

Review



Methods and Potential in Valorization of Banana Peels Waste by Various Extraction Processes: In Review

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Abstract: Over 114 million metric tons of bananas are produced each year. The peel, which accounts for roughly one-third of the fruit's weight, is commonly discarded as waste in the food industry. For centuries, the peel has been prized for its potential to heal a host of ailments. This by-product contains a large concentration of compounds with potent antioxidants linked to several health benefits. Consequently, the extracted valuable components, such as pectin, from this by-product could be applied to the pharmaceutical and food industries. More than 13% of pectin recovery is extracted by current extraction methods, such as ultrasound-assisted extraction. Subcritical water extraction also successfully extracts the pectin with high quality of extract. This review focuses on banana production and the role of pectin. Significant factors affecting its presence within the banana peel, the extraction methods, and current extraction applications are also presented and discussed, highlighting future research into its potential uses.

Keywords: banana peels; waste; pectin; extraction



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1. Introduction

Banana is a prominent tropical fruit with a high nutritional value [1,2]. It is freshly consumed or processed into different products, such as a snack, pesticide, or food colorant [3–6]. The popularity of bananas as functional foods has significantly increased due to their high carbohydrate content and low digestibility [7]. Globally, over 114 million tons of fresh fruit is produced, as shown in Figures 1 and 2 [8]. According to Vu et al. [1], the peel weight accounts for 35% of the total weight of the fruit. Thus, approximately 39.9 million tons of banana peel are produced each year.

Recent initiatives have been taken to substitute plant components with agro-industrial waste as a further step towards the development of greener and more sustainable operations. Research on banana waste, for instance, examined the acceptability of each waste portion, including the seeds and peels [9]. The banana peels as waste have a high antioxidant capacity and antimicrobial properties [10,11]. Burns, diarrhea, ulcers, and inflammation are among several illnesses that the peel has historically been used to treat [12–15]. As a result, it is a raw material with many potentials in the nutraceutical and medicinal industries. However, due to inadequate valorization, the wastes are usually discarded into landfill [16,17]. Furthermore, banana peels are commonly used for livestock feed, especially for cow and buffalo. There are limited studies for the valorization of banana peels in a health and wellness application.



Figure 1. Banana plantation area by years [8].





Meanwhile, high pectin content (9–22%) from bananas contributes to their uniqueness by providing gelatinization, thickening, and stabilization properties [18]. Pectin is primarily found in food, cosmetic, textile, and other industrial fields [19]. The practicality of the pectin compound may be warned and prospected as a new possibility and alternative source of commercial pectin (low and high methoxyl pectin), which is currently derived primarily from citrus peel or apple pomace [20,21]. Pectin extraction was formerly done using conventional extraction methods and a modern extraction method. The conventional extraction methods include maceration and Soxhlet associated with an organic solvent. Nevertheless, a 'green extraction' or modern technology of plant material has become a challenge to particular industrial experts. Demanding green extraction could result in a higher yield and quality of extract at a lower production cost. The formation of toxic residue and the use of an organic solvent could also be reduced [22–25]. Hence, significant advances in green extraction technologies, such as microwaveassisted extraction (MAE) and subcritical water extraction (SWE), also known as superheated water extraction or pressurized hot water extraction (PHWE), were introduced [26–34]. An extraction method has been considered the most vital process to ensure an excellent function of the end product. Although the conventional method resulted in a higher yield, the long-term effects of employing SWE should be considered, especially on our environment and health issues.

The originality of this study is the information on the present and historical valorization of banana peels to generate a high-quality and -quantity extract. The present global banana production as a supply of banana peels and pectin is also examined. The comparison of the sources of banana peels is also highlighted, which is why banana peels are chosen as the pectin source. The latest study on the quality analysis of banana peel extract, including its antioxidant and antibacterial properties, is also highlighted. Therefore, the purpose of this study is to provide an overview of the pectin components in banana peel, followed by a discussion of present extraction techniques, and a focus for future banana peel research.

2. Banana Production

Banana is a *Musaceae* family that includes several varieties in the genus *Musa*, as shown in Table 1 [35,36]. The banana plant is a climacteric fruit and one of the world's most extensive fruit plantations [13]. The plantation, which spans over 2.3 million hectares, is the largest in the world [8,37]. The banana contributes 16.8% of the global fruit supply, followed by the apple and orange, which accounts for 11.4% [8]. The demand has risen from 113 million tons to 117 million tons in the last five years due to its processed products, such as chips, ice cream, jelly, and cake [7,8,11].

Kingdom		Plantae
Subkingdom	=	Tracheobionta
Superdivision	=	Spermatophyta
Division	=	Magnoliophyta
Class	=	Liliopsida
Subclass	=	Zingiberida
Order	=	Zingiberales
Family	=	Musaceae
Genus	=	Musa.L

Table 1. Classification of bananas [36].

Furthermore, population growth, as well as an increase in planted area and productivity, have all contributed to the rise in banana demand. Asian countries provided the most banana production in 2018, with 62.48 million tons, followed by the United States of America and Africa (Figures 1 and 2) [8]. India ranks fourth globally, with a cultivated area of 722 thousand ha and an annual output of 26.51 million tons. Following China are the Philippines, Ecuador, Brazil, and five other countries [8]. Therefore, more research of bananas is necessary; more appropriately, extensive research is necessary.

3. Pectin

This section may be divided by subheadings. It should provide a concise and precise description. In 1790, a pioneer discovered pectin's complex polysaccharides in fruit juices. Before the introduction of new terms such as "pectin", the term "pectos" was derived from the Greek word for coagulated or solidified substances [38]. The majority of pectin research has been focused over the last decade. Pectin sources are typically found in the intermediate layer of the lamella and the primary cell walls of various plants. The American Chemical Society coined the term pectic to describe a complex substance composed of colloidal carbohydrate derivatives found in plants or prepared from them [39].

The methylated ester of 1,4-based galacturonic acid (GA) was compared to rhamnose residues (main chain) and arabinose, galactose, and xylose (side chains) in pectin [40]. 1,2-linked rhamnose contains side branches of either 1,4-linked D-galactose or 1,5-linked L-arabinose. The branched galactose-rich hairy regions of pectin chains promote intertwined complexes, whereas the rhamnose-rich improve cell-cell interactions. Any C-6 carboxyl units in the GA backbone could be esterified with methoxyl groups or exist as uronic acid salts [41]. Depending on the plant source, the GA residues might have methylated to different degrees.

Naturally, the polysaccharides of pectic substance are higher in apples, citrus, blackberries, cranberries, gooseberries, grapes, and plums. Emaga, Andrianaivo, Wathelet, Tchango and Paquot [18] reported that the pectin contents in the banana peel are also higher in the maturity stages compared to other fruits. Moreover, a plentiful source of pectin is also found in various vegetables and fruits, revealing strength and flexibility in the cell wall and the entertaining of biological functions

Basically, the substitution of pectin is made up from the non-sugar elements, namely methanol, acetic acid, phenolic acids and occasionally amide groups. Besides that, they are comprised of reduced carbohydrates, polyhydric alcohols, polyacids, polyesters, some carboxyl groups that are polar, as well as non-polar methyl groups. Thus, some examples of the general composition of pectin are described in Table 2 from different plant sources, such as sugar beet pulp, apple pomace, citrus peels, and pea hulls.

Plant Seeds	Extraction Method	Extraction Conditions	Outcomes	Ref.
Apple pomace	Soxhlet/condensation reflux	Solute/solvent = 1:50 Water-acidic solvent pH = 2.5 Particle size = 250 to 150 µm	Apple peel pectin showed a degree of esterification 68.84%.	[42]
Grapefruit	Ultrasound-microwave	Solute/solvent = 1 g/30 mL intermittent sonication Time of sonication = 30 min; the time of heating = 10 min Power = 0.45 kW	Grapefruit pectin showed a degree of esterification 82.61%.	[43]
	Microwave	Power = 0.9 kW; Time = 6 min; Solute/solvent = 1 g/30 mL	Grapefruit pectin showed a degree of esterification 79.35%.	-
Grapefruit peel	Ultrasound-assisted heating	Power intensity = 12.56 W/cm^2 Temperature = 66.71 °C Sonication time = 27.95 min.	Grapefruit pectin showed a degree of esterification 27.34%	[44]
Lime peel	Microwave	Solvent = hydrochloric Peel-to-extractant ratios = 1:20 and 1:40	Methoxyl content and galacturonic acid content of lime peel pectin was in the range 8.74–10.51%	[45]
Pomelo peel	Subcritical water extraction	Temperature = 90–120 °C Pressure = 30–65 bar	Pectin yield was 19.63%	[46]
Potato peel	Microwave	Optimal conditions of temperature 93 °C, pH 2.0, and time 50 min.	Maximum pectin yield reached 22.86 \pm 1.29%	[47]

Table 2. Recovery of pectin from various sources.

