectin from pitaya [96]. This is attributed to the cavitation force possibly causing fragmentation of the pectin, leading to a decrease in M_w . Time taken for the hydrolysis-extraction process under the influence of high temperature and pressure decreases the molecular weight, which is the PDI value for pectin extracted from apples and sunflowers. At the start of the hydrolysis process, pectin degradation did not start. As it proceeds, the destruction of aggregated pectin occurs through a decrease in the polydispersity, which indicates the enrichment of pectin in the hydrolysis-extraction process by linear homogalacturonan chains [97]. As mentioned earlier, like the effect of extraction conditions, environmental growth can affect the molecular mass and, thereby, the PDI, as reported by Apirattananusorn et al. [94].

Table 3. Polydispersity index for pectin extracted from various sources using different methods.

Source	Extraction method	PDI	Ref.
Sweet lime	Hydrothermal extraction	2.71	[74]
	Conventional solvent extraction	3.42	
Fresh dragon fruit	Cold-Water Extraction	4.81	[95]
	Hot-Water Extraction	4.19	
	Ultrasonic-Assisted Extraction	4.22	
	Enzyme-Assisted Extraction	13.04	
Pulp in Pods of Riang	Extraction using citric acid	4.31	[96]
Apple	Hydrolysis-extraction	8	[97]
Sunflower		5	
Rhubard		2.3	
Pumpkin Peels		2.67	
	Microwave-Assisted Extraction		[98]

2.9. Acetyl Value

The Acetyl groups at O-2 or O-3 on homogalacturonan, which is the backbone of pectin, play a significant role in emulsification. Pectin has a lower acetyl value and shows higher gelling capability. The reported value for pectin extracted from different sources is given in Table 2 [85–89]. The degree of acetylation for pectin extracted from sugar beet flakes by various methods was performed by Dranca et al. [85]. Pectin extracted using pulsed ultrasound-assisted extraction (PUAE) showed a higher degree of acetylation. Yang et al. [86] showed that pectin obtained by acetic acid extraction is more compared to other acid extraction methods. Acetic acid is a weaker acid that can retain more methyl-ester.

2.10. Water and Oil Holding Capacity

Water and oil holding capacity (WHC and OHC) are two essential properties of pectin. It can be defined as the amount of oil/water entrapped by pectin after mixing, incubation, and centrifugation. These properties are controlled by the constituents' hydrophobic/hydrophilic character and the total charge density [98]. Oil holding capacity (OHC) controls its emulsifying property in the food industry. Water holding capacity (WHC) of pectin, due to the presence of -OH in its structure, helps decrease the syneresis rate in food products. The high degree of esterification increases the WHC value of pectin. Details of the WHC and OHC of pectin extracted from different sources are summarized in Table 4 [78,99–115].

Table 4. Summary of the WHC and OHC of pectin extracted from different sources.

Pectin Sources	WHC (water/g powder)	OHC (oil/g powder)	Ref.
Citrus reticulata	8.27	0.10	[78]
Pumpkin peels	1.55	2.51	[99]
Sunflower stalk pith	40.2	40.4	[100]
Tomato pomace	3.57	2.65	[101]
Eggplant	6.02	6.02	[102]
Walnut	5.84	2.22	[103]
By-product from olive oil production	1.87	6.17	[104]
Pistachio green hull	4.11	2.02	[105]
Opuntia ficus indica	4.84	1.01	[106]
Watermelon rind	2	4	[107]
Dragon fruit peel pectin	4.08	2.18	[108]
Watermelon peels	3.65	1.12	[109]
Pumpkin peels	2.65	1.15	
Pomelo peels	4.57	1.49	
Pomegranate peels	2.43	1.17	
Soy hull	6.08	-	[110]
Passion fruit peel	7.57	-	
Orange pomace	6.4	1.6	[111]
Mango	14.6	0.7	
Pineapple	1.58	0.93	
Royal Gala apple	11.05	11.58	[112]
Cocoa Husk	4.19	2.02	[113]
Red chilli peel	8.1	8.5	[114]
Crab apple peel			[115]

3. Structural Characterization

3.1. Fourier Transform Infrared Spectroscopy (FTIR)

The potential capability of IR spectroscopy as a tool for quantitative analysis of pectin was pointed out by the beginning of the 1960s [116]. The peak value from FTIR spectra of commercial pectin is summarized in Table 5. A summary of FTIR results from various literature is given in Table 6 [69–75,85,91,93,117,118]. A significant difference was not observed between extracted and commercial pectin FTIR, as indicated by Wathoni et al. [77]. Also, the peak of the main structure of the pectin was not influenced by the different extraction methods.

Table 5. Summary of the FTIR peaks of pectin.

Wave number (cm ⁻¹)	Group
3200-3500	stretching of oxygen-hydrogen bond (–OH).
2920	C–H bond (including CH, CH ₂ , and CH ₃) vibration.
1730	C=O stretching vibration of ester.
1600-1400	C=O stretching vibration of carboxylate ion.
1200-1000	C–O and C–C vibration bands of glycosidic bonds and pyranoid rings.
1070	–COC– stretching of the galacturonic acid.

Table 6. Summary of FTIR results of pectin obtained from different sources.

Pectin Source	Extraction method	Results	Ref.
Apple pomace	Hot acid extraction	Results revealed that the pyranose was absent due to the extraction procedures. Ultrasound-assisted and Microwave-assisted extraction methods may partially disrupt the covalent bonds between pectin and non-pectic polysaccharides.	[69]
	Extraction using citric acid		
	Microwave-assisted extraction		
	Organic acid mixture		
Citrus sinensis- Poncirus trifoliata	Ultrasound-assisted extraction	Analysis of spectra shows that the heating type does not significantly affect the extracted pectin's structural properties.	[70]
	Hot acid extraction		
	Electromagnetic induction		
Orange peel waste	Extraction using HCl	Spectra of the extracted pectin exhibited consistent functional groups with the standard, although with varying intensities in some instances.	[71]
Sea buckthorn peel	Extraction using citric acid	The peak at 1,745 cm ⁻¹ was stronger than 1,635 cm ⁻¹ , indicating that sea buckthorn contains high methoxy pectin.	[72]
Fälticeni' apple Pomace	Extraction using citric acid	All pectin samples obtained by different extraction methods had a similar transmission pattern to those of commercial apple and citrus pectin samples.	[73]
	Microwave-assisted extraction		
	Ultrasound extraction with and without heat treatment		
Sweet lime	Enzyme-assisted extraction-ultrasound treatment.	The obtained results were compared with conventionally extracted pectin and commercial citrus pectin.	[74]
	Hydrothermal extraction		
Lemon peels	Conventional solvent extraction	FTIR spectra of all samples were nearly identical.	[75]
	Extraction using HCl		
Sugar beet flakes	Extraction using citric acid	In CE pectin, the peak at 1718 cm ⁻¹ was higher when compared with the spectra of MAE and PUAE pectin samples, indicating a higher degree of esterification of the CE sample.	[85]
	Microwave-assisted extraction		
	Pulsed ultrasound-assisted extraction (PUAE)		
Dragon fruit peel	Ultrasound-assisted extraction	Extracted pectin has functional groups similar to commercial-grade pectin and is rich in polygalacturonic acid.	[91]
Saveh Pomegranate Peels	Extraction using citric acid	There is no significant difference between extracted pectin and standard pectin.	[92]
Lemon Waste	Extraction using Citric acid	FTIR spectra were similar to the 55 – 70% degree of esterification of citrus pectin	[93]
Passion	Subcritical Water Extraction		[117]

Fruit Rinds	Pressurized Natural Deep Eutectic Solvents under different temperature	The type of solvent used in the different extraction methods affects the degree of methyl esterification.	
Pumpkin Peels	Microwave Assisted Extraction	The relative intensity of the ester band increased according to the DE of the pectin, while the intensity of the carboxylic stretching band decreased.	[118]

3.2. Thermal Analysis

It's important to conduct thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) for extracted pectin to gather information regarding its thermal behaviour and identify possible target applications.

