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Optimized Extraction of *Passiflora ligularis* Pectins: Characterization and Application in Moisturizing Cosmetic Products

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Abstract

Passiflora ligularis (granadilla), widely cultivated in Colombia, contains secondary metabolites such as flavonoids, phenols, and pectins. Owing to their strong water-retention capacity, pectins are promising candidates for moisturizing cosmetic formulations. This study optimized pectin extraction from fruit peel and mesocarp using aqueous reflux at 90 °C and acid extraction with citric or hydrochloric acid (0.25 N and 0.125 N) at 40–60 °C. The effects of solvent, method (reflux or microwave-assisted), time (15–25 min), and temperature (50–60 °C) were investigated. Extracted pectins were dried, lyophilized, and incorporated into eight gel-type cosmetic formulations subjected to seven-day preliminary stability testing (physicochemical and organoleptic evaluation). Optimal extraction was achieved with citric acid under microwave irradiation at 60 °C for 15 min, yielding 45.23%. The pectin exhibited low moisture (0.13%), acidity (0.42%), methoxyl content (9.05%), and degree of esterification (57.6%), along with high swelling capacity (12.46 mL/g) and water-retention capacity (12.26%). The resulting gel formulation was homogeneous and stable. In vitro assays confirmed significant moisturizing activity. These findings highlight *P. ligularis* pectins as sustainable biopolymers with potential as natural gelling and moisturizing agents in cosmetic products.

Keywords: *Passiflora ligularis*; pectins/; cosmetic/formulation; moisturizers; polymers; natural; agricultural waste

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1. Introduction

Skin aging is a physiological process characterized by the progressive decline of essential skin functions, including its ability to retain water. Reduced hydration compromises the structural integrity of the epidermis, leading to dryness; loss of elasticity; wrinkle formation; and a rough, dull appearance [1,2]. The stratum corneum requires optimal hydration to preserve barrier function, elasticity, and a healthy appearance. Consequently, skin moisturizing is a primary objective in the development of cosmetic products designed to prevent or attenuate visible signs of aging. Insufficient moisturizing also disrupts epidermal renewal and barrier function, further exacerbating uneven skin texture and

tone. To counteract these effects, humectants and moisturizers are commonly incorporated into cosmetic formulations to enhance water retention and maintain optimal skin hydration. Due to their diverse composition, these agents can exert two main cosmetic actions: (i) reducing transepidermal water loss through occlusion (by means of humectant ingredients) and (ii) promoting water retention within the stratum corneum (through moisturizing ingredients). Examples include collagen, elastin, hyaluronic acid, glycerin, propylene glycol, and polyethylene glycols (PEG), among others [3–5].

In recent years, natural ingredients have gained increasing relevance as moisturizing agents due to their functional properties, safety, and compatibility with sustainable formulations [6]. Among them, pectins—a class of plant-derived polysaccharides—are of particular interest. Pectins are known for their water-binding ability, gelling capacity, and contribution to texture and stability in cosmetic systems. Beyond their technological applications, several studies suggest that pectins exert beneficial effects on skin moisturizing and restoration. Their performance as gelling and film-forming agents depends largely on their chemical features, particularly galacturonic acid content and the degree of methoxylation [5,7–9].

Passiflora ligularis (granadilla), a member of the Passifloraceae family, is native to the tropical Andes and is the second most economically important species of the genus after passion fruit. Colombia is the leading global producer, with 4500 hectares cultivated and an annual yield of approximately 20,000 tons [10–13]. This species is characterized by a diverse phytochemical profile, including flavonoids, saponins, phenolic compounds, and carbohydrates such as pectins. Structurally, pectins are complex macromolecules composed of up to 17 different monosaccharides and more than 20 types of linkages. They consist mainly of three domains: homogalacturonan (HG), rhamnogalacturonan I (RG-I), and rhamnogalacturonan II (RG-II). HG is the most abundant, representing around 60% of pectins in plant cell walls. The chemical heterogeneity of RG-I and the highly conserved structure of RG-II, together with the methylation and acetylation patterns of HG, strongly influence the functional properties of pectins [7,8,14–16].

Given its rich composition, *P. ligularis* represents a valuable source of pectins that can be extracted from peel and mesocarp. Previous studies have reported favorable extraction yields and physicochemical properties that support its potential for cosmetic applications, particularly in terms of water retention capacity [17]. However, the use of *P. ligularis* pectins in topical formulations remains limited. Therefore, the aim of this study was to assess the in vitro moisturizing potential of pectins extracted from agro-industrial by-products of *P. ligularis*, as a sustainable and innovative approach for the development of cosmetic formulations intended for skin care.

