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## Article

# The Effect of Ultrasound Pretreatment on Pectin Extraction from Watermelon Rind Using Microwave-Assisted Extraction

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**Abstract:** Ultrasound pretreatment and microwave-assisted extraction (MAE) techniques were used to extract pectin from watermelon rind. This work investigated the effect of the power and time of pretreatment on the yield and characteristics of the extracted pectin. Imaging studies showed that ultrasonic pretreatment changed the morphology of the raw powder, leading to a greater porosity, and facilitated the extraction process. The highest extraction yield of pectin was 19.08% (*w/w*), for 5 min of extraction at 500 W ultrasonic pretreatment. Ultrasonic pretreatment affected the degree of esterification and the galacturonic acid content of pectin. The galacturonic acid content of all samples was higher than 69.0%. Extraction pretreatment reduced the surface tension value of the aqueous solution of pectin and affected the foam capacity of pectin and its monosaccharidic composition. The ultrasonic pretreatment caused significant changes in pectin characteristics, such as intrinsic viscosity, viscosity average, molecular weight, ratio of homogalacturonan to rhamnogalacturonan and the water holding capacity. In addition, kinetic studies showed that a kinetic empirical model adequately describes the effect of ultrasonic pretreatment on foam stability and its half-time.

**Keywords:** pectin; watermelon rind; microwave-assisted extraction; ultrasonication pretreatment



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## 1. Introduction

Watermelon (*Citrullus lanatus*) is a popular fruit with great economic and nutritional value. It is adaptable to most tropical and subtropical regions, such as Iran. According to the statistical database of the Food and Agriculture Organization, China is the largest producer of watermelon, with a production of 60.25 million tons, followed by Turkey (3.47 million tons), India (3.25 million tons) and Iran (1.25 million tons) [1]. The flesh (68%), seeds (2%) and rinds (30%) are three major components of watermelon biomass [2]. Watermelon rind as agro-waste is usually discarded, leading to the generation of environmental waste and loss of plant materials. Nevertheless, it can be used as a source of citrulline as an antioxidant, dietary fibers and phenolic compounds [3], and/or it can be considered a valuable source of pectin because it contains a significant amount of this polysaccharide.

Pectin is an acidic heteropolysaccharide mainly composed of D-galacturonic acid (D-GalA). Pectins consist of three sequences: homogalacturonan (HG), rhamnogalacturonans type I (RG-I) and rhamnogalacturonans type II (RG-II). The RG-I region compositions depend on the plant source, and may contain arabinan, galactan and/or arabinogalactan side chains [4]. RG-II has a very complex structure, with a HG backbone and very complex side chains attached to the GalA residues [5]. Pectin is widely used in the food industry as a gelling and thickening agent and stabilizer, and a fat replacer in jams, dairy products, fruit juices and emulsified meat products [6]. Commercially, pectin is extracted from citrus peels (85%), apple pomace (14%) and sugar beet (less than 1%) [4]. This extraction

process uses acidified water and high temperatures, which is associated with environmental concerns and the generation of waste water from acidic extractions [7]. Moreover, this traditional extraction method has a low efficiency and is time consuming. Therefore, many novel or green extraction methods have been introduced to reduce waste waters and improve the extraction yield. These methods include the application of power ultrasound in directed modification and destruction of pectin, pulsating hydrodynamic function, microwave-assisted extraction, ultrasound-assisted extraction (UAE) and supercritical fluid extraction [8]. Generally, any process with a lower temperature and shorter extraction time is supposed to protect the structure and functionality of the extracted samples. Pectin characteristics, such as molecular weight, the degree of esterification (DE), neutral sugar and galacturonic acid composition, could be affected by the source of feed and the extraction method, and influence the functional properties of this polysaccharide [9]. Previously, MAE was used to extract pectin from lime peels, passion fruit peels and banana peels [8,10]. Microwave heating involves electromagnetic radiation in the microwave frequency range, to produce thermal energy in the solvent. Microwave heating rapidly increases the temperature of the samples and the efficiency of the extraction process. This method has been shown to be more effective than the conventional method for extracting pectin with a better polysaccharide quality [8], lower production cost, shorter processing time and less solvent used.

Foam is defined as a complex of air/gas bubbles (dispersed phase) trapped in the fluid medium (continuous phase) [7]. This type of foam is used in food, cleaning, surface treatment, cosmetics and as a reducing pollution agent [11]. Some hydrocolloids can be used in cosmetic products, such as shampoo, due to their hydrophilicity and stabilizing capacity [6]. Foam products are widely used in the cosmetic industry, including foam-like shampoos, conditioners, aerosol hair dyeing and hair colourants. Cosmetic foam can include complex formulations with surfactants, lubricating agents, polymers, keratolytic and germicide agents [7]. Biopolymers that are mostly used for cosmetic applications include starch, guar gum, xanthan gum, carrageenan, pectin, alginate, gelatin and agar. In cosmetic formulations, these natural polymers are used to promote a variety of functions, such as rheology modifiers, thickeners, foam stabilizers, emulsifiers, conditioning and film formers [8]. Pectin is used as an emulsifier or thickener in cosmetic preparations, i.e., creams and lotions [9]. In the cosmetic and pharmaceutical industries, it is also used as a natural texturizer, thickener and stabilizer, lotion and tonic [3].

Materials with a high foam capacity (FC) and foam stability (FS) could be used in the aerated food product formulation [7]. These dispersion systems are thermo-dynamically unstable. Stability is an important property of food foam, because consumer perception of quality is influenced by the appearance of a product. FS depends on the properties of the foaming agent in the system. Studies reported that the most important surface-active components in foods are proteins and low-molecular-weight emulsifiers [11–13]. As mentioned earlier, pectin is used as a gelling and stabilizing agent; however, we can consider other applications for pectin, such as foaming activity. Therefore, the appropriate extraction method for biopolymers, such as pectin, is selected based on the yield, as well as the desired functional properties.