Plant Seeds	Extraction Method	Extraction Conditions	Outcomes	Ref.
Apple peel	Ultrasound-assisted	Liquid-solid ratio = 10–25 mL/g Time = 10–30 min Temperature = 50–80 °C pH of solution = 1–3	Maximum yield pectin = 8.93%	[48]
<i>Ficus carica</i> l. Skin	Ultrasound-assisted	Frequency = 20 khz Maximum power = 400 W	Maximum yield pectin = 13.9%	[49]
Ponkan peel	Microwave	pH = 1.6 Extraction time = 100 min Liquid: solid ratio = 36 mL/g	Maximum yield pectin = 25.6%	[50]
Melon peel	Soxhlet	Temperature = $35-95 \degree C$ Time = $40-200 \mod P$ pH = $1-3$ Liquid: solid ratio = $10-50 v/w$	The yield and DE-ranged from 2.87 to 28.98% and 1.33–29.33%, respectively	[51]
<i>'apple pomace'</i> apple pomace	Ultrasound	Amplitude = 100% pH = 1.8 Liquid: solid ratio = 1:10 g/mL Time = 30 min	Yield of 9.183% pectin, with a 98.127 g/100 g galacturonic acid content and 83.202% degree of esterification	[52]
Jackfruit waste	Waterbath	Temperature = 50–90 °C Time = 30 to 60 min	Maximum pectin yield was 39.05 ± 0.59 g/g	[53]
Sweet lemon peel	Microwave	Power = 700 W Irradiation time = 3 min pH = 1.5	Highest pectin yield was 25.31%	[54]
Cocoa peel	Microwave	Citric acid solution (pH of 1.5) Power = 180–600 W Time = 10–30 min	Highest pectin yield was 42.3%	[55]

Table 2. Cont.

4. Antioxidant Activities

An outstanding example of a functional advantage that plant extracts may provide is antioxidant activity. Plants are known to contain a range of natural antioxidants that maintain and preserve their physical and metabolic integrity, as well as their heredity via the seeds they produce. Many of these plant extracts and chemicals are showing promise in reducing the symptoms of ageing on the skin by minimizing the metabolic repercussions of oxidation.

Vitamin C, vitamin E, anthocyanin, catechin, and rosmarinic acid (RA) are widely utilized in foods and cosmetics because of their strong antioxidant action, which helps to keep products stable [56–62]. However, the banana peel extract also provides significant antioxidant properties [1]. Reduced oxidation provides an obvious advantage for both the product and the skin, and antioxidants have a favorable consumer impression, making them especially appealing as cosmetic additives. The problem is that a single antioxidant is often marketed as a cure-all. Plant antioxidants vary not just in terms of redox potential and solubility, but also in terms of how they work. Some ROS, such as superoxide, hydroxyl radicals, and singlet oxygen, are quenched [63]. Others decrease oxidative enzyme activity or expression, increase antioxidative compound or enzyme activity or expression, such as catalase, or chelate oxidizing metal ions, or operate via various mechanisms, both known and undiscovered. Given the wide range of chemical structures and biological processes identified for antioxidants found in plants, it is not unexpected that not all antioxidants provide the same level of skin protection. Compounds produced by skin cells or peels, such as glutathione and ubiquinol, as well as those absorbed from plant sources in the diet, such as vitamin E, vitamin C, and retinoids, are among the tiny molecular weight antioxidants

naturally present in skin or peel. They work together in certain instances, but they also work as part of separately controlled systems to handle challenges to the cell's or tissue's redox state.

5. Antimicrobial Activities

The process of destroying or suppressing disease-causing microorganisms is referred to as antimicrobial activity [64]. This is accomplished using a variety of antimicrobial agents. Antimicrobials have antibacterial, antifungal, and antiviral properties. They all have different modes of action by which they act to suppress the infection. Methods for determining antimicrobial activity in food are as ancient as disinfectants and medicines. The antibacterial activity of crushed garlic vapours against Mycobacterium species, Escherichia coli, Serratia marcescens, and *B. subtilus*, for example, was investigated as early as 1936 [65]. On the lid of a Petri dish, crushed banana peels extract was put, and the bottom of the dish with a nutritional medium was inverted over the top. For various durations of exposure, the garlic vapours were allowed to enter agar with the test microorganism and incubated to evaluate inhibition. The majority of techniques for assessing the activity of food antimicrobials have been implemented, either completely or partially. An in vitro or screening test is used to get preliminary information on the antibacterial activity of a chemical that has not been applied to a product under normal usage circumstances. The endpoint tests provide qualitative data on effective concentration levels. A microorganism is challenged for an arbitrary length of time in this technique, and the findings represent the inhibitory power of a chemical during the time period chosen. The descriptive screening techniques, which include periodic sampling to assess changes in viable cell counts over time, provide quantitative information about the growth dynamics. The antibiotic is applied to real food in applied testing, and the antimicrobial's effectiveness is assessed, especially for banana peel extract [10].

6. Sources and Compositions

Apple peels (8.93%), pomelo peels (23.81%), lemon peels (25.31%), and lime peels (10.31%) naturally contain more pectic matter called polysaccharides [45,46,48,54]. According to Lee, Yeom, Ha and Bae [19], the pectin content of mature banana peels is higher than that of other fruits. Pectin is naturally abundant in a wide range of vegetables and fruits, as the cell wall is solid and flexible, and biological functions are presented.

Non-sugar components, such as amide groups, phenolic acids, methanol, and acetic acid, have been used to replace pectin [19]. Besides that, reduced sugars, polyhydric alcohols, polyacids, polyesters, polar and non-polar carboxyl groups, and other carboxyl groups were included. Table 2 shows several examples of pectin origins and extraction methods from various plant sources, including sugar beet pulp, apple pomace, citrus peels, and pea hulls.

Compared to research of Khamsucharit et al. [66], pectin from five different types of banana peels using a citric acid solution was extracted. The pectin of banana peel (15.89% to 24.08%) is higher than grapefruit peel, apple peel and potato peel. Therefore, this substance can be substituted to another pectin source. Although, grapefruit gives the highest pectin recovery (82.61%), the grapefruit cannot be compared with banana peels. This is due to the grapefruit skin not being considered agricultural waste and characterized as main product of agriculture.

7. Method of Extraction

7.1. Soxhlet Extraction

Solvent extraction, also known as "solid-liquid extraction," but more accurately referred to as "leaching" or "lixiviation" to best represent its physical-chemical foundation, is one of the oldest concrete sample preparation techniques. Its goal is to separate the compounds of interest from insoluble high-molecular-weight fractions and other compounds that could interfere with the analytical process in the future. Maceration has historically been the most common type of leaching, relying on the correct solvent approach and heat, either with or without agitation, to improve the solubility of substances and mass transfer rate. Despite its widespread use, particularly for natural product isolation, maceration is characterized by inefficient and time-consuming extraction protocols.

In 1879, Von Soxhlet invented a new extraction method (the Soxhlet extractor), which has long been the most widely used leaching technique [67,68]. For more than a century, Soxhlet extraction has been a popular technique, and methods based on it are still used to analyze current leaching methods. The advantages and disadvantages of Soxhlet extraction have been considered to reduce or eliminate the latter while maintaining or improving the former. The majority of the documented improvements over the last few decades have been aimed at bringing Soxhlet closer to current solid sample preparation techniques, such as using auxiliary energies to reduce leaching times and automating the extraction assembly [69]. In some of the previous studies, the enzyme assisted extraction combined with the Soxhlet extraction to extract the phenolic and flavonoid compounds from agrowaste material was utilized [70].

According to Singh and Prakash [71], the free radical scavenging activity of banana crude extracts was significantly higher in acetone extract than in any of the other extracts tested. A comparison of Soxhlet peel extract and soaking extracts revealed that the former performed better than the latter. All of the extracts had lower antioxidant activity when compared to the control group. Under optimal conditions, Hamid, et al. [72] reported pectin extraction recovery from *Musa aluminata balbisiana* (MBS), *Musa acuminata Cavendish* subgroup (MCS), and *Musa acuminata Colla* (MES) was 39.53%, 62.42%, and 39.53%, respectively, and oil extracted, was 3.6 mL, 5.3 mL, and 3 mL. Morphological examination of banana peel waste revealed the formation of a mixture of follicular gel (pectin), which leads to the presence of oil.

According to Nasir et al. [73], the highest scavenging operation of banana peels was reported at 1000 mg/mL, which was up to $94.13 \pm 0.11\%$, while the lowest was 0.1 mg/mL. During phytochemical analysis, flavonoids, alkaloids, tannins, and glycosides were discovered. On the other hand, GC/MS analysis detected antioxidant compounds, such as pentafluoro propionic acid, 2-pentenoic acid, 4-hexadecyl ester, 3-ethyl-methyl ester, 2-tetradecene, and 1-hexadecene. These compounds are essential in neutralizing free radicals and lowering their ability to kill cells.