2. Materials and Methods

2.1. Plant Material

Organic residues of *Passiflora ligularis* (granadilla), obtained from domestic fruit consumption, were collected in Medellín (Calazans, Unidad Campo de Verano; Chagualo, Unidad Ciudadela Sevilla) and Sabaneta (Barrio San José, Edificio Barcelott), Colombia, and subsequently used for pectin extraction. The residues consisted of peel and mesocarp, which were thoroughly washed with potable water followed by deionized water, oven-dried at 40 °C for 48 h and subsequently milled and sieved to achieve a uniform particle size distribution (<800 µm).

2.2. Exploratory Extraction

Two extraction methods were initially evaluated. The first consisted of a hot-water reflux extraction at 90 °C, using a ratio of 1 g of plant material to 20 mL of solvent. The

second method involved acid extraction with citric acid (0.125 N and 0.250 N) and hydrochloric acid (0.125 N and 0.250 N) at 40 °C and 60 °C. In total, 18 trials were performed, covering all combinations of solvent type, acid concentration, temperature (40–60 °C), and extraction time (30–60 min), including two additional hot-water extractions at 90 °C for 30 and 60 min. The extracted pectins were precipitated with 95% v/v ethanol, dried, and weighed to calculate yield (%) [18].

2.3. Experimental Design

Based on the exploratory stage, a 24 factorial design was implemented to evaluate solvent (water and citric acid), extraction method (reflux and microwave-assisted), temperature (50 °C and 60 °C), and time (15 and 25 min). Sixteen experimental combinations were performed in triplicate, using extraction yield (%) as the response variable. Each pectin extract was filtered and precipitated with two volumes of 95% v/v ethanol. The recovered pectins were lyophilized, milled into a fine powder, and stored for subsequent analyses [19–21].

2.4. Pectin Characterization

Moisture content was determined by drying the samples at 105 °C for 24 h and calculated using Equation (1) [22]. The acidity percentage was measured by acid–base titration with 0.1 N sodium hydroxide, using phenolphthalein as an indicator, and calculated according to Equation (2) [23]. Briefly, 0.2 g of pectin were dissolved in 50 mL of distilled water and titrated with 0.1 N NaOH until a faint pink color appeared (initial titration). The milliequivalents (*meq*) of NaOH consumed in this step were recorded as *meq A NaOH* (*free acidity*). Subsequently, 10 mL of 0.1 N NaOH were added, the solution was shaken, and allowed to stand for 30 min. Thereafter, 10 mL of 0.1 N HCl were added until the pink coloration disappeared. A second titration with 0.1 N NaOH was then carried out until the faint pink color persisted. The milliequivalents of NaOH consumed in this second titration were recorded as *meq B NaOH* (*methoxyl content*). The *methoxyl content* was calculated from the second titration (Equation (3)) [24], while the degree of esterification was determined based on the *meq A NaOH* and *meq B NaOH* consumed in both titrations (Equation (4)) [25].

$$\%\ Moisture = \frac{(Initial\ weight - Final\ weight)}{(Initial\ weight)} \times 100 \quad (1)$$

$$\%\ Acidity = \frac{(meq\ A\ NaOH)}{(mg\ of\ pectin)} \times 100 \quad (2)$$

$$\%\ Methoxyl = \frac{(meq\ B\ NaOH \times 31)}{(mg\ of\ pectin)} \times 100 \quad (3)$$

$$\%\ Esterification = \frac{(meq\ B\ NaOH\ (methoxyl\ content))}{(meq\ A\ NaOH\ (free\ acidity) + meq\ B\ NaOH\ (methoxyl\ content))} \times 100 \quad (4)$$

2.5. Swelling Capacity and Water Retention Capacity

Swelling capacity was determined by measuring the volumetric increase of the sample after 24 h of hydration. Briefly, 0.400 g of dry pectin was gradually dispersed in 10 mL of distilled water in a graduated cylinder [26]. The mixture was allowed to hydrate at room temperature for 24 h, after which the volume of the solid phase and the final volume of the hydrated sample were recorded (Equation (5)).

