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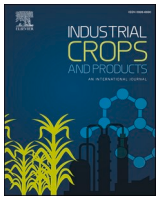

Enhancement of industrial pectin production from sugar beet pulp by the integration of surfactants in ultrasound-assisted extraction followed by diafiltration/ultrafiltration

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ABSTRACT

To recover industrial pectin from sugar beet pulp (SBP), a combined surfactant and ultrasound-assisted extraction, followed by a concentration with ultrafiltration/diafiltration membranes is proposed. First, the operation conditions of the extraction were optimized for synthetic (Tween80, PEG4000) and natural (Saponin) surfactants. Tween 80 and Saponin provided maximum galacturonic acid (GalA) concentrations (5.5 and 5.8 g/L, respectively) under the best extraction conditions (4 $g_{surfactant}$/L, pH = 1, amplitude = 90%, and time = 90 min). The extracted liquid was purified and concentrated through membranes. The final retentate maintained 73% of the initial GalA and eliminated a high percentage of oligosaccharides. Finally, the pectin was precipitated with ethanol, and the precipitate solid contained 57 g GalA/100 g GalA of SBP with a high degree of esterification (DE = 81%) and M_W (930 kDa). The pectin yield for the global process was 24.6%. The ultrasound-assisted extraction with surfactants followed by diafiltration/ultrafiltration could become a promising process for the chemical industry, able to provide pectin-enriched products of commercial interest from sugar beet pulp.

1. Introduction

Pectin is a natural fiber composed of different heteropolysaccharides, which constitute the main component of the cell wall of plants, and pectin is the primary precursor in their growth and development (Ciriminna et al., 2015; Sharma et al., 2021). Pectin consists of up to 300–1000 saccharide units, most of which are polymeric units of D-galacturonic acid (GalA) (Voragen et al., 2009). Industrial pectin is commonly used as a technological adjuvant in the cosmetic, plastic, and pharmaceutical industries, in cancer treatments, agricultural applications, as an absorbent in the textile industry, or as a gelling, stabilizing, or thickening agent in the food industry (Concha Olmos and Zúñiga Hansen, 2012; Mata et al., 2009). Commercial pectin is provided principally by apple pomace and citrus peel, extracted at low pH and high temperature (Ciriminna et al., 2015; Pilgrim et al., 1991; Su et al., 2019). However, in recent years, other agronomic wastes, such as sugar beet pulp (SBP) (Abou-Elseoud et al., 2021; del Amo-Mateos et al., 2022), dragon fruit peels (Chua et al., 2022), or sunflowers (Ezzati et al., 2022) have been used to obtain pectin.

SBP is one of the most abundant residues produced in Europe, around 20 million tonnes per year, and its most common use is as feed for livestock (Joanna et al., 2018; Usmani et al., 2022). However, according to the literature, SBP has a composition range of 20–25% of cellulose, 20–30% of hemicellulose, 15–25% of pectin, and 10–15% of protein (del Amo-Mateos et al., 2022; Usmani et al., 2022). Due to this composition, the SBP valorization to obtain such high-value products as pectin and oligosaccharides can be taken into account within the circular economy framework.

The most common technology used to extract pectin from agronomical wastes is conventional extraction at low pH and high temperatures for a long time, increasing the selectivity and extraction yield. However, these severe working conditions can possibly cause the degradation of valuable compounds (Das and Arora, 2021; Li et al., 2012). Hence, novel techniques are emerging to improve pectin extraction, considering the mass transfer principles of pectin hydrolysis, solubilization, and equilibrium (Adetunji et al., 2017). Some examples

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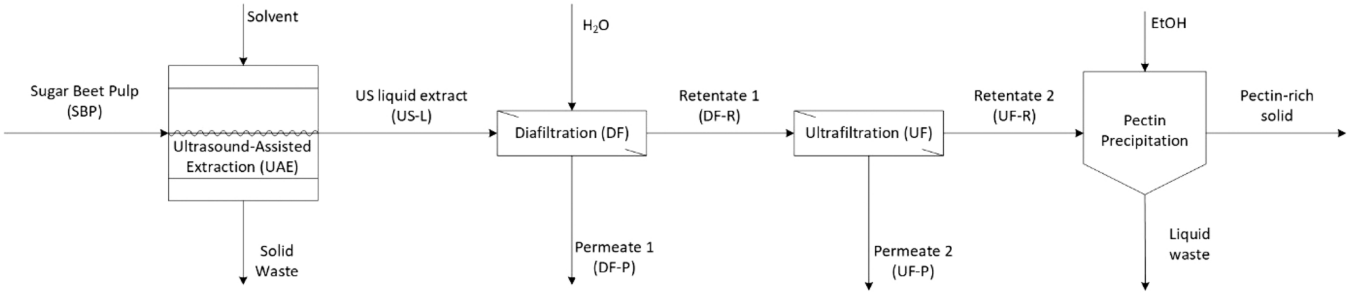

Fig. 1. The integrated proposed process to recover pectin from Sugar Beet Pulp.

of these novel technologies include, enzyme-assisted extraction, microwave-assisted extraction, subcritical water extraction, or ultrasound-assisted extraction (Adetunji et al., 2017; Gerschenson et al., 2021; Mao et al., 2019; Marić et al., 2018).

Ultrasound-assisted extraction (UAE) is mainly characterized by choosing the frequency and wavelength amplitude to perform the solid-liquid extraction (Mao et al., 2019). UAE improves the pectin extraction yield due to a higher biomass cell rupture and the process known as cavitation, a two-step cycle: first the expansion, where bubbles are formed and expanded, and second the compression, where the bubbles collapse because of the negative pressure on the liquid tensile strength (Adetunji et al., 2017; Mao et al., 2019; Selvakumar et al., 2021). This type of extraction has some advantages compared to the conventional method, such as increasing the extraction efficiency while reducing the extraction time and consuming less energy and water (Jovanovic-Malinovska et al., 2015; Shivamathi et al., 2019). However, UAE can suffer from poor pectin uniformity due to the application of ultrasound waves, making it difficult to reproduce the method (Cui et al., 2021).

Surfactant extraction combined with UAE has been proposed to improve the extraction yield and reproducibility (Selvakumar et al., 2021). The use of surfactants in extracting organic compounds is a novel technique that has emerged recently (Su et al., 2019; Wang et al., 2022). Surfactants are amphiphilic molecules with a hydrophilic head and hydrophobic tail (Sharma et al., 2021). When the surfactant concentration exceeds the critical micellar concentration, micelles are formed. These micelles are inclined to establish chemical and physical interactions with the hydrophilic and lipophilic compounds present in the liquid. For instance, organic compounds, such as pectin, remain within the micelle in the lipophilic zone (Sharma et al., 2021; Su et al., 2019). This type of extraction can be suitable for improving pectin extraction without causing compound degradation.

Once the extraction has been performed, a pectin recovery step is necessary. Some studies have reported that alcohol precipitation is an effective method for recovering pectin from liquid extracts (Ai et al., 2020; López-Linares et al., 2020). However, precipitation is a non-selective method. Not only does the pectin precipitate, but other compounds, such as monomeric and oligomeric sugars, decrease the purity of the precipitated pectin (del Amo-Mateos, 2020; Ramos-Andrés et al., 2019).