Okolie et al. [74] discovered that ethanolic extracts of the same banana varieties have higher phenolic and flavonoid content (336.83 mgGAE/100 g and 242 mgRutin/100 g) than methanolic extracts (299.42 mgGAE/100 g and 240.77 mgRutin/100 g). Methanolic extracts have higher 2,2-diphenyl-1-picrylhydrazyl (DPPH) antioxidant activity (30.82 > 25.44%) than ethanolic. The higher activity suggests that antioxidative substances other than phenolics and flavonoids were involved in DPPH radical prevention. Wu et al. [75] found that the following conditions were optimal for banana peel extraction: an ethanol concentration of 75.44%, solid to liquid ratio of 1:35, time of 7.94 h, and temperature of 62.85 °C. The estimated tannin extraction yield under optimal conditions was 58.55%, while the actual was 57.42%, with a relative error of 1.13%.

7.2. Microwave-Assisted Extraction (MAE)

Microwaves are non-ionising electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz between X-rays and infrared rays in the electromagnetic spectrum [76,77]. Microwaves are used primarily for two purposes in contemporary science: connectivity and energy vectoring. The latter application is concerned with the physical interaction of waves with objects, and some of the electromagnetic energy absorbed might be converted into heat energy. Microwaves consist of two perpendicular oscillating fields, viz. an electric and a magnetic field. The electric field is responsible for heating, while the magnetic is responsible for cooling [78].

In contrast to traditional heating methods that rely on conduction and convection, a substantial amount of thermal energy is lost to the environment. However, since MAE is

used in a closed structure, the heating is concentrated and selective, with nearly no heat lost to the environment. Compared to Soxhlet, this heating mechanism could significantly reduce extraction time, generally less than 30 min [79]. Microwave heating involves direct contact with polar materials/solvents and is regulated by two conditions: ionic conduction and dipole rotation that usually occurs in parallel [80,81]. Ionic conduction is the electrophoretic diffusion of ions under the influence of a shifting electric field. Because of the solution's resistance to ion migration, pressure builds up, causing the solution to heat up. Dipole rotation realigns the molecule's dipoles with the changing electric field. Heating is influenced even at 2450 MHz, as the electric portion of the wave varies at 4.9 ± 0.104 times per second [82,83].

As this electrical component of the wave varies rapidly (frequency larger than 2450 MHz), the solvent molecules strain, realign themselves and begin vibrating, producing heat through frictional energy. Thus, the molecules may not have enough time to coordinate with the external environment, resulting in little heating. The electrical portion changes even more slowly when the frequency is less than 2450 MHz, allowing the molecules ample time to align themselves with the electric field to avoid heating. Based on the mechanisms mentioned above, a microwave heats dielectric materials or solvents with permanent dipoles. The dissipation factor $(\tan \delta)$, which defines how effectively different solvents heat up in the microwave, is a measure of the solvent's ability to absorb microwave energy and pass it on to the surrounding molecules as heat [84,85].

Pectin from banana peels was extracted using continuous and intermittent MAE [86]. Swamy and Muthukumarappan [86] used microwave power of 300–900 W, for a period of 100–300 s, and pH of 1–3 in the continuous mode, and microwave power of 300–900 W, pulse ratio of 0.5–1, and pH of 1–3 in the sporadic phase. The continuous method produced the highest pectin content (2.18%) from banana peels with a microwave power of 900 W, a period of 100 s, and a pH of 3. With a microwave power of 900 W, a pulse ratio of 0.5, and a pH of 3, the intermittent method produced the highest pectin content (2.5%). It is believed that the increase in microwave power and pH correlated to the rise in pectin content. The plant tissue softens and the phenolic compound and protein/carbohydrate interface are reduced when microwave power levels are raised. As a result, the solubility of phenolic compounds improves. As a consequence, the diffusion rate is increased, resulting in a significant increase in the extraction rate.

Rivadeneira et al. [87] discovered that by using response surface methodology (RSM), the parameters for microwave-assisted pectin extraction from "Saba" banana peel waste were screened and optimized. Pectin purification and characterization were carried out at pH 3 of hydrochloric acid (HCl), 195 °C, and 8% solid–liquid ratio. These parameters were the best extraction conditions, with predicted and actual yields of 12.8% and 14.2%, respectively. Following purification, pectin's purity increased by 300%. The pectin was discovered to be low-methoxy with an average particle size of 300 nm.

The best pectin extraction conditions for the Box Behnken design were at 75 °C (extraction temperature), 23 min extraction period, and a solid–liquid ratio of 1:33.3 g/mL (Lin, Xia and Liu, [51]). Pectin obtained with or without optimized conditions had a degree of esterification (DE) of 71.921 \pm 0.38% and 76. \pm 0.12%, respectively. Each pectin was discovered to have a high methoxyl content. Pectin with the highest DE content gels rapidly. Based on the findings, pectin yield and gelling time increased after optimization.

MAE was applied in a different study to recover pectin from banana peel waste (Phaiphan [88]). They used a central composite design (CCD) to analyze the effects of processing parameter variables (microwave irradiation, extraction period, and pH). Pectin yield, DE, and galacturonic acid content (GA) extracted from dried banana fruit peel with 0.05 M hydrochloric acid were studied and optimized. Microwave irradiation of 300–600 watts, an extraction period of 5–15 min, and a pH of 1–3 were used as extraction parameters in this analysis. Based on the findings, all of the process parameters had a significant impact on the responses. The optimal conditions for pectin yield (13.47%), DE (92.45%), and GA (87.99%) were at a microwave irradiation of 580 watts, extraction period

of 15.86 min, and pH of 1.71. This study revealed that the experimental and expected values were in close alignment under ideal conditions.

Meanwhile, Khamsucharit, Laohaphatanalert, Gavinlertvatana, Sriroth and Sangseethong [66] extracted pectin from five different types of banana peels using a citric acid solution. They assessed the capacity of banana peels as an alternative source of industrial pectin. Furthermore, the chemical characteristics of banana peel pectin were investigated and compared to citrus peel and apple pomace, which were collected under the same extraction conditions. Based on the analysis, pectin yield from banana peels ranged from 15.89% to 24.08%. Since solid methoxyl pectin was also derived from banana peels, the DE varied from 63.15% to 72.03%, equivalent to those present in citrus peel (62.83%) and apple pomace (72.03%). The study also reported anhydrouronic acid (AUA) concentrations in banana peel pectin that ranged between 34.56% and 66.67%.

7.3. Ultrasound-Assisted Extraction (UAE)

Applying ultrasound has become a critical concern to achieve long-term "green" chemistry and extraction technique. Ultrasound has long been known to accelerate chemical and food systems. Complete extractions could be completed in minutes with high reproducibility, which reduces solvent consumption, simplifies manipulation and work-up, improves finished product purity, eliminates wastewater post-treatment, and uses a fraction of the fossil oil in traditional extraction processes. Soxhlet extraction, maceration, and evaporation are examples of these.

Natural product using UAE has been thoroughly studied [89–92]. However, processes that lead to extraction changed due to the ultrasound application. This situation is barely discussed in these reviews and the literature. Only a few reference papers [93,94] described the effects of ultrasound propagation in a solid/liquid media.

As a consequence of the cavitation phenomenon, the media is exposed to substantial shear powers. Micro-jetting is caused by the implosion of cavitation bubbles on a product's surface, which resulted in surface peeling, corrosion, and particle fragmentation. The implosion causes macro-turbulence and micro mixing. Surprisingly, the yield in some natural products has increased when employing the UAE due to cavitation effects during ultrasonic irradiation.

Meanwhile, to further explain and demonstrate the ultrasound impact on a vegetal matrix during UAE, several studies were analyzed. Toma et al. [95] reported that irradiation caused matrix fragmentation and that ultrasound caused matrix hydration to increase. They have found that sonicated samples had a higher extraction index than the non-sonicated. They also discovered that ultrasound extraction works by a variety of separate or combination processes, including capillarity, sonoporation, detexturation, separation, and degradation. The following segment focuses on the physical effects of ultrasound on a vegetal matrix, which can be attributed to an improvement in extraction yield. All of the experiments used high-powered ultrasound with frequencies of 20 to 25 kHz.

Acetone concentration significantly impacted the recovery yields of phenolic compounds, proanthocyanidins, flavonoids, and antioxidant properties, besides other extraction parameters (Vu, Scarlett and Vuong [1]). The optimum conditions were 30 °C ultrasonic temperature, 5 min ultrasonic duration, 150 W ultrasonic strength, 8:100 g/mL sample to solvent ratio, and 60% acetone concentration. A total of 1 g of banana (*M. cavendish*) peel could yield 23.49 mg of phenolic compounds, 39.46 mg of flavonoids, and 13.11 mg of proanthocyanidins in these conditions.