We hypothesized that pectin extraction using microwave-assisted extraction could be improved using ultrasonic pretreatment and that ultrasonic could (favourably or unfavourably) affect the structure. Therefore, the objective of this study was to investigate the effect of ultrasonic pretreatment under microwave heating on the extraction yield and structure of pectin from watermelon rind. It aims also to evaluate the effect of this method on techno-functional characteristics of extracted pectin, such as foaming properties, water holding capacity (WHC) and colour parameters. These parameters are essential for pectin utilisation in food, pharmaceuticals and cosmetics.

## 2. Materials and Methods

### 2.1. Sample Preparation

Watermelon (*Citrullus lanatus*) was obtained from a local market in Isfahan, Iran. Watermelon rinds were removed from the flesh and blanched by saturated steam for 28 min, and washed in flowing water to remove free sugar. The blanched slices were dried in a hot-air oven at 50 °C for approximately 24 h, to reach a constant weight. The dried watermelon rinds were powdered by a grinder (KG-250 model, Korea) and passed through a 60-mesh sieve. The fine powder was transferred to an airtight bag and stored in a dry place until further use.

### 2.2. Pectin Extraction

#### Ultrasonic Pretreatment

Ultrasonic pretreatment was performed using an ultrasonic probe system (10 mm horn tip) with a frequency of 20 kHz (topsonic-UHP-400, Iran, Tehran). The watermelon rind powder was exposed to distilled water (150 mL) at a ratio of 1:40. Ultrasonic pretreatment was carried out at two ultrasonic powers, i.e., 150 and 300 W, for 10 and 20 min. The pulse duration (5 s) and pulse interval (5 s ON/5 s OFF) were kept constant during the pretreatment.

### 2.3. Microwave-Assisted Extraction

An experimental microwave oven (Panasonic, model NN-SM330W, China) was used for the extraction process. The pH of the extraction effluent was adjusted to 1.5 adding HCl. The temperature of each extraction treatment was set at 90 °C ± 1.5 °C. The net microwave exposure times were 3, 4, and 5 min at 500 W. After cooling the glass container, the mixture was filtered through a cloth filter and the supernatant was centrifuged at 7500 rpm (5280 × g at 4 °C for 10 min). The pectin was precipitated at 4 °C by adding ethanol 96% (v/v), at a 1:1 (v/v) ratio during the 24 h period. Wet pectin was collected by centrifugation at 10,000 rpm (9390 × g at 4 °C in 10 min). The polymer was dried using a vacuum oven at 50 °C, until a constant weight was obtained. The yield of the watermelon rind pectin (WRP) was calculated as follows [14]:

$$\text{Pectin Extraction Yield (\%)} = \frac{\text{WRP (g)}}{\text{dried watermelon rind powder (g)}} \times 100 \quad (1)$$

### 2.4. Morphology of Watermelon Rind Powder

The morphology of watermelon rind powder before and after ultrasonication was examined via SEM (XI30 model-Philips). The micrographs of the samples, coated with gold, were taken at an accelerating voltage of 10 kV. The obtained micrographs (scale of 50 μm) were analysed by the ImageJ software (National Institutes of Health, Bethesda, MD, USA). The thresholding tools (Otsu and the manual) were used for image segmentation [15], then the porosity and pore size were measured.

### 2.5. Pectin Characteristics

A titration method was used to determine the degree of esterification (DE) value of the samples, as described by Wai et al. [16]. The carbazole-sulfuric acid method, introduced by Taylor in 1993, was used to measure the GalA content of pectin [17]. The protein content of WRP was estimated using the Kjeldahl method (N × 6.25) [7].

The WHC of samples was evaluated in triplicate at room temperature. The samples (0.3 g) were added to 10 mL of distilled water, the mixture was vortexed for 1 min and incubated for 30 min before centrifugation at 7500 rpm (5280 × g) for 10 min at 25 °C. The pellet was weighted after the supernatant removal. The WHC value of samples was expressed as grams of water retained by 1 g pectin [18].

### 2.6. Monosaccharide Composition of Pectin

The monosaccharide composition was determined using a high-performance anion-exchange chromatography-pulsed amperometric detector (HPAEC-PAD). WRP monosaccharide composition was analyzed by HPAEC with an ICS 3000 system (Dionex Corporation, Sunnyvale, CA, USA), equipped with pulsed amperometric detection (PAD) and an AS 50 autosampler. Twenty-five  $\mu\text{L}$  of samples was injected in the system and eluted into a pre-column CarboPac PA1-column ( $4 \times 50$  mm) and an analytical CarboPac PA1-column ( $4 \times 250$  mm), equilibrated for 15 min with 18 mM NaOH. Data were collected and analysed using the Dionex Chromeleon 6.80 software (Sunnyvale, CA, USA). L-rhamnose, L-arabinose, D-xylose, D-galactose and D-GalA were used as standards.

### 2.7. Surface Tension and Foaming Properties

The equilibrium surface tension was measured by the du Nouy ring method, using a Kruss K20 Easy Dyne Tensiometer. For this test, the sample solutions (1%  $w/w$ ) were prepared by dissolving 0.3 g of WRP in 30 mL distilled water at 60 °C. Sample solutions were kept at 4 °C for 24 h before the measurements. Fresh Milli-Q water surface tension, 71.0 mN/m, was used as a reference.

The foaming properties of WRP solutions were expressed as the foam capacity (FC) and foam stability (FS), and were determined by the method explained by Kazemi et al., with slight modifications [19]. The foam was prepared by the homogenization of the pectin solution (1%  $w/v$ ) at 11,000 rpm for 1 min at 25 °C, using T10 basic Ultraturax (IKA). The foam volume was recorded every 5 min to 35 min using a digital camera, and then analysed by the ImageJ software 1.46r. The FC and FS were determined by measuring the ratio of foam height to total height immediately after foam formation and after 35 min, respectively.