Pectin extracted by UAE is characterized by a relatively high molecular weight (M_W) (> 50 kDa) (Gerschenson et al., 2021; Mao et al., 2019). Therefore, one way to concentrate and purify the liquid extract obtained is to use ultrafiltration/diafiltration (UF/DF) membranes, which can separate the pectin from the low M_W compounds that lack interest. In this way, the precipitated pectin obtained after a process of membrane purification is expected to have a higher degree of purity. A further advantage of the previous concentration step of the liquid extracts is that the amount of ethanol needed for subsequent pectin precipitation is considerably reduced. Some studies have reported different configurations to purify the extract liquids. For example, Ramos-Andrés et al. (2019) proposed first performing ultrafiltration (UF) to concentrate the extract, followed by diafiltration (DF) to purify the hemicellulose fractions obtained by the hydrothermal treatment of discarded carrots. However, Thuvander et al. (2016) proposed starting with a DF process, subsequently carrying out a UF step to recover hemicelluloses and lignin from the thermomechanical pulp.

Based on a literature review, no studies analyze the effect of surfactants on the UAE to extract pectin from agronomical wastes, such as SBP. Furthermore, none of them include the use of UF/DF membranes to separate and purify pectin-rich liquids. The main goals of this paper are: (1) to optimize pectin extraction based on UAE technology using different synthetic (Tween80, PEG4000) and natural (Saponin) surfactants. For this purpose, a Factorial Design to select the best conditions (surfactant concentration, pH, amplitude, and time) was proposed; (2) to study the sequence of concentration and purification of pectin based on the use of DF/UF membranes, to obtain a pectin-rich liquid of M_W and purity with potential commercial use.

The development of green and energy-saving technologies for the valorization of agri-food by-products to obtain products with high-added value is of considerable importance for the chemical industry. Developments such as those proposed in this work (pectin-enriched product from SBP), contribute significantly to progress in achieving sustainable development goals: Ensuring sustainable consumption and production patterns (SDG 12), as well as affordable and clean energy (SDG 7).

2. Materials and methods

2.1. Raw material

A sugar company graciously provided sugar beet pulp (SBP), frozen at − 20 ºC until experiment use. Before the test runs, the SBP was washed, dried in an oven at 70 ºC, and ground ($d_p < 1$ mm).

2.2. Ultrasound-assisted extraction

The extraction was performed in the ultrasound equipment with a direct sonification (20 kHz) (Hielscher Ultrasound Technology UIP1000hd transducer, Hielscher Ultrasonics GmbH, Germany) under the established experimental conditions. For UAE, the standard method was as follows: S:L extraction was carried out using a stainless steel hermetically sealed reactor in which the necessary amount of SBP and 150 mL of the solvent were combined (Fig. 1). Following extraction, the mixture was centrifuged for 10 min at 10,000 g using a Centrifuge Sorvall legend RT from Thermo Fisher Scientific in Spain. A filtration (Filter-lab 1300/80 0.45 mm, Filters AOIA S.A., Spain) was used to

Table 1
Factorial Design for UAE. Experimental GalA concentration for each run carried out with different surfactants: Tween80, PEG4000, and Saponin. Fixed operating conditions: S/L ratio: 1/20 (g/mL) and without temperature control.

Run	X_1: Surfactant Concentration (g/L)	X_2: pH	X_3: Amplitude (%)	X_4: Time (min)	Y: GalA concentration (g/L)		
					Tween80	PEG4000	Saponin
1	0	1	50	30	1.49	1.49	1.49
2	0	1	50	90	1.74	1.74	1.74
3	0	1	90	30	1.62	1.62	1.62
4	0	1	90	90	1.81	1.81	1.81
5	0	4	50	30	1.38	1.38	1.38
6	0	4	50	90	1.82	1.82	1.82
7	0	4	90	30	1.61	1.61	1.61
8	0	4	90	90	1.90	1.90	1.90
9	4	1	50	30	3.51	1.84	2.97
10	4	1	50	90	3.82	2.04	4.26
11	4	1	90	30	4.86	2.78	5.50
12	4	1	90	90	5.50	2.85	5.84
13	4	4	50	30	1.47	1.50	1.60
14	4	4	50	90	2.35	1.81	1.89
15	4	4	90	30	1.90	1.70	2.61
16	4	4	90	90	2.42	2.44	3.35

Note: Data shown as the mean value with less than 5% of relative error.

carefully recover the supernatants. They were then kept at 4 ºC until further product recovery and analysis.

2.3. Factorial design for ultrasound-assisted extraction

A literature review of the recovery of pectin was carried out to establish the extraction conditions (Karbuz and Tugrul, 2020; Jafarzadeh-Moghaddam et al., 2020; Maran et al., 2017; Su et al., 2019; Wang et al., 2016; Zaid et al., 2020). Six critical factors and their operation ranges were identified from this review: the S:L ratio (1/5–1/40), pH (1−5), the extraction time (30–180 min), and temperature (30–85 ºC), amplitude (50 −100%), and the use or not of surfactants.

The Factorial Design was performed to maximize the GalA concentration in the extracted liquid. The Factorial Design chosen is a type 2^4, so it analyzed the effect of four critical extraction factors with two levels on the recovery of pectin, measured as GalA in the liquid extracts. The factors and levels were: surfactant concentration (X_1: 0–4; g/L), pH (X_2: 1–4), amplitude (X_3: 50–90; %), and time (X_4: 30–90, min). The chosen surfactants to compare were: Tween80, PEG4000, and Saponin (Table 1). The proposed runs were performed under the following unvaried conditions: The S/L ratio was set at 1/20 (g/mL) to ensure that the S/L mixture was as homogeneous as possible without compromising the extraction performance (Ezzati et al., 2020; Shivamathi et al., 2019), and the temperature was not controlled due to the equipment design. The total number of experimental runs was 32.

A second-order polynomial model was suggested to compare the experimental and expected responses of the GalA concentration. Eq. (1) is an example of the second-order polynomial model used to analyze the experimental findings.

$$Y = b_0 + \sum_{i=1}^{4} b_i X_i + \sum_{i=1}^{4} b_{ii} X_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{4} b_{ij} X_i X_{ij} \quad (1)$$

where Y is the response value, b_0 is the offset term, b_i is the linear effect, b_{ii} is the squared effect, b_{ij} is the interaction effect, and X_i and X_j are independent variables.

2.4. Diafiltration and ultrafiltration (DF/UF)

The equipment used to perform the DF/UF process was the Labscale TFF system (Millipore Corporation, United States). The UAE extracted liquid passed through a membrane with an M_W cut-off (MWCO) of 50 kDa and a filtration area of 50 cm^2 (Pellicon XL 50 Biomax membrane).

In the DF/UF configuration (Fig. 1), a continuous DF process was first performed to purify the extracted liquid provided by the UAE (US-L) using distilled water, as marked by the manufacturer. Then, the retentate obtained by DF (DF-R) was subjected to a UF process to concentrate it and obtain a GalA-rich liquid (UF-R). In this instance, a manually regulating valve on the retentate side kept the transmembrane pressure (TMP) with the range of 1.5–2.0 bar. The range for the feed flow rate was 0.8–1.0 mL/min/cm^2. After approximately an hour of operation, the procedure was completed when the feed volume had been lowered by 50% (v/v). The UF/DF membranes underwent a cleaning and flushing stage following each experiment, as advised by the manufacturer.

After the HPLC composition analysis, GalA and other by-products in the samples were confirmed by mass balance.