3.2.1. Thermogravimetric Analysis (TGA)

For pectin, temperature has a greater influence on the loss of mass, structural changes, conformational changes, changes of state, chemical reactions, degradation, etc. The thermal stability and mass loss of pectin can be measured using TGA as a function of temperature. Pectin typically undergoes a three-step degradation process: The first step below 100°C corresponds to water loss [98], and the second step is between 200 and 400°C due to pyrolytic decomposition and breaking of hydrolytic bonds. It consists of a primary and secondary decarboxylation involving the acid side group and a carbon in the ring. Due to oxidative reactions, the third stage was postponed, particularly for linoleic residues, as expected for the oxygen-scavenging effect of the double bonds [75,119]. Like commercial pectin, the pectin extracted from Citrus Limetta peels showed three-step degradations [74].

3.2.2. Differential Scanning Calorimeter (DSC)

For pectin, the DSC thermogram shows two prominent peaks: One for the endothermic property of pectin, which can be affected by extraction temperature, and another for exothermic property, affected by its constituents and raw material [120]. The first endothermic peak, between 50 and 150°C, is due to the evaporation of water molecules [98], followed by an exothermic peak between 210 and 270°C, which is associated with pectin degradation. DSC analysis for pectin extracted from pomelo peel with different extraction techniques was reported by Yu et al. [67]. Irrespective of the extraction method, all pectin showed exothermic peaks between 240 and 265°C, where the pectin degradation begins, and a significant difference was not observed for exothermic peak values. Notably, the pectin obtained by microwave-assisted extraction had a broader exothermic peak, indicating that the pectin had a higher thermal stability. In contrast, Mahmoud et al. [69], showed that pectin extracted by citric acid exhibited an endothermic peak that shifted slightly towards lower temperatures due to greater water content and altered pectin structure.. Another study by Dranca et al. [85] found varying thermal profiles in pectin samples: sugar beet flake pectins showed one or two endothermic peaks and one exothermic peak, while pectin from Malus domestica 'Fälticeni' apple pomace showed no endothermic peaks. Specifically, for CE and MAE pectin from sugar beet flakes, the first endothermic peak appeared at 156.42°C and 160.45°C respectively, with the second peak at 183.97°C and 190.08°C respectively. These endothermic peaks were linked to pectin melting, potentially caused by conformational changes and chemical bond cleavage preceding polysaccharide degradation [121]. The absence of endothermic peaks in the apple pomace pectin suggests no elimination of bound water from these samples. The pectin extracted from sea buckthorn peel also showed a melting temperature of 133.48°C, and the enthalpy of degradation was 269.03°C [72].

3.3. Scanning Electron Microscopy (SEM)

The particle size greatly influences the characteristics and end applications of pectin. Pectin with smaller particle sizes can quickly disperse in solutions, enhancing solubility and interaction with other food components. Scanning Electron Microscopy (SEM) can be used to investigate the effect of

extraction methods and conditions on the morphology of the pectin [73–77,79,117,122,123]. Pereira et al. [117] studied the morphology of pectin extracted from passion fruit rinds using conventional, sub-critical water and pressurized natural deep eutectic solvents. The pectin extracted using water showed a uniform surface, while the acidified mixtures led to more heterogeneous surfaces.

The effect of the extraction method (MAE, UAE, and CE) on the morphology of apple pomace was studied by Dranca et al. [73]. The CE pectin had a homogeneous, smooth and porous structure compared to MAE pectin. Rapid increases in temperature and pressure in the MAE process can cause a rough surface for the pectin. The SEM of UAE pectin showed higher fragmentation but was closely packed. Similar morphology was reported for potato pectin extracted by combined ultrasound microwave-assisted acid extraction [122]. The pectin extracted from lemon peel also exhibited roughness and irregularity in morphology [75]. Das et al. [74] reported a visible structural difference between pectin obtained through hydrothermal (HT) and conventional solvent extraction with commercial samples. The morphology of HT extracted pectin closely resembled that of commercial pectin, which has a loose surface and some fractural changes. SEM of pectin extracted from feijoa using a conventional heating method showed relatively compact with a honeycomb surface, despite several cracks and potholes on the surface [76]. The surface of the apple waste using an organic acid mixture and ultrasonication was more compact, multilaminated, and flaky and looked extremely hard [79]. The high amount of neutral sugar in the pectin extracted from cocoa pod resulted in heterogeneous morphology [123]. The morphology of pectin extracted from mangosteen rind was compared with commercial pectin by Wathoni et al. [77]. The extracted pectin had a surface with relatively smaller particles than standard pectin.

3.4. X-Ray Diffraction (XRD)

X-ray Diffraction (XRD) can analyze the variation in amorphous and crystalline nature attributed to the source and extraction methods and conditions. A comprehensive summary of XRD results of pectin obtained from different sources is given in Table 7 [76,78,89,124–131]. Mahmoud et al. [69] used XRD to study how different extraction methods affect pectin's crystalline and amorphous nature from apple waste. The peaks below 2θ less than 20° indicate the pectin's semi-crystalline nature. A sharp peak disappeared for MIC, HC and ORG-extracted pectin due to the decrease in its molecular weight. A similar XRD pattern was reported for the pectin obtained from Indonesian mangosteen and standard pectin by Wathoni et al. [77]. In contradiction to the above, Duggal et al. [78] revealed that commercial pectin is more crystalline than pectin extracted from kinnow peel, having 2θ values at 14.31, 37.93 and 43.16°.

Table 7. Summary of XRD results of pectin obtained from different sources.

Pectin Source	Extraction method	2θ values	Ref.
Feijoa (Acca sellowiana) fruit	Conventional Heating (CHE)	Four characteristic peaks at 2θ = 13.14°, 21.05°, 43.41° and 50.39° were observed in pectin obtained by CHE method. CHE exhibited much broader and stronger diffraction peaks at 12° than MEAE and EAE samples. Besides, the peaks around about 2θ = 21.05°, 43.41°, and 50.39° in MEAE and EAE samples exhibited less sharp, and the peak at around 2θ = 13.14° was not detected.	[76]
	Microwave-enzyme-assisted extraction (MEAE)		
	Enzyme-assisted extraction (EAE)		
Citrus Reticulata peels	Microwave-assisted extraction	Kinnow pectin is less crystalline compared to commercial pectin. Peak values were observed at 2θ = 14.31°, 37.93° and 43.16° while for commercial citrus pectin at 21.33° and 22.57°.	[78]
Coffee pulp pectin	Acid extraction using sulfuric acid.	Coffee pulp powder is more amorphous compared to the coffee pulp pectin. Coffee pulp pectin exhibited sharp peaks at 2θ = 15.72, 19.52, 20.90, 22.94, 25.02, 26.88, 28.44, 29.59, 31.59, 32.94, 35.88, 38.32, 44.5, 46.66, 48.2, and 51.44°. Coffee pulp	[89]

		pectin has a higher degree of crystallinity than coffee pulp powder.	
Sweet lemon peel pectin	Microwave-assisted extraction	Peaks observed at $2\theta = 12.36^\circ, 13.96^\circ, 14.91^\circ, 19.61^\circ, 18.91^\circ, 21.36^\circ, 32.46^\circ,$ and 36.66°	[124]
Orange peels	Extraction using HCl.	Peaks observed at $2\theta = 13.222^\circ, 15.37^\circ, 21.78^\circ, 25.28^\circ, 27.87^\circ$ and has crystallinity value, 68.97%.	[125]
Watermelon rind	Extraction using acetic acid.	Shows a weak characteristic diffraction peak at $2\theta = 12.1^\circ$ and 21.3° .	[126]
Orange waste	Extraction using H_2SO_4 .	Pectin shows crystalline peaks in its diffractogram at $2\theta = 9.5, 11.5, 20.8, 28.9, 30.9,$ and 31.5° , which indicates that it is crystalline.	[127]
Apple pomace	Extraction using HCl.	Pectin extracted from the Royal (<i>Malus pumila</i>) apple was more crystalline than Golden (<i>Spondias dulcis</i>).	[128]
Taiwan's Citrus depressa Hayata Peels	Ultrasonic method	Peaks were observed at $2\theta = 16.06, 18.92,$ and 21.19° XRD pattern was similar to commercial pectin, only that commercial pectin showed intense peaks at $20.84, 25.59$ and 30.3° .	[129]
Passion Fruit Peel	Acid Extraction (AE) Ultrasonic-Assisted Acid Extraction (UA) Steam Explosion Pretreatment Combined with Acid Extraction (SEA) Ultrasonic-assisted steam Explosion Pretreatment Combined with Acid Extraction (USEA)	The crystal structures of pectin extracted by AE and SEA are similar but different from those of UA and USEA. The ultrasonic-assisted process changes the crystal structure of pectin.	[130]
Orange peel pectin	Alcohol precipitation method	Peaks observed at $2\theta = 12^\circ, 16^\circ, 18^\circ, 27^\circ$ and 29° while by enzymatic method was $16^\circ, 26^\circ, 29^\circ,$ and 30° .	[131]

3.5. Nuclear Magnetic Resonance (NMR) Spectroscopy

Pectin is a complex structure with diverse heterogeneous polysaccharide groups usually found on the surface of the plant's cell and in the cementing layer between the primary walls of adjacent cells, mainly responsible for the pectin's mechanical strength and flexibility. By interacting with other components' cell walls, these also significantly influence their industrial applications. NMR spectroscopy is a valuable technique for component characterization and structure elucidation. It is a promising device for quantitatively determining functional groups of complicated structures where all the equivalent nuclei possibly bring about signals with equal intensity despite their chemical background. This NMR technique has been well established for determining pectin structure related to the macromolecule chain (Table 8). The preparation of solutions for NMR analysis is complex. However, it is easily dissolved in water, making it more viscous, which could complicate NMR solution preparation for its analysis.