Regarding the kinetics modelling of foam stability, the empirical models (Equation (2)) were used to correlate experimental data of the foam ratio, which was expressed as the foam height to the total height ratio from  $t = 0$  to  $t = 35$  min. The half-time ( $t_{1/2}$ ) that represents the stability of the foam was calculated as shown below (Equation (3)). The kinetics parameter,  $k_1$  (foam volume per minute), and constant term,  $V_r$ , were determined through non-linear regression using the curve fitting toolbox of MATLAB R2008a (The Mathworks, Natick, MA, USA).

$$V_r = V_{r0} - k_1 t^{0.1} \quad (2)$$

$$t_{1/2} = \left( \frac{V_{r0}}{2 \times k_s} \right)^{10} \quad (3)$$

### 2.8. Intrinsic Viscosity and Viscosity Average Molecular Weight

The intrinsic viscosity of the dilute pectin solutions (5 mg/mL) was measured with a capillary viscometer (Ostwald, haian Co., Shiraz, Iran) immersed in a water bath at 25 °C. Pectin samples were dissolved in 0.2 mol  $\text{L}^{-1}$  NaCl solution. The intrinsic viscosity was measured using the equations described below.  $t_0$  and  $t_s$  were noted as the time required by the sample solution and solvent to flow out of the capillary, and  $[\eta]$  was the intrinsic viscosity [20].

$$[\eta] = \frac{1}{c} \sqrt{2 \left( \frac{t_s - t_0}{t_0} - \ln \left( \frac{t_s}{t_0} \right) \right)} \quad (4)$$

The Mark–Houwink equation was used to determine the relationship between molecular mass and intrinsic viscosity. The  $a$  and  $k$  values were selected from previous research, in which the intrinsic viscosity was measured at various molecular weights. “ $\alpha$ ” was measured from the slope of the log–log graph between intrinsic viscosity and molecular weights, and “ $k$ ” was from the intercept of the graph. The  $\alpha$  and  $K$  values were 1.34 and  $1.4 \times 10^{-6}$ , respectively [21].

$$[\eta] = k \times M_W^\alpha \quad (5)$$

### 2.9. Colour

The colour parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) of the WRP were analysed with a colourimeter (ZE-6000 model, Nippon, Japan) based on the CIE LAB colour system, using illuminant D65 and a  $10^\circ$  observer angle. The total colour differences were calculated using Equation (6).  $L_0$ ,  $a_0$  and  $b_0$  were the colour properties of the sample obtained from the MAE.

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (6)$$

### 2.10. Statistical Analysis

The experimental data were analysed using the statistical software SPSS. The significant difference between treatments was determined using an ANOVA and a Tukey–Kramer post hoc test, at  $p < 0.05$ .