2.5. Pectin recovery by ethanol precipitation

Following the extraction and concentration of GalA, the GalA-rich liquid (UF-R) was combined with two volumes of 96% ethanol. Overnight, the mixture was maintained at 4 °C. Next, the precipitated solid was centrifuged, filtered, and dried at 60 °C. Ethanol from the supernatant was recovered using a rotary evaporator.

The pectin yield was calculated as follows (Eq. (2)):

$$Pectin\ yield(\%) = \frac{Weight\ of\ dried\ recovery\ pectin(g)}{Weight\ of\ dried\ initial\ SBP(g)} x100 \quad (2)$$

The resulting precipitated solids were stowed for further evaluation by Fourier Transform Infrared spectroscopy (FTIR) and by the degree of esterification.

The pectin-rich solids were resuspended in water (1 g/L) at 35 °C and 200 rpm for 24 h in an orbital shaker. The composition of mono- and oligosaccharides, GalA, and the MW distribution of the solid suspension were all determined.

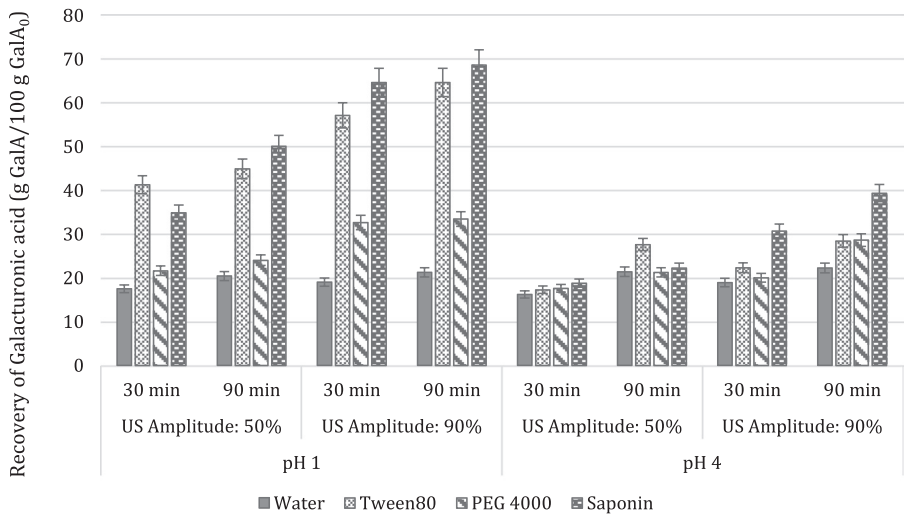

Fig. 2. Galacturonic acid (GalA) recovery (g GalA/100 g initial GalA) for each UAE run.

2.6. Analytical methods

2.6.1. Galacturonic acid concentration

GalA was used as a reference to quantify the pectin content and determine it using the colorimetric method (Melton and Smith, 2001). In a UV–VIS spectrophotometer, absorbance was measured at 525 nm (Uvmini-1240, Shimadzu Suzhou Instruments Wfg. Shimadzu Corporation, Kyoto, Japan). del Amo-Mateos et al. (2022) define this strategy thoroughly.

2.6.2. Degree of esterification (DE)

First, 0.2 g of precipitated material was combined with 3 mL of 96% ethanol and diluted in 20 mL of distilled water to calculate the pectin DE. After adding two phenolphthalein drops, the solution was titrated with 0.1 M NaOH (V1). The mixture was hydrolyzed with 10 mL of 0.1 M NaOH, and the pink coloration was removed with 10 mL of 0.1 M HCl after 15 min. The samples were then titrated with 0.1 M NaOH (V2). Pectin DE was calculated as follows (Eq. (3)):

$$Degree\ of\ esterification(\%) = V_2/(V_1 + V_2)\ x\ 100 \quad (3)$$

2.6.3. Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR was used to examine the functional-group composition of the precipitated solid (Alpha model, with a Platinum ATR single reflection diamond module, Bruker, USA). Between 4000 and 400 cm^{-1} in wavelength range, absorbance spectra were measured.

2.6.4. High-Performance Liquid Chromatography (HPLC) analysis

HPLC with an Aminex HPX-87 H column and a refractive index detector (Waters 2414, USA) was used to analyze the concentration of sugars (glucose, arabinose and galactose). The operational conditions were described by del Amo-Mateos et al. (2022).

2.6.5. Molecular Weight (M_w) analysis

The M_W distribution of solid suspensions was determined by High-Performance Size Exclusion Chromatography (HPSEC) using a refractive index detector (Waters 2414, USA) and an Ultrahydrogel 250 column (Waters, Japan). The operational conditions were thoroughly described by López-Linares et al. (2020). Dextran standards (Sigma-Aldrich, USA) were used with MW of 1, 5, 12, 50, and 670 kDa. Before analysis, all the samples were filtered via 0.22 μm nylon filters. Every analysis was performed twice.

2.7. Data analysis

The Statgraphics Centurion IXX version was used for an ANOVA test and a Factorial Design experimental design. ANOVA and Tukey's multiple-range tests determined the statistically significant differences at a 95% confidence level ($p < 0.05$).

3. Results and discussion

3.1. Composition of raw material

The complete characterization of the SBP used in this study is shown in del Amo-Mateos et al. (2022). In short, the SBP had the following composition (% dry weight basis): Pectin as GalA: 17.0; glucose: 22.4; galactose: 7.7; and arabinose: 32.3. According to this composition, the valorization of the SPB for the recovery of pectin is of interest.

On the other hand, the type of pectin in SBP might be known. Pectin is composed principally of homogalacturonan (HG) and rhamnogalacturonan I (RG-I). HG is an esterified and acetylated linear galacturonic acid polymer methyl. RG-I is a mix of galacturonic acid and rhamnose, which has many-ramified arabinan, galactan, or arabinogalactan side chains (del Amo-Mateos et al., 2022; Martínez et al., 2009). The SBP pectin used in this work is mainly made up of RG-I pectin due to the high hemicellulose content present in the raw material used (Mao et al., 2019).

3.2. Galacturonic acid extraction by UAE

3.2.1. UAE model and statistical analysis

A green and novel approach is proposed based on UAE mediated by surfactants to obtain a GalA-rich solution from SBP. For this purpose, the Factorial Design was planned to assess the influence of critical extraction parameters, such as the surfactant concentration (X_1: 0–4; g/L), pH (X_2: 1–4), amplitude (X_3: 50–90; %), and time (X_4: 30–90, min) on the GalA concentration in the extracts. As detailed above, the S/L ratio was fixed at 1/20 without temperature control. Table 1 shows the experiment set provided by the Factorial Design and the experimental GalA concentrations for each surfactant used.

A second-order polynomial model to predict the GalA concentration was developed through the experimental data for Tween80 (Eq. (4)), PEG4000 (Eq. (5)), and Saponin (Eq. (6)), considering only the significant operation parameters ($p < 0.05$). As seen in Eqs. (4)–(6), not all the equation terms are significant for predicting the GalA concentration.

