

Article

Pectin Extraction from *Opuntia* spp. Cladodes: Process Optimization, Characterization and Films Development for Food Packaging

Carolina Rodrigues ¹ , Bilge Sayın ² , Victor Gomes Lauriano Souza ^{1,3} , Ana Gabriela Azevedo ⁴ , Isabel Coelho ⁵  and Ana Luísa Fernando ^{1,5,*} 

¹ METRICs, Departamento de Química, NOVA School of Science and Technology (NOVA FCT), Universidade NOVA de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal; cpe.rodrigues@campus.fct.unl.pt (C.R.); v.souza@fct.unl.pt (V.G.L.S.)

² Department of Gastronomy and Culinary Arts, School of Tourism and Hotel Management, Ardahan University, 75000 Ardahan, Turkey; bilgesayin@ardahan.edu.tr

³ GeoBioTec, Departamento de Ciências da Terra, NOVA School of Science and Technology (NOVA FCT), Universidade NOVA de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal

⁴ International Iberian Nanotechnology Laboratory (INL), Av. Mestre José Veiga s/n, 4715-330 Braga, Portugal; ana.azevedo@inl.int

⁵ LAQV-REQUIMTE, Departamento de Química, NOVA School of Science and Technology (NOVA FCT), Universidade Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal; imrc@fct.unl.pt

* Correspondence: ala@fct.unl.pt

Abstract

This study investigated the extraction and characterization of pectin from the peel and the pulp of *Opuntia ficus-indica* (OFI) cladodes, aiming to define sustainable and optimized extraction conditions and to evaluate the applicability of the extracted pectin in film development for food packaging. Cladodes were chemically characterized, confirming their richness in sugars, dietary fiber, and bioactive compounds. Different solvents (citric acid, acetic acid, and acidified water) and pH values (1.5–7) were evaluated, with citric acid (1% *w/v*) selected as the most suitable solvent due to its extraction efficiency and food-grade nature. Process optimization was performed using response surface methodology (RSM), considering liquid-to-solid ratio (5–15 *v/w*), extraction time (40–60 min), and temperature (70–90 °C). The regression models showed good fit, with R^2 values of 88.79% for peel and 89.20% for pulp. Extraction yield was mainly influenced by liquid-to-solid ratio, time, and temperature, with optimal conditions defined as 10 *v/w*, 40 min, and 80 °C. Pectin obtained under optimized conditions was characterized by Fourier-transform infrared (FTIR) spectroscopy, showing functional groups consistent with commercial citrus pectin, while galacturonic acid content and degree of esterification confirmed its purity and classification as low-methoxyl pectin, supporting its suitability for further film production. Additionally, the extracted pectin was successfully incorporated into blended films with commercial pectin, resulting in films with improved water resistance and water vapor barrier performance. Overall, OFI cladodes represent a promising and sustainable source of pectin for biodegradable food packaging applications.

Keywords: *Opuntia ficus-indica*; pectin extraction; response surface methodology; polysaccharide-based films; food packaging application



Academic Editor: Vasiliki (Viki) Evageliou

Received: 31 December 2025

Revised: 8 March 2026

Accepted: 31 March 2026

Published: 7 April 2026

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1. Introduction

Opuntia ficus-indica (OFI) is a plant that belongs to the *Cactaceae* family, genus *Opuntia*, that is well adapted to arid and semi-arid lands and widespread around the world (e.g., Mexico and Mediterranean area) [1–5]. The fruits, known as cactus pear or prickly pear, have gained further attention due to their high nutritional content and health beneficial effects, due to their antioxidant, anticancer and anti-inflammatory properties [6–8]. Cladodes, the stems, are mostly used for human consumption but also animal feed [2,9], and they are rich in water, carbohydrates and fiber (namely, cellulose, hemicellulose, pectin, lignin and mucilage) [10–14]. The chemical composition of cladodes depends on several factors such as maturity stage, environmental conditions, harvest season, post-harvest treatment and the type of species [15,16]. Cladodes also find application in several industries such as cosmetics, pharmaceuticals and nutraceuticals [17–19]. Cladodes have also been applied as adsorbent materials in the textile industry, as flocculant agents in water and wastewater treatment, as additives in building construction and have been valorized for energy, such as the production of hydrochars and biogas [20–26].

OFI cladodes are rich in mucilage, a mixture of heterogenous polysaccharides with high molecular weight constituted mostly by arabinose, galactose, galacturonic acid, rhamnose and xylose [27–29]. Due to its elastic flow characteristic and ability to form a molecular network, OFI mucilage can be used in the food industry as edible films/coatings [30–33]. Cactus cladodes also contain pectin, which exhibits gelling properties in the presence of Ca^{2+} [7]. Pectin is a complex polysaccharide mainly constituted by α -D-galacturonic acids [34], commonly used in the food industry as a gelling and stabilizing agent, e.g., in the formulation of jellies and jams [13,35,36]. Pectin can also be used to produce films which present some positive features, such as: their biodegradability, low-cost production, excellent mechanical properties and the possibility to extend the shelf-life of the packaged food [37]. Pectin is richer in galacturonic acid than mucilage, but both hydrocolloids show great similarities in terms of chemical structures, and therefore, mucilage can be indistinctly referred to as pectin [28].

Moreover, the cactus needs to be pruned annually, generating an enormous amount of cactus stems/cladodes, which are a waste product/by-product capable of numerous applications, such as those aforementioned [38,39]. Thus, pruning cladodes residues is a significant source of low-cost biomass for mucilage/pectin extraction, and their exploitation would in part help to solve the problem of disposal/waste materials. The industrial exploitation of this crop generates by-products between 20% and 45% of the fresh weight of cladodes and fruits, respectively [40,41]. For instance, in Mexico, the biggest OFI producer, around 40,000 tons of OFI cladode waste are lost annually [30].

Pectin extraction is commonly performed under acidic conditions with high temperatures (60–100 °C) [37,42]. High temperatures facilitate the solvent diffusion into the plant matrix, helping the extraction and also allowing the polymer's solubilization. However, the use of high temperatures can cause pectin depolymerization, ultimately reducing the extraction yield [43]. An acidic pH is fundamental for the extraction process since it promotes the hydrolysis of protopectin [44]. The use of strong acids as solvents, such as sulfuric acid and hydrochloric acid, is commonly employed but their use is associated with high costs and significant environmental impacts [42]. To overcome this challenge, mild acids, including citric acid, malic acid, and acetic acid, are valuable alternatives to use as solvents in pectin extraction [45]. Despite these mild acids exhibiting lower hydrolyzing efficiency compared to strong acids, they are considered food grade, making them more suitable for food applications [42,45,46]. Moreover, several other factors are of extreme importance to obtain the best results for pectin extraction such as pH, time, agitation, surface area and solid–liquid ratio [47,48].

Some other techniques have been employed to enhance pectin extraction and the most innovative and green technologies include enzymatic extraction, ultrasonic-assisted extraction (UAE), microwave-assisted extraction, and the use of non-thermal technologies such as subcritical fluids, high hydrostatic pressure, pulsed electric field and deep eutectic solvents [30,44,49–52].

In general, the functional properties of pectins are linked to the degree of esterification (DE), and they are classified as low-methoxyl (LM) pectin (DE < 50%) and high-methoxyl (HM) pectin (DE > 50%). The DE of the pectin is directly related to the source and has a fundamental influence on future applications [53]. Pectin from OFI is generally classified as low-methoxyl pectin [13,35,36,53].

An example of pectin extraction from OFI cladodes was done by Chaouch et al. [54]. In this study three different extraction conditions were used: acidic (pH = 2, 2 h, 40 °C), neutral (pH = 6, 2 h, 40 °C) and basic (pH = 10, 2 h, 4 °C) and samples were submitted to 10 min of ultrasound prior to extraction. In conclusion, the highest yield of extraction was obtained with neutral pH, however, acidic conditions presented a higher galacturonic acid content [54]. Moreover, in the work of Zamboi et al. [51], pectin from OFI was extracted using UAE, maintaining the power and frequency of the ultrasound bath constant and varying the solid–liquid ratio (SL), temperature, pH and time. The authors found the optimal operating conditions were obtained using a solid–liquid ratio of 1:10 *w/v*, pH = 2.5, temperature of 65 °C and time of extraction of 20 min. The extraction yield was around 11% dry weight (dw), and pectin was mainly composed of galacturonic acid, arabinose and galactose, with a 42.82% degree of esterification (DE), thus it was characterized as low-methylation pectin [51].

Hence, the present study is focused on the optimization of the extraction method of pectin from cladodes of *Opuntia ficus-indica*. It combines the use of more eco-friendly acids and optimized extraction parameters with a specific focus on pectin extraction for films development for food packaging applications. To achieve this, cladodes were first characterized in terms of physical, chemical and bioactive properties. Focusing on the sustainability of the extraction process and thorough literature review, three different solvents were tested (water, citric acid aqueous solution and acetic acid aqueous solution), avoiding the use of strong acids. Additionally, the impact of pH on extraction was investigated by testing a range of pH values (1.5, 2, 2.5, 3, 4, and 7). Once the optimal solvent and pH were determined based on extraction yield, further parameters, including liquid-to-solid ratio, extraction time, and temperature, were optimized using response surface methodology (RSM). RSM allows minimization of the number of experiments and gathering more information in order to optimize various parameters and the effect of their interaction on the desired goal [55]. The best extraction conditions resulting from the optimization were then used to obtain pectin that was further characterized in terms of degree of esterification, galacturonic acid content and identification of functional groups. This pectin was further used in the production of films. Ultimately, this study seeks to identify sustainable and efficient methods for pectin extraction from an alternative source and to evaluate the potential of the extracted pectin through the development of films, envisaging food packaging applications.