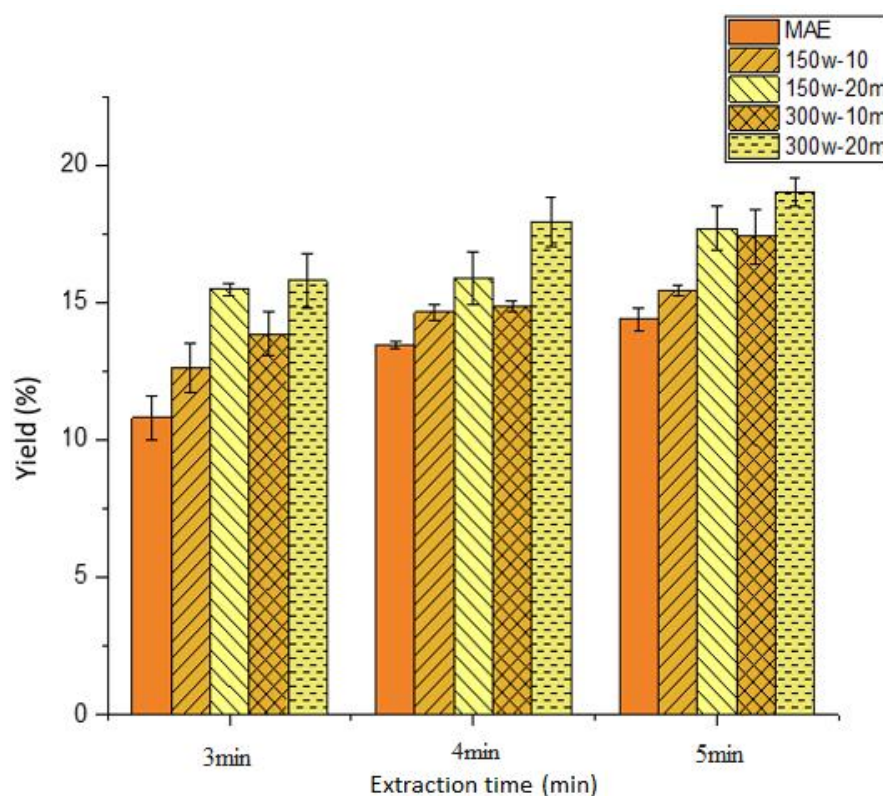
## 3. Results and Discussion

### 3.1. Extraction Yield

The yield values of the extracted pectin, using MAE and MAE with ultrasonic pretreatment (MAUPE), are shown in Figure 1. The yield of extracted pectin significantly increased from  $10.85 \pm 0.7$  to  $14.4 \pm 0.3\%$ , when the duration of microwave heating (MAE) increased from 3 to 5 min. In the extraction process, microwave heating creates significant pressure inside a watermelon rind and it can alter the raw material physical properties, such as porosity, and improve the penetration of the solvent into the tissue, energy transfer to the mixture and the release of target compounds in the extraction medium [22]. According to the obtained results and analysis of variance, MAUPE can significantly increase the extraction yield, and the pretreatment time showed a greater effect on pectin yield than ultrasonic power. The maximum yield of pectin ( $19.08\% \pm 0.5$ ) was achieved using pretreatment for 20 min and a power of 300 W before the microwave-assisted extraction (at 500 W for 5 min). Jiang et al. (2012) showed that the extraction yield of pectin from watermelon rind was 19.6% by MAE at a power of 500 W, pH of 1.5 and extraction time of 7 min [23]. A similar magnitude of extraction yield (19.75%) was reported by Korish et al. using the conventional method to extract pectin from watermelon rind (pH 2,  $80^\circ\text{C}$  for 60 min) [4]. Moreover, Jiang et al. optimized the conventional extraction from watermelon rind, with a temperature of  $90^\circ\text{C}$ , a pH of 1.0, a time of 150 min and an SLR of 1:20. The extraction yield was 17.6% [5], which was lower than the yield of pectin extraction with the ultrasonic pretreatment. Therefore, pretreatment could reduce the extraction time.

The extraction rate can be assessed by dividing the yield value by time. In the early stages (from  $t = 0$  to  $t = 3$  min) of pectin extraction, the process rate was higher than in later ones. This is probably due to the lower viscosity of extraction media at this stage compared to the latter. Ultrasonic pretreatment (300 W for 20 min) increased the extraction rate from 2.88 to 3.8% per minute.

This non-thermal pretreatment affects the cell wall of the raw material, and promotes the interaction between the substrate and the solvent by generating cavitation in the mixed solution [5], finally increasing the mass transfer coefficient [24]. The cavitation effect includes a high temperature and local pressure, turbulence in the fluid, high stress near the air bubble and microjets near the solid surface, which affect the raw material and result in the facility of the extraction process [25]. Therefore, MAUPE can be used to improve the pectin extraction yield. Compared to other sources of pectin, such as passion fruit and banana, the pectin yields were 14.8 and 11.63%, respectively [26,27], and the watermelon rind showed a higher extraction yield of pectin.



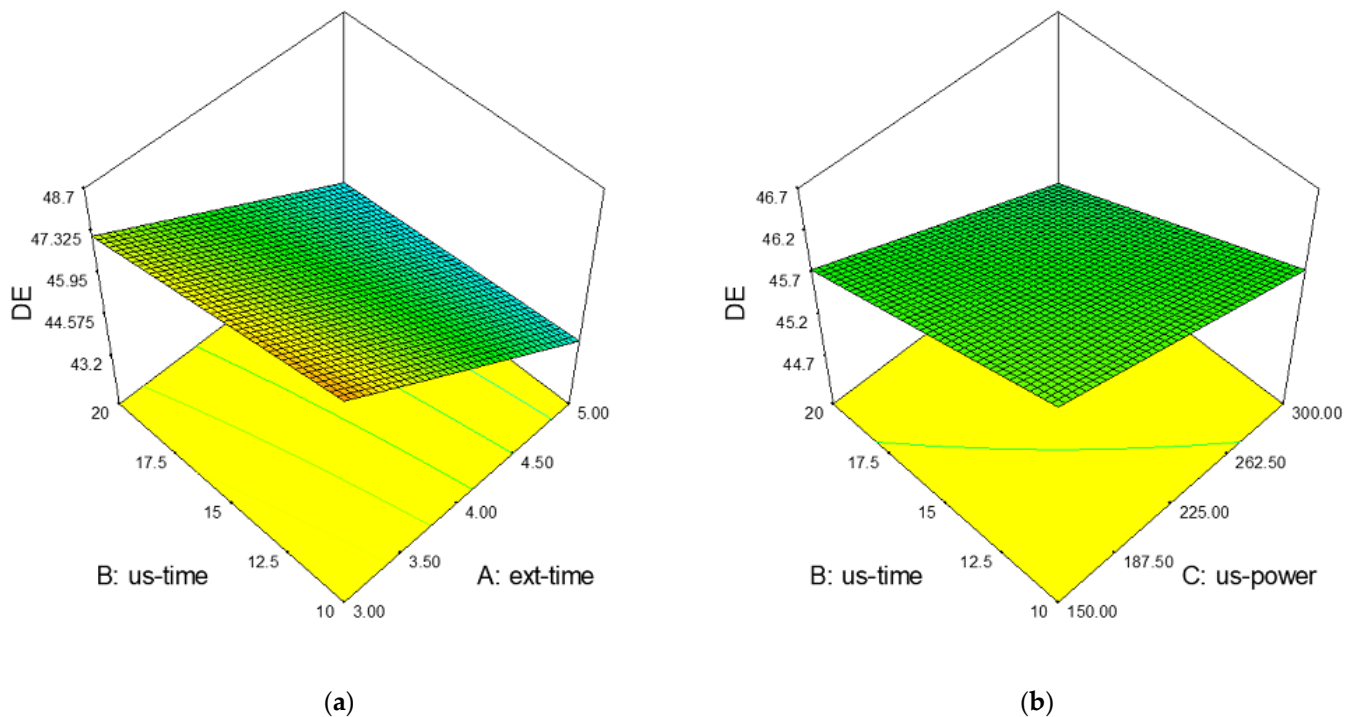
**Figure 1.** Yield values of extracted pectin using microwave-assisted extraction (MAE) and microwave-assisted extraction with ultrasonic pretreatment (MAUPE), which were shown using ultrasonication pretreatment power and time.