Maran et al. [96] extracted pectin from forest banana industrial waste using an ultrasound-assisted citric acid-mediated extraction process. The best extraction conditions were an ultrasound capacity of 323 w, a pH of 3.2, an extraction period of 27 min, and a solid–liquid ratio of 1:15 g/mL. The mean of the experimental pectin yield ($8.99 \pm 0.018\%$) was in good agreement with those expected (9.02%). Another research has generated similar results when extracting high tannin content and antioxidant activity from an unripe *Musa acuminata* peel (Cavendish). The overall tannin content of crude extract of

the unripe peel was 119.2 mg TAE per gram of the sample, and the optimum pectin yield processing parameter was 14.9%. The flavonoid content was also 29.0 mg/gram of sample, with a DPPH and scavenging activity of 80.8% and 84.7%, respectively [97].

Since the analysis of pectin recovery through UAE is minimal, filling this research gap is crucial. Grape pomace, tomato, apple peel, dragon fruit peel, grapefruit, pomegranate peel, and passion fruit are typical, and commonly, are sources of pectin recovery using the UAE [48,98–103]. Therefore, to achieve the optimum pectin yield from banana peels, it is necessary to select the appropriate extraction processes, process parameters, and solvent used, to name a few.

7.4. Subcritical Water Extraction (SWE)

Subcritical water is defined as hot water under sufficient pressure to maintain a liquid state at a temperature between 100 °C (the boiling point of water) and 374 °C (the critical point of water) under pressure between 1 and 22.1 MPa [104–108]. The dielectric constant, viscosity and surface tension decreased when the temperature rises. At high temperatures, an adequate pressure will keep the water warm since it has a dielectric constant of 80 at 25 °C. Water has the same properties as organic solvents under these conditions and could remove a wide range of medium and low polarity substances [109–113].

The advantage of SWE is that the dielectric constant could be varied over a wide range of temperatures and pressures [114]. SWE also could induce mass transfer via diffusion and convection [115]. During the desorption process, low activation energy is required. However, it would disrupt the adhesive (solute–matrix) and cohesive (solute–solute) relationships of the subcritical water's energy [116]. Meanwhile, increased pressure may aid extraction by forcing water into the matrix (pores), which would be difficult under normal pressure [117].

A shift in temperature and pressure significantly impacts the properties and polarity of water. As a result, non-polar, low-polar, medium-polar, and polar substances could be distinguished, followed by a decrease in viscosity and improved diffusivity, allowing greater matrix particle penetration. Water is constantly inflowed through the complex extraction phase of subcritical water, improving mass transfer performance and extraction yield. At elevated temperatures and pressures, a substance's surface may be dissolved. The solute–matrix interaction, which is caused by hydrogen bonding, van der Waals forces, active sites in the matrix, and dipole attraction of solute molecules, could be overcome by increasing the temperature.

The SWE process is divided into four stages. The first step is to desorb the solute at high temperatures and pressures at various active positions in the sample matrix. The second stage focuses on extract diffusion into the matrix. In the third step, the solutes could partition themselves from the sample matrix into the extraction fluid. The sample fluid is eluted and extracted from the extraction cell using a chromatograph [69,118]. Previous research has shown that the SWE process follows the thermodynamic paradigm [119]. Finally, two steps are necessary to separate a compound from a matrix in this model. (1) The compounds must be desorbed from the sample matrix's initial binding position, and (2) the compounds must be extracted from the sample using a method equivalent to front elution chromatography.

A peak hold test and an interfacial double wall ring were used to evaluate the gelation properties of banana peel pectin, as reported by Rasidek et al. [120]. The best extraction conditions for pectin yield were a temperature of 140 °C, a period of 5 min, and a particle size of 1.18 mm. The Fourier-transform infrared spectroscopy (FTIR) spectrum displayed a high concentration of free esterified carboxylic groups, indicating a low in methoxyl pectin. The most increased torque (168.97 N.m) and viscosity values (0.005 Pa.s) were obtained using a gelation interaction combined with pectin extract and 80 mM/L Ca²⁺ methods, respectively. These methods have led to a better gel by increasing the elastic (*G*') and viscous (*G*'') moduli to 0.170 and 0.018 Pa, respectively.

Banana peel pectin is extracted from its waste using hot compressed water (140–160 °C, 5 min, particle size 1.18 mm) [121]. Its moisture (7.44–8.47%), ash (3.45–4.98%), protein (1.08–1.92%), fat (0.04–3.42), starch (83–86%), total sugar (1.77–3.41%), energy (353–369 kcal/100g), and heat (1.42–1.62 kJ/kg °C) are in a close range as industrial pectin. Pectin is commonly isolated from cacao, apple pomace, citrus peel, pomelo peels, jackfruit peels, and passion fruit by SWE [21,46,122–124]. However, since research on pectin recovery via SWE is scarce, more research is needed to understand the processes and applications better.

8. Summary of Various Extraction Method to Valorize the Banana Peels

Table 3 provides a review of the advantages and disadvantages of different extraction techniques to valorize the banana peels. Comparing Soxhlet to subcritical water, the traditional Soxhlet process delivers a greater yield but of worse quality. Microwave-assisted extraction (MAE) is superior than Soxhlet extraction in terms of extraction time and the quality of the extract. This is because the MAE used a shorter extraction time at a lower temperature.

Ultrasound-assisted extraction (UAE) is also an improvement over the Soxhlet extraction process, offering less energy usage, a quicker extraction time, and a better quality banana peel extract. However, ultrasonic waves have been shown to degrade certain phenolic acids and generate extremely reactive hydroxyl radicals inside the gas, which are drawbacks of this approach.

In terms of a green and sustainable extraction process, subcritical water extraction (SWE) is a simple way to remove banana peels. This is because water is used to extract pectin from banana peels using green solvent. In addition, the extraction time is reduced compared to earlier techniques, such as Soxhlet, MAE, and UAE. Therefore, energy consumption may be minimized. The downside of SWE is that it is unsuitable for thermolabile chemicals. This is because of the high temperatures.

Extraction Methods	Advantages	Disadvantages
Soxhlet	 Using auxiliary energies to reduce leaching times. Less consumption of solvent. Offer high quantity of global yield. 	Commonly, this method uses the toxic solvent. High temperature condition based on the bubble point of each solvent and long extraction time. The quality of extract is low due to long extraction time with high temperature.
Microwave-Assisted Extraction (MAE)	 Shorter extraction time, increase in yield of extracted components, Less solvent consumption. improvement of the quality of extracts compared to Soxhlet. 	This method commonly uses the toxic solvent (methanol, ethanol and hexane) and high temperature condition based on the bubble point of each solvent.

Table 3. Summary of advantages and disadvantages in pectin extraction.

Extraction Methods	Advantages	Disadvantages
Ultrasound-Assisted Extraction (UAE)	 Low energy consumption. Less extraction times and active compound damage High extraction yields as compared with conventional extraction (Soxhlet) methods. Faster leaching compared with MAE. In acid digestions, the ultrasonic procedure is safer as it requires no high pressure or temperature [68]. In many cases, the whole procedure is simpler as it involves fewer operations and is thus less prone to contamination. 	Ultrasonic waves have been reported to result in the degradation of some phenolic acids and the creation of highly reactive hydroxyl radicals within the gas.
Subcritical Water Extraction (SWE)	 Relatively new technique for extracting less-polar compounds. Short extraction time in 30 min. Water at a higher temperature has a lower dielectric constant, which weakens the hydrogen bonds and makes subcritical water more similar to less-polar organic solvents such as methanol and ethanol. The solubility of less polar phenolics increased when the temperature of subcritical water was increased. SWE is an environmentally friendly and efficient extraction method that does not require the use of an organic solvent to extract phenolics and flavonoids. There has been an increasing interest in the use of ecofriendly technologies. SWE can provide high biological activities of extracts while precluding any toxicity solvents. 	High temperature condition; therefore, this method is not suitable for extraction of thermo-labile compounds.

Table 3. Cont.

9. Future Perspectives and Conclusions

Pectin is a polymer present in the cell walls of non-woody plant cells. It is commonly used in the food business as a hydrocolloid because it can absorb water and form gels at low concentrations. Additionally, it is fast expanding into other industries, with new uses being found on a regular basis [121]. These applications are connected with structural and functional features of extracted polysaccharides. Current pectin extraction techniques are well-established. To satisfy increased demand, however, the process must be enhanced in terms of efficacy, predictability, and consistency of product quality.

As noted in this study, Soxhlet, microwave, ultrasound, and subcritical water are among the successful and dependable innovative tactics being researched for incorporation into the pectin extraction procedure; although to various degrees of effectiveness. Although these procedures are quantitatively and qualitatively adequate for laboratory usage, a lack of knowledge hinders their typical industrial use. It cannot be used for scale-up, when the continuous technique is still favored.

Some of these advances are too costly for new and small manufacturers; however, this may not be the case for the most notable specialized chemical/ingredient producers. However, it is necessary to carefully optimize the process parameters of these most recent

approaches. Eventually, market participants will adopt one or more of these techniques to produce customized pectin, most likely using microwave heating for fast mass transfer. Several research institutions and labs have concentrated on discovering and using banana peels as pectin processing raw materials, in addition to the industrially manufactured banana peels described above. In addition, minimal study has been conducted on pectin recovery from banana peels utilizing UAE and SWE. Pectin was often extracted using conventional procedures, such as Soxhlet extraction and MAE. As a result, there is a technology gap in pectin extraction methods that are greener and more sustainable.