Furthermore, regression coefficients are shown; R^2 is the adjustment considering all variables, and the adjusted R^2 considers only the significant variables ($p < 0.05$).

$$GalA(g/L) = 0.2264 + 0.8307X_1 - 0.0796X_2 + 0.0158X_3 + 0.0094X_4 - 0.1791X_1X_2$$
$$R^2 : 0.9430 \; ; \; R^2adjust : 0.9074 \quad (4)$$

$$GalA(g/L) = 1.1260 + 0.0043X_1 - 0.0042X_2 + 0.0032X_3 + 0.0052X_4 - 0.0440X_1X_2 + 0.0032X_1X_3$$
$$R^2 : \; 0.9112 \; ; \; R^2adjust : 0.8520 \quad (5)$$

$$GalA(g/L) = 1.0880 + 0.2455X_1 - 0.0125X_2 + 0.0019X_3 + 0.0076X_4 - 0.1869X_1X_2 + 0.0098X_1X_3$$
$$R^2 : \; 0.9722 \; ; \; R^2adjust : 0.9537 \quad (6)$$

Eqs. (4)–(6) show that with increasing amplitude, operation time, and concentration of the surfactant, a positive effect on the concentration of GalA was obtained. The surfactant concentration is the one that has a more significant effect, especially in the case of Tween80 and Saponin. However, in the case of the pH, the opposite effect occurs, indicating that higher concentrations of GalA were obtained under acidic conditions. On the other hand, if the interactions between parameters are considered, it can be seen that the interaction between the pH and surfactant concentration is negative, so the pH and surfactant concentration should not take high values in any case. On the contrary, the interaction between the surfactant concentration and the amplitude of US indicates that both values need to be high so that higher GalA concentrations can be reached. This behavior can also be observed in Fig. 2, which shows the GalA recovery concerning the initial GalA for each run of the experimental design. As shown in Fig. 2, there is a significant improvement in GalA recovery yields with time and amplitude. In addition, this Figure shows that the surfactants significantly improve the extraction yields of GalA compared to water (control). As can be seen in Fig. 2, the best yields are obtained with Tween80 (41 – 65 g GalA/100 g initial GalA) and Saponin (35 – 69 g GalA/100 g initial GalA), reaching up to 2-times higher than those obtained by PEG4000 (21 – 65 g GalA/100 g initial GalA).

The three surfactants tested obtained the same best extraction conditions: 4 g/L of surfactant, pH 1, amplitude: 90%, and 90 min. With these values, the predicted GalA concentrations were: 5.02 g/L for Tween80, 2.86 g/L for PEG4000, and 5.69 g/L for Saponin. These predicted values vary concerning the experimental ones of 8.7%, 0.2%, and 2.5%, respectively. The previous second-order polynomial equations (Eqs. (4)–(6)) can explain the interactions between the operation parameters and the GalA concentration for each surfactant used. Using higher-order models is unnecessary because the experimental and predicted data have less than 10% variation, and R^2 is over 90% (Kazemi et al., 2019; Maran et al., 2014).

3.2.2. Influence of operation parameters on Galacturonic Acid recovery

The operation parameters, such as surfactant concentration, pH, US amplitude, and operation time, were analyzed through Factorial Design. Depending on the operation conditions, the GalA concentration ranged from 1.38 to 5.84 g/L.

3.2.2.1. Effect of surfactant concentration.

It can be observed in Table 1 how the use of surfactants enhances the GalA concentration in the extracted liquid compared to the control runs. Fig. 2 shows that the surfactants had from 1.1 to 3.3 times higher GalA recoveries than the water (control) for each operating condition studied in the experimental design. In this case, Tween80 and Saponin are the surfactants with better results, obtaining between 5.5 and 5.9 g/L of GalA in the most extreme conditions. These results may be due to the structure of each surfactant and the balance between the hydrophobic and hydrophilic forces. This structure can form micelles to protect the GalA and the by-products from UAE degradation, increasing the final concentration, as indicated by Sharma et al. (2021) and Su et al. (2019).

3.2.2.2. Effect of the pH.

This parameter is essential for extracting GalA because, as shown in Table 1, under the same conditions of amplitude, time and surfactant, the GalA concentration was higher when pH was acid, reaching 2.3 times higher when using Tween80. These results are in concordance with previous studies, which concluded that a low pH (< 2) was necessary to increase GalA recovery (Chua et al., 2020; Minjares-Fuentes et al., 2014). If the pH chosen is acid (< 3), the insoluble pectin can hydrolyze and convert into soluble pectin, increasing the amount of GalA extracted (Shivamathi et al., 2019).

3.2.2.3. Effect of the amplitude.

Amplitude is another key parameter for GalA extraction. In this case, when surfactants were used, an increase in amplitude from 50% to 90% significantly improved the concentration of GalA (Table 1) and their respective yields (Fig. 2). For example, Tween80 has the most remarkable effect since it can increase up to 1.45 times the concentration of GalA with higher amplitudes, increasing from 3.82 g/L (50%) to 5.5 g/L (90%) when the time is 90 min and pH 1 (Table 1). When the ultrasound waves are of a high amplitude, the collapse of the cavitation bubble can occur nearby and cause microfractures in the solid (Vilkhu et al., 2008). By improving the cavitation, the surface area between the solid and the liquid increases, improving the diffusion of solutes, enabling a greater penetration of the liquid and thus improving the extraction of the compounds of interest (Cassiana Frohlich et al., 2022; Yusoff et al., 2022). These facts agree with

Table 2
Concentration and mass of galacturonic acid and oligosaccharides (galactose and arabinose) of the streams of the diafiltration/ultrafiltration process.

	Volume (mL)	Concentration (g/L)			Mass (mg)		
		GalA	Galactose	Arabinose	GalA	Galactose	Arabinose
US – L	200	5.50	1.08	6.91	1128	221	1382
DF – R	182	6.00	1.04	2.86	1092	190	521
DF – P	120	0.30	0.14	4.46	35	16	535
UF – R	70	11.76	1.97	3.51	823	138	246
UF – P	112	0.31	0.12	1.83	27	13	205

Note: Data shown as the mean value with less than 5% of relative error.

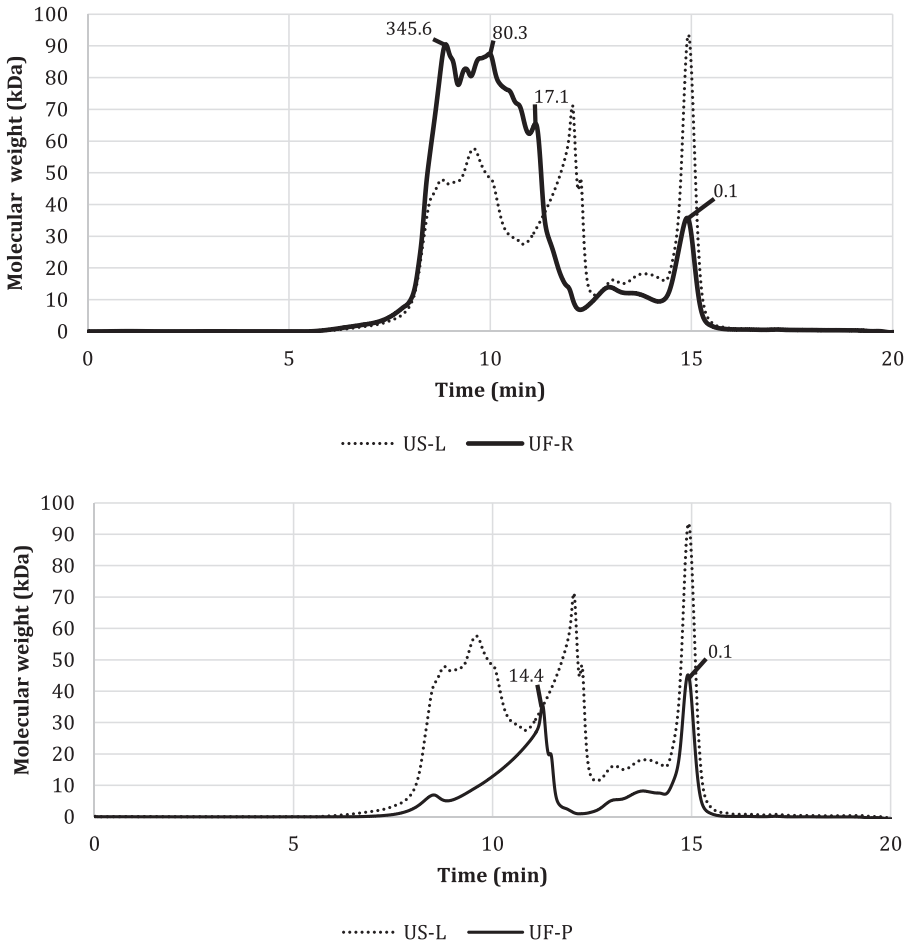

Fig. 3. Molecular weight distribution for extracted liquid (US-L), final retentate (UF-R) and final permeate (UF-P).