### 3.2. Degree of Esterification

The application of pectin largely depends on its degree of esterification (DE). The results showed that the DE value of pectin from MAE (3,4 and 5 min of extraction) varied between 47.5 and 48.5%. No significant difference was observed in the DE value of pectin obtained with different extraction durations; they all belonged to low-methoxy pectin, as evidenced by the DE values, which were lower than 50%. According to this result and Figure 2, the effect of extraction time on DE values depends on the pretreatment used. The DE values of the samples obtained by MAUPE decreased from  $44.7 \pm 0.5\%$  to  $42.9 \pm 0.5\%$ . Cavitation and reaction by ionized groups produced during ultrasonic pretreatment can reduce the pectin DE [11]. Statistical analysis of the three factors, i.e., extraction time, ultrasonication time and ultrasonication power, showed that the DE value decreased with an increase in extraction pretreatment time and power (Figure 2). Moreover, the DE reduction was more substantial with the increase in extraction time when MAUPE was used. In this case, the time and power of extraction pretreatment had an equal effect on the DE value. It was previously reported that the DE value depends on the nature of the pectin source and extraction conditions [11]. Jiang et al. reported that the DE value of watermelon rind pectin extracted by MAE was 44.3–48.7% [21].

### 3.3. Pectin Compositions (Monosaccharide, Galacturonic Acid Content and Protein Content)

The MAE and MAUPE pectins had a GalA content of 73.5% and 69.5–72.5%, respectively. The pretreatment influenced the GalA content of pectin. The total GalA content indicates the pectin purity [28]. From Table 1, it is understood that the decrease in GalA content of pectin corresponded to the rise in extraction pretreatment power and time. Therefore, this result is probably related to the existence of higher impurity, according to the protein content and monosaccharides composition.



**Figure 2.** Interaction effect of ultrasonication and extraction time (a) and time and power of ultrasonication (b) on the DE of extracted pectin. The vertical axis represents the DE value of the extracted pectin. us: ultrasonication, ext-time: extraction time.

**Table 1.** Monosaccharide composition (%) of WRP extracted by MAE and MAUPE.

	MAE	MAUPE			
		150 W–10 min	150 W–20 min	300 W–10 min	300 W–20 min
Rhamnose (%)	0.29	0.47	0.41	0.39	0.31
Arabinose (%)	0.55	0.70	0.65	0.86	0.77
Galactose (%)	3.28	6.04	3.85	6.01	5.51
Glucose (%)	2.50	3.23	2.95	3.28	4.82
Xylose (%)	0.13	0.15	0.18	0.12	0.15
Galacturonic acid (%)	73.5	72.5	71.6	70.0	69.5
HM (%)	73.40	72.08	71.19	69.51	69.19
RG-I (%)	4.37	7.68	5.32	7.64	6.92
R1 (%)	31.10	24.66	25.85	23.53	15.79
Protein (%)	5.84	8.22	8.57	8.37	8.81

Pretreatment increased the total neutral sugar content of the samples. The total amount of rhamnose, arabinose and galactose, which form the side chain of the pectin polymer, was significantly lower than GalA. Results showed that the HG index (GalA–Rha) [29] of pectin from MAE and MAUPE was 73.40%, and varied between 69.19% and 72.08%, respectively. Galactose and arabinose from the rhamnagalacturonan side chains, and rhamnose, which form the branching point of the main chain, are involved in creating the branching parts of the pectin polymer [30]. Petkowicz et al. (2017) reported that pectin isolated from watermelon rind contained HG and RG-I fractions [31].

From Table 1, it can be seen that the samples from MAUPE were richer in RG-I (5.32% to 7.68% of RG-I). However, in both methods, the proportion of HG was dominant. Based on the literature, pectin is physically trapped in the cellulose matrix, or strongly interacts with cellulose. The linkage between pectin and cellulose was found in RG-I rather than HG [32]. Broxterman et al. proposed that in the RG-I side chains, galactose or arabinose units are covalently linked to cellulose [33]. According to these results, ultrasonic pretreatment can



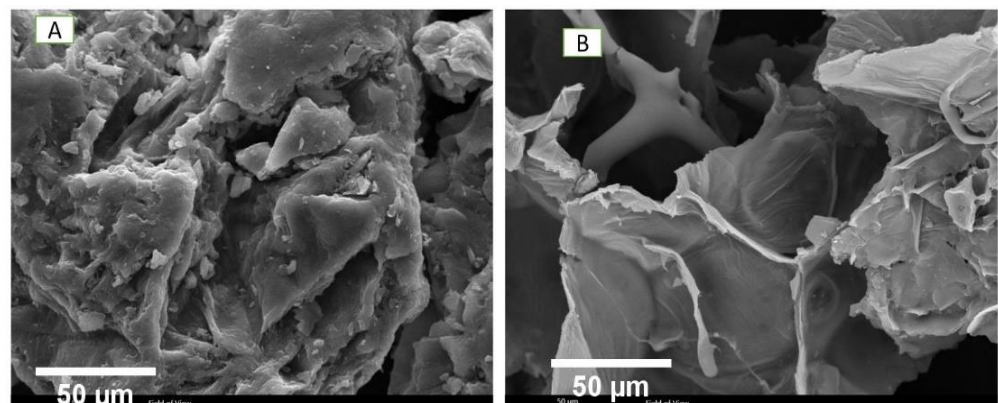
have the ability to increase the extraction yield and affect pectin monosaccharides which introduce the pectin with a higher RG-I content. Therefore, the higher amount of RG-I side chains obtained by MAUPE can probably be explained by the ultrasonic effect on covalent bonds (celloles–RG-I) in raw materials. Xylose was detected at a small percentage, which is due to the low presence of rhamnogalacturonan-II [7,19] in the structure of the samples. Thus, WRP is predominantly composed of HG and a small fraction of RG-I.

The presence of glucose can be explained as a result of non-pectin polysaccharid degradation during extraction [30]. A lower glucose content of the sample from MAE than MAUPE indicates the lower amounts of co-extracted hemicelluloses, cellulosic oligomers or starch (impurity) in this sample compared to other samples. Moreover, the (Rha + Ara + Gal + GalA)–Glc ratio reveals the pectin purity [29]; it compares the amount of pectin components to any co-extractants. As shown in Table 1, when a harsher extraction pretreatment was used, the extract becomes richer in impurities. These results were in agreement with the high protein content of the sample obtained from MAUPE (300 W).