Table 8. NMR information for the characterization of pectin.

Instrumentation	Results	Ref.
Bruker Avance III (400 MHz) was used and applied with 400.15 MHz for 1H and 100.63 MHz for ^{13}C , equipped with a mass probe of 7 mm H/S to collect spectral information. The NaCl powder	<ul style="list-style-type: none">• The reference, C_1, C_2, C_3, C_4, C_5 and C_6 intensities were very strong.• The absorption peaks at 171 ppm as COOH (protonated), 174 ppm as COOCH₃, and 176 ppm as COO⁻ (ionized form) in galacturonic acid.• The signals at 101 ppm were C_1 and 79 ppm as C_4 of glycosidic bonds.	[132]

was mixed with the sample of about 170 ±1 mg into the cylindrical zirconium dioxide rotors with an outer diameter of about 7 mm with silicon rubber tubing.	<ul style="list-style-type: none">• The signals between 67–72 ppm were the pyranoid ring's C₂, C₃, and C₅ carbon.• The peak at 53 ppm was methyl carbon of COOCH₃ (methyl ester).• The peak at 0 ppm represents -CH₃ from the dimethyl silicone rubber tubing.• The % recovery between 94.33–102.77% with %RSD < 2.32 %	
The ¹³ C high-resolution NMR manufactured by Bruker (Avance AV 400), applied at 400 MHz and equipped with a 4 mm outer diameter, included 62.5 kHz rf-field strength and 15 kHz as spin rate. The carbonyl peak at 176.05 ppm of glycine was a reference for all spectral data. The software MestReNova 6.1.1 was used for the analysis & interpretation.	<ul style="list-style-type: none">• Resonances assign galacturonic units of C₆ carbon at 176–168 ppm.• Glycosidic bonds C₁ and C₄ carbon were indicated at 101 and 79 ppm, respectively.• The pyranoid ring's other carbons C₂, C₃ and C₅ at 67-72 ppm.• The peak at 175 ppm represents the ionized form (COO⁻) and methyl ester pectinate.• At 53 ppm, it was assigned as methyl ester's methyl carbons.• The average % recovery was 92.5%, with % RSD within the 4.4–4.9% range.	[133]
The Bruker (Germany) 400 Advance Ultra 9.4 T was controlled at 400 MHz with a broad band inverse probe at 303 K. D ₂ O was used to prepare the pectin samples (0.5%, w/w) and reference as perdeuterated 3-(trimethylsilyl) propionate sodium salt. The acquisition time was 1.98 s with a pulse of 301 and a relaxation delay time of 8 s with scans of 128, including an 8278.15 Hz as sweep width.	<ul style="list-style-type: none">• A large doublet at 1.14 ppm was assigned as isopropanol, used to separate pectin from aqueous solutions as a precipitate. At 4 ppm, it also contributed significantly.• The methoxy group containing proton in the esterified pectin showed at 3.78 ppm, which enhanced the degree of esterification.• The proton H-2 and H-3 were assigned at 3.7 and 3.96, respectively.• The two signals at 4.9 and 3.96 ppm are H-5 protons near galacturonic acid. At 4.6 ppm for H-5, protons adjacent to ester groups shifted downhill to about 5.0 ppm.	[134]

4. Eco-Friendly Extraction Techniques

In recent years, the demand for pectin—a versatile polysaccharide widely used in food, pharmaceutical, and cosmetic industries—has surged due to its gelling, thickening, and stabilizing properties. Traditional pectin extraction methods typically rely on acid hydrolysis and extensive thermal processing, both of which consume significant amounts of energy and generate chemical waste, raising environmental and economic concerns [135]. Researchers have increasingly turned to eco-friendly extraction techniques that improve efficiency while reducing environmental impact to address these challenges. These greener methods minimize the use of harmful solvents and excessive energy consumption and maintain the extracted pectin's structural integrity and quality. Innovations such as ultrasound-assisted extraction, pulsed ultrasound-assisted extraction, pulsed electric field, moderate electric field mediated extraction, microwave-assisted extraction, subcritical water extraction, enzyme-assisted extraction, ohmic heating-assisted extraction, ultrasound-assisted microwave extraction, ultrasound-assisted ohmic heating extraction, hydrothermal processing using water, high-pressure processing extraction, and dielectric barrier discharge extraction present promising pathways for achieving more efficient and environmentally responsible pectin production.

4.1. Ultrasound-Assisted Extraction (UAE)

This method increases mass transfer and extraction efficiency by using ultrasonic waves. Ultrasound waves cause cavitation bubbles in the solvent to abruptly burst, releasing shock waves that degrade cell walls and facilitate extraction. The sound waves here have frequencies higher than human hearing; they usually fall between 20 and 40 kHz for pectin extraction. Ultrasound

irradiation of raw materials fulfils several goals. On the one hand, ultrasonic irradiation increases cell disintegration by cavitation, exposing the cells' inner structure to the solvent. On the other hand, sonication speeds the rehydration of dry materials, boosting solvent penetration. Nevertheless, extended ultrasonic irradiation with heat should be avoided since it may promote fast pectin degradation, resulting in a decreased yield [21,136–141]. Overall, the UAE mechanism maximizes mass transfer, cavitation, and cell disruption, thereby improving extraction yield and efficiency. The mechanism (Figure 2) is described as follows:

- As ultrasound waves travel through a liquid, they produce alternating high and low-pressure cycles. The low-pressure phase of the liquid is when bubbles, which are tiny holes filled with vapour, form. As the pressure rises, these bubbles suddenly pop. Cell walls and tissues might be harmed by shock waves from the collapse's concentrated high temperatures and pressures.
- The energy produced during cavitation causes plant cell walls to rupture, allowing solvents to enter and dissolve the required ingredients. When the cell wall breaks down, the solvent may more readily penetrate the material, improving contact and interaction between the solvent and the target molecule. Furthermore, mechanical movement can make the solventless viscous and extraction easier.
- Cavitation can produce localized heating, increasing the solubility of some substances, even though UAE is often conducted at mild temperatures to preserve sensitive compounds. The type of material removed and the ultrasonic frequency, power, and duration can all impact UAE performance.

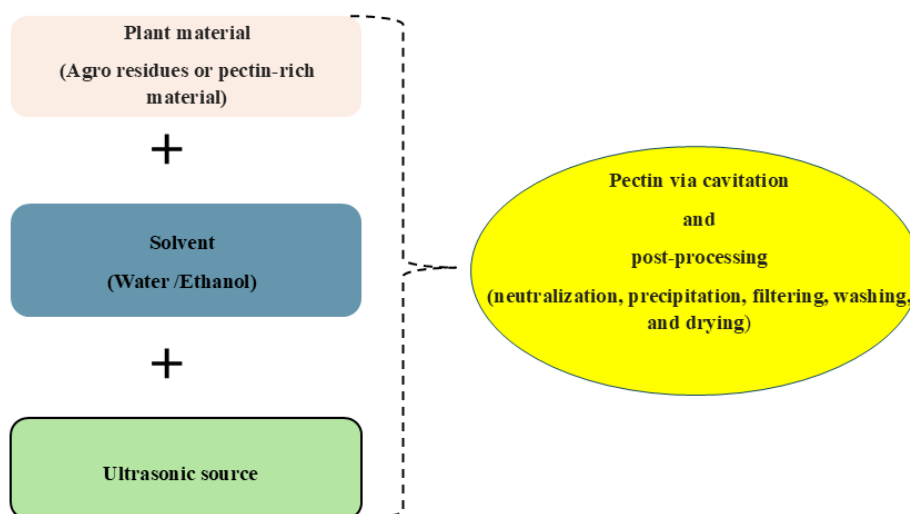


Figure 2. Schematic representation of Ultrasound-Assisted Extraction technique.