Jafarzadeh-Moghaddam et al. (2020), whose optimized amplitude was 96%. Furthermore, high yields need high amplitudes (Alves et al., 2022).

3.2.2.4. Effect of the operation time. The irradiation time is essential in UAE to obtain a high GalA concentration. The effect of time for UAE shows how, as time increases in all the cases studied, the recovery of GalA (Fig. 2) and its concentration (Table 1) also increase. This is more pronounced when UAE is performed in acid conditions and with surfactants. Eqs. 4–6 show that time has a similar effect in all cases and less than the other variables studied. By leaving ample extraction time, the cavitation bubbles can affect a more considerable amount of solid, decreasing its size and increasing the surface area. Two steps are necessary for UAE to perform this. The first step is the liquid penetration in the matrix, and the second is the transfer diffusion of soluble compounds (Selvakumar et al., 2021). Fick's law of diffusion states that improving the GalA recovery in a short time is caused by the equilibrium between solute concentrations in the solid matrix and the solvent (Mokrani and Madani, 2016). However, in 30 min, the two UAE stages may not have been performed thoroughly. On the contrary, at 90 min, the solid matrix can be entirely destroyed and decomposed, which causes the GalA recovery efficiency to improve, reaching almost 70% of GalA extracted (Fig. 2). Similarly, Minjares-Fuentes et al. (2014) and Pinheiro et al. (2008) concluded the necessity of using high extraction times to obtain higher efficacies, between 60 and 90 min.

The proposed Factorial Design for UAE is only a screening design that allows the most important variables (surfactant concentration, pH, amplitude and time) and their effects on the response variable (GalA concentration) to be determined. However, an extended design of the experiments would be necessary to allow the optimization of the variables and thus be able to establish an optimal value of the operating parameters.

3.3. Purification and concentration by diafiltration/ultrafiltration

The main objective of the membrane process is to purify, concentrate, and separate the GalA as a stage prior to its ethanol precipitation. The best liquid extracts obtained after the extraction step were selected for purification and concentration by the DF/UF membrane process. In this sense, Tween80 and Saponin provided the best results with a surfactant concentration of 4 g/L, pH 1, 90% amplitude, and 90 min. If significant differences between these runs are analyzed using a Tukey's test, the GalA recovery is not significantly different ($p > 0.05$). This means that the use of either of the two surfactants would provide the best GalA recovery values. However, from a technical point of view, Tween80 is better than Saponin due to the liquid extract recovery after the UAE process. Tween80 could recover around 55% of the initial aqueous solution, while Saponin could only recover up to 40% (data not shown). Due to this, the UAE with Tween80 needs fewer runs than the corresponding extraction with Saponin to reach the amount of liquid necessary to perform the DF/UF, significantly reducing operating costs.

The DF/UF configuration selected for GalA purification and concentration consists of a continuous DF process to purify US-L using distilled water, and a subsequent step of UF to concentrate the DF-R to obtain a GalA-rich liquid (UF-R) (Fig. 1). The composition of GalA, galactose, and arabinose in all the liquids of the DF/UF process is shown in Table 2.

As previously mentioned, US-L was treated by DF using a UF

Table 3
Characterization of final precipitated solid.

Characteristic	Unit	Value
Process yield	%	24.6
Final GalA recovery	g GalA of precipitated solid/100 g GalA of SBP	57
GalA	g/100 g of precipitated solid	39.1
Neutral Sugars	g/100 g of precipitated solid	3.3
DE	%	80.5
M_W	kDa	930

membrane with a nominal cut-off of 50 kDa to purify GalA from non-interest compounds with a small M_W. As expected, practically all the GalA (97% concerning the initial one) remains in the retentate (DF-R) due to its high M_W (Gerschenson et al., 2021). Regarding the concentration of galactose and arabinose, Table 2 shows how galactose is not affected by DF and remains in the DF-R as GalA. On the other hand, approximately 50% of the arabinose present in US-L goes to the permeate (DF-P), since it will be short-chain with a low M_W (<50 kDa).

After that, the DF-R was treated by UF with the same membrane to concentrate the purified GalA, which will later be precipitated with ethanol. The GalA present in the UF-R, concentrated almost 2 times, reached 11.76 g/L without significant losses of GalA in the permeate (UF-P). In the same way, galactose was principally found in the retentate (UF-R). However, as DF, arabinose is split between the retentate (UF-R) and the permeate (UF-P).

After the DF/UF process, the final goal is to obtain a liquid (UF-R) with a high amount of GalA and a low amount of by-products, such as oligo galactose and arabinose. In this way, the solid precipitated subsequently will have a high purity of GalA and, therefore, of pectin. Generally, the total GalA recovery in the final liquid (UF-R) was around 73 g GalA of UF-R/100 g GalA of US-L, and galactose and arabinose mass reduction was 37.6% and 82.2%, respectively.

Fig. 3 shows the M_W distribution of the UF-R and UF-P fraction compared to the initial liquid US-L, and the separation between M_W fractions is visible. As can be seen, in the UF-R (Fig. 3a), the high M_W fractions principally appeared in a range between 80.3 and 345.6 kDa and can be attributed to GalA (del Amo-Mateos et al., 2022). Although this liquid (UF-R) has some peaks representing M_W of 0.1 and 17.1 kDa, the separation by membranes, although efficient, has not been complete. This may be because these low M_W compounds may have adhered to the high M_W ones, and their separation by membranes has not been possible (Ramos-Andrés et al., 2019). On the other hand, Fig. 3b shows that only low M_W compounds (0.1–14.4 kDa) remain in the permeate (UF-P) below the cut-off weight of the membrane (50 kDa). The solid degradation by acid extraction provided these compounds, such by-products as formic and acetic acid or short-chain sugars (Gerschenson et al., 2021).

3.4. Analysis of precipitated pectin

Conventional ethanol precipitation was used for the purification of pectin from UF-R. The main characteristics of the precipitated solid are shown in Table 3. First, the overall pectin yield of the process was 24.6%. This value is consistent with previous investigations. For instance, the pectin yield was 25% in SBP pretreated with UAE (Chen et al., 2015). On the other hand, when SBP was treated with high-voltage electrical discharges, the process yield was 25.3% (Almohammed et al., 2017).