2. Materials and Methods

2.1. Materials and Reagents

Opuntia ficus-indica cladodes were harvested in September 2019 and kindly provided by the company *Pepe Aromas*, located in Vale Pereiro, Évora, Portugal.

The water used was purified using a Milli-Q system (Millipore, Belford, MA, USA) and all chemicals were of analytical reagent grade. 2,2-Diphenyl-1-picrylhydrazyl (DPPH),

ascorbic acid, catechin, aluminum chloride, sulfuric acid, 3,5-dimethylphenol, D-(+)-galacturonic acid monohydrate and pectin from citrus peel were purchased from Sigma-Aldrich (Steinheim, Germany). Glacial acetic acid, gallic acid and glycerol were acquired from Alfa Aesar (Kandel, Germany). Folin–Ciocalteu reagent, citric acid, sodium chloride, copper sulfate pentahydrate, phenolphthalein, potassium hexacyanoferrate II trihydrate, sodium hydroxide, sodium carbonate anhydrous, and magnesium nitrate hexahydrate were obtained from PanReac (Barcelona, Spain). 2,6-Dichlorophenol-indophenol, hydrochloric acid, and ammonium molybdate tetrahydrate were purchased from Fisher Chemical (Loughborough, UK). Potassium antimony tartrate, sodium nitrite, and zinc sulfate heptahydrate were purchased from Merck (Darmstadt, Germany). Ethanol absolute, nitric acid, and sodium sulfate were purchased from Riedel-de-Haen (Seelze, Germany). Potassium sodium tartrate tetrahydrate was purchased from Scharlau (Barcelona, Spain). Potassium dihydrogen phosphate was purchased from Chem-Lab (Zedelgem, Belgium).

2.2. Cladode Characterization

Cladodes were manually separated into inner (pulp) and outer (peel) layers using a stainless steel knife and characterized in terms of the following methods: pH, titratable acidity, total protein content, moisture and ash content according to Association of Official Analytical Chemists (AOAC) methods [56]. The moisture content was determined by drying the samples at 103 ± 2 °C until constant weight in a WTB Binder type E28 oven (Binder, Tuttlingen, Germany) and the ash content by using a Heraeus Eletronik KR 170 E muffle furnace (Heraeus, Hanau, Germany) at 550 ± 25 °C and the results expressed as a percentage on a dry basis. In order to assess the pH and titratable acidity, an extract of 5 g of cladode in 10 mL of water was made. The pH values were measured using a HI 2210 potentiometer (Hanna Instruments, Woonsocket, RI, USA) and titratable acidity was evaluated by titrating 10 mL of extract with 0.1 N NaOH. The results of titratable acidity were expressed as a percentage of citric acid. The ascorbic acid content was determined by the 2,6-dichlorophenol indophenol titrimetric method according to AOAC [56]. Fiber content was assessed through sequential extraction of the various fiber fractions (hemicellulose, cellulose and lignin) [57] and the results were expressed in g per 100 g on a dry basis. The total sugar composition, reducing and non-reducing sugar, was evaluated through the Munson and Walker method [58]. Protein content was measured by nitrogen content using the Kjeldahl method and a conversion factor of 6.25 was used to obtain the total protein content [59]. The phosphorus (P) content was determined by digestion with acid followed by the determination of the phosphates in the digestates by molecular absorption spectrophotometry, through the formation of a colored complex with an ammonium molybdate solution, in the presence of ascorbic acid and potassium and antimony tartrate [60] and the samples were read at 880 nm in an E-1000UV UV-Vis spectrophotometer (Peak Instruments, Shanghai, China). The P content was obtained by using a calibration curve with known concentrations of P (0–0.25 mg/L) and read in the UV-Vis spectrophotometer at 880 nm. Results were expressed in mg of P per 100 g. Total phenolic content (TPC), antioxidant activity and total flavonoid content of the cladodes were quantified by the Folin–Ciocalteu method [61], DPPH assays [62], and aluminum complexation in the presence of nitrite method [63], respectively. Ethanolic extracts in a cladodes/ethanol proportion of 1/10 g/mL were used in the analysis. The TPC was obtained by using a calibration curve with known concentrations (0–120 mg/L) of gallic acid solution and the sample's absorbance at 760 nm read in the UV-Vis spectrophotometer. Results were expressed in mg of gallic acid equivalent (GAE) per 100 g of cladodes. To evaluate the antioxidant activity, a DPPH assay was carried out and the absorbance was read at 517 nm in a UV-Vis spectrophotometer and the results were expressed in mg of Trolox equivalent per kg. For

total flavonoid content, the samples were read at 510 nm in a UV-Vis spectrophotometer and the results were expressed in mg of catechin (CE) per 100 g of cladodes. The results were obtained by using a calibration curve of catechin.

All the analyses were performed in triplicate.

2.3. Pectin Extraction and Characterization

To optimize the conditions for pectin extraction based on extraction yield, a series of experiments were conducted. The general procedure for pectin extraction is detailed in Table 1, while specific experiments were designed to evaluate and optimize the key extraction parameters. In order to define the solvent to be used, three different solvents were tested, namely: deionized water, aqueous solutions of acetic acid (10% *w/v*) and citric acid (1% *w/v*) at pH 2 adjusted with HCl 0.1 N—experimental set 1 [64,65]. Then, experiments were conducted to study the impact of pH by testing different pH values (1.5, 2, 2.5, 3, 4 and 7)—experimental set 2. The remaining parameters for these experiments were defined as shown in Table 1. After these two parameters (pH and solvent) were defined, liquid–solid ratio, time and temperature were optimized using RSM as described in Table 2. The extracted pectin was subsequently characterized following the procedures outlined in Section 2.3.3.

Table 1. Experimental design for the extraction of pectin from *Opuntia ficus-indica* cladodes.

	Solvent	pH	L/S Ratio (v/w)	Time (min)	Temperature (°C)
First set	Deionized water, citric acid (1% <i>w/v</i>), acetic acid (10% <i>v/v</i>)	2	10	50	80
Second set	Deionized water	1.5, 2, 2.5, 3, 4, 7	10	50	80

Table 2. Experimental range and levels of each factor used in the response surface design.

Variables	Factor	Coded Levels		
		−1	0	+1
L/S (% <i>v/w</i>)	X1	5	10	15
Time (min)	X2	40	50	60
Temperature (°C)	X3	70	80	90

2.3.1. Extraction

The extraction general procedure is described as follows. Cladodes were previously washed and separated into peel and pulp and then cut into small pieces. The extraction was performed according to Espino-Díaz and others [66] with modifications. The extraction step was performed in a WB01 thermostatic water bath (Labbox Labware, Barcelona, Spain) with the parameters described in Table 1.

Samples were centrifuged at 10,000 rpm/15 min/0 °C. The supernatant was left overnight at 4 °C in ethanol (ratio of 1:2 (*v/v*)) to precipitate. After precipitation, the samples were again centrifuged and dried in an oven at 45 °C for 2 h and then placed in a desiccator containing silica. The final samples were reduced into powder for further analysis. All the extractions were performed in triplicate.

2.3.2. Experimental Design

The effects of the studied factors on extraction yields were evaluated using response surface methodology (RSM). A central composite design (CCD, $\alpha = 1$), corresponding to a face-centered design, was employed to optimize the extraction yield. The coded levels of the independent variables were set at −1, 0, and +1, with 0 representing the center point, as shown in Table 2. The parameters (L/S, time and temperature) and the levels

chosen were selected based on preliminary experiments and the literature [51,67]. A total of 20 experimental runs were conducted according to the CCD, including six replicates at the center point to estimate experimental error and assess model reproducibility.

The dependent variable was pectin extraction yield, and the data were analyzed using Minitab statistical software, version 19.2 (Minitab, State College, PA, USA). The complete second-order model was initially tested (Equation (1)). Model adequacy was determined based on the non-significance of the lack-of-fit test ($p > 0.05$) and the statistical significance of the regression ($p < 0.05$).

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad (1)$$

where Y represents the yield of extraction, X_1 is the real variable L/S ratio, X_2 is the real variable for time, X_3 is the real variable for temperature, and $\beta_0, \beta_1, \beta_2, \beta_3, \beta_{11}, \beta_{22}, \beta_{33}, \beta_{12}, \beta_{13}, \beta_{23}$ represent the equation coefficients.

The statistical significance of the terms in the regression equation was determined with a 95% confidence level.