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Nomenclature

AUA	anhydrouronic acid
CCD	central composite design
DE	degree of esterification
DPPH	2,2-diphenyl-1-picrylhydrazyl
GA	galacturonic acid
GC	gas chromatography
HCL	hydrochloric acid
MAE	microwave-assisted extraction
MBS	Musa aluminata balbisiana
MCS	Musa acuminata Cavendish subgroup
MES	Musa acuminata Colla
RSM	response surface methodology
SWE	subcritical water extraction
PHWE	pressurized hot water extraction

References

- 1. Vu, H.T.; Scarlett, C.J.; Vuong, Q.V. Optimization of ultrasound-assisted extraction conditions for recovery of phenolic compounds and antioxidant capacity from banana (Musa cavendish) peel. *J. Food Process. Preserv.* **2017**, *41*, e13148. [CrossRef]
- Gómez, A.V.; Tadini, C.C.; Biswas, A.; Buttrum, M.; Kim, S.; Boddu, V.M.; Cheng, H. Microwave-assisted extraction of soluble sugars from banana puree with natural deep eutectic solvents (NADES). LWT 2019, 107, 79–88. [CrossRef]
- Farahmandfar, R.; Mohseni, M.; Asnaashari, M. Effects of quince seed, almond, and tragacanth gum coating on the banana slices properties during the process of hot air drying. *Food Sci. Nutr.* 2017, 5, 1057–1064. [CrossRef]
- Majaliwa, N.; Kibazohi, O.; Alminger, M. Optimization of process parameters for mechanical extraction of banana juice using response surface methodology. J. Food Sci. Technol. 2019, 56, 4068–4075. [CrossRef]
- 5. Yan, L.; Fernando, W.M.; Brennan, M.; Brennan, C.S.; Jayasena, V.; Coorey, R. Effect of extraction method and ripening stage on banana peel pigments. *Int. J. Food Sci. Technol.* **2016**, *51*, 1449–1456. [CrossRef]
- 6. Sartori, R.; Higino, M.; Bastos, L.; Mendes, M. Supercritical extraction of pesticides from banana: Experimental and modeling. *J. Supercrit. Fluids* **2017**, *128*, 149–158. [CrossRef]
- 7. Mohapatra, D.; Mishra, S.; Sutar, N. Banana and its by-product utilisation: An overview. J. Sci. Ind. Res. 2010, 69, 323–329.
- 8. FAO. Production of Banana. Available online: http://www.fao.org/faostat/en/#data/QC (accessed on 11 July 2021).

- Zuorro, A.; Iannone, A.; Natali, S.; Lavecchia, R. Green Synthesis of Silver Nanoparticles Using Bilberry and Red Currant Waste Extracts. *Processes* 2019, 7, 193. [CrossRef]
- 10. Ibrahim, H.M. Green synthesis and characterization of silver nanoparticles using banana peel extract and their antimicrobial activity against representative microorganisms. *J. Radiat. Res. Appl. Sci.* **2015**, *8*, 265–275. [CrossRef]
- 11. Padam, B.S.; Tin, H.S.; Chye, F.Y.; Abdullah, M.I. Banana by-products: An under-utilized renewable food biomass with great potential. *J. Food Sci. Technol.* **2014**, *51*, 3527–3545. [CrossRef] [PubMed]
- 12. Arun, K.; Persia, F.; Aswathy, P.; Chandran, J.; Sajeev, M.; Jayamurthy, P.; Nisha, P. Plantain peel-a potential source of antioxidant dietary fibre for developing functional cookies. *J. Food Sci. Technol.* **2015**, *52*, 6355–6364. [CrossRef] [PubMed]
- Khoozani, A.A.; Birch, J.; Bekhit, A.E.-D.A. Production, application and health effects of banana pulp and peel flour in the food industry. J. Food Sci. Technol. 2019, 56, 548–559. [CrossRef] [PubMed]
- Amorim, J.C.; Vriesmann, L.C.; Petkowicz, C.L.; Martinez, G.R.; Noleto, G.R. Modified pectin from Theobroma cacao induces potent pro-inflammatory activity in murine peritoneal macrophage. *Int. J. Biol. Macromol.* 2016, 92, 1040–1048. [CrossRef] [PubMed]
- 15. Vu, H.T.; Scarlett, C.J.; Vuong, Q.V. Phenolic compounds within banana peel and their potential uses: A review. *J. Funct. Foods* **2018**, 40, 238–248. [CrossRef]
- Hernández-Carranza, P.; Ávila-Sosa, R.; Guerrero-Beltrán, J.; Navarro-Cruz, A.; Corona-Jiménez, E.; Ochoa-Velasco, C. Optimization of antioxidant compounds extraction from fruit by-products: Apple pomace, orange and banana peel. *J. Food Process. Preserv.* 2016, 40, 103–115. [CrossRef]
- 17. Castillo-Israel, K.; Baguio, S.; Diasanta, M.; Lizardo, R.; Dizon, E.; Mejico, M. Extraction and characterization of pectin from Saba banana [*Musa 'saba'* (*Musa acuminata x Musa balbisiana*)] peel wastes: A preliminary study. *Int. Food Res. J.* **2015**, *22*, 202–207.
- 18. Emaga, T.H.; Andrianaivo, R.H.; Wathelet, B.; Tchango, J.T.; Paquot, M. Effects of the stage of maturation and varieties on the chemical composition of banana and plantain peels. *Food Chem.* **2007**, *103*, 590–600. [CrossRef]
- 19. Lee, E.-H.; Yeom, H.-J.; Ha, M.-S.; Bae, D.-H. Development of banana peel jelly and its antioxidant and textural properties. *Food Sci. Biotechnol.* **2010**, *19*, 449–455. [CrossRef]
- Methacanon, P.; Krongsin, J.; Gamonpilas, C. Pomelo (*Citrus maxima*) pectin: Effects of extraction parameters and its properties. *Food Hydrocoll.* 2014, 35, 383–391. [CrossRef]
- Wang, X.; Chen, Q.; Lü, X. Pectin extracted from apple pomace and citrus peel by subcritical water. *Food Hydrocoll.* 2014, 38, 129–137. [CrossRef]
- Mandana, B.; Russly, A.; Farah, S.; Noranizan, M.; Zaidul, I.; Ali, G. Antioxidant activity of winter melon (*Benincasa hispida*) seeds using conventional Soxhlet extraction technique. *Int. Food Res. J.* 2012, 19, 229–234.
- 23. Putra, N.R.; Yunus, M.A.C.; Ruslan, M.S.H.; Idham, Z.; Idrus, F.N. Comparison extraction of peanut skin between CO₂ supercritical fluid extraction and soxhlet extraction in term of oil yield and catechin. *Pertanika J. Sci. Technol.* **2018**, *26*, 799–810.
- Salgın, U.; Salgın, S.; Ekici, D.D.; UludaL, G. Oil recovery in rosehip seeds from food plant waste products using supercritical CO₂ extraction. J. Supercrit. Fluids 2016, 118, 194–202. [CrossRef]
- Hasmida, M.; Liza, M.; Nur Syukriah, A.; Harisun, Y.; Mohd Azizi, C.; Fadzilah Adibah, A. Total Phenolic Content and Antioxidant Activity of Quercus infectoria Galls Using Supercritical CO₂ Extraction Technique and Its Comparison with Soxhlet Extraction. *Pertanika J. Sci. Technol.* 2015, 23, 287–295.
- Sodeifian, G.; Saadati Ardestani, N.; Sajadian, S.A.; Ghorbandoost, S. Application of supercritical carbon dioxide to extract essential oil from Cleome coluteoides Boiss: Experimental, response surface and grey wolf optimization methodology. *J. Supercrit. Fluids* 2016, 114, 55–63. [CrossRef]
- Milala, J.; Grzelak-Błaszczyk, K.; Sójka, M.; Kosmala, M.; Dobrzyńska-Inger, A.; Rój, E. Changes of bioactive components in berry seed oils during supercritical CO₂ extraction. *J. Food Process. Preserv.* 2018, 42, e13368. [CrossRef]
- De Melo, M.; Şen, A.; Silvestre, A.J.; Pereira, H.; Silva, C.M. Experimental and modeling study of supercritical CO₂ extraction of Quercus cerris cork: Influence of ethanol and particle size on extraction kinetics and selectivity to friedelin. *Sep. Purif. Technol.* 2017, 187, 34–45. [CrossRef]
- 29. Pour Hosseini, S.R.; Tavakoli, O.; Sarrafzadeh, M.H. Experimental optimization of SC-CO₂ extraction of carotenoids from Dunaliella salina. *J. Supercrit. Fluids* **2017**, 121, 89–95. [CrossRef]
- Chan, Y.H.; Yusup, S.; Quitain, A.T.; Chai, Y.H.; Uemura, Y.; Loh, S.K. Extraction of palm kernel shell derived pyrolysis oil by supercritical carbon dioxide: Evaluation and modeling of phenol solubility. *Biomass Bioenergy* 2018, 116, 106–112. [CrossRef]
- 31. Taheri, S.; Brodie, G.; Gupta, D. Fluidisation of lentil seeds during microwave drying and disinfection could prevent detrimental impacts on their chemical and biochemical characteristics. *LWT* **2020**, *129*, 109534. [CrossRef]
- 32. Sarip, M.S.M.; Yamashita, Y.; Morad, N.A.; Yunus, M.A.C.; Aziz, M.K.A. Modeling and Optimization of the Hot Compressed Water Extraction of Palm Oil Using Artificial Neural Network. *J. Chem. Eng. Jpn.* **2016**, *49*, 614–621. [CrossRef]
- Hans, N.; Naik, S.N.; Malik, A. Platform Molecules from Algae by Using Supercritical CO₂ and Subcritical Water Extraction. In Handbook of Algal Technologies and Phytochemicals; CRC Press: Boca Raton, FL, USA, 2019; pp. 229–243.
- 34. Aguiló-Aguayo, I.; Walton, J.; Viñas, I.; Tiwari, B.K. Ultrasound assisted extraction of polysaccharides from mushroom byproducts. *LWT* **2017**, *77*, 92–99. [CrossRef]
- 35. Armstrong, W. Wayne's Word: An On-Line Textbook of Natural History; Palomar College: San Marcos, CA, USA, 2008.