Water retention capacity was assessed by centrifuging the hydrated samples at 3000 rpm for 10 min at room temperature, followed by gravimetric analysis. The parameter was calculated according to Equation (6), where *W1* corresponds to the weight of

the hydrated material after centrifugation or draining, and *W0* to the initial dry weight of the sample.

$$Swelling\ capacity = \frac{(Final\ volume - Initial\ volume)}{Sample\ weight} \quad (5)$$

$$Water\ retention\ capacity = \frac{(W1 - W0)}{W0} \times 100 \quad (6)$$

2.6. ATR-FTIR and NMR Spectroscopy

ATR-FTIR spectra were acquired using a Thermo Nicolet iS50 spectrophotometer equipped with a diamond crystal. Measurements were recorded in the 4000–400 cm^{-1} range at a resolution of 4 cm^{-1}, averaging 64 scans per sample. Spectral data were subsequently processed with ATR corrections and baseline adjustments to enhance signal quality.

For NMR analysis, 10 mg of pectin were dissolved in 0.5 mL of deuterium oxide (D_2O). Proton (^1H), DEPT-135, and HSQC spectra were obtained on a Bruker Ascend III HD 600 MHz spectrometer at room temperature. Chemical shifts were expressed in parts per million (ppm).

2.7. Preparation of Cosmetic Formulations

Table 1 summarizes the exploratory formulation, designed to obtain a hydrating gel with light consistency, acceptable stability, and pleasant organoleptic properties. Based on this initial approach, eight hydrating gel formulations (Table 2) were developed using pectin extracted from *P. ligularis*, aiming to optimize physicochemical stability, texture, sensory attributes, and skin compatibility. All cosmetic-grade ingredients used in the formulations were obtained from LyM and LyF Chemicals (Medellín, Colombia) and verified in the European Commission's CosIng database to ensure their safety, authorized cosmetic use, and compliance with permitted concentration limits [27].

Table 1. Base formulation containing pectin.

Ingredient (INCI Name)	Concentration (% *w*/*w*)	Main Function
Aqua	93.1	Solvent
Pectin (*P. ligularis*)	5.0	Binder/Emulsion stabilizer
Phenoxyethanol, Ethylhexylglycerin	1.0	Antimicrobial/Preservative
Carbomer	0.5	Rheology modifier
Ascorbic acid	0.4	Antioxidant
Sodium Hydroxide	q.s.	pH adjuster

The formulation process was carried out in two phases. Phase A consisted of dispersing Carbomer or guar gum in deionized water, followed by pH adjust with the selected base (triethanolamine or sodium hydroxide) until reaching a pH of 5.5, 6.0 or 6.5 (Table 2). Phase B contained pectin, the antioxidant, and the preservative, which were dispersed separately to prevent premature interactions that could compromise system stability. Both phases were then combined under controlled stirring and subsequently homogenized at 300 rpm using a rotor–stator system (Ultra-Turrax T25 Basic, IKA, Staufen, Germany), ensuring uniform ingredient distribution and final pH adjustment to 5.5.

Table 2. Modifications in the pectin-based formulations and functional classification according to the European Commission (CosIng database).

INCI Name	Concentration (% *w*/*w*)								
		Main Function	**F1**	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**
Aqua		Solvent	q.s. to 100%							
Pectin (*P. ligularis*)	5.0	Moisturizing/ Gelling agent	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Phenoxyethanol and Ethylhexyglycerin	1.0	Preservative	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Ascorbic Acid	0.4	Antioxidant	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Sodium Hydroxide (1 M)	q.s.	pH adjuster	q.s. *	q.s. *	q.s. *	0.0	0.0	0.0	0.0	0.0
Carbomer	0.5/1.0	Rheology modifier	0.0	0.5	1.0	1.0	0.0	0.0	1.0	1.0
Triethanolamine (TEA)	2.8	pH adjuster	0.0	0.0	0.0	2.4 (pH6.5)	2.4 (pH5.5)	2.4 (pH5.5)	2.2 (pH6.0)	2.0 (pH5.5)
Guar Hydroxypropyltrimonium Chloride	0.4/1.0	Rheology modifier	0.0	0.0	0.0	0.0	0.4	1.0	0.0	0.0

* Quantity sufficient to pH c.a.5.5.

2.8. Preliminary Stability

The formulation was stored for seven days under the conditions described in Table 3 to simulate thermal and environmental stress. Gel formulations were evaluated for the attributes of color, odor, appearance, and texture by five individually selected sensory evaluators. The gel samples were spread on white paper sheets and evaluated under white light. Descriptive and acceptance analysis of each of the attributes described was carried out, under the criteria of acceptability of a cosmetic product. In the evaluation of the appearance and texture, all the mechanical, geometric, and surface attributes of the formulations, perceptible through touch and vision, were evaluated [28–30]. Application-related parameters, including drying time and residual skin feel, were also recorded. In addition, pH was monitored to evaluate chemical stability and potential skin compatibility.