Commercial pectin must contain at least 65% of GalA [34]. Thus, the extracted pectin from MAE and MAUPE fulfils this requirement. WRP had a protein content in the range of  $5.84 \pm 0.47$  to  $8.57 \pm 0.20\%$ , which was higher than the pistachio green hull pectin (6.3%) or eggplant peel pectin (2.53%) [7]. FAO suggests that the protein content of pectin should not be higher than 15.6% [35]. Hence, the extracted pectin from MAE and MAUPE fulfils this requirement. As can be seen in Table 1, the protein content of samples was increased as the power and duration of pretreatment were increased.

### 3.4. Morphology of the Watermelon Rind Powder

Watermelon rind powder, before (Figure 3A) and after ultrasonic pretreatment (Figure 3B), was scanned and the effect of ultrasonic pretreatment, which produced the highest extraction yield (300 W for 20 min), is shown in Figure 3. As can be seen, the physical structure of the raw powder was extremely affected by ultrasonic pretreatment, which resulted in an extensive decomposition of the raw materials. After the ultrasonic pretreatment, the porosity of the samples was increased from 33 to 44%, and the average pore size from 13 to 42  $\mu\text{m}$ . This effect of pretreatment on the watermelon rind was attributed to cavitation. This phenomenon improved the heat and mass transfer, followed by increasing the extraction efficiency of pectin.



**Figure 3.** Watermelon rind powder before (A) and after ultrasonic pretreatment (B) at 300 W for 20 min.

### 3.5. Water Holding Capacity and Surface Tension

WHC can be effective for viscosity, texture and the bulk volume of formulated food products. As can be seen in Table 2, the WHC of the WRP powder obtained from MAE and MAUPE was in the range of 11.72 to 12.43 g/g, which showed proper values for WHC as compared to the WHC of commercial citrus pectin (10.3 g water/g pectin) [36]. According to experimental results (Table 3), the WHC value of pectin from MAE was higher than that

obtained by MAUPE, while the only significant difference was observed between samples from MAE and MAUPE when using pretreatment for 20 min at 300 W. The pectin physical structure and chemical composition could be effective for WHC [37]. Therefore, ultrasonic pretreatment (300 W for 20 min) can change the physical structure or chemical composition of pectin, which affects this parameter and reduces the hydrophilic property of the samples.

**Table 2.** Water holding capacity, foam properties and surface tension of 1% (*w/v*) pectin solutions at 25 °C.

	MAE	MAUPE			
	0 W	150 W–10 min	150 W–20 min	300 W–10 min	300 W–20 min
Surface tension	45.75 ± 0.21 <sup>b</sup>	45.5 ± 0.14 <sup>b</sup>	44.6 ± 0.28 <sup>a</sup>	44.4 ± 0.28 <sup>a</sup>	44.2 ± 0.20 <sup>a</sup>
WHC	12.43 ± 0.19 <sup>b</sup>	11.95 ± 0.32 <sup>ab</sup>	11.94 ± 0.5 <sup>ab</sup>	11.97 ± 0.27 <sup>ab</sup>	11.72 ± 0.30 <sup>a</sup>
FC	81.5 ± 0.7 <sup>a</sup>	83.9 ± 0.5 <sup>ab</sup>	86.3 ± 0.8 <sup>bc</sup>	86.5 ± 0.8 <sup>bc</sup>	87.25 ± 0.7 <sup>c</sup>
FS	30.0 ± 0.8 <sup>a</sup>	30.5 ± 0.7 <sup>ab</sup>	31.2 ± 1.2 <sup>b</sup>	31.8 ± 0.7 <sup>b</sup>	31.5 ± 0.9 <sup>b</sup>

Values in the same row with different letters are significantly different ( $p < 0.05$ ).