- 36. United States Department of Agriculture. 2020. Available online: https://plants.usda.gov/java/ClassificationServlet?source=display&classid=MUSA2 (accessed on 15 August 2022).
- Cordenunsi, B.R.; Lajolo, F.M. Starch Breakdown during Banana Ripening: Sucrose Synthase and Sucrose Phosphate Synthase. J. Agric. Food Chem. 1995, 43, 347–351. [CrossRef]
- 38. Kertesz, Z.I. The Pectic Substances; Interscience Publishers: New York, NY, USA, 1951; Volume 1.
- 39. Moslemi, M. Reviewing the recent advances in application of pectin for technical and health promotion purposes: From laboratory to market. *Carbohydr. Polym.* 2020, 254, 117324. [CrossRef]
- 40. Ciriminna, R.; Chavarría-Hernández, N.; Inés Rodríguez Hernández, A.; Pagliaro, M. Pectin: A new perspective from the biorefinery standpoint. *Biofuels Bioprod. Biorefining* **2015**, *9*, 368–377. [CrossRef]
- 41. Salma, M.; Jahan, N.; Islam, M.; Hoque, M. Extraction of Pectin from lemon peel: Technology development. J. Chem. Eng. 2012, 27, 25–30. [CrossRef]
- Canteri-Schemin, M.H.; Fertonani, H.C.R.; Waszczynskyj, N.; Wosiacki, G. Extraction of pectin from apple pomace. *Braz. Arch. Biol. Technol.* 2005, 48, 259–266. [CrossRef]
- Bagherian, H.; Zokaee Ashtiani, F.; Fouladitajar, A.; Mohtashamy, M. Comparisons between conventional, microwave- and ultrasound-assisted methods for extraction of pectin from grapefruit. *Chem. Eng. Processing Process Intensif.* 2011, 50, 1237–1243. [CrossRef]
- 44. Wang, W.; Ma, X.; Xu, Y.; Cao, Y.; Jiang, Z.; Ding, T.; Ye, X.; Liu, D. Ultrasound-assisted heating extraction of pectin from grapefruit peel: Optimization and comparison with the conventional method. *Food Chem.* **2015**, *178*, 106–114. [CrossRef]
- 45. Rodsamran, P.; Sothornvit, R. Microwave heating extraction of pectin from lime peel: Characterization and properties compared with the conventional heating method. *Food Chem.* **2019**, 278, 364–372. [CrossRef]
- Liew, S.Q.; Teoh, W.H.; Tan, C.K.; Yusoff, R.; Ngoh, G.C. Subcritical water extraction of low methoxyl pectin from pomelo (*Citrus grandis* (L.) Osbeck) peels. *Int. J. Biol. Macromol.* 2018, 116, 128–135. [CrossRef]
- Yang, J.-S.; Mu, T.-H.; Ma, M.-M. Optimization of ultrasound-microwave assisted acid extraction of pectin from potato pulp by response surface methodology and its characterization. *Food Chem.* 2019, 289, 351–359. [CrossRef]
- Shivamathi, C.; Moorthy, I.G.; Kumar, R.V.; Soosai, M.R.; Maran, J.P.; Kumar, R.S.; Varalakshmi, P. Optimization of ultrasound assisted extraction of pectin from custard apple peel: Potential and new source. *Carbohydr. Polym.* 2019, 225, 115240. [CrossRef]
- 49. Gharibzahedi, S.M.T.; Smith, B.; Guo, Y. Ultrasound-microwave assisted extraction of pectin from fig (*Ficus carica* L.) skin: Optimization, characterization and bioactivity. *Carbohydr. Polym.* **2019**, 222, 114992. [CrossRef]
- 50. Colodel, C.; Vriesmann, L.C.; Teófilo, R.F.; de Oliveira Petkowicz, C.L. Extraction of pectin from ponkan (*Citrus reticulata Blanco* cv. Ponkan) peel: Optimization and structural characterization. *Int. J. Biol. Macromol.* **2018**, *117*, 385–391. [CrossRef]
- Raji, Z.; Khodaiyan, F.; Rezaei, K.; Kiani, H.; Hosseini, S.S. Extraction optimization and physicochemical properties of pectin from melon peel. Int. J. Biol. Macromol. 2017, 98, 709–716. [CrossRef]
- 52. Dranca, F.; Oroian, M. Ultrasound-assisted extraction of pectin from Malus domestica 'Fălticeni'apple pomace. *Processes* **2019**, 7, 488. [CrossRef]
- 53. Sundarraj, A.A.; Vasudevan, R.T.; Sriramulu, G. Optimized extraction and characterization of pectin from jackfruit (Artocarpus integer) wastes using response surface methodology. *Int. J. Biol. Macromol.* **2018**, *106*, 698–703. [CrossRef]
- 54. Rahmani, Z.; Khodaiyan, F.; Kazemi, M.; Sharifan, A. Optimization of microwave-assisted extraction and structural characterization of pectin from sweet lemon peel. *Int. J. Biol. Macromol.* **2020**, *147*, 1107–1115. [CrossRef]
- Sarah, M.; Hanum, F.; Rizky, M.; Hisham, M. Microwave-assisted extraction of pectin from cocoa peel. In Proceedings of the IOP Conference Series: Earth and Environmental Science, Banda Aceh, Indonesia, 26–27 September 2018; p. 012079.
- Abdul Aziz, A.H.; Putra, N.R.; Kong, H.; Che Yunus, M.A. Supercritical carbon dioxide extraction of sinensetin, isosinensetin, and rosmarinic acid from Orthosiphon stamineus leaves: Optimization and modeling. *Arab. J. Sci. Eng.* 2020, 45, 7467–7476. [CrossRef]
- Daud, N.M.; Putra, N.R.; Jamaludin, R.; Norodin, N.S.M.; Sarkawi, N.S.; Hamzah, M.H.S.; Nasir, H.M.; Zaidel, D.N.A.; Yunus, M.A.C.; Salleh, L.M. Valorisation of plant seed as natural bioactive compounds by various extraction methods: A review. *Trends Food Sci. Technol.* 2022, 119, 201–214. [CrossRef]
- Rizkiyah, D.N.; Jusoh, W.M.S.W.; Idham, Z.; Putra, N.R.; Che Yunus, M.A. Investigation of Phenolic, Flavonoid and Antioxidant Recovery and Solubility from Roselle Using Supercritical Carbon Dioxide: Experimental and Modelling. *J. Food Process. Preserv.* 2022, e16670. [CrossRef]
- 59. Putra, N.R.; Wibobo, A.G.; Machmudah, S.; Winardi, S. Recovery of valuable compounds from palm-pressed fiber by using supercritical CO₂ assisted by ethanol: Modeling and optimization. *Sep. Sci. Technol.* **2020**, *55*, 3126–3139. [CrossRef]
- Idham, Z.; Putra, N.R.; Aziz, A.H.A.; Zaini, A.S.; Rasidek, N.A.M.; Mili, N.; Yunus, M.A.C. Improvement of extraction and stability of anthocyanins, the natural red pigment from roselle calyces using supercritical carbon dioxide extraction. *J. CO2 Util.* 2022, 56, 101839. [CrossRef]
- Idham, Z.; Putra, N.R.; Nasir, H.; Yian, L.N.; Idrus, N.F.M.; Yunus, M.A.C. Extraction and Solubility Modeling of Anthocyanins Rich Extract from Hibiscus sabdariffa L. using Supercritical Carbon Dioxide. *Malays. J. Fundam. Appl. Sci.* 2021, 17, 720–730. [CrossRef]