2.3.3. Characterization of the Extracted Pectin

The yield of extraction was performed through a gravimetric method and the results were expressed as a percentage (%) of dry weight. Fourier-transform infrared (FTIR) spectra of the resulting materials were acquired in the range 400–4000 cm^{-1} using a PerkinElmer Spectrum Two FTIR spectrometer (Perkin Elmer, Waltham, MA, USA). The powder was directly analyzed, with 4 scans and a resolution of 4 cm^{-1} . The FTIR spectra analyzed were used to calculate the degree of esterification (DE), using the peak areas corresponding to COO-asymmetric stretching and at C=O using Equation (2) [68]:

$$DE(\%) = \frac{Abs(C = O_{esterified})}{(Abs(C = O_{esterified}) + Abs(COO^{-}_{asymmetricstretching}))} \times 100 \quad (2)$$

The determination of galacturonic acid content followed the methodology of Van Hung et al. [69] and the results are expressed as (%).

A commercial pectin from citrus (Sigma-Aldrich, Steinheim, Germany) was used to compare with the experimental results.

2.4. Film Preparation

Films were prepared by the solvent casting method using the pectin extracted from *Opuntia ficus-indica* cladodes pulp (Section 2.3) blended with pristine commercial pectin [66]. Preliminary assays using *Opuntia* pectin as the sole polymeric matrix (2% w/v , plasticized with 50% w/w glycerol) resulted in continuous but mechanically fragile films, limiting their practical applicability. Therefore, to improve film cohesion and mechanical integrity, a polymer blending strategy was adopted. The optimized formulation consisted of a total polymer concentration of 3% (w/v), with equal proportions (50:50, w/w) of extracted *Opuntia* pectin and commercial pectin, plasticized with 50% (w/w) glycerol relative to total pectin content. Films were obtained by casting the filmogenic suspension onto polystyrene Petri dishes and dried at room temperature until complete solvent evaporation. Films were stored in a desiccator at 52% RH (with supersaturated solution of magnesium nitrate hexahydrate $[Mg(NO_3)_2 \cdot 6H_2O]$) until further use. Pristine commercial films were prepared and used for comparison.

Film Characterization

The developed films were characterized in terms of optical, mechanical, physical, barrier, and morphological properties in order to evaluate the effect of blending pectin

extracted from *Opuntia ficus-indica* cladodes with pristine commercial pectin (OFIPC), in comparison with commercial pectin (PC) films.

Optical properties were assessed through color parameters (L^* , a^* , b^*) using the CIELab color space, chroma (C^*), opacity, and transparency. Chroma was calculated from the a^* and b^* coordinates (Equation (3)), while opacity was determined from absorbance values at 600 nm normalized by film thickness. The transparency of the films was made through a spectrum scan in a range of 200–900 nm in a UV/Vis spectrophotometer, expressed as transmittance (%) [70–72].

$$C^* = (a^2 + b^2)^{0.5} \quad (3)$$

Thickness was measured by using an electronic micrometer (Mitutoyo, Tokyo, Japan) at five random different points of each film. The results were expressed in micrometers (μm). Mechanical performance was evaluated by tensile testing according to ASTM D882 [73], allowing the determination of tensile strength (TS), Young's modulus (YM), and elongation at break (EAB) from stress–strain curves. The physical properties of the films were evaluated through moisture content and water solubility. Moisture content was determined gravimetrically as the percentage of mass loss after drying. Water solubility was assessed by immersing pre-dried films (M_i) in ultrapure water at room temperature for 24 h, followed by recovery of the insoluble fraction and re-drying to constant mass (M_f). Film solubility was expressed as the percentage of soluble material relative to the initial dry mass (Equation (4)) [71].

$$\text{Film water solubility (\%)} = \frac{M_i - M_f}{M_i} \times 100 \quad (4)$$

Barrier properties were assessed by determining the water vapor permeability (WVP) using a gravimetric method in accordance with ASTM E96/E96M-10 [74]. The water vapor transmission rate (WVTR) was calculated from the mass gain over time under controlled relative humidity conditions, and WVP was obtained considering film thickness (e) and the difference of water vapor partial pressure gradient across the film (ΔP) (Equation (5)).

$$WVP = WVTR \times \frac{e}{\Delta P} \quad (5)$$

Finally, the surface morphology of the films was examined by scanning electron microscopy (SEM) to evaluate film homogeneity, surface integrity, and the presence of structural defects or phase separation.

2.5. Statistical Analysis

A statistical analysis of data was performed through a one-way analysis of variance using Minitab, version 19.2 (Minitab, State College, PA, USA), and differences among mean values were processed by the Tukey test. Significance was defined at $p < 0.05$.

3. Results

3.1. Cladode Characterization

The composition of cladodes varies depending on several factors such as species, cultivar, maturity stage, climatic, soil, and water conditions, geographical location, and harvest/post-harvest conditions. These variables contribute to the differences observed in the literature regarding cladodes composition [15,75]. The chemical composition of peel and pulp from *O. ficus-indica* cladodes is shown in Table 3.

Table 3. Chemical composition of peel and pulp of OFI cladodes.

	Peel	Pulp	Literature
	Average $\pm$ SD	Average $\pm$ SD	
Moisture % (wb)	89.7 $\pm$ 0.4 ^b	94.7 $\pm$ 0.2 ^a	OFI cladodes powder—94.1% db [76] OFI 89.9% [77]
Ash content % (db)	19.8 $\pm$ 0.4 ^a	16.5 $\pm$ 0.2 ^b	OFI cladodes powder—23.3% db [76] OFI 24.9% [77] OFI <i>f. amyloceae</i> —25.7% db [78]
Crude protein content (% db)	4.0 $\pm$ 0.2 ^a	3.4 $\pm$ 0.3 ^b	OFI 0.30% FW [79] OFI 8.4% [77] OFI 7.1% db [12]
P content (mg P/100 g)	28 $\pm$ 2 ^a	20 $\pm$ 4 ^a	OFI—16.4 mg/100 g FW [79]
pH	4.96 $\pm$ 0.01 ^a	4.88 $\pm$ 0.03 ^b	4.4 [15] 4.64 [79]
Total titratable acidity (% w/w of citric acid)	0.44 $\pm$ 0.02 ^a	0.17 $\pm$ 0.01 ^b	0.286 g/100 g FW [79]
Total sugars (% w/w) db	15 $\pm$ 3 ^b	30 $\pm$ 1 ^a	41.2% [80]
Total reducing sugars (%w/w db)	7.9 $\pm$ 0.6 ^b	21.3 $\pm$ 0.0 ^a	2.87% [80]
Total fiber content (g/100 g db)	26 $\pm$ 3 ^a	30 $\pm$ 2 ^a	56.7% [76]
Cellulose (g/100 g db)	10.9 $\pm$ 0.4 ^a	11.1 $\pm$ 1.6 ^a	21.6% dry weight [81] 26.7% db [76]
Hemicellulose (g/100 g db)	8.8 $\pm$ 1.7 ^b	13.1 $\pm$ 0.8 ^a	15.3% db [76]
Lignin (g/100 g db)	6.8 $\pm$ 0.8 ^a	5.4 $\pm$ 0.9 ^a	3.6% dry weight [81] 2.5% [76]
Ascorbic acid content (mg/100 g)	5.7 $\pm$ 1.0 ^a	4.8 $\pm$ 1.0 ^a	26.8—77.4 mg/100 g [82] 10–15 mg/100 g [83]
Total phenolic content (mg GAE/100 g)	5.3 $\pm$ 0.1 ^a	1.6 $\pm$ 0.1 ^b	260 mg GAE/100 g fresh weight [84] 16.1 mg/100 g [85]
Antioxidant activity (mg Eq Trolox/kg)	9.2 $\pm$ 1.5 ^a	2.3 $\pm$ 0.4 ^b	45.1 mg Eq Trolox/kg [85]
Total flavonoids (mg Eq catechin/100 g)	4.7 $\pm$ 0.4 ^a	1.2 $\pm$ 0.7 ^b	12.6 mg/100 g [85]

Superscript letters (a, b): different superscripts within the same row indicate significant differences among samples ($p < 0.05$). wb—wet basis; db—dry basis. GAE—gallic acid equivalent.

Peel and pulp from OFI cladodes showed no differences in terms of P content, total fiber content, cellulose and lignin content and ascorbic acid content. On the other hand, other parameters demonstrated statistically significant differences ($p < 0.05$). This was especially evident in terms of bioactive compounds, showing higher values in the external part, specifically the total phenolic content, antioxidant activity and total flavonoids. A higher amount of bioactive compounds is generally observed in the outer layer of fruits and vegetables when compared to other parts of the plants such as pulp and seeds [86].