4.2. Pulsed Ultrasound-Assisted Extraction (PUAE)

A sophisticated method called Pulsed Ultrasound-Assisted Extraction (PUAE) uses sporadic ultrasonic pulses to separate pectin from plant components. This technique maximizes extraction efficiency, minimizes possible heat-induced deterioration, and takes advantage of ultrasounds. The mechanism of the PUAE technique (Figure 3) could be elaborated as below [136,140,142,143]:

- PUAE alternates between periods of rest and sound application using short bursts or pulses of ultrasonic energy. This pulse allows for cooling periods, which aid in temperature regulation and reduce the danger of heat-induced damage to sensitive molecules.
- Cavitation during ultrasonic pulses causes microbubbles to expand and collapse. This activity breaks down the plant material's cell walls, making pectin easier to release.

- By using ultrasonic radiation and pulsed application to promote solvent diffusion into the plant matrix, pectin extraction is accomplished more effectively.
- The technique prevents excessive temperature buildup by pulsing the ultrasonic, which is crucial for maintaining the integrity of pectin and other sensitive bioactive components.

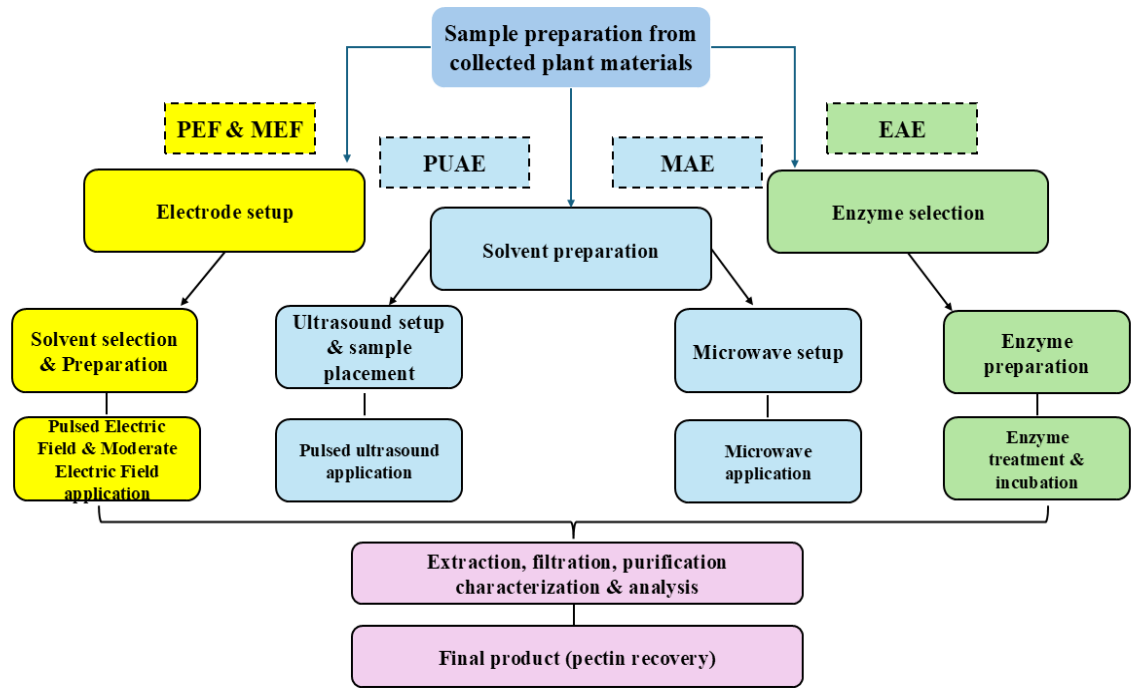


Figure 3. Schematic representation of PUAE, PEF, MAE and EAE extraction techniques.

4.3. Pulsed Electric Field (PEF) and Moderate Electric Field (MEF) Mediated Extraction

Two cutting-edge methods that use electric fields to improve the extraction of bioactive chemicals from plant materials are pulsed electric field (PEF) and moderate electric field (MEF) extraction [139,140,144–148]. The possible extraction mechanism of PEF (Figure 3) is as follows:

- Plant tissues are subjected to brief, high-voltage electric field bursts by PEF. The term "electroporation" describes how electric fields cause cell membranes to break down electrically.
- Temporary membrane ruptures brought on by cell membrane disruption let intracellular substances, like pectin, phenolics, and essential oils, leak out into the surrounding solvent.
- Solvents may enter the matrix more rapidly due to the more porous cell walls, which facilitates the extraction of the required molecules.

Meanwhile, MEF employs lower-intensity electric fields than PEF. The basic approach relies on constantly producing an electrical field to improve the extraction process without creating a lot of electroporation. Charged molecules and ions may travel more freely in an electric field, increasing chemical solubilization and cell wall breakdown.

4.4. Microwave-Assisted Extraction (MAE)

The Microwave-Assisted Extraction (MAE) technique uses microwave radiation to enhance the extraction of bioactive compounds from solid materials, including plant tissues. This approach consists of oscillating electric and magnetic fields perpendicular to one other. Microwave radiation exposes plant skin tissues quickly and extensively because it breaks down the cell wall matrix and ruptures parenchymal cells [8,21]. As a result, the extracting agent and the source material employed in the extraction process will interact more; the extracting agent will penetrate deeper into more

pectin [139,140]. The following extraction procedures (Figure 3) could be followed for MAE [149–154]:

- When subjected to microwave electromagnetic radiation, polar molecules—like water—vibrate quickly, producing heat inside the sample.
- The pressure generated by the quick heating and evaporation of intracellular water leads to cell rupture and increased release of target molecules.
- The interaction of heat and solvent enhances the extracted chemicals' solubility and diffusion, hence boosting extraction efficiency.
- Rapid heating and effective energy transfer lead to short extraction time.

4.5. Subcritical Water Extraction (SWE)

Subcritical water (SCW) is a rapidly growing approach for extracting bioactive compounds from different food sources [21]. Water is maintained at temperatures between 100°C and 374°C and pressure high enough to conserve the liquid state of water (less than the critical pressure of 22 MPa). This technique uses water under subcritical temperatures and pressures with a dielectric constant and a higher ion product change to extract valuable compounds from biomass. It takes advantage of the unique properties of water when heated under pressure [21,120], allowing it to act as both a solvent and a reactant. SCW usually follows the following extraction mechanism (Figure 4) [139,140,155,156]:

- Water is heated to subcritical temperatures (100°C–374°C) while under pressure to avoid boiling.
- Water dissolves nonpolar compounds more easily at subcritical temperatures because its dielectric constant decreases. As a result, it can potentially extract a wider range of polar and nonpolar compounds.
- Many compounds become more soluble at higher temperatures, allowing mass transfer from the solid matrix to the solvent.
- Heat and pressure can aid in releasing bioactive compounds from the plant matrix by dissolving the cell walls.

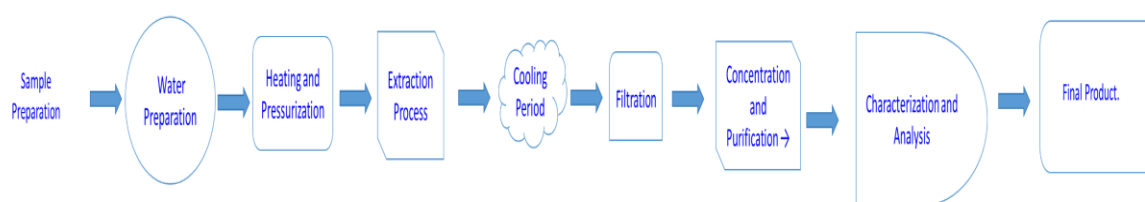


Figure 4. Schematic representation of the SWE technique.