Table 3. Conditions for preliminary stability testing.

Condition	Parameter
Centrifugation	3000 rpm, 30 min
High temperature	40 °C, 24 h
Room temperature	25 °C, 24 h
Refrigeration	2–8 °C, 24 h
Freezing	−5 °C, 24 h
Sunlight (UVA, UVB, Visible)	2 h

Photostability was assessed by exposing the formulation, contained in a beaker, for 2 h in a solar simulator (Solarbox 1500e; Erichsen, Hemer, Germany) equipped with a 1500 W xenon arc lamp and UV glass filters to block radiation below 290 nm. Irradiance was maintained at 325 W/m^2 in accordance with global solar spectral standards. Sensory analysis was subsequently performed to detect color changes indicative of oxidation [31–33].

2.9. Statistical Analysis

Multiple range tests were performed using Fisher's least significant difference (LSD) procedure, and a four-way ANOVA was applied to assess possible interactions among variables. Statistical significance was considered at $p < 0.05$. All tests were performed in triplicate and results are reported as mean ± standard deviation (SD). Data were analyzed using Statgraphics Centurion software version 19 (Statgraphics Technologies, Inc., The Plains, VA, USA).

3. Results and Discussion

3.1. Exploratory Extraction

The exploratory extraction revealed significant differences in pectin yield under the evaluated conditions. The highest yields were obtained using distilled water at 90 °C for 60 and 30 min, with values of 4.54% and 4.08%, respectively. Similarly, extraction with hydrochloric acid (0.125 N) at 60 °C for 60 min yielded 3.81%. For citric acid, the maximum yield was achieved with 0.125 N at 60 °C for 60 min, resulting in 3.08%.

When comparing solvents, water exhibited the highest average yield (4.31%), followed by hydrochloric acid (3.13%) and citric acid (2.14%). These results confirm that solvent type, temperature, and extraction time significantly influenced ($p < 0.05$) both yield and physicochemical properties of the extracted pectins.

However, pectins extracted with hydrochloric acid displayed a rigid and brittle texture, attributed to hydrolysis by this strong acid, which promotes demethylation and markedly reduces the degree of esterification (DE) and galacturonic acid content. A lower DE results in low-methoxyl pectins, which form harder gels through divalent ion interactions [34].

Conversely, citric acid (a weak acid) yielded pectins with more suitable physical characteristics for topical formulations. This mild hydrolysis preserved the polysaccharide backbone more effectively and favored higher methoxyl content [34]. As a result, the gels obtained were softer, more flexible, and easier to handle, particularly in the presence of sugar and acid, thereby fulfilling cosmetic formulation requirements [35–37].

Although citric acid extractions produced slightly lower yields, the quality and functional performance of the gels were prioritized over quantity. Therefore, only water and citric acid were selected as solvents for the subsequent experimental design to ensure the stability and efficacy of the final formulation.

3.2. Experimental Design

A 2^4 factorial design was used to evaluate the effect of four factors on the yield of pectin extracted from *P. ligularis*: solvent, extraction method, extraction time, and temperature. ANOVA revealed that the most influential factors were the extraction method, solvent type, and their interaction, all statistically significant ($p < 0.05$). The determination coefficient (R^2) of 95.01% confirmed the robustness and accuracy of the model.

Microwave-assisted extraction consistently yielded higher percentages than reflux. The maximum average yield (47.37%) was obtained with citric acid at 50 °C for 15 min (E15), followed by E13 (citric acid, 60 °C, 15 min; 45.97%) and E14 (citric acid, 60 °C, 25 min; 32.85%). In contrast, reflux extractions with citric acid (E5–E8) produced negligible yields (<2%), confirming their low efficiency (Table 4).

Table 4. Experimental design results for reflux and microwave extraction methods.

Experiment	Method	Solvent	Temperature (°C)	Time (min)	Yield (%)
E1	Reflux	Water	60	15	6.387 ± 0.400
E2	Reflux	Water	60	25	6.149 ± 1.400
E3	Reflux	Water	50	15	6.067 ± 1.100
E4	Reflux	Water	50	25	5.847 ± 1.800
E5	Reflux	Citric acid	60	15	0.499 ± 0.300
E6	Reflux	Citric acid	60	25	0.513 ± 0.080
E7	Reflux	Citric acid	50	15	1.886 ± 0.200
E8	Reflux	Citric acid	50	25	0.965 ± 0.300
E9 *	Microwave	Water	60	15	19.300 ± 0.100
E10 *	Microwave	Water	60	25	22.435 ± 3.500
E11	Microwave	Water	50	15	13.544 ± 6.800
E12 *	Microwave	Water	50	25	14.530 ± 0.300
E13	Microwave	Citric acid	60	15	45.970 ± 5.500
E14 *	Microwave	Citric acid	60	25	32.855 ± 0.700
E15	Microwave	Citric acid	50	15	47.373 ± 3.500
E16	Microwave	Citric acid	50	25	25.496 ± 2.200

* Pectin precipitation was not achieved.