Results showed a high moisture content in both peel (89.7%) and pulp (94.7%), similar to those reported by other authors of 89.90% in OFI cladodes [77] and 94.12% for cladodes powder [76]. The ash content is slightly lower than the ones presented in the literature for OFI of 23.3% [76], 24.9% [77] and 25.7% [78]. The protein content obtained (4.0% for peel and 3.4% for pulp) is slightly lower than values reported in the literature such as 8.4% [77] and 7.1% [12]. The phosphorus content results were slightly higher than the 16.4 mg/100 g reported by Pérez Méndez et al. [79] in *O. ficus-indica* cladodes. The pH values and titratable acidity obtained are considered as low (acidic), most likely because of the presence of organic acid such as malic, oxalic and citric acids due to the crassulacean acid metabolism (CAM) performed by the cactus and these values align with previous studies [10,79]. Sugars are one of the main components of cladodes, as demonstrated by the total sugar values obtained of 15% w/w in the peel and 30% w/w in the pulp. The reducing sugar content was 7.9% w/w in the peel and 15.3% in the pulp. These values are consistent

with those reported Mounir et al. [80], who found total sugar and reducing sugar values of 41.2% and 2.87%, respectively. Fibers represent a major fraction in both the peel (26 g/100 g) and the pulp (30 g/100 g), with no significant differences observed ($p \geq 0.05$). The lignocellulosic composition of cladodes revealed considerable amounts of lignin and hemicellulose, along with cellulose, further highlighting their potential for lignocellulosic compound extraction [76]. The ascorbic acid content in the peel and pulp was lower than the values reported for fresh OFI cladodes from four different cultivars (26.8–77.4 mg/100 g) [82] but similar to the 10–15 mg/100 g reported for young OFI cladodes [83]. OFI cladodes are also a source of phenolic compounds and flavonoids [87,88]. However, the total phenolic content obtained in this study was lower than the values reported for OFI cladodes in Spain [85,88] and significantly lower to the 260 mg GAE/100 g reported by other authors [84]. The antioxidant activity of the peel (9.2 mg Eq Trolox/kg) and pulp (2.3 mg Eq Trolox/kg) was notably lower than values found in the literature [85]. Similarly, total flavonoid content was lower than the results obtained in a study with OFI cladodes (12.6 mg/100 g) [85]. Thus, the chemical composition confirms that cladodes are a rich source of dietary fiber, sugars, and lignocellulosic compounds, supporting their potential use in various applications. In addition, slight compositional differences were observed between peel and pulp of the cladodes, with the pulp showing lower ash and protein but higher sugar contents and peel showing higher bioactive activity. These variations likely reflect their distinct physiological functions, with the peel accumulating more structural components and minerals, while the pulp mainly stores water and soluble sugars [12,15,87]. Furthermore, *Opuntia ficus-indica* cladodes are recognized as a valuable source of polysaccharides, particularly mucilage rich in pectic substances. Reported yields of pectin range from about 5 to 12% of the dry cladode weight, with galacturonic acid contents between 30 and 80%, while mucilage represents approximately 6 to 15% of the dry mass [13,35,36,89]. These components, rich in uronic acids and neutral sugars, impart the characteristic hydration and gelling behavior of cladodes, reinforcing their potential as natural hydrocolloids. Although this work focused on the extraction of pectin, the close association and overlapping composition of pectin and mucilage suggest that both contribute to the functional properties that are investigated in the following section. Moreover, the presence of significant amounts of insoluble fibers, particularly lignin and cellulose shown in Table 3, suggests a complex lignocellulosic matrix. These components can act as a physical barrier to solvent penetration, obstructing the pectin release and thus the need to optimize the extraction parameters such as temperature and time to maximize yield [90].

3.2. Pectin Extraction

3.2.1. Experimental Set 1—Yield of Extraction with Different Solvents

Opuntia ficus-indica cladodes have an appreciable content in macromolecules such as pectin and mucilage, which exhibit high water retention capacity [91]. The results obtained for the extraction with different solvents (all adjusted to pH 2.0 with 0.1 N HCl) are summarized in Figure 1. According to these results, the yield of extraction obtained ranged from 3.69 to 14.8%, all performed under the same conditions (time of extraction—50 min, liquid/solid ratio—10 v/w, pH—2 and temperature—80 °C). Overall, the extraction yield from the pulp was consistently higher than from the peel, regardless of the solvent used. In the peel, statistically significant differences ($p < 0.05$) were observed between citric acid extractions and those performed with the other solvents. The highest extraction yield obtained in pulp ($14.8 \pm 1.0\%$) was obtained using deionized water acidified to a pH of 2 when compared to citric acid 1% ($10.4 \pm 1.2\%$) and acetic acid 10% (9.54%). In the pulp, significant differences were found between deionized water acidified to a pH of 2 and the other solvents. The highest extraction yield ($7.6 \pm 0.8\%$) was achieved by using 10% (v/v)

acetic acid as the solvent, whilst only $6.0 \pm 0.7\%$ was obtained for deionized water acidified to a pH of 2, and $3.7 \pm 0.5\%$ was obtained with 1% citric acid.

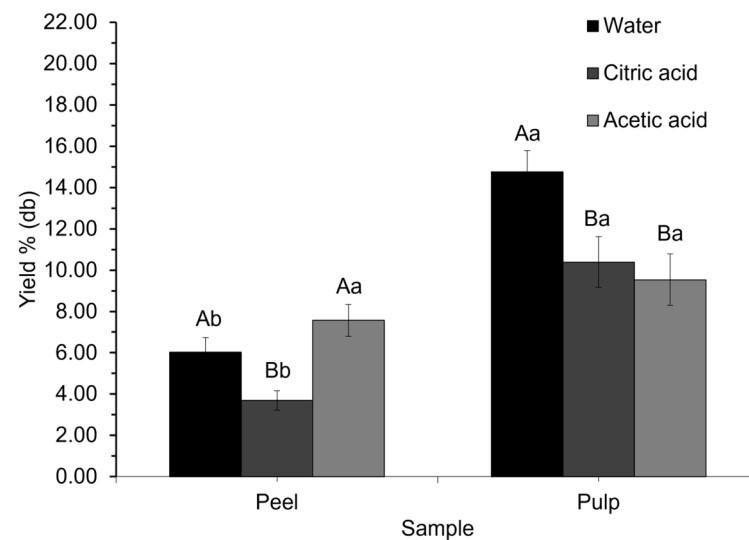


Figure 1. Yield of extraction (% db) obtained with different solvents. Superscript letters (a, b): different superscripts indicate significant differences between peel and pulp ($p < 0.05$) for each extraction solution. Superscript letters (A, B): different superscripts indicate significant differences between solvents ($p < 0.05$) for each fraction of the OFI cladodes.

These results are aligned with those reported by Quintero-Garcia et al. [92] for fresh cladodes [92] and are slightly lower than the yields reported by Contreras-Padilla et al. [93]. Specifically, the extraction yield using deionized water acidified to a pH of 2 (14.8% DW in the pulp) is comparable to the 12.0% DW reported by Dick et al. [94] for *O. monocantha* and higher than the 6.2% DW obtained by Kalegowda et al. [95] in *O. dilenii* cladodes. Similarly, other authors reported a value of 6.13% DW for the pectic fraction from cladodes that were extracted with water under a pH of 2.8 [96].

The variation in extraction yield reported in the literature may be attributed to several factors, including differences in extraction conditions, maturity stage of the cladodes, species, and experimental methods [92]. Moreover, the choice of solvent plays a crucial role in optimizing extraction yield. The use of acids plays crucial role in the extraction since, by facilitating cell wall degradation, there is increased access to cellular contents and enhanced efficiency of the extraction process [69]. Organic acids, such as acetic and citric acids, were explored in this study due to their cost-effectiveness and environmental friendliness compared to inorganic acids (e.g., hydrochloric acid and sulfuric acid) [47,97]. Focusing exclusively on the performance of the two organic acids tested, citric acid achieved a higher yield on the pulp. Due to the fact that the difference between the two solvents in pulp yield was not statistically significant ($p \geq 0.05$), citric acid was ultimately chosen as the solvent to proceed with the pectin extraction from OFI. This decision highlights the importance of optimizing solvent selection to maximize yield while maintaining the process's sustainability.

3.2.2. Experimental Set 2—Yield of Extraction Under Different pH Values

pH is one of the most important features of the extraction process and it is stated that acid conditions favor pectin extraction [47]. The results of extraction with different pH values are presented in Figure 2.

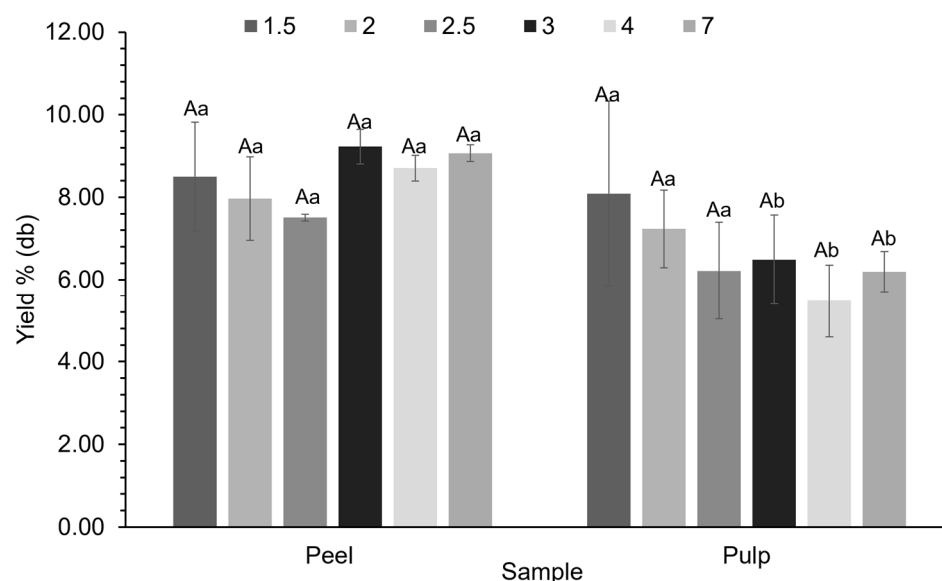


Figure 2. Yield of extraction (% db) obtained at different pH (1.5, 2, 2.5, 3, 4 and 7) using water as solvent. Lowercase letters (a, b): different lowercase letters indicate significant differences between peel and pulp for the same pH ($p < 0.05$). Uppercase letter (A): no significant differences between pH values were observed in each fraction of the cladode ($p > 0.05$).

