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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 (1246.26%) and water-retention capacity (1225.77%). 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*; pectin extraction; cosmetic formulation; moisturizing agent; Natural polymers; agro-industrial by-products

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 widely incorporated into cosmetic formulations to enhance water retention and maintain skin hydration [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. Among them, pectins—a class of plant-derived polysaccharides—are of particular interest. Pectins are known for

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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 [5–8]

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 4,500 hectares cultivated and an annual yield of approximately 20,000 tons [9–12]. 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 [6,7,13–15].

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 [16]. However, the use of P. ligularis pectins in topical formulations remains limited. Therefore, the present study aims to evaluate the moisturizing potential of cosmetic formulations incorporating pectins extracted from agro-industrial by-products of P. ligularis, proposing a sustainable and innovative alternative for skin care.

2. Materials and Methods

2.1. Plant Material

Organic residues of Passiflora ligularis (granadilla), obtained from domestic fruit consumption, were employed 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.

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 by varying solvent type, acid concentration, temperature, and extraction time. The extracted pectins were precipitated with 95% v/v ethanol, dried, and weighed to calculate yield (%).

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 [17–19].

2.4. Pectin Characterization

Moisture content was determined by drying the samples at 105 °C for 24 h and calculated using Equation 1 [20]. The acidity percentage was measured by acid–base titration with 0.1 N sodium

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hydroxide, using phenolphthalein as an indicator, and calculated according to Equation 2 [21]. 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). 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 (final titration). The methoxyl content was calculated from the second titration (Equation 3), while the degree of esterification was determined based on the milliequivalents of NaOH consumed in both titrations (Equation 4).

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

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

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

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

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 were gradually dispersed in 10 mL of distilled water in a graduated cylinder [22]. 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 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.

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

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

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 Passiflora ligularis, aiming to optimize physicochemical stability, texture, sensory attributes, and skin compatibility. All ingredients were verified in the European Commission's CosIng database to ensure safety, permitted concentrations, and authorized cosmetic use [23].

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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

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	q.s. to 100%	Solvent	X	X	X	X	X	X	X	X
Pectin (*P. ligularis*)	5.0	Moisturizing/Gelling agent	X	X	X	X	X	X	X	X
Phenoxyethanol and Ethylhexylglycerin	1.0	Preservative	X	X	X	X	X	X	X	X
Ascorbic Acid	0.4	Antioxidant	X	X	X	X	X	X	X	X
Sodium Hydroxide (1M)	q.s.	pH adjuster	X	X	X	X	—	—	X	X
Carbomer	0.5/1.0	Rheology modifier	—	X	X	X	—	—	X	X
Triethanolamine (TEA)	2.8 %	pH adjuster	—	—		X	X	X	X	X
Guar Hydroxypropyltrimonium Chloride	0.4/1.0	Rheology modifier	—	—	—	—	X	X	—	—

X: Ingredient present in the formulation, —: Ingredient not included in the formulation. **Formulation 1 (F1):** Base formulation. **Formulation 2 (F2):** Incorporation of Carbomer at 0.5%. **Formulation 3 (F3):** Adjustment of Carbomer concentration to 1.0%. **Formulation 4 (F4):** Adjustment of ascorbic acid concentration to 0.5% and addition of triethanolamine (TEA) as neutralizer. **Formulation 5 (F5):** Replacement of Carbomer with guar gum at 0.4%. **Formulation 6 (F6):** Replacement of Carbomer with guar gum at 1.0%. **Formulation 7 (F7):** Evaluation of Formulation 4 at different pH values (5.5, 6.0, 6.5). **Formulation 8 (F8):** Selection of Formulation 7 at pH 5.5, with TEA at 2.8%.

The formulation process was carried out in two phases. Phase A consisted of dispersing Carbomer in deionized water, followed by neutralization with the selected base (triethanolamine or sodium hydroxide) until reaching an approximate pH of 6.0. 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, Germany), ensuring uniform ingredient distribution and final pH adjustment to 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. Visual and tactile evaluations were performed to assess color, homogeneity, phase separation, texture, flow, and viscosity. Olfactory analysis was carried out to detect signs of degradation or microbial contamination. 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.

Photostability was assessed by exposing the formulation, contained in a beaker, for 2 h in a solar simulator (Solarbox 1500e; Erichsen, 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/m² in accordance with

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global solar spectral standards. Sensory analysis was subsequently performed to detect color changes indicative of oxidation [24–26].

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

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 results are reported as mean ± standard deviation (SD). Data were analyzed using Statgraphics Centurion software (Statgraphics Technologies, Inc., 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 minutes, with values of 4.54% and 4.08%, respectively. Similarly, extraction with hydrochloric acid (0.125 N) at 60 °C for 60 minutes yielded 3.81%. For citric acid, the maximum yield was achieved with 0.125 N at 60 °C for 60 minutes, 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. The highest yields were achieved with water at 90 °C and hydrochloric acid at 60 °C.

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 [27].

Conversely, citric acid (a weak acid) yielded with more suitable physical characteristics for topical formulations. This mild hydrolysis preserved the polysaccharide backbone more effectively and favored higher methoxyl content [27]. 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 [28–30].

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.