Water-based extractions yielded intermediate values, ranging from 5.84% to 22.43%. Within this group, the highest results were obtained with E10 (water, 60 °C, 25 min; 22.43%) and E12 (water, 50 °C, 25 min; 14.53%). However, under some microwave conditions with water or citric acid (E9, E10, E12, E14), effective pectin precipitation was not achieved, possibly due to the co-extraction of soluble solids (low-molecular-weight carbohydrates or phenolic compounds), which can interfere with alcohol-induced pectin precipitation. In these cases, no measurable solid fraction was recovered; therefore, the yield was calculated by drying the extract–alcohol mixture. This observation underscores the importance of evaluating not only extraction yield but also extract quality when selecting optimal processing conditions.

These findings are consistent with previous studies demonstrating that microwave-assisted extraction enhances efficiency by generating rapid and selective volumetric heating, promoting cell wall disruption and polysaccharide release while minimizing thermal degradation [38–40]. Conversely, reflux extraction is less effective due to limited energy transfer and potential degradation of plant material under prolonged heat exposure.

The theoretical optimal yield was estimated at 45.23% (citric acid, microwave, 60 °C, 15 min). Nevertheless, no statistically significant differences were found compared to 50 °C and 15 min, based on multiple range tests. Therefore, 50 °C was selected as the definitive condition to reduce energy consumption and prevent excessive degradation of the pectin. Main effects analysis showed positive slopes for all factors except time, with extraction method exerting the greatest impact. Interaction plots confirmed that the combination of microwave assistance and citric acid solvent was the most effective condition for pectin recovery.

The yields obtained in this study (up to 47.37%) are comparable to or even higher than those reported for pectins extracted from conventional sources such as citrus peel (20–35%) and apple pomace (10–20%) [19–21]. They are also superior to yields described for passion fruit (*Passiflora edulis*) by-products, which generally range from 10% to 25% depending on the extraction method [16]. These results highlight *P. ligularis* peel and mesocarp as a promising, underutilized agro-industrial residue for pectin production. Beyond yield, the selection of citric acid under microwave-assisted conditions provides an environmentally

friendly and efficient alternative that aligns with sustainable development goals while ensuring functional properties suitable for cosmetic applications.

3.3. Pectin Characterization

Characterization of *P. ligularis* pectin included the determination of moisture content, degree of esterification, acidity, methoxyl content, swelling capacity, and water retention capacity, with results summarized in Table 5.

Table 5. Pectin characterization results.

Parameter	Result ± SD
Moisture (%)	0.13 ± 0.01
Acidity (%)	0.42 ± 0.01
Methoxyl content (%)	9.05 ± 0.01
Degree of esterification (%)	57.6 ± 0.03
Swelling capacity (mL/g)	12.46 ± 0.01
Water retention (%)	12.26 ± 0.01

The moisture content of the extracted pectin was very low (0.13 ± 0.01%), well below the maximum limit of 12.0% established for commercial pectin [41,42], highlighting the efficiency of the lyophilization process. Such low moisture is advantageous because it reduces the risk of hydrolytic degradation and microbial growth during storage, thereby improving physicochemical stability and shelf life [42,43]. These results indicate that the extracted pectin meets quality criteria for use in food, pharmaceutical, and cosmetic applications. The acidity percentage fell within the range reported for moderately acidic pectins (0.3–1.0%), indicating that *P. ligularis* pectin exhibits moderate acidity [23]. This parameter is influenced by galacturonic acid content, degree of esterification, and extraction conditions, particularly pH and processing temperature [23].

The methoxyl content confirmed that *P. ligularis* pectin is classified as high-methoxyl pectin (>7% methoxyl, DE > 50%) [37]. Such classification is associated with strong gelling capacity in acidic media with high sugar concentrations, which is advantageous for both food and cosmetic applications [38]. In cosmetics, these properties contribute to the formation of stable gels with suitable viscosity and spreadability, while also enabling the formation of a protective film on the skin that enhances water retention and improves sensory perception.