4.6. Enzyme-Assisted Extraction (EAE)

Enzyme-Assisted Extraction (EAE) is a technique that employs enzymes to extract bioactive compounds from plant or animal tissues. Enzyme extraction and ultrasonic extraction both make use of enzymes' ability to catalyze processes, resulting in shorter extraction times and less alcohol volume in the precipitation step. EAE offers significant benefits for extracting pectin from plant feeds, wastes, and byproducts. This method improves pectin extraction yield while improving energy consumption. The mechanism (Figure 3) of EAE is as follows [157–161]:

- The kind of biomass and target compounds are considered while selecting enzymes.

- The biomass is treated with the selected enzymes under certain pH, temperature, and time conditions to maximize their activity.
- Cell walls' proteins and polysaccharides are hydrolyzed by enzymes, which permits solvents to enter and remove the required ingredients.

Generally, the above-mentioned techniques are excellent for extracting pectin from different sources in terms of physical properties and showed low, medium, and higher percentages of yield and other valuable contents [139,140,142,154–162]. Tables 9 and 10 below represent a general comparison of these extraction methods.

Table 9. Comparison considering different parameters: yield, color, moisture, protein content, galacturonic acid and methoxyl content.

Extraction Method	Yield	Color	Moisture	Protein Content	Galacturonic Acid	Methylxyl Content
UAE	M-to-H	MR	H	V	V	M-to-H
PUAE	H	HR	L	H	H	H
REF	M	MR	H	V	V	V
MEF	H	MR	M	M	H	M
MAE	H	MR	H	H	M	H
SWE	M-to-H	MR	H	V	H	M
EAE	H	MR	H	H	M	H

L=Low, M= Medium, H=High, VH=Very High, V=Variable, L-to-M= Low to Medium, M-to-H= Medium to High, MR= Medium Retention, HR=High Retention.

Table 10. The comparison considers different parameters: degree of esterification, polydispersity index, equivalent weight, molecular weight, acetyl value, oil holding capacity, and water holding capacity.

Extraction Method	Degree of Esterification	Polydispersity Index	Equivalent Weight	Molecular Weight	Acetyl Value	Oil Holding Capacity	Water Holding Capacity
UAE	M	L-to-M	V	V	V	M	H
PUAE	M	L	V	H	V	H	H
REF	H	H	H	H	H	M	M
MEF	M	L-to-M	H	V	V	M-to-H	M
MAE	M	L	V	L	V	H	H
SWE	V	L	V	V	L	M	H
EAE	H	L	V	H	H	H	VH

4.7. Ohmic Heating-Assisted Extraction (OHAE)

Ohmic Heating-Assisted Extraction (OHAE) uses electrical resistance to achieve rapid and uniform heating of the plant matrix, facilitating the extraction of pectin. Applying an electric current generates heat within the sample, leading to cell wall breakdown and improving bioactive compounds' solubility [163,164]. Figure 5 shows a schematic representation of the extraction process.

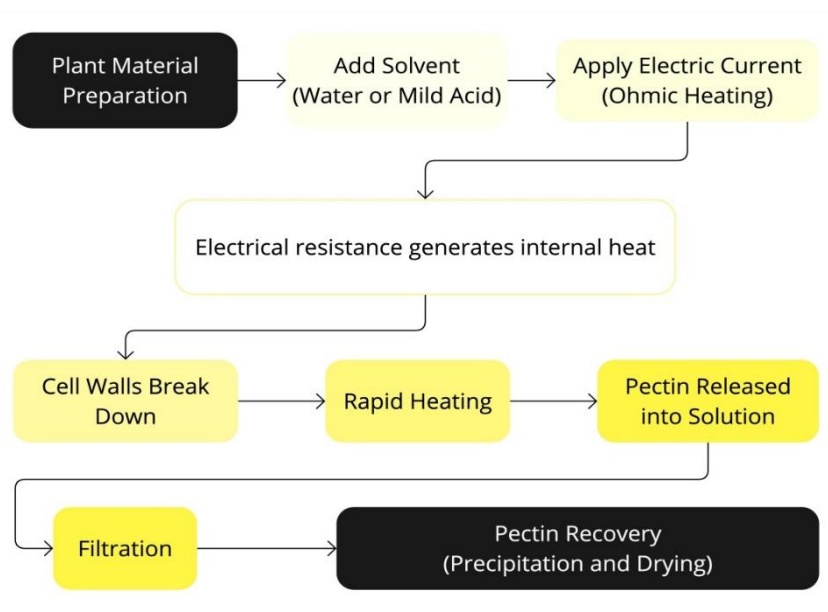


Figure 5. Schematic representation of the Ohmic Heating-Assisted Extraction (OHAE) process.

This method is particularly advantageous due to its uniform heating, which minimizes the thermal degradation of pectin. Additionally, OHAE offers significant energy efficiency compared to traditional heating methods and reduces the overall processing time. The rapid heating process enhances extraction yields, making it suitable for industrial applications. However, challenges remain in the adoption of OHAE. The initial investment in specialized equipment is high, which may limit its availability to smaller operations. Optimization of electrical parameters, such as voltage and current, is critical to prevent overheating and pectin degradation. Furthermore, scalability to industrial levels is complex due to the technical intricacies of maintaining consistent heating across large volumes [163]. Despite the above challenges, Gavahian and Chu successfully used the techniques for pineapple core valorization [165]. Sharifi et al. developed optimal conditions for extracting pomegranate pectin by ohmic heating. Extraction yield and galacturonic acid percentage were 8.16% and 82.86%, respectively [166].

4.8. Ultrasound-Assisted Microwave Extraction (UAME)

Building on the efficiency of OHAE, Ultrasound-Assisted Microwave Extraction (UAME) introduces a dual mechanism that enhances extraction further by combining ultrasound waves and microwave heating. UAME combines ultrasonic waves with microwave radiation to enhance the pectin extraction process; the synergistic effects of these two technologies result in efficient cell disruption and heating, leading to improved mass transfer and pectin release. Microwaves increase the internal temperature and pressure of plant cells, while ultrasound waves disrupt the cell walls, facilitating the release of pectin molecules [167–169]. Sonication before microwave results in efficient cell disruption, giving a better quality of pectin.

This method significantly reduces solvent usage and shortens extraction time, making it both cost-effective and environmentally friendly. The quality and purity of the extracted pectin are often superior due to the mild processing conditions. Despite its advantages, UAME presents several challenges. The need for specialized dual-function equipment increases initial costs. Furthermore, pectin degradation may occur if the heat generated by microwaves is not carefully controlled. Optimizing the balance between microwave power and ultrasound intensity is crucial to achieving efficient extraction without compromising pectin integrity [168]. Yang and coworkers applied Box–Behnken design to optimize the UAME process for potato pectin extraction. Under optimal conditions, the pectin yield and galacturonic acid percentage were 23% and 42%, respectively. As depicted in Table 11, the potato pectin had an Mw of 1.537×10^5 g/mol. It was classified as a low

methoxyl (DM, 32.58%), but highly acetylated (DA, 17.84%) pectin [170]. Lasunon and Sengkhamparn studied the effect of ultrasound-assisted, microwave-assisted, and ultrasound-microwave-assisted extraction on pectin extraction from industrial tomato waste [171]. They found that the combined ultrasound and microwave techniques gave a better pectin yield of 34%. Although some researchers subjected the raw sample to microwave heating before applying ultrasound, as depicted in Figure 6, Forouhar and coworkers showed that ultrasonic pretreatment changed the morphology of the raw powder, leading to better extraction efficiency. They reported a 19% extraction yield of pectin from watermelon rind and a galacturonic acid content of 69% [172].