Pectin yield in peel varied between 7.5% (pH 2.5) and 9.2% (pH 3) and in pulp the yields were between 5.5% (pH 4) and 8.1% (pH 1.5). No statistical differences were observed among different pH values in the pectin extracted from OFI cladode peel ($p > 0.05$), showing that pH did not have a significant effect on the pectin extraction from peel under the tested conditions. Similarly, the same behavior was observed in the pectin extracted from OFI cladodes pulp, within the test pH range. Comparing the yield of extraction between peel and pulp at each pH level, at pH 1.5, 2, and 2.5, no significant differences ($p > 0.05$) were found between peel and pulp. On the other hand, at pH 3, 4, and 7, significant differences ($p < 0.05$) were observed. The values of the extraction at a pH of 2 ($8.0 \pm 1.0\%$ w/w db—peel; $7.2 \pm 0.9\%$ w/w db—pulp) are similar to the ones reported by Chaouch et al. [54] ($8.8 \pm 0.7\%$ w/w db) at the same pH. At a pH of 6, the same authors reported a yield of extraction of $24.5 \pm 0.5\%$ w/w db and, at a pH of 10, a value of $4.6 \pm 0.4\%$ w/w db which reflects the fact that under basic conditions the yield of extraction starts to decrease [54]. This behavior is in agreement with the behavior observed in the extractions from pulp, where the results suggested that more acidic conditions (pH of 1.5, 2, 2.5, 3) allowed the hydrolysis of the insoluble pectin into soluble pectin, increasing the yield of extraction which is in agreement with [89]. Based on these previous studies, the solvent and pH were fixed for subsequent experiments. Citric acid 1% (w/v) was selected as the extraction solvent without pH adjustment, as its natural pH was approximately 2.2–2.3. This pH value aligns with the findings presented in Figure 2, indicating that, within the tested range, pH had no significant impact on the extraction process.

3.2.3. Experimental Design

A total of 20 experiments, following the methodology described in Section 2.3.2, were conducted in a randomized form, including six replicates at the central point to validate the model, in order to optimize the extraction conditions of pectin.

The experimental conditions tested and the results of yield of extractions for each experiment are described in Table 4, expressed as percentages on a dry basis.

Table 4. Yield of pectin extraction (% on a dry basis) from OFI peel and pulp of cladodes at pH around 2, using 1% citric acid solution, as affected by L/S ratio, time and temperature.

L/S	Time (min)	Temperature (°C)	Yield (%) Peel	Yield (%) Pulp
10	50	80	13.74	23.38
15	40	90	16.65	24.08
15	50	80	10.78	18.93
15	40	70	8.52	19.34
10	40	80	17.88	24.56
10	50	80	12.75	22.94
5	50	80	16.27	17.26
10	50	90	15.80	26.32
10	50	80	14.62	23.73
10	60	80	13.79	18.86
10	50	70	13.84	19.70
5	40	90	19.28	21.79
5	60	90	7.70	13.98
15	60	90	11.27	17.29
10	50	80	14.45	21.21
5	40	70	16.75	14.00
5	60	70	13.81	14.16
10	50	80	14.00	23.34
10	50	80	12.76	21.11
15	60	70	6.18	12.09

The statistically non-significant terms were removed from the initial model, and the experimental data were re-fitted using only the significant parameters ($p < 0.05$). The model adjustments representing the variations in yield of pectin extraction from OFI peel and pulp of cladodes in response to the different extraction conditions are presented in Table 5.

Table 5. Model adjustment with the respective determination coefficients for yield of pectin extraction from OFI peel and pulp of cladodes at pH ~ 2, using 1% citric acid solution, as affected by L/S ratio (X_1), time (X_2) and temperature (X_3).

Sample	Adjusted Model	R ²
Yield (%) Peel	$-9.0 - 2.46 X_1 + 0.905 X_2 + 0.426 X_3 - 0.0657 X_1^2 + 0.0420 X_1 \cdot X_3 - 0.0146 X_2 \cdot X_3$	88.79%
Yield (%) Pulp	$-6.13 + 4.39 X_1 - 0.274 X_2 + 0.242 X_3 - 0.209 X_1^2$	89.20%

Pectin yields ranged from 6.2 to 19.3% in peel and 12.1 to 26.3%, on a dry basis, in pulp (Table 4), suggesting that pulp contains more extractable pectin than the peel. This difference can be directly linked with the differences in the total sugar content, showing higher values in pulp than in the peel of the cladodes (Table 3). The fitted models were able to explain 88.79% in the extraction from peel and 89.20% in the extraction from pulp. Figure 3 presents the 2D response surface plots obtained from the RSM analysis, illustrating the effects of extraction parameters on pectin yield from *Opuntia ficus-indica* cladodes. Plots (a–c) correspond to peel, and plots (d–f) correspond to pulp.

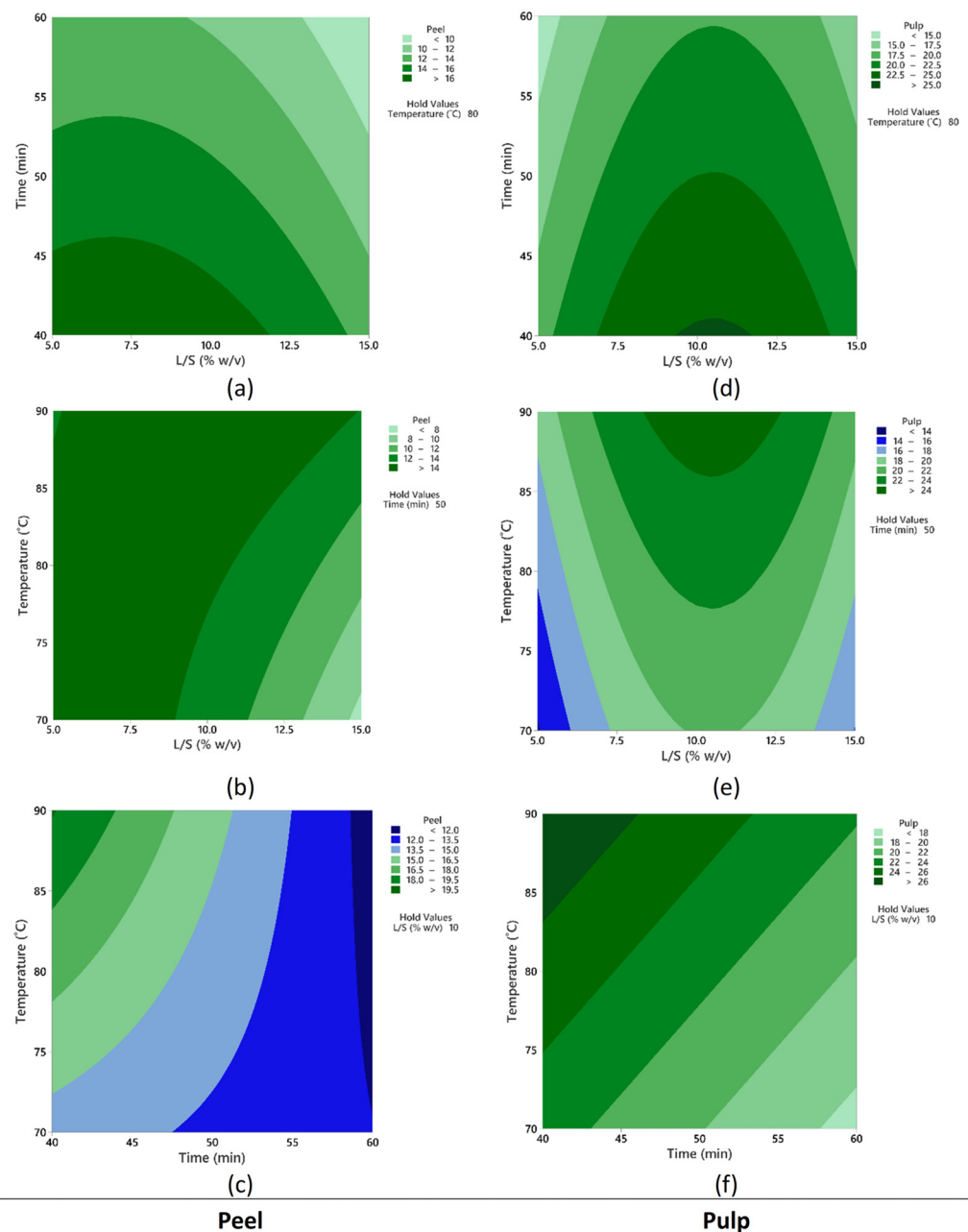


Figure 3. 2D response surface plots showing the effects of extraction parameters on pectin yield from *Opuntia ficus-indica* cladodes. Plots (a–c) correspond to peel, and (d–f) to pulp. (a,d) represent the correlation between extraction time and liquid-to-solid (L/S) ratio at the middle temperature; (b,e) show the correlation between temperature and L/S ratio at the middle extraction time; and (c,f) depict the correlation between temperature and extraction time at the middle L/S ratio.

