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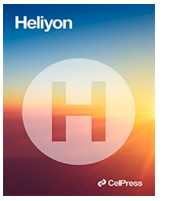

Research article

Physicochemical characterization of pectin and mango peel (*Mangifera indica* L.) from Mexican cultivars

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ABSTRACT

In Mexico, about 40 % of the mango harvest is lost due to marketing problems. Moreover, the mango industry generates peel and seed waste that ranges from 35 to 60 % of the total weight of processed fruits. This unexploited mango biomass represents a potential resource for producing value-added by-products. A market alternative is exploiting the mango peel as a source of bio-functional compounds, such as pectin. This hydrocolloid has applications in the pharmaceutical, cosmetic, and food industries. This study quantified the peel components of the Ataulfo, Panameño, Manila, and Haden cultivars. The mango peel showed a considerable input of dietary fiber (37–45 % DM), minerals (1018–2156 mg/100 g DM), phenols (2123–4851 mg gallic acid equivalent/100 g DM), flavonoids (0.74–2.7 mg quercetin equivalent/g DM) and antioxidant capacity (375–937 μM Trolox equivalent/g DM). The four cultivars presented high methoxyl pectins (66–71 %). The molecular weight of the pectins analyzed was from 957 to 4859 kDa. The Panameño cultivar showed the highest amount of pectin and viscosity concerning the peel of the other cultivars and a higher content of glucomannans (≈28.21 %). The pectin of the Haden cultivar was the only one with arabinoxylans since xylose was not detected in the pectin of the other cultivars. The chemical characteristics of the studied mango peels are promising for their industrialization.

1. Introduction

Mango (*Mangifera indica* L.) is the third fruit with the highest production in tropical countries and the fourth with the highest demand and consumption worldwide, making it one of the most cultivated due to its economic and nutritional importance [1,2]. Globally, Mexico ranks fourth in mango production and is positioned as the largest exporter, supplying 21 % of world demand with around 24 % of the national output, mainly to the USA with more than 476,000 tons in 2021, which provided 66 % of the internal market of that country [1–3]. In 2021, Mexico produced 2,156,040 tons of mango, with a value of 535,729,687.22 USD and an average

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rural price (ARP) of 248.45 USD per ton. In addition, an accumulated growth of 42.7 % in national production and an increase in world demand for mangoes from 3.23 to 4.06 million tons are projected for 2030 [4,5].

At the national level, most of the mango production is marketed fresh; only about 16 % is processed by the industry to make juices, jellies, frozen mango, dehydrated mango, purees, nectars, and preserves, among others. However, despite the low percentage of mango processing, large amounts of peel and seed are generated, representing 35–60 % of the total weight of processed mango [6,7], becoming a source of contamination due to the lack of proper handling of this waste. Furthermore, the losses generated by fruits that do not meet commercial quality and those derived during post-harvest handling account for 40 % of the mango production in Mexico [8].

Various studies reveal that the industry can use mango waste to obtain by-products with nutraceutical, prebiotic, and pharmaceutical potential [9–14]. Therefore, waste from the mango industry and lagging mangoes (fruits that do not meet the quality attribute to be processed or marketed) represent this crop's great economic potential and valorization. A viable alternative is the extraction of pectin from the mango peel since the demand for this polymer has shown an annual increase of 6 % in the last decade, producing around 40,000 tons with a value of 319 million dollars [15]. In the next five years (2022–2027), the global pectin market will have an annual growth rate of 7.27 % [16].

Previously, our working group published a review article in which 47 scientific articles were analyzed. These articles reported that the peel of 21 of the 46 mango cultivars analyzed contained between 14 and 32 % of pectin in cultivated fruits in Cameroon, China, Thailand, Vietnam, Jamaica, India, Kenya, Pakistan, Brazil, Peru, Australia, Colombia, and Burma [17]. However, only some authors characterized the pectin they extracted, reporting a degree of esterification of 82 %, a content of methoxyl groups of 12 %, and 82 % of galacturonic acid [18], and a degree of esterification of 56 %–66 % [19]. Industrial-level methods have yet to be established to extract pectin from mango waste, probably because laboratory-level studies have not provided higher extraction yields without increasing costs. It is worth mentioning that, in the 47 articles analyzed above, the authors used very varied extraction techniques and conditions, which allowed us to compare and select the extraction conditions with the highest yield, such as pH and type of solvent, hydrolysis time, the mass-solvent ratio, the temperature, the particle size of the sample, among other characteristics. Additionally, the state of ripeness of the fruits was not specified or did not coincide with that used by the industry, which is important in our study to provide novel information on waste from the mango industry through the extraction and characterization of both the peel like pectin in ripe fruits. For this reason, this study focuses on characterizing the mango peel of the most economically important cultivars in Mexico and extracting the pectin with different techniques and characterizing it, contributing to valuing the mango peel as a source of pectin. This can help satisfy the growing demand for this polymer, reduce environmental pollution generated by the waste of this fruit, and increase the economic utilities of the mango industry.