The polymer also demonstrated excellent water absorption and swelling capacities, with a swelling value of 12.46 ± 0.01 mL/g and a water retention capacity of 12.26 ± 0.01%. These values reflect a highly hydrophilic structure, indicating a polymeric network with functional groups readily available for interaction with water. Such features are beneficial for applications requiring moisture stabilization or the development of viscous, film-forming systems and it is correlate with the signals found in the FT-IR analysis [42–46].

The FT-IR spectrum of *P. ligularis* pectin (Figure 1) exhibited a broad band at 3377 cm^{-1} corresponding to –OH stretching, partly from polysaccharide hydroxyl groups and absorbed water. The band at 2918 cm^{-1} was assigned to CH, CH_2, and CH_3 stretching vibrations of the methyl esters in galacturonic acid [40]. A strong peak at 1718 cm^{-1} was attributed to ester groups ($COOCH_3$), whose intensity indicated high methoxyl content, corroborating the NaOH titration results. Bands at 1610 cm^{-1} and 1403 cm^{-1} correspond to –OH and $–COO^-$ stretching, while signals between 1015 and 1100 cm^{-1} arise from C–O–C and C–O stretching vibrations of galacturonic acid units [47]. These findings are consistent with previous studies on pectins from Passifloraceae species, which display similar spectral profiles with variations in relative peak intensities due to differences in extraction method, processing conditions, and plant fraction used (peel or mesocarp) [48].

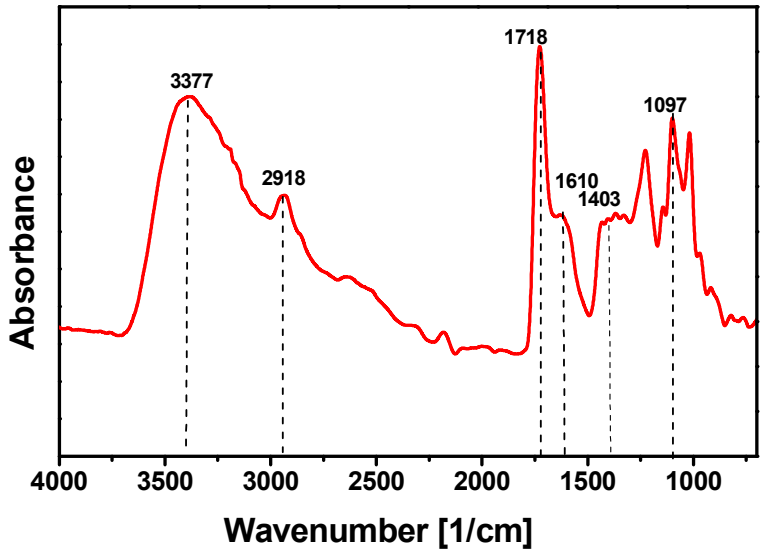

Figure 1. FTIR Spectrum of granadilla pectin shows the main functional groups. The broad band at 3377 cm^{-1} corresponds to –OH stretching vibrations from polysaccharide hydroxyl groups and absorbed water. Peaks at 2918 cm^{-1} and 1718 cm^{-1} are attributed to C–H stretching and ester ($COOCH_3$) groups, respectively. Bands at 1610 cm^{-1} and 1403 cm^{-1} correspond to –OH and $–COO^-$ stretching, while signals between 1015 and 1100 cm^{-1} arise from C–O–C and C–O stretching vibrations of galacturonic acid units [47,49].

^{1}H NMR analysis (Figure 2A) showed a sharp signal at 3.57 ppm corresponding to the $–OCH_3$ protons of esterified galacturonic acid. Four characteristic signals of D-galacturonic acid were observed: H-1 at 4.75 ppm, H-2 at 3.29 ppm, H-3 at 3.93 ppm, and H-4 at 4.05 ppm, in agreement with previously reported values. Weak peaks at 1.98 and 2.08 ppm corresponded to O-acetyl groups ($–COCH_3$). A minor resonance at 7.86 ppm was attributed to residual phenolic compounds [43], which are common in Passiflora extracts due to the peel's richness in flavonoids and polyphenols [50]. The ^{13}C NMR spectrum (Figure 2B) showed a strong peak at 57.14 ppm assigned to the methyl carbons linked to galacturonic acid carboxyl groups, along with characteristic signals at 100.40, 67.76, 71.10, 78.51, and 173.04 ppm for C-1, C-2, C-3, C-4, and C-6 of D-GalA, respectively [44,51].