In addition, Table 3 details the solid composition, where the concentration of GalA was 39.1 g/100 g of precipitated solid, recovering 57 g GalA/100 g GalA of SBP in the precipitated solid. This outcome is consistent with the concentrations that del Amo-Mateos et al. (2020) and Chen et al. (2015) obtained by recovering GalA from SBP using microwave and ultrasound treatments, respectively, ranging between 23 and 60 g/100 g. Additionally, the recovery is similar to that found by Huang et al. (2018) because, in their study, recovery was up to 60% through a conventional extraction method. The total amount of sugars precipitated in the solid should also be considered. Its concentration is 3.3 g/100 g of precipitated solid, as shown in Table 3. Compared to other studies, this value is too low. For example, del Amo-Mateos et al. (2020) had a total sugar concentration of 27.2 g/100 g. This difference, however, can be explained by the use of membranes. Membranes can separate and purify the galacturonic acid and other main interest compounds of monosaccharides and other short-chain sugars, as seen in Table 2. So, it stands to reason that the precipitated solid has a low concentration of total sugars.

If other characteristics are compared, the solid M_W is 930 kDa, which is in line with that shown by Muñoz-Almagro et al. (2020) and Shivamathi et al. (2019), who reported M_W between 710 and 1175 kDa for pectin extracted by UAE for other agronomic wastes, such as custard apple peel. However, the M_W was possibly higher than expected because the Tween80 favored the GalA extraction and maintained the molecular structure (Su et al., 2019).

On the other hand, the DE of this solid is 80.5% (Table 3), being higher than those reported in the literature if only UAE is performed. For instance, Jafarzadeh-Moghaddam et al. (2021) reported a pectin DE of 63% after using UAE for treating SBP. Additionally, compared with other raw materials treated with UAE, Ezzati et al. (2020) reported that pectin obtained from sunflower had a DE of 34%, and Zaid et al. (2020) reported a DE of 56% for the extraction of *Hylocereus polyrhizus*. However, the DE of this study is similar to others that use membranes to concentrate and purify the extracted liquids, such as Jin et al. (2022),

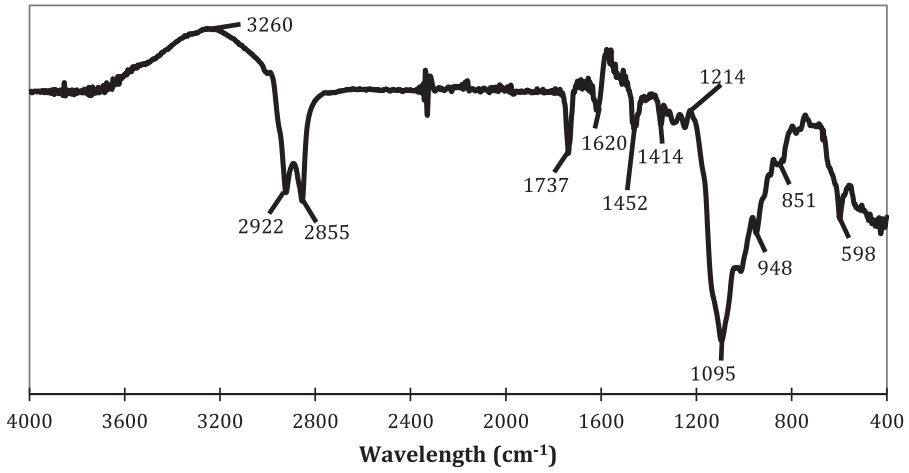

Fig. 4. FTIR spectra of precipitated solid obtained from final retentate (UF-R).

who reported a DE of 78% for their pectin using UF membranes.

Finally, the FTIR spectrum, in a range of 4000–400 cm^{-1} of precipitate, is shown in Fig. 4. This spectrum shows the distinct chemical groups to which the pectin belongs (Concha Olmos and Zúñiga Hansen, 2012). The bands between 3260 and 2855 correspond to the hydroxyl groups (-OH) and the C-H bonds in all the variants (-CH, $-CH_2$, and $-CH_3$), which may be due to the methyl ester groups present in pectin. Between 1800 and 1600 cm^{-1}, the type of pectin can be known because this band is related to DE. Since DE is greater than 50% (80.5%, Table 3), the pectin could be considered high-methoxyl pectin (Gerschenson et al., 2021; Marić et al., 2018). This type would be similar to the pectins obtained from SBP reported by del Amo-Mateos et al. (2022) and Concha-Olmos & Zúñiga Hansen (2012). Furthermore, the band 1737 cm^{-1} represents the esterified carboxyl groups, and the band 1620 cm^{-1} corresponds to non-esterified carboxyl groups. On the other hand, the bands presented between 1600 and 1500 cm^{-1} represented such groups as protein amide and aromatics rings. This indicates the presence of proteins and lignin in the precipitate (Concha Olmos and Zúñiga Hansen, 2012), which could explain the percentage of GalA in the precipitate (39.1 g/100 g of precipitated solid; Table 3). Moreover, the bands between 1452 and 1214 cm^{-1} could derive from the asymmetric vibrations of C-O-C and CH_3 bending (Santos et al., 2020). The spectrum between 1100 and 800 cm^{-1} is considered the region for carbohydrates (Jin et al., 2022). This part of the spectrum corroborates the presence of sugars in the pectin (3.3 g/100 g of precipitated solid; Table 3).

4. Conclusions

The integrated surfactant-mediated ultrasound-assisted extraction process followed by diafiltration/ultrafiltration is suitable for recovering and purifying pectin from SBP. The pectin yield for the global process was 24.6%. Moreover, the type of recovered pectin, high methoxyl pectin with high molecular weight (930 kDa), is indicative of good gelation properties with potential commercial applications. This work represents a significant advance in the research for the enhancement of industrial pectin production from sugar beet pulp. However, further research is required to optimize the integrated UAE/membranes process and to determine the functional properties of the final pectin-enriched product.

CRediT authorship contribution statement

Marina Fernández Delgado: Investigation, Methodology, Supervision, Writing – original draft. **Esther del Amo Mateos**: Investigation, Methodology, Visualization. **Mónica Coca**: Conceptualization, Formal analysis, Supervision. **Juan C. López-Linares**: Investigation, Methodology, Supervision. **María Teresa García-Cubero**: Conceptualization, Formal analysis, Supervisión. **Susana Lucas**: Conceptualization, Writing – review & editing, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

Abou-Elseoud, W.S., Hassan, E.A., Hassan, M.L., 2021. Extraction of pectin from sugar beet pulp by enzymatic and ultrasound-assisted treatments. Carbohydr. Polym. Technol. Appl. 2, 100042 https://doi.org/10.1016/j.carpta.2021.100042.

Adetunji, L.R., Adekunle, A., Orsat, V., Raghavan, V., 2017. Advances in the pectin production process using novel extraction techniques: A review. Food Hydrocoll. 62, 239–250. https://doi.org/10.1016/j.foodhyd.2016.08.015.

Ai, C., Meng, H., Lin, J., Zhang, T., Guo, X., 2020. Combined membrane filtration and alcohol-precipitation of alkaline soluble polysaccharides from sugar beet pulp: Comparision of compositional, macromolecular, and emulsifying properties. Food Hydrocoll. 109, 106049 https://doi.org/10.1016/j.foodhyd.2020.106049.

Almohammed, F., Koubaa, M., Khelfa, A., Nakaya, M., Mhemdi, H., Vorobiev, E., 2017. Pectin recovery from sugar beet pulp enhanced by high-voltage electrical discharges. Food Bioprod. Process. 103, 95–103. https://doi.org/10.1016/j.fbp.2017.03.005.