2. Material and methods

2.1. Material

Ataulfo, Panameño, Manila, and Haden mango cultivars were harvested at the ripening stage from Guerrero, Mexico, in 2020.

2.2. Disinfection and processing of mango fruits

The fruits were washed with drinking water and disinfected by immersing them for 3 min in sodium hypochlorite at 200 ppm. They were rinsed with drinking water to remove chlorine residues and left to dry at 20 °C. Mango pulp was manually separated from the peel and the seed, and the weight and percentage of each part of the fruit were determined and stored for further evaluation. The proximate analysis was obtained from the frozen peels (−20 °C). The pectin extraction and the total phenols, total flavonoids, and antioxidant capacity evaluations were carried out from the lyophilized and pulverized peel using a Thomas Scientific mill model 3383-L10 (Swedesboro, NJ, USA).

2.3. Proximate analysis and mineral composition of mango peel

The AOAC Official Methods [20] used were moisture (925.10), ash (942.05), lipids (920.39), protein (984.13 conversion factor 6.25), and dietary fiber (total, soluble, and insoluble) (991.43/AACC 32 -07.01). The equipment used for proximate analysis were Yamato convection oven (model DKN602C, U.S.A.), Thermolyne muffle (model FD1530M, U.S.A.), 125 mL Soxhlet PYREX extractor, Labconco micro digester (model 6030000, U.S.A.), RapidStill I Labconco (model 6500000I, U.S.A.) and Fibertec FOSS (model 1023, China). In addition, the minerals Na, K, Ca, Mg, Fe, Zn, Cu, and Mn were quantified by atomic absorption spectrometry (AA FS flame AA 280FS + SIPS 20, Agilent Technologies, Santa Clara, CA, USA) according to AOAC Method 955.06 (1998).

2.4. Determination of total phenols, total flavonoids, and antioxidant capacity in mango peel

The content of phenolic compounds was determined by the Folin-Ciocalteu method in a Synergy HTX Microplate Reader (BioTek, Inc, USA) [21]. A standard curve of gallic acid was used at concentrations from 0 to 0.4 mg/mL; the results were expressed in mg equivalents of gallic acid 100 g^{-1} of dry weight (mg GAE 100 g^{-1}) [22].

The quantification of total flavonoids in the sample was done with some modifications to the method reported by Ghasemi, Ghasemi, & Ebrahimzadeh [23], adjusted to a smaller volume for microplate reading, absorbance at 415 nm was read on a Synergy HTX microplate reader (BioTek, Inc. USA). The results were expressed in mg quercetin equivalents per gram of dry extract (mg QE

g^{-1}), using a standard curve of quercetin from 0 to 0.4 mg/mL (different from the original method).

The TEAC method was followed to determine the mango peels' antioxidant capacity. The extraction was carried out with 80 % methanol, and it was shaken at 200 rpm for 2 h and centrifuged at 15,000 rpm for 20 min at 4 °C [22]. The results were expressed in equivalent micromoles of Trolox per gram of sample (μM TE g^{-1}) [24].

2.5. Pectin extraction in mango peel

The pectin extraction in Ataulfo mango peel was determined using the techniques of Guo et al. [25], Azad, Ali, Akter, Rahman, & Ahmed [26], and Güzel & Akpinar [27], using 5 g of peel instead of 10 g as indicated by the original technique. The pectin extraction process of the three methods used is described below.

The lyophilized mango peel was mixed with the extraction solvent with the proportions shown in Table 5. It was incubated in a water bath at 80 °C for 1 h (100 °C for [26]), filtered with organza, and the supernatant was mixed with ethanol to precipitate pectin (see the volume of ethanol for each technique in Table 5). The extract was incubated overnight at 4 °C for [25] and at room temperature for [26], and filtered with organza after incubation, while for [27], it was filtered immediately without incubation. The pectin was washed three times with 20 mL of ethanol [25], twice with 20 mL of 70 % ethanol, and a third wash with 20 mL of ethanol [27], and three washes with 20 mL of 75 %, 85 %, and absolute ethanol [26]. The pectin obtained by the three techniques was dried in a Yamato model DKN602C convection oven at 40 °C for 24 h.

The modified Güzel & Akpinar [27] technique was chosen, as it provides a higher extraction yield and a lower process cost, with which the pectin of the Ataulfo, Panameño, Manila, and Haden cultivars was extracted for characterization. The yield of pectin extraction was calculated using Equation (1).

$$Yield = \frac{grams\ of\ dry\ pectin}{grams\ of\ dry\ mango\ peel} \times 100 \quad (1)$$

2.6. Partial characterization of mango peel pectin

2.6.1. Analysis of particles by dynamic light scattering (DLS)

The parameters of molecular weight, intrinsic viscosity, polydispersity, and hydrodynamic radius were determined in the peel pectin of the four mango cultivars by high-performance size exclusion chromatography (HPSEC) at room temperature with a system consisting of a Shodex column OH SB-G (Showa Denko, Tokyo Japan) serially (Shodex OHpak SB-804 HQ and Shodex OHpak SB-804 HQ). In addition, 50 mM sodium nitrate (NaNO3) with 0.02 % (w/v) sodium azide (NaN3) was used as eluent at a flow rate of 0.7 mL/min. The detectors used were.