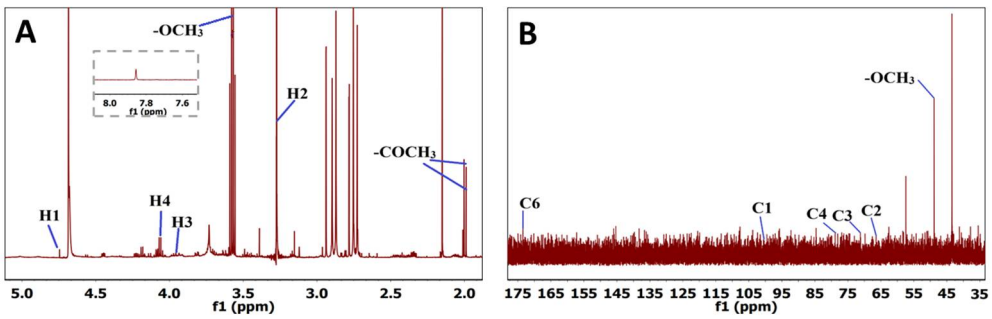

Figure 2. (**A**) ^{1}H NMR and (**B**) ^{13}C NMR of spectra of granadilla pectin. ^{1}H NMR (**A**) shows characteristic D-galacturonic acid protons (H1–H4) and $–OCH_3$ and $–COCH_3$ signals, confirming methyl-esterified and acetylated structures. ^{13}C NMR (**B**) displays typical D-GalA carbons (C1–C6) and a strong $–OCH_3$ resonance at 57 ppm.

Overall, the physicochemical and structural profile of *P. ligularis* pectin (low moisture, high methoxyl content, and moderate acidity) confirms its classification as a high-methoxyl pectin with strong gelling potential. The combination of excellent swelling, water-binding capacity, and gel-forming properties suggests broad applicability, ranging from food sys-

tems that require gel stability and moisture control to cosmetic formulations designed to enhance moisturizing, viscosity, and sensory attributes.

3.4. Cosmetic Gel Formulations and Preliminary Stability

To identify the most stable formulation, a preliminary physical and sensory stability study was conducted on eight cosmetic gels designed with different gelling systems and pH ranges. Distinct behavioral patterns were observed under stress conditions of heating, refrigeration, time, and light exposure (Table 3). The results of the physicochemical and organoleptic characteristics of the evaluated formulations are summarized in Table 6.

Table 6. Physicochemical and sensory characteristics of preliminary gel formulations under stress conditions.

Formulation	Gelling System	pH Range	Centrifugation (Day 1)	Heat Stability (40 °C)	Cold Stability (2–8 °C)	Viscosity Change	Sensory Changes (Color/Odor/Texture)
F1	Pectin only	4.5–5.0	Stable	Significant loss	Moderate precipitation	↓↓	Color/odor altered
F2	Carbomer (pH 6.0)	6.0–6.2	Stable	Moderate stability	Strong precipitation	↓↓	Odor decreased, texture affected
F3	Carbomer (pH 6.2)	6.0–6.3	Stable	Moderate stability	Strong precipitation	↓↓	Odor decreased, pH fluctuation
F4	Carbomer (pH 6.5)	6.3–6.5	Stable	Moderate stability	Strong precipitation	↓↓	Texture/graininess
F5	Guar gum	5.0–5.5	Stable	Major instability	Precipitation	↓↓↓	Residues on skin, odor altered
F6	Guar gum	5.5–6.0	Stable	Major instability	Precipitation	↓↓↓	Fluid consistency, odor loss
F7	Carbomer (neutralized, pH $\geq$ 6.5)	6.5–6.7	Stable	High stability	Mild precipitation	↓	Good color/odor retention
F8	Carbomer (neutralized, pH $\geq$ 6.5)	6.6–6.8	Stable	High stability	Mild precipitation	↓	Best preserved texture & odor

Legend: ↓ = slight decrease, ↓↓ = moderate decrease, ↓↓↓ = strong decrease.

In the initial centrifugation test (day 1), all formulations were physically stable, with no evidence of phase separation, indicating good initial resistance to mechanical forces simulating accelerated aging. However, differences among formulations became more evident under extreme storage conditions. At elevated temperatures (40 °C), the formulation prepared exclusively with pectin and no carbomer (F1), as well as those containing guar gum (F5 and F6), showed a marked loss of viscosity accompanied by sensory changes such as alterations in color and odor. These results suggest that these gelling systems alone do not provide a sufficiently robust matrix to resist thermal degradation, likely due to the disruption of the polymeric network.