Yield of pectin extraction from peel was influenced by the linear effect of L/S, time, and temperature as well as the interaction of L/S and temperature and time and temperature and the quadratic effect of L/S ($p < 0.05$) according to the analysis of regression coefficients of the response function. The linear regression coefficient of L/S was negative, indicating that a lower L/S led to a higher yield of pectin extraction in peel, while the positive coefficient of time and temperature indicated that extraction yield could increase with longer extraction time and high temperatures. The positive coefficient value of the interaction between L/S and temperature indicated that increasing both variables simultaneously enhanced the extraction efficiency. On the other side, the negative coefficient of the interaction between time and temperature suggested that longer extraction times

combined with high temperatures reduced the pectin extraction yield. For pectin extraction from OFI cladodes, the best extraction conditions should involve a moderate L/S ratio and extraction time, combined with a sufficiently high temperature.

In pectin extracted from pulp, the yield of extraction was only affected by the linear effects of time, temperature and L/S, as well as by quadratic effect of L/S ($p < 0.05$). The negative coefficient of time indicated that increasing the extraction time leads to a slight decrease in pectin yield, while the positive coefficients of L/S and temperature showed that higher values of these parameters favor the extraction, up to an optimum point, and beyond that the yield decreases due to the negative quadratic effect of L/S. This suggests that prolonged extraction time may cause thermal degradation, reducing the pectin extraction or changing the pectin characteristics [46]. Even though both models showed a good fit, they indicated that longer extraction times led to a decrease in yield of extraction.

Based on both models, the optimized extraction conditions should balance the most significant parameters affecting yield in both pulp and peel from OFI cladodes. Thus, the L/S ratio should be kept moderate (10 v/w), since very low ratios may limit solubilization while high ratios reduce yield, particularly in peel and due to the quadratic effect observed in both models. The extraction time should be short to moderate (40–45 min), as prolonged extraction periods negatively affect yield, especially in pulp. Concerning temperature, it should be moderate to high (75–85 °C), as it enhances pectin extraction when combined with a relatively low L/S ratio. Therefore, in this work and, based on the resulting models, the selected optimized pectin extraction conditions were L/S of 10 v/w , time of 40 min and temperature of 80 °C using citric acid 1% w/v at a pH around 2.

Bayar et al. [96] reported a value of 6.1% of dry weight for pectin extraction from OFI cladodes, using HCl as solvent, a pH of 2.8, and an L/S of 15 at 90 °C for 2 h. Also, Sevgi et al. [53] conducted acidified hot water extraction using HCl under conditions of L/S of 15, pH of 2.8, temperature of 90 °C, and extraction time of 4 h, achieving an extraction yield of 0.06–0.18% fw. The results of the present study are relatively higher than those reported by these authors. Despite the similarity in extraction conditions and the use of a different solvent, no optimization of the extraction parameters was performed. In the extraction of pectin from banana peels using a 1:20 w/v solid-to-liquid ratio with citric acid, the optimal conditions were found to be 87 °C, 160 min, and pH 2, resulting in a pectin yield of 14.2 g/100 g, showing similarities to the optimal conditions obtained in the present study [98]. In this case, only the time of extraction was not in accordance with the results obtained. A prolonged time of extraction can result in a degradation of methyl ester groups into carboxyl acid, leading to pectin with a lower degree of esterification [46]. Zamboi et al. [51] used ultrasound-assisted extraction to extract pectin from *Opuntia* cladodes, obtaining optimal conditions of L/S of 10 v/w , pH of 2.5, time of 20 min and temperature of 65 °C with the model validated with a 11.3% extraction yield. Even though the use of ultrasound was not contemplated in this study and therefore it is more difficult to compare the results, the optimal conditions for L/S ratio identified closely aligns with the results obtained in the present work.

3.3. Characterization of the Extracted Pectin

The pectin extracted under the optimized conditions was subsequently characterized based on its functional groups, degree of esterification and galacturonic acid content.

3.3.1. Fourier-Transform Infrared (FTIR)

To obtain a more comprehensive understanding of the extracted material from OFI cladode peel and pulp, FTIR spectra were acquired and compared with a commercial pectin from citrus peel (CP). The peaks and the respective assignments are presented in Figure 4.

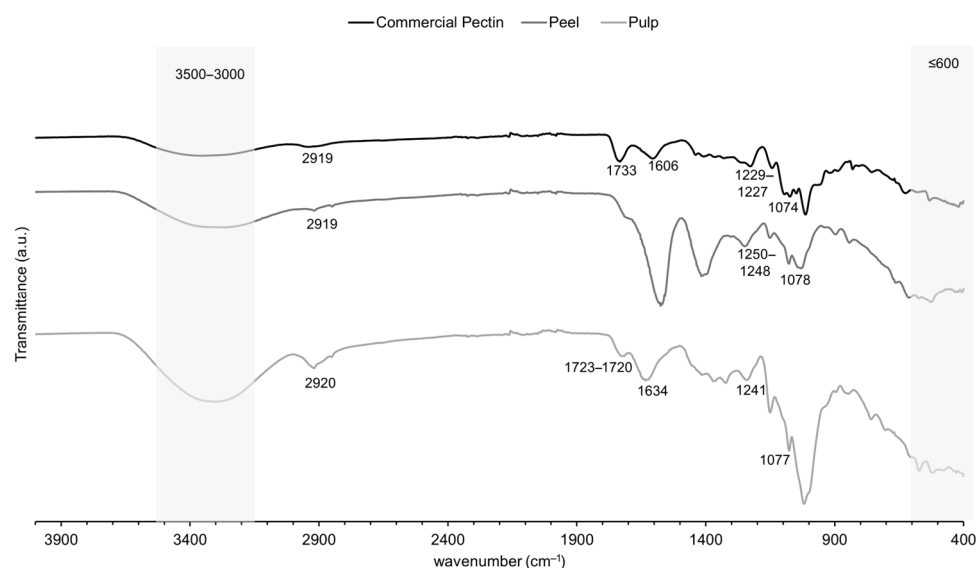


Figure 4. FTIR assignments' wavenumbers (cm^{-1}) of CP and pectin extracted from peel and pulp of OFI.

In both figures a band from $3500\text{--}3000\text{ cm}^{-1}$ corresponding to the stretching of hydroxyl groups (-OH) is observed [51]. Bands in the region of $3000\text{--}2840\text{ cm}^{-1}$ are related to C-H (methyl) stretching bands [51] and are observed in both figures. In peel, a peak at 2919 cm^{-1} revealed the symmetric stretching vibration of the C-H group of the methyl ester of galacturonic acid [96]. In pulp, a peak at 2920 cm^{-1} showed the same behavior. A peak at 1733 cm^{-1} (commercial pectin) and at $1720\text{--}1723\text{ cm}^{-1}$ (pulp) correspond to vibration of the esterified carboxyl group (C=O) [36,99]. Bands around $1600\text{--}1650\text{ cm}^{-1}$ and $1400\text{--}1450\text{ cm}^{-1}$ correspond to free carboxylic groups (COO^-) and are characteristic of pectin, belonging to the class of carboxy polysaccharides [36]. The band around 1600 cm^{-1} was observed in commercial pectin and pulp samples [96], while the band in the region of $1400\text{--}1450\text{ cm}^{-1}$ was specifically detected in pectin extracted from peel. The same behavior is not observed for samples from pulp, which revealed a band around 1370 cm^{-1} which may correspond to the same functional groups. Bands from $1200\text{--}800\text{ cm}^{-1}$ correspond to a carbohydrate fingerprint region [96]. A peak around 1200 cm^{-1} is detectable in commercial pectin, peel and pulp, and it is assigned to O-acetyl-ester [51]. Peaks around 1150 cm^{-1} are linked to C-O-H bonds of primary alcoholic groups and are found for peel and pulp. All samples showed a band around 1070 cm^{-1} , which corresponds to glycosidic linkage (C-O and C-C stretching bond) that is typical from backbone vibrations of pectin [36]. Possibly linked with this connection, another band around $1010\text{--}1040\text{ cm}^{-1}$ was detected. More specifically, C-O-H bonds of primary D-arabinose were visible at 1013 cm^{-1} (commercial pectin) and 1019 cm^{-1} in pulp [53]. A peak at 1030 in peel is assigned as glucan units [51]. Peaks and bands around $850\text{--}900\text{ cm}^{-1}$ represent the α -glycosidic linkage [96] and are detected in commercial pectin (831 cm^{-1}), and in peel (843 ; $896\text{--}898\text{ cm}^{-1}$). In pulp samples, there were no observations around those wavenumbers, but peaks are detected around 760 cm^{-1} . Bands and peaks below 600 cm^{-1} are related to the vibration of monosaccharide and oligosaccharide molecules that are present in all samples [53]. Although the spectra are characteristic of pectin, bands associated with non-pectin substances were also identified. The broad band around $3540\text{--}3125\text{ cm}^{-1}$ may include amide N-H stretching contributions from residual proteins [51]. Furthermore, the region around $1600\text{--}1650\text{ cm}^{-1}$ (related to COO^- stretching) likely overlaps with the amide I band ($\sim 1655\text{ cm}^{-1}$), while weak signals around 1526 cm^{-1} (amide II) confirm the presence of protein traces [51]. In the fingerprint region, peaks observed at $1027\text{--}1045\text{ cm}^{-1}$ and 1077 cm^{-1} are attributed to glucan units

and neutral glycans, respectively, indicating the co-extraction of hemicellulose or cellulose fractions [51,96].