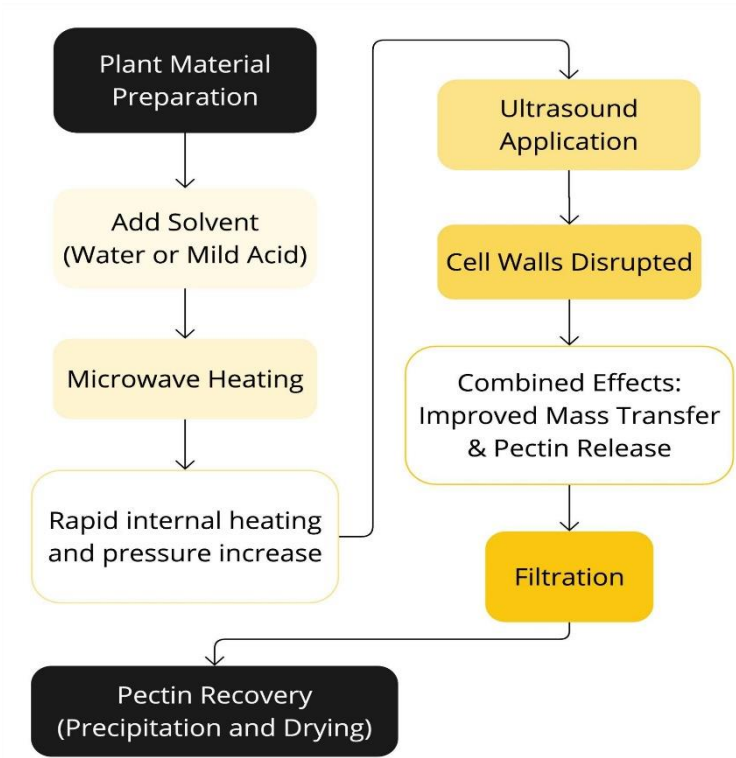


Figure 6. Schematic representation of the Ultrasound-Assisted Microwave Extraction (UAME) process.

4.9. *Ultrasound-Assisted Ohmic Heating Extraction (UAOHE)*

In a similar vein, Ultrasound-Assisted Ohmic Heating Extraction (UAOHE) leverages ultrasound technology but replaces microwaves with ohmic heating. Ultrasound waves effectively disrupt cell walls, while ohmic heating ensures rapid and uniform heating, facilitating mass transfer and enhancing extraction yields [173,174]. This combination method offers several advantages, including reduced energy consumption, higher yields, and shorter processing times. The dual-action mechanism improves pectin extraction's overall efficiency and quality, making it a promising technique for large-scale production. However, the complexity of integrating ultrasound and ohmic heating systems poses significant challenges. The cost of equipment capable of performing both functions is high, which can be a barrier to widespread adoption. Additionally, precise control of process parameters, such as temperature and ultrasound intensity, is necessary to avoid pectin degradation. Prolonged ultrasound irradiation with heat leads to pectin degradation. These factors complicate the scalability of UAOHE for industrial applications.

In spite of some drawbacks, researchers employed UAOHE to extract pectin. For example, Wang et al. used the technique successfully for the extraction of pectin from grapefruit peel achieving a yield of 27%. The study showed that pectin extracted by this method possessed lower viscosity and molecular weight than the traditional heating extraction method [175]. Xu studied the effects of ultrasound and/or heating on the extraction of pectin from grapefruit peel. The study found that

UAOHM significantly enhanced pectin's extraction rate, leading to an improved yield in shorter extraction time and at a lower temperature (Figure 7) [176].

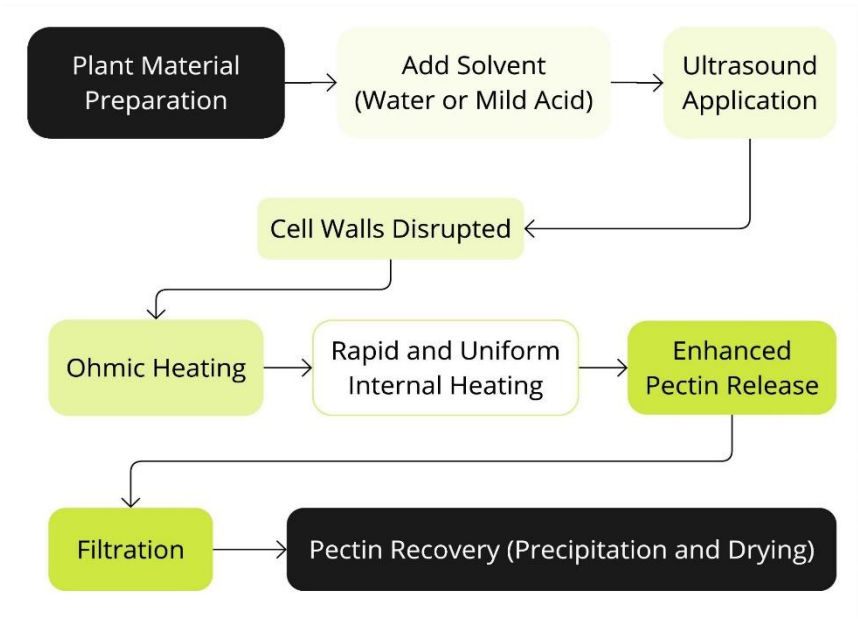


Figure 7. Schematic representation of the process of Ultrasound-Assisted Ohmic Heating Extraction (UAOHE).

4.10. Hydrothermal (HT) Processing

For a solvent-free approach, hydrothermal (HT) processing provides an alternative that relies solely on water. The hydrothermal (HT) process uses subcritical water—water heated to temperatures between 100°C and 200°C under pressure—as a solvent for pectin extraction. This technique takes advantage of water’s altered physical properties under high temperatures and pressures to enhance its solvent capabilities [135]. Figure 8 depicts a simplified representation of the HT process. The HT process is environmentally friendly, using water instead of chemical solvents, reducing the risk of contamination and making the process safer for operators and consumers. Additionally, the simplicity and cost-effectiveness of the process make it attractive for sustainable extraction practices. Nevertheless, the HT process is energy-intensive due to maintaining high temperatures and pressures. Thermal degradation of pectin is a concern if the temperature is not carefully controlled. Moreover, the process can be challenging to optimize, as slight variations in temperature and pressure can significantly impact pectin yield and quality.

Extraction of pectin using HT is relatively rare, mainly if it is done in supercritical water. Recently, Pińkowska and coworkers studied the Hydrothermal Extraction of pectin from sugar beet pulp [177]. The study focused on the effect of extraction temperature and holding time on the course of hydrothermal extraction of pectin, showing that yield, degrees of methoxylation, and acetylation varied with varying temperatures and holding times. Das and Arora applied a one-stage hydrothermal (HT) process using water as an extraction medium to extract pectin from sweet lime peels [74]. As shown in Table 11, the extract was rich in galacturonic acid (70.6±1.3%) and high in degree of esterification (71.2±1.0%).

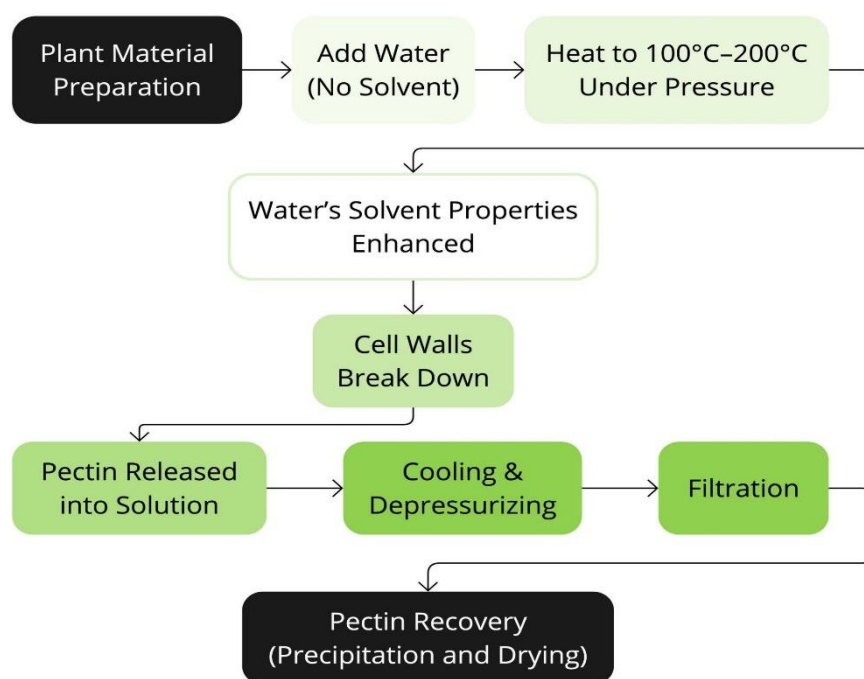


Figure 8. Schematic representation of the Hydrothermal Extraction process (HTE).