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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 minutes (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	Yield (%)
E1	6.387 ±0.400
E2	6.149 ±1.400
E3	6.067±1.100
E4	5.847 ±1.800
E5	0.499 ±0.300
E6	0.513±0.080
E7	1.886±0.200
E8	0.965±0.300
E9*	19.300 ±0.100
E10*	22.435 ±3.500
E11	13.544 ±6.800
E12*	14.530 ±0.300
E13	45.970 ±5.500
E14*	32.855 ±0.700
E15	47.373 ±3.500
E16	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 interfering soluble solids, which highlights the need to assess not only yield but also extract quality.

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 [31–33]. 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 microwave–citric acid combination 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%) [17–19]. 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 [15]. 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.

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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.

The moisture content of the extracted pectin was significantly lower than the maximum limit of 12.0% established for commercial pectin [34,35], highlighting the efficiency of the lyophilization process. Such a low moisture level enhances physicochemical stability and minimizes the risk of microbial deterioration during storage [35,36]. The acidity percentage fell within the range reported for moderately acidic pectins (0.3–1.0%), indicating that *P. ligularis* pectin exhibits moderate acidity. This parameter is influenced by galacturonic acid content, degree of esterification, and extraction conditions, particularly pH and processing temperature [21].

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 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 creation 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 [35–39].

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. The band at 1610 cm^{-1} was associated with –OH stretching, the peak at 1403 cm^{-1} with symmetric stretching of $–COO^-$ and CH_3 groups, and absorptions between 1015–1100 cm^{-1} with C–O vibrations [41]. 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) [42].

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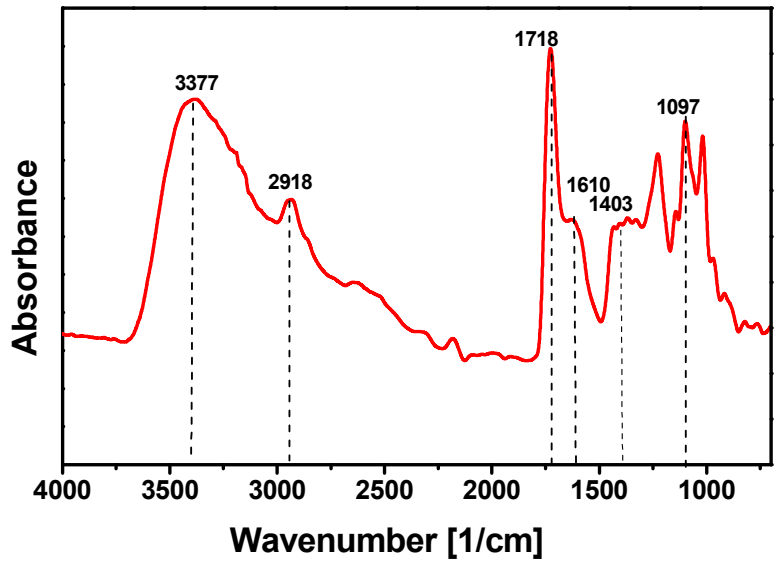

Figure 1. FTIR Spectrum of granadilla pectin.

^{1}H NMR analysis (Figure 2A) revealed a sharp signal at 3.57 ppm, assigned to the –OCH_3 protons of esterified galacturonic acid, reinforcing the classification as high-methoxyl pectin. Four characteristic signals were identified for D-GalA protons: 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, consistent with previous reports. 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 [37]. 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 [39,44].

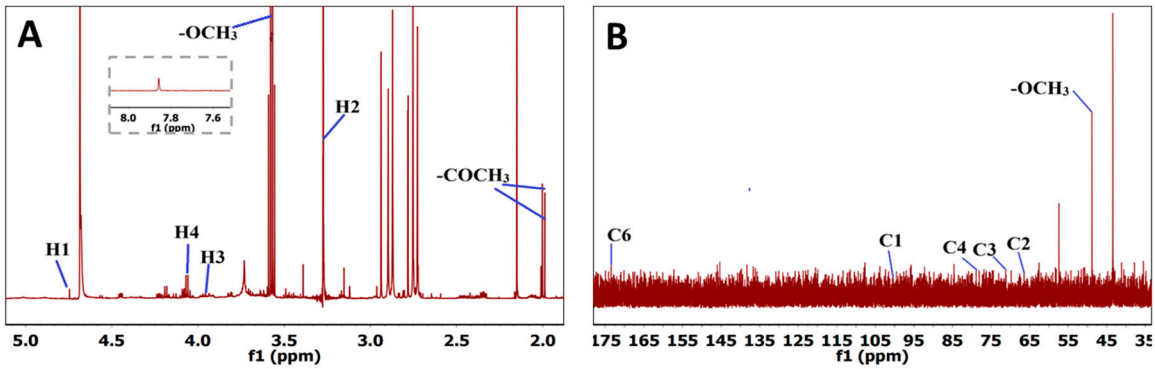

Figure 2. 1H NMR spectrum (A) and 13C NMR of (B) of granadilla pectin.

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

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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 ≥6.5)	6.5–6.7	Stable	High stability	Mild precipitation	↓	Good color/odor retention
F8	Carbomer (neutralized, pH ≥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 ≥ 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 intensity, 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.

Olfactory analysis showed a generalized loss of aromatic intensity (citrus, fruity, and honey notes) under both hot and cold stress conditions. Nevertheless, formulations F7 and F8 preserved

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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, highlighting the need for opaque or UV-protective packaging, as well as storage instructions to avoid direct sunlight, in order to preserve integrity and efficacy during consumer use [45].

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 minutes, 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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