**Table 3.** Kinetics parameters for the foaming characteristics of WRP.

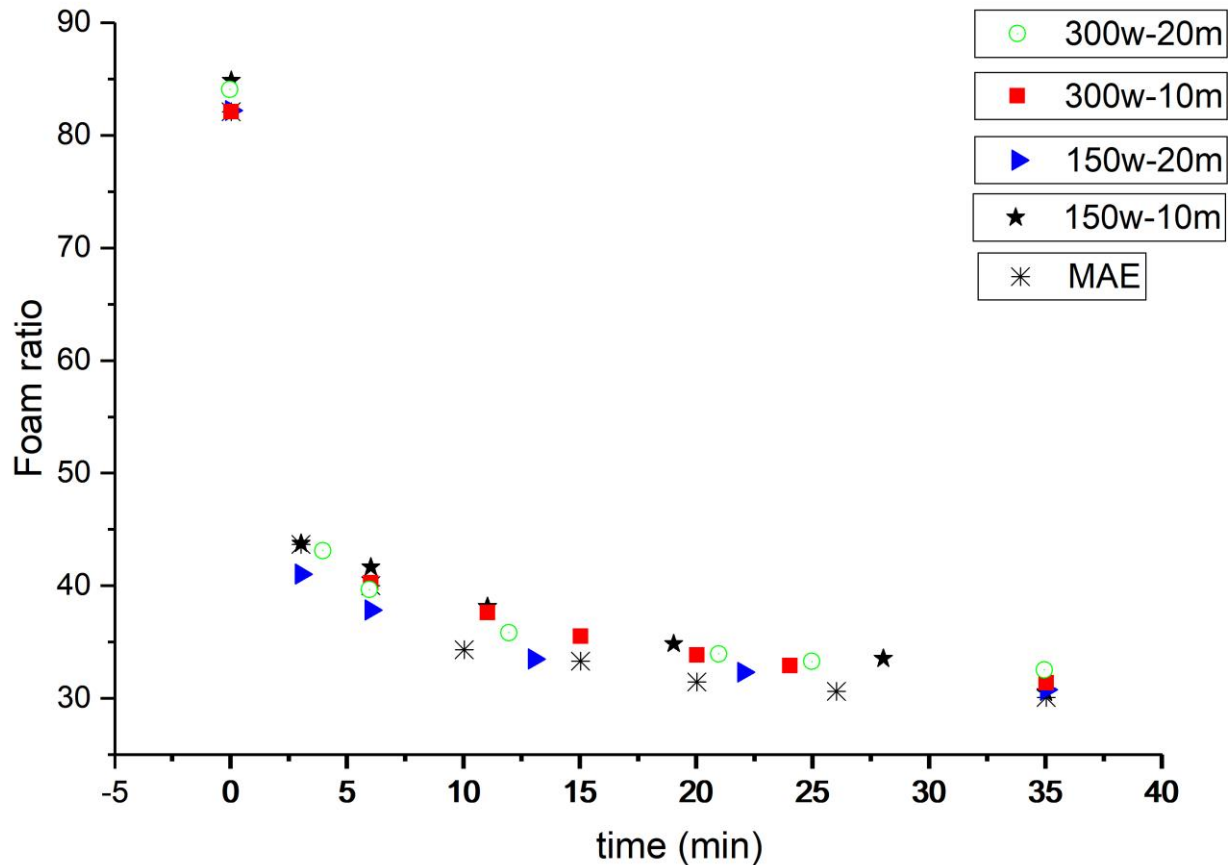
	MAE	MAUPE			
		150 W–10 min	150 W–20 min	300 W–10 min	300 W–20 min
a	82.56	85.12	86.43	86.5	86.98
k	37.04	37.24	39.52	37.91	39.47
$t_{1/2}$	2.78	3.69	2.49	3.73	2.64
$R^2$	0.99	0.99	0.99	0.99	0.99
RMSE	1.135	0.68	0.86	0.52	1.06

The WHC value of watermelon pectin, with or without pretreatment, was higher than the WHC value of guar gum (4.8 g/g) and carboxymethyl cellulose (10.0 g/g) [10] used in cosmetic products as a thickener and to reduce the phenomenon of water loss [11]. Because pectin is classified as a thickening agent and has a WHC value, watermelon pectin can be suggested for uses in cosmetic products. There is a large demand in the food and cosmetic industries to obtain products with better foam properties [12]. Easy and fast foam formation and foam stability are desirable for cosmetic products [7]. Anionic surfactants are more popular for cosmetic applications, which may be due to the hair charge being negative, and consequently, anionic detergents absorb the positively charged oils of hair. As a result, the surfactant can be washed off easily. The viscosity and foaming capability of the cosmetic product (shampoo) is a desirability factor, and it is generally accepted that the higher the foaming obtained, the better the quality of the shampoo [13]. Therefore, due to its anionic composition, the thickening and stabilizing effect, and the foaming properties of watermelon pectin make it a suitable candidate for cosmetic uses. The surface tension of deionized water—the reference—was measured at 71.0 mN/m. As shown in Table 3, the values of surface tension of the WRP solution, 1% (*w/v*), were less than 71.0 mN/m, indicating that WRP can reduce the surface tension of the solution. This parameter is one of the most important physical properties of biopolymers for foam formation: the lower the surface tension, the greater the ability to form a foam [38–40]. The surface tension values of the WRP extracted by MAE, at 45.75 ± 0.21 mN/m, was higher than the value from MAUPE, which ranged from 44.2 ± 0.20 to 45.5 ± 0.14 mN/m. However, the surface tension value of the WRP solution (1% *w/v*) was less than that of commercial pectin (50.8 mN/m) derived from a citrus pectin solution (1% (*w/v*)), reported by Schmidt et al. [41].

### 3.6. Foaming Property

The FC and FS of aqueous solutions of WRP are shown in Figure 4 and Table 2. The FC of the WRP solution was in the range of 81.5 ± 0.7% to 87.25 ± 0.7%. Statistical analysis

revealed that the power and duration of the extraction pretreatment could significantly affect the FC. The pectin obtained using MAUPE (ultrasonic power 300 W) showed a significant difference in the FC and FS from the MAE sample. These results for the FC of WRP solutions agreed with the surface tension measurement and protein data. The FS of the samples was in the range of  $30.5 \pm 0.7\%$  to  $31.8 \pm 0.7\%$ .



**Figure 4.** Foam ratio (%). Samples obtained from MAUPE were shown using pretreatment power and time.

In the foam formation process, the foaming agent adsorbs at the air–water interface [11]. Then, the foam was stabilized by inter-molecular interactions of the foaming agent to create a cohesive viscoelastic film [42]. The branching of the RG-I domains in some pectin molecules can provide a steric stabilization of some dispersions [43]. The molecular weight reduction probably leads to a faster adsorption of pectin molecules to the interface of dispersion. It is also possible that hydrophobic–hydrophilic groups, such as protein groups, were more accessible by pectin depolymerization [41]. Therefore, according to the molecular weight results (Section 3.7) and increasing of the sample protein content, ultrasonic pretreatment probably affects the FC. Basak et al. also reported an increase in the foaming capacity due to a reduction in particle size of apple pectin after plasma treatment [44].

The results showed that the FS of pectin from MAUPE at 300 W for 20 min ( $31.8 \pm 0.7$ ) was higher than that of pectin from MAE ( $30.5 \pm 0.7$ ). No significant difference was observed among the samples obtained from MAUPE. Figure 4 shows that the foam instability rate is very fast in the early stages of storage and slows down with increasing storage time. The results of the kinetic evaluation (Table 3) revealed that the empirical model fit the foam properties properly, by evaluating the highest  $R^2$  and the least RMSE values. A decrease in the kinetics rate ( $k$ ) indicates a decrease in the foam destruction rate. Therefore, the prolonged pretreatment had the greatest effect on the half-time and

the foam destruction rate. The half-time of the foam system using MAUPE ranged from 2.4 to 3.93 min. An increase in  $k$  and a decrease in  $t_{\frac{1}{2}}$  showed a decrease in viscosity after pretreatment.