3.3.2. Degree of Esterification and Galacturonic Acid Content

The values obtained for the degree of esterification and galacturonic acid content of commercial pectin and pectin from OFI peel and pulp are presented in Table 6.

Table 6. Degree of esterification and galacturonic acid content of CP and pectin from OFI peel and pulp.

Sample	DE (%)	Galacturonic Acid Content (%)
CP	52.5 ± 0.7	88.7 ± 0.3
Peel	n.d.	41.4 ± 12.7
Pulp	35.6 ± 4.4	76.9 ± 3.6

CP: Commercial Pectin; DE: Degree of Esterification. n.d.: not determined.

The degree of esterification of pectin extracted from OFI cladodes pulp was $35.6 \pm 4.4\%$ and $52.5 \pm 0.7\%$ for commercial pectin (Table 6). It was not possible to calculate the degree of esterification in OFI cladodes peel due to the absence of peaks in the region of carboxyl groups. Thus, the pectin from OFI pulp can be classified as low-methoxyl (LM) pectin whereas the commercial pectin can be categorized as high-methoxyl pectin [36,51,96]. Those values are comparable with others reported, such as 38.5% for OFI cladodes pectin [53]. Through the analysis of degree of esterification and considering that the pectin obtained in this study had a low degree of esterification, it is also possible to compare with a commercial citrus pectin with low methoxyl content with a value of 45.20% [53]. The values obtained in the present study are in line with other research on pectin extracted from OFI cladodes that show that pectin from OFI is mostly LM [44,51,100].

Galacturonic acid is the major component present in pectin and the presence of high percentages of galacturonic acid suggests the presence of pectin polymers [53]. The results obtained revealed a considerable amount of galacturonic acid content with values of $76.9 \pm 3.6\%$ from pectin extracted from OFI pulp. The CP galacturonic acid content was $88.7 \pm 0.3\%$, which is consistent with the product specification sheet that indicates a content of over 74%. The results aligned with findings from other studies: Ref. [44] reported 68.9% using ultrasonic extraction; Ref. [53] reported 56.2% using acidified hot water extraction; Ref. [100] reported 68.8% using ultrasonic extraction and 60.7% using microwave extraction; Ref. [51] found 25.6% galacturonic acid content by using ultrasound-assisted extraction.

3.4. Film Characterization

Figure 5 illustrates the visual appearance of the pristine commercial pectin (PC) and OFIPC films, and their respective characterization is going to be addressed in this subsection.

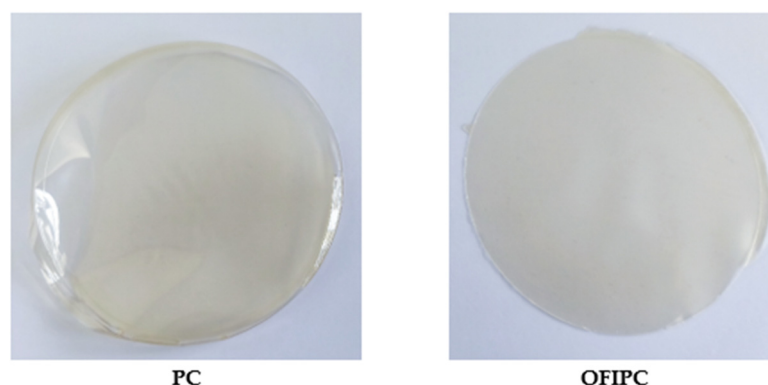


Figure 5. Visual appearance of the films. PC: Pristine Commercial Pectin film; OFIPC: *Opuntia ficus-indica* cladode pectin blended with Commercial Pectin film.

The optical properties of the films were significantly influenced by the incorporation of OFI pectin. OFIPC films exhibited higher L^* values and lower chroma than PC films, indicating increased lightness and reduced color saturation. These characteristics are advantageous for packaging applications, as they limit visual interference with the packaged product [101]. In contrast, OFIPC films showed higher opacity, which may be associated with increased light scattering within the blended polymer matrix and can contribute to enhanced protection against light-induced degradation. OFIPC films exhibited lower light transmittance across the UV–visible range than PC films, indicating reduced transparency, in agreement with their higher opacity values, as can be seen in Figure 6. This behavior is attributed to increased light scattering within the blended pectin matrix and may be advantageous for applications requiring protection against light-induced degradation [102,103]. The thickness of the developed films ranged from $101 \pm 9 \mu\text{m}$ (OFIPC) to $116 \pm 12 \mu\text{m}$ (PC), with no statistically significant differences ($p > 0.05$) observed between the samples. Regarding mechanical properties, OFIPC films exhibited lower Young's modulus values than PC films, indicating reduced stiffness and a more flexible polymeric network. Although OFIPC films displayed lower mean tensile strength values when compared to PC films, these differences were not statistically significant, suggesting that blending did not compromise film cohesion or resistance to rupture, which may be related to the composition of the film-forming dispersion [104]. A slight reduction in elongation at break was also observed for OFIPC films, indicating a moderate decrease in extensibility, yet without significance. Overall, the mechanical behavior of OFIPC films reflects a balance between flexibility and strength, supporting their suitability for handling and food packaging applications [105].

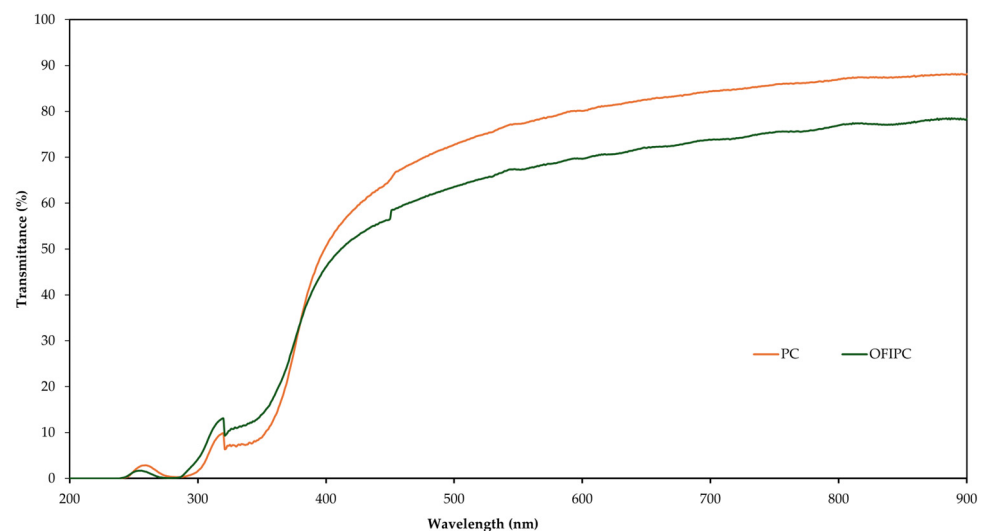


Figure 6. UV-Vis transmittance spectra of PC and OFIPC films.

OFIPC films exhibited a higher moisture content (22%) compared to PC films (17%), which can be attributed to the increased hydrophilic character of the blended polymeric matrix [106]. The use of pectin from *Opuntia cladodes* (OFIPC—79%) highly reduced solubility of the films when compared to PC films (100% soluble in water), indicating their hydrophilic nature [106]. This reduction is likely attributed to the presence of non-pectin substances, such as residual proteins and cell wall polysaccharides (cellulose and hemicellulose), identified in the FTIR analyses [36,96]. Unlike commercial pectin, which is highly purified, these residual hydrophobic or less soluble fractions can strengthen the film matrix through intermolecular interactions (e.g., protein–pectin crosslinks), delaying water diffusion and disintegration [36]. Furthermore, the presence of those substances contributes to the increased opacity and the yellowish-brown color of the OFIPC films, as proteins and phenolic compounds scatter light [96]. PC films showed the highest WVP

values of all films ($29.7 \pm 4.6 \times 10^{-11}$ g/s.m.Pa) in this study. The combination of pectin from *Opuntia* cladodes and pristine commercial pectin significantly improved the WVP ($p < 0.05$) when compared to pristine pectin films, resulting in OFIPC films with a value of $20.4 \pm 1.5 \times 10^{-11}$ g/s.m.Pa. To contextualize these results, the WVP obtained for OFIPC films is lower than values commonly reported for several polysaccharide-based films, including sodium alginate (22.1×10^{-11} g/s.m.Pa) [107], chitosan (28.4×10^{-11} g/s.m.Pa), and pectin films (29.6×10^{-11} g/s.m.Pa) [105]. However, the WVP of OFIPC films remains higher than that of petroleum-derived PVC films (2.47×10^{-13} g/s.m.Pa) [108]. Nevertheless, these results demonstrate a clear enhancement in water vapor barrier performance within biodegradable polysaccharide-based film systems.