- 62. Putra, N.R.; Rizkiyah, D.N.; Zaini, A.S.; Machmudah, S.; Yunus, M.A.C. Solubility of catechin and epicatechin from Arachis Hypogea skins wastes by using supercritical carbon dioxide-ethanol and its optimization. *J. Food Meas. Charact.* **2021**, *15*, 2031–2038. [CrossRef]
- Putra, N.R.; Aziz, A.H.A.; Yian, L.N.; Ramli, W.D.; Yunus, M.A.C. Optimization of supercritical carbon dioxide and co-solvent ethanol extraction of wasted peanut skin using response surface methodology. In Proceedings of the MATEC Web of Conferences the 24th Regional Symposium on Chemical Engineering, Semarang, Indonesia, 15–16 November 2017; p. 02005.
- 64. Mohd-Nasir, H.; Putra, N.R.; Chuo, S.C.; Daud, N.M.; Hartati, H.; Bakeri, N.; Ruslan, M.S.; Mohd-Setapar, S.H.; Ahmad, A.; Md Salleh, L. Optimization of the supercritical carbon dioxide extraction of Quercus infectoria galls extracts and its bioactivities. *J. Food Process. Preserv.* **2021**, *45*, e15156. [CrossRef]
- Baldevraj, R.S.M.; Jagadish, R.S. 14-Incorporation of chemical antimicrobial agents into polymeric films for food packaging. In *Multifunctional and Nanoreinforced Polymers for Food Packaging*; Lagarón, J.-M., Ed.; Woodhead Publishing: Thorston, UK, 2011; pp. 368–420.
- 66. Khamsucharit, P.; Laohaphatanalert, K.; Gavinlertvatana, P.; Sriroth, K.; Sangseethong, K. Characterization of pectin extracted from banana peels of different varieties. *Food Sci. Biotechnol.* **2018**, *27*, 623–629. [CrossRef]
- Zuorro, A. Enhanced Lycopene Extraction from Tomato Peels by Optimized Mixed-Polarity Solvent Mixtures. *Molecules* 2020, 25, 2038. [CrossRef]
- 68. De Castro, M.L.; Priego-Capote, F. Soxhlet extraction: Past and present panacea. J. Chromatogr. A 2010, 1217, 2383–2389. [CrossRef]
- Hawthorne, S.B.; Grabanski, C.B.; Martin, E.; Miller, D.J. Comparisons of Soxhlet extraction, pressurized liquid extraction, supercritical fluid extraction and subcritical water extraction for environmental solids: Recovery, selectivity and effects on sample matrix. J. Chromatogr. A 2000, 892, 421–433. [CrossRef]
- 70. Zuorro, A.; Lavecchia, R.; González-Delgado, Á.D.; García-Martinez, J.B.; L'Abbate, P. Optimization of Enzyme-Assisted Extraction of Flavonoids from Corn Husks. *Processes* **2019**, *7*, 804. [CrossRef]
- Singh, S.; Prakash, P. Evaluation of antioxidant activity of banana peels (*Musa acuminata*) extracts using different extraction methods. *Chem. Sci. Trans* 2015, 4, 158–160.
- 72. Hamid, H.; Abdollah, M.; Masripan, N.; Hasan, R. Characterization of raw and ripen of banana peel wastes and it's oils extraction using soxhlet method. *Int. J. Appl. Chem.* 2016, *12*, 1–5.
- Nasir, N.A.H.A.; Roslly, N.A.L.; Rosli, N.M.; Razali, Z. Phytochemical Screening and Potential DPPH Radical Scavenging Activity of Banana Peel Extract. In *Charting the Sustainable Future of ASEAN in Science and Technology*; Springer: Berlin/Heidelberg, Germany, 2020; pp. 249–258.
- 74. Okolie, J.A.; Henry, O.E.; Epelle, E.I. Determination of the antioxidant potentials of two different varieties of banana peels in two different solvents. *Food Nutr. Sci.* 2016, *7*, 1253.
- 75. Wu, T.R.; Wang, H.L.; Jiang, S.W.; Liu, D.D.; Wei, F. Optimization of Extraction of Tannins from Banana Peel Using Response Surface Methodology. *Appl. Mech. Mater.* **2014**, *678*, 566–571. [CrossRef]
- 76. Mandal, V.; Mohan, Y.; Hemalatha, S. Microwave assisted extraction—An innovative and promising extraction tool for medicinal plant research. *Pharmacogn. Rev.* 2007, 1, 7–18.
- 77. Letellier, M.; Budzinski, H. Microwave assisted extraction of organic compounds. Analusis 1999, 27, 259–270. [CrossRef]
- 78. Latha, C. Microwave-assisted extraction of embelin from Embelia ribes. Biotechnol. Lett. 2007, 29, 319–322. [CrossRef]
- 79. Huie, C.W. A review of modern sample-preparation techniques for the extraction and analysis of medicinal plants. *Anal. Bioanal. Chem.* **2002**, *373*, 23–30. [CrossRef]
- 80. Letellier, M.; Budzinski, H.; Charrier, L.; Capes, S.; Dorthe, A. Optimization by factorial design of focused microwave assisted extraction of polycyclic aromatic hydrocarbons from marine sediment. *Fresenius J. Anal. Chem.* **1999**, *364*, 228–237. [CrossRef]
- Yan, M.-M.; Liu, W.; Fu, Y.-J.; Zu, Y.-G.; Chen, C.-Y.; Luo, M. Optimisation of the microwave-assisted extraction process for four main astragalosides in Radix Astragali. *Food Chem.* 2010, 119, 1663–1670. [CrossRef]
- Egizabal, A.; Zuloaga, O.; Etxebarria, N.; Fernandez, L.; Madariaga, J. Comparison of microwave-assisted extraction and Soxhlet extraction for phenols in soil samples using experimental designs. *Analyst* 1998, 123, 1679–1684. [CrossRef]
- 83. Zuloaga, O.; Etxebarria, N.; Fernández, L.A.; Madariaga, J.M. Optimisation and comparison of microwave-assisted extraction and Soxhlet extraction for the determination of polychlorinated biphenyls in soil samples using an experimental design approach. *Talanta* **1999**, *50*, 345–357. [CrossRef]
- 84. Karthikeyan, S.; Balasubramanian, R.; See, S.W. Optimization and validation of a low temperature microwave-assisted extraction method for analysis of polycyclic aromatic hydrocarbons in airborne particulate matter. *Talanta* **2006**, *69*, 79–86. [CrossRef]
- Routray, W.; Orsat, V. Microwave-assisted extraction of flavonoids: A review. *Food Bioprocess Technol.* 2012, *5*, 409–424. [CrossRef]
 Swamy, G.J.; Muthukumarappan, K. Optimization of continuous and intermittent microwave extraction of pectin from banana
- peels. *Food Chem.* 2017, 220, 108–114. [CrossRef]
 87. Rivadeneira, J.P.; Wu, T.; Ybanez, Q.; Dorado, A.A.; Migo, V.P.; Nayve, F.R.P.; Castillo-Israel, K.A.T. Microwave-Assisted Extraction of Pectin from "Saba" Banana Peel Waste: Optimization, Characterization, and Rheology Study. *Int. J. Food Sci.* 2020, 2020, 8879425. [CrossRef]
- 88. Phaiphan, A. Optimisation of pectin extraction assisted by microwave from banana (*Musa sapientum* L.) fruit peels using response surface methodology. *Carpathian J. Food Sci. Technol.* **2019**, *11*, 127–140.