Alves, T.P., Triques, C.C., Palsikowski, P.A., da Silva, C., Fiorese, M.L., da Silva, E.A., Fagundes-Klen, M.R., 2022. Improved extraction of bioactive compounds from Monteverdia aquifolia leaves by pressurized-liquid and ultrasound-assisted extraction: Yield and chemical composition. J. Supercrit. Fluids 181, 105468. https://doi.org/10.1016/j.supflu.2021.105468.

del Amo-Mateos, E., López-Linares, J.C., García-Cubero, M.T., Lucas, S., Coca, M., 2022. Green biorefinery for sugar beet pulp valorisation: Microwave hydrothermal processing for pectooligosaccharides recovery and biobutanol production. Ind. Crops Prod. 184, 115060 https://doi.org/10.1016/j.indcrop.2022.115060.

Cassiana Frohlich, P., Andressa Santos, K., Din Mahmud Hasan, S., Antônio da Silva, E., 2022. Evaluation of the ethanolic ultrasound-assisted extraction from clove (Syzygium aromaticum) leaves and chemical characterization of the extracts. Food Chem. 373, 131351 https://doi.org/10.1016/j.foodchem.2021.131351.

Chen, H.M., Fu, Z., Luo, Z.G., 2015. Properties and extraction of pectin-enriched materials from sugar beet pulp by ultrasonic-assisted treatment combined with subcritical water, 302-210 Food Chem. 168. https://doi.org/10.1016/j.foodchem.2014.07.078.

Chua, B.L., Tang, S.F., Ali, A., Chow, Y.H., 2020. Optimisation of pectin production from dragon fruit peels waste: Drying, extraction and characterisation studies. SN Appl. Sci. 2. https://doi.org/10.1007/s42452-020-2415-y.

Ciriminna, R., Chavarría-Hernández, N., Inés Rodríguez Hernández, A., Pagliaro, M., 2015. Pectin: A new perspective from the biorefinery standpoint. Biofuels, Bioprod. Bioref. 9, 368–377. https://doi.org/10.1002/bbb.1551.

Concha Olmos, J., Zúñiga Hansen, M.E., 2012. Enzymatic depolymerization of sugar beet pulp: Production and characterization of pectin and pectic-oligosaccharides as a potential source for functional carbohydrates. Chem. Eng. J. 192, 29–36. https://doi.org/10.1016/j.cej.2012.03.085.

Cui, J., Zhao, C., Feng, L., Han, Y., Du, H., Xiao, H., Zheng, J., 2021. Pectins from fruits: Relationships between extraction methods, structural characteristics, and functional properties. Trends Food Sci. Technol. 110, 39–54. https://doi.org/10.1016/j.tifs.2021.01.077.

Das, I., Arora, A., 2021. Kinetics and mechanistic models of solid-liquid extraction of pectin using advance green techniques- a review. Food Hydrocoll. 120, 106931 https://doi.org/10.1016/j.foodhyd.2021.106931.

Ezzati, S., Ayaseh, A., Ghanbarzadeh, B., Heshmati, M.K., 2020. Pectin from sunflower by-product: Optimization of ultrasound-assisted extraction, characterization, and functional analysis. Int. J. Biol. Macromol. 165, 776–786. https://doi.org/10.1016/j.ijbiomac.2020.09.205.

Gerschenson, L.N., Fissore, E.N., Rojas, A.M., Idrovo Encalada, A.M., Zukowski, E.F., Higuera Coelho, R.A., 2021. Pectins obtained by ultrasound from agroindustrial by-products. Food Hydrocoll. 118, 106799 https://doi.org/10.1016/j.foodhyd.2021.106799.

Huang, X., Li, D., Wang, L.J., 2018. Effect of particle size of sugar beet pulp on the extraction and property of pectin. H. Food Eng. 218, 44–49. https://doi.org/10.1016/j.jfoodeng.2017.09.001.

Jafarzadeh-Moghaddam, M., Shaddel, R., Peighambardoust, S.H., 2020. Sugar beet pectin extracted by ultrasound or conventional heating: A comparison. J. Food Sci. Technol. 58, 2567–2578. https://doi.org/10.1007/s13197-020-04763-1.

Jin, L., Zuo, F., Gao, Y., Sui, S., Zhang, D., 2022. Purification of pectin by ultrafiltration in combination with sodium citrate. J. Food Eng. 335, 111158 https://doi.org/10.1016/j.jfoodeng.2022.111158.

Joanna, B., Michal, B., Piotr, D., Agnieszka, W., Dorota, K., Izabela, W., 2018. Sugar beet pulp as a source of valuable biotechnological products. In: Advances in Biotechnology for Food Industry. Elsevier, pp. 359–392. https://doi.org/10.1016/b978-0-12-811443-8.00013-x.

Jovanovic-Malinovska, R., Kuzmanova, S., Winkelhausen, E., 2015. Application of ultrasound for enhanced extraction of prebiotic oligosaccharides from selected fruits and vegetables. Ultrason. Sonochem. 22, 446–453. https://doi.org/10.1016/j.ultsonch.2014.07.016.

Karbuz, P., Tugrul, N., 2020. Microwave and ultrasound assisted extraction of pectin from various fruits peel. J. Food Sci. Technol. 58, 641–650. https://doi.org/10.1007/s13197-020-04578-0.

Kazemi, M., Khodaiyan, F., Hosseini, S.S., 2019. Eggplant peel as a high potential source of high methylated pectin: Ultrasonic extraction optimization and characterization. LWT 105, 182–189. https://doi.org/10.1016/j.lwt.2019.01.060.

Li, D., Jia, X., Wei, Z., Liu, Z., 2012. Box–Behnken experimental design for investigation of microwave-assisted extracted sugar beet pulp pectin. Carbohydr. Polym. 88, 342–346. https://doi.org/10.1016/j.carbpol.2011.12.017.

López-Linares, J.C., Lucas, S., García-Cubero, M.T., Jiménez, J.J., Coca, M., 2020. A biorefinery based on brewer's spent grains: Arabinoxylans recovery by microwave assisted pretreatment integrated with butanol production. Ind. Crops Prod. 158, 113044 https://doi.org/10.1016/j.indcrop.2020.113044.

Mao, G., Wu, D., Wei, C., Tao, W., Ye, X., Linhardt, R.J., Orfila, C., Chen, S., 2019. Reconsidering conventional and innovative methods for pectin extraction from fruit and vegetable waste: Targeting rhamnogalacturonan I. Trends Food Sci. Technol. 94, 65–78. https://doi.org/10.1016/j.tifs.2019.11.001.

Maran, J.P., Priya, B., Manikandan, S., 2014. Modeling and optimization of supercritical fluid extraction of anthocyanin and phenolic compounds from Syzygium cumini fruit pulp. J. Food Sci. Technol. 51, 1938–1946. https://doi.org/10.1007/s13197-013-1237-y.

Maran, J.P., Priya, B., Al-Dhabi, N.A., Ponmurugan, K., Moorthy, I.G., Sivarajasekar, N., 2017. Ultrasound assisted citric acid mediated pectin extraction from industrial waste of Musa balbisiana. Ultrason. Sonochem. 35, 204–209. https://doi.org/10.1016/j.ultsonch.2016.09.019.

Marić, M., Grassino, A.N., Zhu, Z., Barba, F.J., Brnčić, M., Rimac Brnčić, S., 2018. 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. 76, 28–37. https://doi.org/10.1016/j.tifs.2018.03.022.