PC films showed a homogenous, non-porous surface and cross-section as demonstrated in Figures 7A and 7B, respectively. Similar uniform morphologies were reported by several authors for pectin-based films, evidencing that pectin matrices generally form continuous and compact structures [71,109,110]. This homogeneity can also be enhanced by the presence of glycerol, which acts as a plasticizer, reducing internal stress and promoting chain mobility during film formation [109].

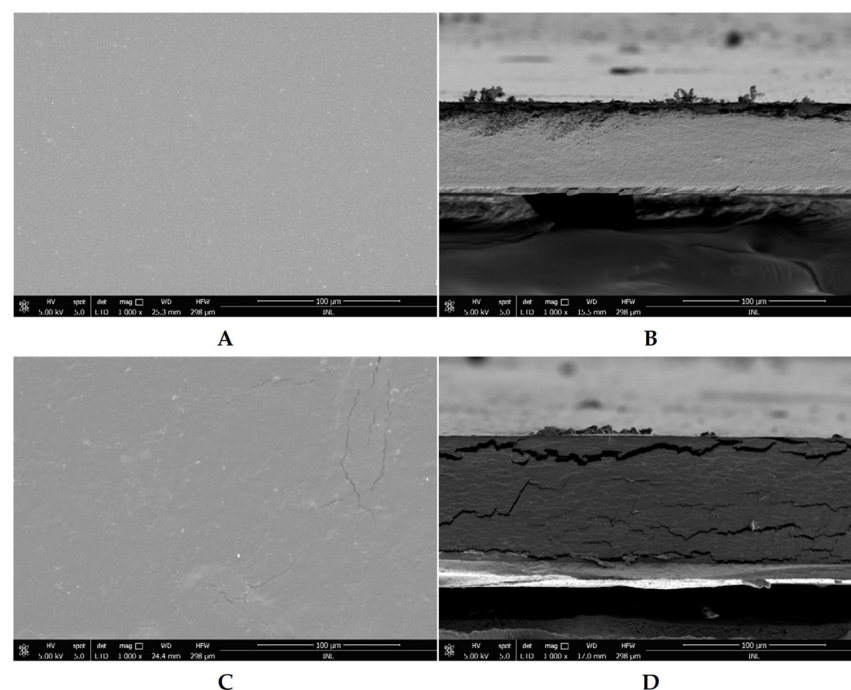


Figure 7. SEM surface images and Cross-section images of PC and OFIPC films: (A)—PC film surface image; (B)—PC film cross-section; (C)—OFIPC film surface image; (D)—OFIPC film cross-section.

On the other hand, the blend of commercial pectin with *Opuntia* cladodes pectin (OFIPC films) leads to a more heterogenous microstructure, with visible agglomerates (Figure 7C), with some cracks and pores that can be visible in the cross-section of the film (Figure 7D). These characteristics observed for OFIPC films may be related to chemical composition of the extracted pectin compared to the commercial pectin. As shown in FTIR analysis (Figure 4), the pectin extracted from *Opuntia* cladodes as a constituent of the OFIPC film contains residual insoluble cell wall polymers (such as cellulose and hemicellulose) and proteins [36,96]. These components tend to aggregate or undergo phase separation from the continuous pectin network during film drying, creating the observed surface irregularities and disrupting the matrix uniformity. Similar microstructural features were also reported by Van Rooyen et al. [104], who reported that cactus pear mucilage films presented irregular and non-homogeneous surfaces with cracks and pores, in contrast to

the smoother and more compact morphologies of pectin and alginate films [104]. Such morphological heterogeneity is consistent with the lower tensile strength observed in the OFIPC films (Table 7), suggesting that structural irregularities directly affected their mechanical integrity. These results are also related to the opacity of the films (Table 7), where OFIPC films were more opaque than the PC films possibly due to the agglomerates observed in SEM images.

Table 7. Optical Properties, Mechanical properties, Moisture, Solubility and Water Vapor Permeability of PC and OFIPC films.

Parameter	PC	OFIPC
L	82.9 ± 0.4 ^b	85.8 ± 0.4 ^a
Chroma	7.0 ± 0.3 ^a	4.8 ± 0.3 ^b
Opacity (mm ^{−1})	1.0 ± 0.0 ^b	1.6 ± 0.1 ^a
Thickness (μm)	116 ± 12 ^a	101 ± 9 ^a
YM (MPa)	2.2 ± 0.8 ^a	0.9 ± 0.1 ^a
TS (MPa)	22.9 ± 2.9 ^a	8.3 ± 1.2 ^a
EAB (%)	20.8 ± 6.1 ^a	12.5 ± 3.7 ^a
Humidity (%)	17 ± 1 ^b	22 ± 2 ^a
Solubility (%)	100 ± 0 ^a	79 ± 1 ^b
WVP (10 ^{−11} g/s.m.Pa)	29.7 ± 4.6 ^a	20.4 ± 1.5 ^b

a, b: values in the same row not sharing lower case superscript letters indicate significant differences among films ($p < 0.05$).

4. Conclusions

This study successfully optimized the extraction of pectin from the peel and pulp of *Opuntia ficus-indica* cladodes using a sustainable approach. Citric acid (1% *w/v*) was selected as the most suitable solvent, and response surface methodology defined the optimal conditions as a liquid-to-solid ratio of 10 *v/w*, 40 min extraction time, and 80 °C. Under these conditions, the extracted pectin from pulp showed a low degree of esterification (35.6%) and high galacturonic acid content (76.9%), confirming its quality and structural integrity comparable to commercial pectin.

Furthermore, the extracted pectin was effectively utilized in the development of films blended with commercial pectin (OFIPC film). These films exhibited balanced optical, mechanical, and barrier properties, including reduced water solubility and significantly improved water vapor barrier performance when compared to pristine commercial pectin films. The results demonstrated that pectin from OFI cladodes has feasibility to be integrated as a food packaging material. To further enhance extraction efficiency, future research could explore advanced techniques such as ultrasound and pulsed electric fields, which may be valuable to improve yield of extraction. In order to improve the film's performance, future studies should be focused on formulation optimization, crosslinking strategies, or incorporation of active compounds and nanoreinforcements, as well as evaluate the performance of these materials in real food packaging systems.

Author Contributions: Conceptualization, C.R., V.G.L.S., I.C. and A.L.F.; methodology, C.R., B.S., V.G.L.S., A.G.A., I.C. and A.L.F.; formal analysis, C.R., B.S., V.G.L.S. and A.G.A.; investigation, C.R., B.S., V.G.L.S. and A.G.A.; data curation, C.R., V.G.L.S., I.C. and A.L.F.; writing—original draft preparation, C.R. and V.G.L.S.; writing—review and editing, C.R., B.S., V.G.L.S., A.G.A., I.C. and A.L.F.; supervision, V.G.L.S., I.C. and A.L.F.; resources, I.C. and A.L.F.; funding acquisition, I.C. and A.L.F. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by national funding by Foundation for Science and Technology (FCT), through the individual research grant (<https://doi.org/10.54499/2020.04441.BD>) of C.R. V.G.L.S thanks FCT/MECI (Portugal) for the individual contract (<https://doi.org/10.54499/2023.09446.CEECIND/CP2>

836/CT0011). This work was supported by Fundação para a Ciência e Tecnologia (FCT)/MECI through the METRICs research unit (UID/04077/2025, <https://doi.org/10.54499/UID/04077/2025>, Mechanical Engineering and Resource Sustainability Center) and by national funds from Fundação para a Ciência e a Tecnologia (FCT), I.P., under the scope of the projects UID/50006/2025, UID/PRR/50006/2025 and LA/P/0008/2020 of the Associated Laboratory for Green Chemistry—LAQV REQUIMTE (<https://doi.org/10.54499/UID/50006/2025>, <https://doi.org/10.54499/UID/PRR/50006/2025> and <https://doi.org/10.54499/LA/P/0008/2020>). This work also received funds from FCT/MECI through project ERANETMED/0001/2017—MediOpuntia (Portugal).

Data Availability Statement: All the data is presented within the manuscript itself in the form of figures and tables.

Acknowledgments: The authors acknowledge Pepe Aromas for the cladodes supplied to conduct this study.

Conflicts of Interest: The authors declare that this study received cladodes (raw material) as support from Pepe Aromas (company). The company was not involved in the study design, collection, analysis, interpretation of data, the writing of this article or the decision to submit it for publication.

Abbreviations

The following abbreviations are used in this manuscript:

CCD	Central composite design
CP	Commercial pectin
DE	Degree of esterification
EAB	Elongation at break
FTIR	Fourier-transform infrared spectroscopy
GAE	Gallic acid equivalent
HM	High methoxyl
LM	Low methoxyl
L/S	Liquid-to-solid ratio
OFI	<i>Opuntia ficus-indica</i>
OFIPC	<i>Opuntia ficus-indica</i> pectin/commercial pectin
PC	Pristine commercial pectin
RH	Relative humidity
RSM	Response surface methodology
SEM	Scanning electron microscopy
TS	Tensile strength
WVP	Water vapor permeability
YM	Young's modulus

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