- 89. Shirsath, S.; Sonawane, S.; Gogate, P. Intensification of extraction of natural products using ultrasonic irradiations—A review of current status. *Chem. Eng. Process. Process Intensif.* **2012**, *53*, 10–23. [CrossRef]
- 90. Tiwari, B.K. Ultrasound: A clean, green extraction technology. TrAC Trends Anal. Chem. 2015, 71, 100–109. [CrossRef]
- 91. Vilkhu, K.; Mawson, R.; Simons, L.; Bates, D. Applications and opportunities for ultrasound assisted extraction in the food industry—A review. *Innov. Food Sci. Emerg. Technol.* **2008**, *9*, 161–169. [CrossRef]
- 92. Chemat, F.; Khan, M.K. Applications of ultrasound in food technology: Processing, preservation and extraction. *Ultrason. Sonochem.* **2011**, *18*, 813–835. [CrossRef]
- 93. Mason, T.J.; Lorimer, J.P. *Applied Sonochemistry: The Uses of Power Ultrasound in Chemistry and Processing*; Wiley-Vch Weinheim: Weinheim, Germany, 2002; Volume 10.
- 94. Suslick, K.S.; Price, G.J. Applications of ultrasound to materials chemistry. Annu. Rev. Mater. Sci. 1999, 29, 295–326. [CrossRef]
- 95. Toma, M.; Vinatoru, M.; Paniwnyk, L.; Mason, T.J. Investigation of the effects of ultrasound on vegetal tissues during solvent extraction. *Ultrason. Sonochem.* 2001, *8*, 137–142. [CrossRef]
- 96. Maran, J.P.; Priya, B.; Al-Dhabi, N.A.; Ponmurugan, K.; Moorthy, I.G.; Sivarajasekar, N. Ultrasound assisted citric acid mediated pectin extraction from industrial waste of Musa balbisiana. *Ultrason. Sonochem.* **2017**, *35*, 204–209. [CrossRef]
- Ishak, N.A.; Razak, N.A.A.; Dek, M.S.P.; Baharuddin, A.S. Production of High Tannin Content and Antioxidant Activity Extract from an Unripe Peel of Musa acuminata (Cavendish) Using Ultrasound-Assisted Extraction (UAE). *BioResources* 2020, 15, 1877–1893.
- Minjares-Fuentes, R.; Femenia, A.; Garau, M.; Meza-Velázquez, J.; Simal, S.; Rosselló, C. Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach. *Carbohydr. Polym.* 2014, 106, 179–189. [CrossRef]
- Marić, M.; Grassino, A.N.; Zhu, Z.; Barba, F.J.; Brnčić, M.; Brnčić, S.R. An overview of the traditional and innovative approaches for pectin extraction from plant food wastes and by-products: Ultrasound-, microwaves-, and enzyme-assisted extraction. *Trends Food Sci. Technol.* 2018, *76*, 28–37. [CrossRef]
- Grassino, A.N.; Brnčić, M.; Vikić-Topić, D.; Roca, S.; Dent, M.; Brnčić, S.R. Ultrasound assisted extraction and characterization of pectin from tomato waste. *Food Chem.* 2016, 198, 93–100. [CrossRef]
- 101. Lin, C.B.; KAI, N.; Ali, A. Ultrasound assisted extraction of pectin from dragon fruit peels. J. Eng. Sci. Technol. 2018, 13, 65–81.
- 102. Moorthy, I.G.; Maran, J.P.; Muneeswari, S.; Naganyashree, S.; Shivamathi, C. Response surface optimization of ultrasound assisted extraction of pectin from pomegranate peel. *Int. J. Biol. Macromol.* **2015**, *72*, 1323–1328. [CrossRef]
- de Oliveira, C.F.; Giordani, D.; Lutckemier, R.; Gurak, P.D.; Cladera-Olivera, F.; Marczak, L.D.F. Extraction of pectin from passion fruit peel assisted by ultrasound. *LWT-Food Sci. Technol.* 2016, *71*, 110–115. [CrossRef]
- 104. Ju, Z.; Howard, L.R. Subcritical water and sulfured water extraction of anthocyanins and other phenolics from dried red grape skin. *J. Food Sci.* 2005, *70*, S270–S276. [CrossRef]
- Ramos, L.; Kristenson, E.M.; Brinkman, U.T. Current use of pressurised liquid extraction and subcritical water extraction in environmental analysis. J. Chromatogr. A 2002, 975, 3–29. [CrossRef]
- Munir, M.; Kheirkhah, H.; Baroutian, S.; Quek, S.Y.; Young, B.R. Subcritical water extraction of bioactive compounds from waste onion skin. J. Clean. Prod. 2018, 183, 487–494. [CrossRef]
- 107. Zhang, J.; Wen, C.; Chen, M.; Gu, J.; Zhou, J.; Duan, Y.; Zhang, H.; Ma, H. Antioxidant activities of Sagittaria sagittifolia L. polysaccharides with subcritical water extraction. *Int. J. Biol. Macromol.* **2019**, *134*, 172–179. [CrossRef]
- Zaini, A.; Putra, N.; Idham, Z.; Norodin, N.M.; Rasidek, N.M.; Yunus, M.C. Mini Review: Extraction of Allicin from Allium sativum using Subcritical Water Extraction. *Mater. Sci. Eng.* 2020, 923, 012023. [CrossRef]
- Nastić, N.; Švarc-Gajić, J.; Delerue-Matos, C.; Barroso, M.F.; Soares, C.; Moreira, M.M.; Morais, S.; Mašković, P.; Srček, V.G.; Slivac, I. Subcritical water extraction as an environmentally-friendly technique to recover bioactive compounds from traditional Serbian medicinal plants. *Ind. Crops Prod.* 2018, 111, 579–589. [CrossRef]
- 110. Lachos-Perez, D.; Baseggio, A.M.; Mayanga-Torres, P.; Junior, M.R.M.; Rostagno, M.; Martínez, J.; Forster-Carneiro, T. Subcritical water extraction of flavanones from defatted orange peel. *J. Supercrit. Fluids* **2018**, *138*, 7–16. [CrossRef]
- Todd, R.; Baroutian, S. A techno-economic comparison of subcritical water, supercritical CO₂ and organic solvent extraction of bioactives from grape marc. J. Clean. Prod. 2017, 158, 349–358. [CrossRef]
- 112. Liu, X.; Wang, Y.; Zhang, J.; Yan, L.; Liu, S.; Taha, A.A.; Wang, J.; Ma, C. Subcritical water extraction of phenolic antioxidants with improved α-amylase and α-glucosidase inhibitory activities from exocarps of Castanea mollissima Blume. *J. Supercrit. Fluids* 2020, 158, 104747. [CrossRef]
- 113. Sarfarazi, M.; Jafari, S.M.; Rajabzadeh, G.; Feizi, J. Development of an environmentally-friendly solvent-free extraction of saffron bioactives using subcritical water. *LWT* **2019**, *114*, 108428. [CrossRef]
- Gbashi, S.; Adebo, O.A.; Piater, L.; Madala, N.E.; Njobeh, P.B. Subcritical water extraction of biological materials. *Sep. Purif. Rev.* 2017, 46, 21–34. [CrossRef]
- 115. Pinto, D.; Vieira, E.F.; Peixoto, A.F.; Freire, C.; Freitas, V.; Costa, P.; Delerue-Matos, C.; Rodrigues, F. Optimizing the extraction of phenolic antioxidants from chestnut shells by subcritical water extraction using response surface methodology. *Food Chem.* 2021, 334, 127521. [CrossRef]
- 116. Kovačević, D.B.; Barba, F.J.; Granato, D.; Galanakis, C.M.; Herceg, Z.; Dragović-Uzelac, V.; Putnik, P. Pressurized hot water extraction (PHWE) for the green recovery of bioactive compounds and steviol glycosides from Stevia rebaudiana Bertoni leaves. *Food Chem.* 2018, 254, 150–157.

- Pillot, M.; Lebeau, B.; Nouali, H.; Daou, T.J.; Patarin, J.; Ryzhikov, A. High pressure intrusion of water and LiCl aqueous solutions in hydrophobic KIT-6 mesoporous silica: Influence of the grafted group nature. *Microporous Mesoporous Mater.* 2019, 280, 248–255. [CrossRef]
- 118. Ong, E.S.; Cheong, J.S.H.; Goh, D. Pressurized hot water extraction of bioactive or marker compounds in botanicals and medicinal plant materials. *J. Chromatogr. A* 2006, 1112, 92–102. [CrossRef]
- 119. Holgate, H.R.; Tester, J. Oxidation of hydrogen and carbon monoxide in sub-and supercritical water: Reaction kinetics, pathways, and water-density effects. 2. Elementary reaction modeling. J. Phys. Chem. **1994**, *98*, 810–822. [CrossRef]
- Rasidek, N.A.M.; Nordin, M.F.M.; Tokuyama, H.; Nagatsu, Y.; Mili, N.; Zaini, A.S.; Idham, Z.; Yunus, M.A.C. Subcritical water-based pectin from banana peels (*Musa Paradisiaca* Cv. Tanduk) as a natural gelation agent. *Mater. Today Proc.* 2021, 47, 1329–1355. [CrossRef]
- 121. Rasidek, N.A.M.; Nordin, M.F.M.; Yusoff, Y.A.; Tokuyama, H.; Nagatsu, Y. Effect of temperature on rheology behaviour of banana peel pectin extracted using hot compressed water. *Jurnal Teknologi*. **2018**, *80*, 3. [CrossRef]
- Muñoz-Almagro, N.; Valadez-Carmona, L.; Mendiola, J.A.; Ibáñez, E.; Villamiel, M. Structural characterisation of pectin obtained from cacao pod husk. Comparison of conventional and subcritical water extraction. *Carbohydr. Polym.* 2019, 217, 69–78. [CrossRef]
- 123. Li, W.J.; Fan, Z.G.; Wu, Y.Y.; Jiang, Z.G.; Shi, R.C. Eco-friendly extraction and physicochemical properties of pectin from jackfruit peel waste with subcritical water. *J. Sci. Food Agric.* **2019**, *99*, 5283–5292. [CrossRef]
- Klinchongkon, K.; Khuwijitjaru, P.; Adachi, S. Properties of subcritical water-hydrolyzed passion fruit (*Passiflora edulis*) pectin. *Food Hydrocoll.* 2018, 74, 72–77. [CrossRef]