4.11. High-Pressure Processing Extraction (HPPE)

Shifting the focus from temperature to pressure, High-Pressure Processing Extraction (HPPE) offers a low-temperature method that maintains pectin integrity by applying high pressure. HPPE employs pressures between 100MPa and 600MPa to disrupt plant cell walls and facilitate pectin release. The pectin extraction process using HPPE is schematically shown in Figure 9. Unlike traditional thermal methods, HPPE can be conducted at low temperatures, preserving the integrity of heat-sensitive pectin molecules [178,179]. This method offers several advantages, including increased extraction yield, reduced processing time, and minimal solvent use. The ability to maintain the quality and purity of pectin makes HPPE particularly suitable for food-grade applications. However, the implementation of HPPE is hindered by high equipment costs and maintenance requirements. The process requires skilled operators to manage and optimize pressure conditions. Additionally, scaling up HPPE to industrial levels poses challenges due to the complexity of maintaining consistent high-pressure conditions [179].

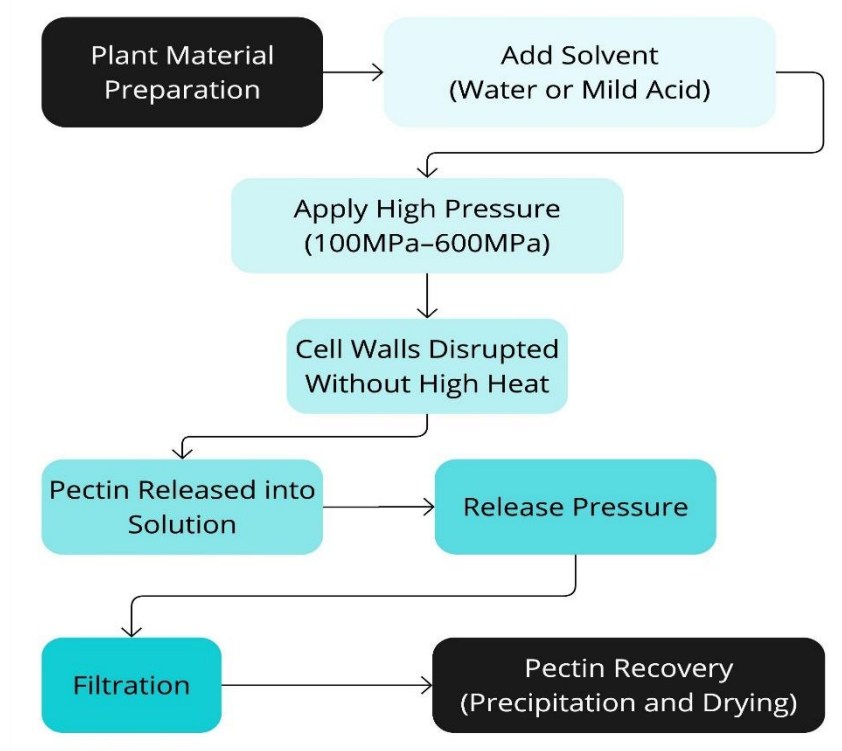


Figure 9. Schematic representation of the High-Pressure Processing Extraction technique (HPPE).

4.12. Dielectric Barrier Discharge Extraction (DBDE)

Dielectric Barrier Discharge Extraction (DBDE) uses plasma technology to extract pectin solvent-free and energy-efficiently to further explore non-thermal methods. DBDE uses plasma generated by dielectric barriers to produce reactive species that facilitate the extraction of pectin. This non-thermal method operates at atmospheric pressure and does not require solvents, making it an eco-friendly option [180,181]. DBDE preserves the quality of heat-sensitive compounds and offers energy efficiency compared to thermal methods. The enhanced cell wall disruption leads to higher pectin yields and reduced processing times. Figure 10 depicts the DBDE process.

Despite its potential, DBDE faces challenges regarding equipment complexity and cost. The precise control of plasma parameters, such as the types and concentrations of reactive species, is critical to avoid pectin degradation. Additionally, scaling up DBDE for industrial use remains challenging due to the need for specialized equipment and process optimization [179]. The DBDE method is usually used to modify the structure of the extracted pectin or pre-treat its extraction. Recently, the DBDE was successfully applied to extract pectin from watermelon. Forouhar et al. studied the effects of high-voltage dielectric barrier discharge on the extraction and properties of pectins from watermelon rinds [182]. In this study, the extracted yield of pectin was reported to be 18.5% and pectin was rich in galacturonic acid content (79-80%).

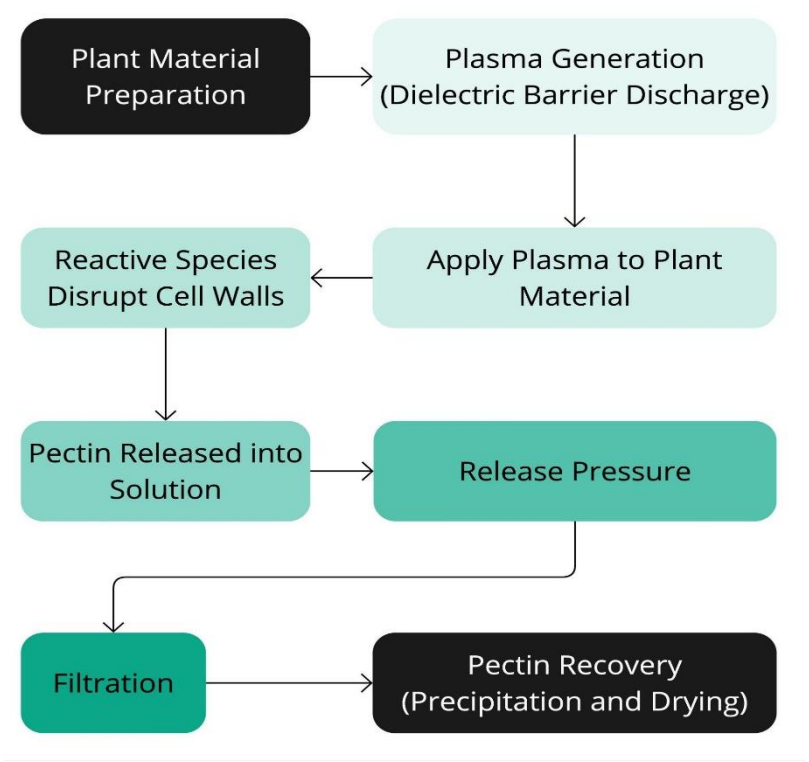


Figure 10. Schematic representation of the Dielectric Barrier Discharge Extraction (DBDE) process.

The brief data provided in Table 11 clearly indicates that the extraction yield and the galacturonic acid (GalA) content vary greatly depending on the pectin source and the technique used to extract it. Adopting greener pectin extraction techniques offers promising avenues for sustainable and efficient production. Each technique provides unique advantages in terms of yield, energy efficiency, and environmental impact. However, equipment costs, process optimization, and scalability challenges must be addressed to facilitate their broader industrial application. Continued research and development are essential to overcome these barriers and enhance the feasibility of these eco-friendly methods. Tables 12 and 13 summarize the advantages, disadvantages and applications of the above techniques.

Table 11. Non-conventional techniques are used for the extraction of pectin from different food sources.

Technique	Pectin source	Yield (%)	GalA (%)	Ref.
OHAE	Pomegranate	8.1	83	[166]
UAME	Potato	23	42	[170]
	Tomato	34	69	[171]
	Watermelon	19	68	[172]
UAOHE	Grape	27		[176]
HT	Sugar beet	13.6		[177]
	pulp			
	lime peels	23.8	70.6	[74]
DBDE	Watermelon	18.5	80	[182]
	rinds			

Table 12. Comparison between different non-conventional extraction techniques in terms of advantages and disadvantages.