Martínez, M., Gullón, B., Schols, H.A., Alonso, J.L., Parajó, J.C., 2009. Assessment of the production of oligomeric compounds from sugar beet pulp. Trends Food Sci. Technol. 48, 4681–4687. https://doi.org/10.1021/ie8017753.

Mata, Y.N., Blázquez, M.L., Ballester, A., González, F., Muñoz, J.A., 2009. Sugar-beet pulp pectin gels as biosorbent for heavy metals: Preparation and determination of biosorption and desorption characteristics. Chem. Eng. J. 150, 289–301. https://doi.org/10.1016/j.cej.2009.01.001.

Melton, L.D., Smith, B.G., 2001. Determination of the uronic acid content of plant cell walls using a colorimetric assay. Curr. Protoc. Food Anal. Chem. 00. https://doi.org/10.1002/0471142913.fae0303s00.

Minjares-Fuentes, R., Femenia, A., Garau, M.C., Meza-Velázquez, J.A., Simal, S., Rosselló, C., 2014. Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach. Carbohydr. Polym. 106, 179–189. https://doi.org/10.1016/j.carbpol.2014.02.013.

Mokrani, A., Madani, K., 2016. Effect of solvent, time and temperature on the extraction of phenolic compounds and antioxidant capacity of peach (Prunus persica L.) fruit. Sep. Purif. Technol. 162, 68–76. https://doi.org/10.1016/j.seppur.2016.01.043.

Muñoz-Almagro, N., Prodanov, M., Wilde, P.J., Villamiel, M., Montilla, A., 2020. Obtainment and characterisation of pectin from sunflower heads purified by membrane separation techniques. Food Chem. 318, 126476 https://doi.org/10.1016/j.foodchem.2020.126476.

Pilgrim, G.W., Walter, R.H., Oakenfull, D.G., 1991. Jams, Jellies, and Preserves. In: The Chemistry and Technology of Pectin. Elsevier, pp. 23–50. https://doi.org/10.1016/b978-0-08-092644-5.50007-8.

Pinheiro, E.R., Silva, I.M.D.A., Gonzaga, L.V., Amante, E.R., Teófilo, R.F., Ferreira, M.M. C., Amboni, R.D.M.C., 2008. Optimization of extraction of high-ester pectin from passion fruit peel (Passiflora edulis flavicarpa) with citric acid by using response surface methodology. Bioresour. Technol. 99, 5561–5566. https://doi.org/10.1016/j.biortech.2007.10.058.

Ramos-Andrés, M., Andrés-Iglesias, C., García-Serna, J., 2019. Production of molecular weight fractionated hemicelluloses hydrolyzates from spent coffee grounds combining hydrothermal extraction and a multistep ultrafiltration/diafiltration. Bioresour. Technol. 292, 121940 https://doi.org/10.1016/j.biortech.2019.121940.

Santos, E.E., Amaro, R.C., Bustamante, C.C.C., Guerra, M.H.A., Soares, L.C., Froes, R.E.S., 2020. Extraction of pectin from agroindustrial residue with an ecofriendly solvent: Use of FTIR and chemometrics to differentiate pectins according to degree of methyl esterification. Food Hydrocoll. 107, 105921 https://doi.org/10.1016/j.foodhyd.2020.105921.

Selvakumar, P., Karthik, V., Kumar, P.S., Asaithambi, P., Kavitha, S., Sivashanmugam, P., 2021. Enhancement of ultrasound assisted aqueous extraction of polyphenols from waste fruit peel using dimethyl sulfoxide as surfactant: Assessment of kinetic models. Chemosphere 263, 128071. https://doi.org/10.1016/j.chemosphere.2020.128071.

Sharma, P., Gautam, K., Pandey, A.K., Gaur, V.K., Farooqui, A., Younis, K., 2021. Pectin. In: Biomass, Biofuels, Biochemicals. Elsevier, pp. 101–128. https://doi.org/10.1016/b978-0-12-821888-4.00020-4.

Shivamathi, C.S., Moorthy, I.G., Kumar, R.V., Soosai, M.R., Maran, J.P., Kumar, R.S., Varalakshmi, P., 2019. Optimization of ultrasound assisted extraction of pectin from custard apple peel: Potential and new source. Carbohydr. Polym. 225, 115240 https://doi.org/10.1016/j.carbpol.2019.115240.

Su, D.-L., Li, P.-J., Quek, S.Y., Huang, Z.-Q., Yuan, Y.-J., Li, G.-Y., Shan, Y., 2019. Efficient extraction and characterization of pectin from orange peel by a combined surfactant and microwave assisted process. Food Chem. 286, 1–7. https://doi.org/10.1016/j.foodchem.2019.01.200.

Thuvander, J., Oinonen, P., Jönsson, A.-S., 2016. Enzymatic treatment of hemicelluloses and lignin isolated from thermomechanical pulp mill process water. Chem. Eng. J. 296, 141–145. https://doi.org/10.1016/j.cej.2016.03.087.

Usmani, Z., Sharma, M., Diwan, D., Tripathi, M., Whale, E., Jayakody, L.N., Moreau, B., Thakur, V.K., Tuohy, M., Gupta, V.K., 2022. Valorization of sugar beet pulp to value-added products: A review. Bioresour. Technol. 346, 126580 https://doi.org/10.1016/j.biortech.2021.126580.

Vilkhu, K., Mawson, R., Simons, L., Bates, D., 2008. Applications and opportunities for ultrasound assisted extraction in the food industry — A review. Innov. Food Sci. Emerg. Technol. 9, 161–169. https://doi.org/10.1016/j.ifset.2007.04.014.

Voragen, A.G.J., Coenen, G.-J., Verhoef, R.P., Schols, H.A., 2009. Pectin, a versatile polysaccharide present in plant cell walls. Struct. Chem. 20, 263–275. https://doi.org/10.1007/s11224-009-9442-z.

Wang, M., Huang, B., Fan, C., Zhao, K., Hu, H., Xu, X., Pan, S., Liu, F., 2016. Characterization and functional properties of mango peel pectin extracted by ultrasound assisted citric acid. Int. J. Biol. Macromol. 91, 794–803. https://doi.org/10.1016/j.ijbiomac.2016.06.011.

Wang, Z., Pan, H., Xu, J., Chang, Y., Liu, C., Zhang, Y., Yang, H., Duan, C., Huang, J., Fu, Y., 2022. A sustainable and integrated natural surfactant mediated microwave-assisted extraction technique enhances the extraction of phytochemicals from plants. Ind. Crops Prod. 184, 115043 https://doi.org/10.1016/j.indcrop.2022.115043.

Yusoff, I.M., Mat Taher, Z., Rahmat, Z., Chua, L.S., 2022. A review of ultrasound-assisted extraction for plant bioactive compounds: Phenolics, flavonoids, thymols, saponins and proteins. Food Res. Int. 157, 111268 https://doi.org/10.1016/j.foodres.2022.111268.

Zaid, R.M., Mishra, P., Siti Noredyani, A.R., Tabassum, S., Ab Wahid, Z., Mimi Sakinah, A.M., 2020. Proximate characteristics and statistical optimization of ultrasound-assisted extraction of high-methoxyl-pectin from Hylocereus polyrhizus peels. Food Bioprod. Process. 123, 134–149. https://doi.org/10.1016/j.fbp.2020.06.011.