In contrast, formulations containing carbomer at pH $\geq$ 6.5 (particularly F7 and F8) demonstrated superior thermal resistance. These samples more consistently preserved their organoleptic characteristics, including viscosity, color, and odor, with less deterioration under heat stress, indicating improved colloidal stability when carbomer is adequately neutralized.

Cold storage (2–8 °C) also revealed critical challenges. Refrigeration generally induced clumping or precipitation in most formulations, negatively impacting texture and spreadability. Formulations F2, F3, and F4, which contained carbomer at slightly acidic pH (6.0–6.5), were the most affected, showing decreased viscosity, reduced odor inten-

sity, and pH fluctuations. This finding reinforces the observation that slightly acidic pH values can compromise the integrity of polymeric systems, leading to both physical and sensory instability.

With respect to pH, most formulations remained within the cosmetically acceptable range (4.5–6.5). However, significant fluctuations were observed in formulations with higher pH values (F7 and F8), indicating lower compatibility and formulation control under these conditions. This is particularly relevant since pH influences both gel stability and the safety/tolerability of the final product. Despite pH fluctuations observed in the evaluated formulations, all values remained within the range of 4.5–6.8 (slightly acidic), which is consistent with the physiological pH of the skin, approximately 4.7–5.7.

A general reduction in aromatic intensity (citrus, fruity, and honey notes) was observed under both hot and cold stress conditions. Nevertheless, formulations F7 and F8 preserved their olfactory profile more effectively, an important attribute for consumer perception and product freshness. Similarly, texture and ease of application were severely compromised in guar gum-based gels (F5 and F6), which tended to leave residues on the skin or became excessively fluid after stress cycles. In contrast, F7 and F8 maintained a stable, homogeneous sensory profile without clumping or loss of spreadability, even at the end of the observation period.

Among these, formulation F8 can be considered the most robust and sensorially acceptable, as it combined physical stability, favorable thermal performance, and positive sensory perception under simulated storage conditions (Tables 6 and 7). For this reason, it was selected for photostability testing under simulated light exposure. After two hours, noticeable darkening, the formation of a denser and more elastic texture, and visible volume loss were observed, changes consistent with radiation-induced degradation, likely involving ascorbic acid and the gelling system. These results indicate that the formulation is photosensitive. Therefore, opaque or UV-protective packaging is required, along with clear storage instructions to prevent direct sunlight exposure and preserve integrity and efficacy during consumer use [52].

Table 7. Overall stability and sensory performance of selected formulations.

Formulation	Thermal Stability	Cold Stability	pH Stability	Sensory Attributes (Odor, Texture, Spreadability)	Overall Performance
F1	Low	Moderate	Stable	Altered color/odor, rigid texture	Poor
F2–F4	Moderate	Low	Unstable	Reduced odor, precipitation, texture changes	Limited
F5–F6	Very low	Low	Stable	Residues on skin, loss of viscosity, odor loss	Poor
F7	High	Moderate	Moderate	Good texture/odor retention	Good
F8	High	Moderate	Moderate	Excellent texture, odor, and spreadability	Best (selected)

4. Conclusions

The methodological strategy implemented allowed the optimization of the pectin extraction process from *Passiflora ligularis* residues, demonstrating that the use of citric acid as a solvent, in combination with microwave-assisted extraction at 50 °C for 15 min, maximizes yield while preserving the physicochemical properties of the biopolymer. This condition not only showed reproducibility but also favored the recovery of high-methoxyl

pectins, suitable for cosmetic applications due to their proven moisturizing capacity associated with excellent water retention. The factorial design and ANOVA analysis confirmed that the extraction method was the most influential factor, establishing a robust basis for future production scaling and applications in sustainable natural products. Preliminary stability tests on moisturizing formulations enabled the selection of a final version (Formulation 8) that met criteria of homogeneity, physical stability, acceptable rheological behavior, and skin compatibility. This formulation maintained its organoleptic characteristics, viscosity, and pH within optimal ranges under thermal and environmental stress conditions, without phase separation or sensory deterioration. The incorporation of extracted pectins as a gelling agent proved functional and effective, positioning this biomolecule as a viable, natural-origin cosmetic ingredient. Overall, these findings highlight the potential of *P. ligularis* pectin as a multifunctional biopolymer for cosmetic applications, combining gelling capacity, water retention, and consumer-acceptable sensory attributes. Future studies should address long-term stability, scalability of the extraction process, and clinical evaluations to further validate its effectiveness and safety in topical formulations.

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