Techniques	Advantages	Disadvantages
UAE	<ul style="list-style-type: none">Processing times are reduced since UAE extraction methods are more efficient than traditional methods.The yields of extracting bioactive components can be increased by UAE's improved solvent penetration.UAE is more cost-effective and advantageous for the environment as it uses less solvent.The UAE approach preserves the purity of delicate chemicals by minimizing heat exposure.The UAE's efficacy could be preserved when scaled up for industrial use.	<ul style="list-style-type: none">Initially, purchasing ultrasonic equipment might be expensive, and adjusting parameters like frequency, power, and duration to achieve the required results could be challenging.Sensitive materials may degrade when exposed to high-intensity ultrasound.Despite its ability to speed up processing, the UAE may use significant energy when operating.It might not be compatible with all materials or substances. May result in the degradation of some phenolic acids.
PUAE	<ul style="list-style-type: none">When compared to traditional extraction techniques, PUAE usually delivers larger pectin yields because of its superior solubilization and release from cell walls.The method greatly cuts down on extraction time, increasing process efficiency.Because pulsed ultrasound is mild, pectin's quality and functional qualities are preserved, allowing it to continue gelling and thickening.It may be more energy-efficient because the pulsed technique permits regulated energy delivery instead of continuous exposure.	<ul style="list-style-type: none">The hefty initial cost of ultrasonic equipment may deter smaller labs and organizations.To achieve ideal results with PUAE, rigorous parameter adjusting (such as solvent type, frequency, and pulse duration) is required, which can take time.Not every material or contaminant may be eliminated with PUAE. Some people may respond negatively to ultrasonography.
PEF	<ul style="list-style-type: none">PEF may greatly enhance the yield of extracted chemicals and is quicker than traditional procedures, resulting in more efficient processing.PEF can help to protect the integrity and purity of delicate substances since it functions at lower temperatures.The dry solid content increased over the lengthier "OFF" time as compared to the control studies.	<ul style="list-style-type: none">Investing in specialized equipment might be costly for small businesses.The usefulness of PEF to some plant sources may be limited since not all substances respond well to it.Excessive exposure to electric fields may damage sensitive components, reducing the quality of the extract.
MEF	<ul style="list-style-type: none">The moderate electric field strength may use less energy than higher-intensity techniques.MEF can be used on fragile materials and chemicals since it is less abrasive than PEF.	<ul style="list-style-type: none">When it comes to extracting chemicals, especially those that are difficult to remove, MEF may not be as effective as PEF.MEF still needs certain equipment, which not all facilities may have, even if it is less complex than PEF.The use of MEF may be restricted to particular plant matrices or substances since not all materials may react properly with it.
MAE	<ul style="list-style-type: none">MAE increases the release and solubility of bioactive compounds, resulting in increased yields.It maintains the integrity of fragile materials by shielding them from light and heat.Removes airborne contaminants.It needs very little training.	<ul style="list-style-type: none">Factors such as solvent type, time, and microwave power must be carefully controlled to achieve the optimum results.Certain compounds may degrade under microwave conditions, and not all materials may respond well to microwave extraction.

	<ul style="list-style-type: none">• Prevents the loss of volatile chemicals.• Minimal costs for investments.	<ul style="list-style-type: none">• Trained staff are required to correctly operate and utilize the equipment for the most demanding operations.
SWE	<ul style="list-style-type: none">• It is an environmentally friendly and sustainable extraction technique because SWE employs water as its main solvent instead of hazardous organic solvents.• The technique's improved solubility and diffusion often lead to better extraction yields than traditional approaches.• SWE often extracts faster, increasing processing efficiency overall.• Certain substances can be extracted selectively by adjusting the temperature and pressure according to their solubility properties.• Because it reduces solvent waste, the method is economical and ecologically safe.	<ul style="list-style-type: none">• Specialized high-pressure equipment might be costly initially.• The temperature and pressure parameters must be carefully adjusted to get the best extraction results.• This approach requires careful monitoring and is inappropriate for extracting thermo-labile chemicals since sensitive molecules may break down at higher temperatures.• Not all compounds can be extracted using SWE; some may need a different solvent solution.
OHAE	<ul style="list-style-type: none">• Uniform heating minimizes thermal degradation.• Lower maintenance costs.• Shorter overall processing time.• Improved extraction yields.• Reduced solvent requirement.• Shorter extraction time.	<ul style="list-style-type: none">• The initial investment is high.• Need for carefully controlled voltage and current.• Scalability is complex.
UAME	<ul style="list-style-type: none">• Higher extraction efficiency and improved yield.• Cost-effective and environmentally friendly.• Low energy consumption.• Shorter extraction times.	<ul style="list-style-type: none">• Initial investment in specialized equipment is high.• There is a need for carefully controlled heating to avoid degradation.• Potential limitations in scaleup.
UAOHE	<ul style="list-style-type: none">• Less solvent consumption.• Improved yield.	<ul style="list-style-type: none">• High equipment cost.• Complexity in the integrated system.• Need for precise control of temperature and ultrasound intensity.
HT	<ul style="list-style-type: none">• Environmentally friendly.• Safer and more sustainable.• Simple and overall cost-effective.	<ul style="list-style-type: none">• Energy-intensive.• Low extraction efficiency.• Risk of thermal degradation of pectin.• Sensitive to variations in temperature and pressure.
HPPE	<ul style="list-style-type: none">• Increased extraction yield.• Minimal solvent use.• Better quality and purity of pectin.• Solvent-free and eco-friendly.	<ul style="list-style-type: none">• High operation and maintenance costs.• Sensitive to variations in pressure.• Scaling up is challenging.• High equipment cost.
DBDE	<ul style="list-style-type: none">• Suitable for keeping the integrity of pectin.• Improved yield and purity.	<ul style="list-style-type: none">• Need for precise control of plasma parameters.• Industrial scaling up is challenging.

Table 13. Applications of various non-conventional extraction techniques.

Industry Type	Applications
Food Industry	<ul style="list-style-type: none">• Extraction of flavours and aromas.• Used widely in the food industry to extract biological components, tastes, and juices from fruits and vegetables.• Eliminating fruits and vegetables' flavours, hues, and antioxidants.

Pharmaceuticals	<ul style="list-style-type: none">• Extracting the nutritious ingredients, flavours, and aromas from fruits, vegetables, and grains.• Isolation of active ingredients.• Extracting bioactive compounds with potential health benefits.• Vitamins, pigments, and other bioactive compounds in a range of industries, including food, medicine, and cosmetics.• To create new medications, the active components in medicinal plants are separated.• Extracting biological components, flavours, and antioxidants from herbs, fruits, and vegetables.• Separating active components in medicinal plants to create new medications.• Isolation of the bioactive ingredients of medicinal plants to make new medications.
Cosmetics and Personal Care	<ul style="list-style-type: none">• Extraction of bioactive ingredients.• Extracting bioactive compounds with potential health benefits.• Extraction of the bioactive ingredients found in skincare and cosmetic products.• Extraction of natural ingredients for skincare and cosmetics.• Extraction of natural ingredients for use in skincare and cosmetics products.
Environmental Applications	<ul style="list-style-type: none">• Pollutant Extraction.• Elimination of pollutants from soil and water samples.
Nutraceuticals	<ul style="list-style-type: none">• Functional Ingredients.• Extraction of oils.
Biofuels	<ul style="list-style-type: none">• Extracting oils and fats from biomass to create biodiesel.• Producing biofuel by separating the oils and sugars in biomass.

5. Future Perspectives and Conclusions

Pectin contains a distinct structure with versatile polysaccharides. It is in high demand because of its gelling, stabilizing, and thickening properties in the food, pharmaceutical, and cosmetic industries. However, extraction methods can significantly impact the backbone structure of pectin. Therefore, understanding the degradation mechanism is crucial when modifying polysaccharides to achieve desired functional properties and predict their behaviour. Conventional pectin extraction, primarily relying on acid hydrolysis and extensive thermal treatment, poses significant environmental and economic challenges due to high energy consumption and chemical waste generation. Moreover, the scarcity of commercial sources and limited knowledge of pectin occurrence in various plant species necessitates the exploration of alternative sources and modification of existing extraction methods. Accordingly, researchers have gradually shifted to sustainable extraction techniques that could improve efficacy while lowering negative ecological impacts. The

thirteen discussed technologies could be an alternative to conventional methods in order to decrease harmful solvents, excessive energy consumption and uphold the extracted pectin's structural reliability and quality. Each one of the techniques above presents distinct advantages and limitations that must be carefully evaluated, with selection based on specific applications. The pectin exhibits a large water and oil-holding capacity with potential emulsifying properties, which makes it viable as a textural ingredient and emulsifier in different food products and pharmaceutical enhancements. It may also potentially be used as a replacement for fat and sugar in low-calorie foods. Therefore, further research into advanced extraction and analytical methods is essential for expanding the production and applications of pectin. This research will likely uncover new potential applications for this versatile natural compound and ultimately contribute to a circular economy.

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