### 3.7. Intrinsic Viscosity and Viscosity Average Molecular Weight

The intrinsic viscosity is used to specify the volume occupied by single-sample molecules in dilute solutions and is also relevant to the molecular weight of the polymer in the solution [45,46]. The intrinsic viscosity of watermelon rind pectins from MAE and MAUPE ranged from  $28.9 \pm 0.4$  to  $37.7 \pm 0.3$  mL/g (Table 4). Pretreatment had a significant effect on intrinsic viscosity and the viscosity average molecular weight (MW), except for 150 W–10 min. A decrease in intrinsic viscosity was associated with a decrease in molecular weight. Regarding intrinsic viscosity, this study concludes that MAUPE had more degradative effect on pectin structure than MAE.

**Table 4.** Molecular characteristics of pectins from watermelon rind.

	MAE	MAUPE			
	0 W	150 W–10 min	150 W–20 min	300 W–10 min	300 W–20 min
Intrinsic viscosity (mL/g)	$37.7 \pm 0.3^b$	$33.1 \pm 0.3^b$	$29.6 \pm 0.4^a$	$30.4 \pm 0.3^a$	$28.9 \pm 0.4^a$
Mw (KDa)	$322.8 \pm 2.0^b$	$318.1 \pm 2.3^b$	$292.8 \pm 2.7^a$	$298.1 \pm 2.1^a$	$287.2 \pm 2.7^a$

Values in the same row with different letters are significantly different ( $p < 0.05$ ).

The Mark–Houwink–Sakurada equation was used to calculate the MW. The MW of pectin from MAUPE was lower than the sample obtained from MAE. This result was in line with that previously reported by Ma et al., which showed a reduction in MW of pectin (from 451 KDa to 210 KDa) extracted by enzymes with ultrasonic pretreatment [47]. Bagharian previously reported that continued heating of pectin may lead to the disintegration of pectin networks [48]. According to Section 3.1, ultrasound pretreatment significantly accelerated the extraction of pectin. Molecules extracted from MAUPE were then suspected to be heated more. From the results of pectin MW, it can be concluded that the pectin chain of MAUPE was partially degraded. However, these fragments were large enough to be separated by alcohol precipitation, and retained a significant portion of the RG-I side chains. The MW of WRP was lower than the values of 432.8–564.0 kDa mango peel pectins [21].

### 3.8. Colour Attributes

The colour attributes, including lightness ( $L^*$ ), redness ( $a^*$ ) and yellowness ( $b^*$ ) values of watermelon pectin, are shown in Table 5. Samples obtained using MAUPE had a darker appearance than samples obtained from MAE, with less  $L^*$ .

**Table 5.** Colour property of pectin powder.

	MAE	MAUPE			
		150 W–10 min	150 W–20 min	300 W–10 min	300 W–20 min
$L^*$	$66.4 \pm 0.2^b$	$62.5 \pm 1.0^a$	$62.4 \pm 0.5^a$	$60.6 \pm 0.2^a$	$61.1 \pm 0.2^a$
$a^*$	$0.3 \pm 0.06^a$	$0.2 \pm 0.1^a$	$0.7 \pm 0.2^a$	$0.4 \pm 0.2^a$	$0.7 \pm 0.1^a$
$b^*$	$17.3 \pm 0.7^a$	$18.4 \pm 0.5^a$	$18.6 \pm 0.8^a$	$22.3 \pm 0.8^b$	$25.3 \pm 0.7^b$
$\Delta E$	-	4.05	4.22	7.65	9.60

Values in the same row with different letters are significantly different ( $p < 0.05$ ).

The colour of pectin powder and or solution of pectin is one of the most important factors affecting the appearance of pectin gel. Indeed, a dark colour can limit the application of the products in some formulations. The ultrasonic pretreatment before the extraction affected the colour of the extracted pectin, which showed a darker appearance, and decreased the  $L^*$  value of these samples. Moreover, the results did not show a difference in the redness value of the samples. The sample yellowness value was obtained by using

MAUPE with ultrasonication at 300 W, which was more than the other. The  $L^*$  value of watermelon pectin powder was higher than the  $L^*$  value of sugar beet pectin (41.27) and tomato pectin (50.96), which were previously extracted using MAE [27,28]. As can be seen, the colour difference, was increased using MAUPE as an increase in  $\Delta E$  values. The power of extraction pretreatment was a dominant factor affecting this parameter. The colour difference between the samples indicated the magnitude of the colour difference between samples obtained from the MAE and MAUPE methods. These results were probably due to the degradation of the side chains, followed by a higher monosaccharide content, and a partial demethylation of the samples using intense extraction pretreatment (300 W) that influenced the colour properties of the samples, as reported by Ramos-Aguilar [29].

#### 4. Conclusions

Ultrasonic pretreatment can be used to improve the extraction yield of pectin, due to its effect on the rind powder porosity. WRP can reduce the surface tension of the solution and, therefore, be used as a foaming agent in different formulations. The FC was increased and WHC values decreased by increasing the power and duration of extraction pretreatment. Pretreatment changed the linearity of the pectin chain, also partially degrading it. The kinetics studies showed that the empirical model introduced in this research matched the foam ratio well, and demonstrated the stability kinetics of the foam.

The results of this study show that combining an ultrasonic pretreatment with microwave gives a higher extraction yield than the MAE method. The properties of the extracted pectin make it a good candidate as a thickening and foaming agent, with a high potential in formulations within the food and cosmetic industries.

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