a) UV 1260 Infinity at 280 nm (Agilent, USA), extinction coefficient 0.660 mL/(mg cm),
b) Light scattering model DAWN8+ (Wyatt, Santa Barbara, USA), constant calibration 3.0519×10^{-5} 1/(V cm),
c) On-line differential viscometer model ViscoStar II (Wyatt, Santa Barbara, USA), dilution factor 0.4954,
d) Differential refractometer with extended range model Optilab T-rEX (Wyatt, Santa Barbara, USA), refractive index 1.330.

The ASTRA software version 6.1.2.84 (Wyatt, Santa Barbara, USA) was used to analyze and calculate the variables. The refractive index differential (dn/dc) used was 0.146 mL/g for pectin [28].

2.6.2. Determination of the profile of neutral sugars

The neutral-sugar profile of the pectin of the four mango cultivars was determined by gas chromatography (GC) according to Blakeney, Harris, Henry, & Stone [29], and Hemery et al. [30] with modifications; hydrolysis of the polysaccharides was carried out followed by a reduction and acetylation. The derivatized samples were injected into a gas chromatograph model PerkinElmer Clarus 580. A 0.32 μm × 30 m type BD225 column (50 % cyanipropyl phenyl-dimethylpolysiloxane) was used with a 0.15 μm internal layer. The oven temperature was 140–230 °C, the injector temperature was 220 °C, and the detector temperature was 260 °C. Commercial standards were used to quantify arabinose, xylose, rhamnose, galactose, glucose, and fructose (Sigma, Mexico).

2.6.3. Fourier-transform infrared spectroscopy (FT-IR) and determination of the degree of methyl-esterification (DME)

Absorption bands associated with chemical bonds in pectins were determined by the Attenuated Total Reflectance (ATR) technique on a Fourier-transform infrared spectrophotometer model Thermo Scientific TM Nicolet iS50 (Madison, WI. USA). Spectra were recorded from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}, a scan speed of 0.475 cm s^{-1}, and 100 scans [31,32]. The DME was calculated using Equation (2):

$$DME = \frac{Area\ of\ esterified\ carboxyl\ groups}{(Area\ of\ esterified\ carboxyl\ groups + Area\ of\ nonesterified\ carboxyl\ groups)} \times 100 \quad (2)$$

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The DME value was linearly correlated with the calibration curve obtained of the relationship area of esterified carboxyl groups and the area of nonesterified carboxyl groups of the commercial citrus pectin (Sigma), with its corresponding known DME value [31,33].

2.7. Statistical analysis

A completely randomized design with three replicates for each response variable (total phenols, flavonoids, TEAC, moisture, ashes, lipids, protein, minerals, soluble fiber, insoluble fiber, and total fiber) was used for the characterization of the peel of mango of the four cultivars. The results were subjected to an analysis of variance (ANOVA) and a mean comparison test using the Tukey method with a significance of $p < 0.05$ in the statistical software NCSS 2022, Mexico. All data were expressed as mean and standard deviation ($n = 3$). In addition, a descriptive analysis of the pectin characteristics (molecular weight, intrinsic viscosity, polydispersity, hydrodynamic radius, neutral sugars, and degree of methyl-esterification) extracted from the peel of the four mango cultivars was carried out.

3. Results and discussion

3.1. Anatomical composition of mango fruits

The proportion of peel, pulp, and seed of the Ataulfo, Panameño, Manila, and Haden cultivars is shown in Table 1. The mango fruits presented from 14 to 17 % of peel, 66–73 % of pulp, and seed weight from 11 to 18 %. These results indicate the potential of the fruits as a source of pulp and peel, like what was reported by Kansci, Koubala, & Lape [34], for the cultivars Améliorée, Keitt, Mango and Palmer from Cameroon, in which the peel represents 16–19 %, the pulp 65–72 % and the seed 12–17 % of the total weight of the ripe fruits.

On the other hand, Vijayanand, Deepu, & Kulkarni [35] reported an average weight of 237.1, 429.8, and 396.1 g in ripe mango fruits for the cultivars Sindura, Mallika, and Totapuri from India, respectively, and proportions of peel (14.8–19.6 %), pulp (62.5–75.3 %) and seed (10.7–17.9 %) comparable to the Mexican cultivars analyzed in this study. While for Ataulfo, 10.2 % of seed, 71.1 % of pulp, and 19.0 % of peel are reported [36]. San Martín-Hernández, Pérez-Rubio, Muy-Rangel, Vargas-Ortiz, & Quintana-Obregón [37] reported 15.47 % of seed, 61.54 % of pulp and 22.14 % of peel in Ataulfo mango in the ripening stage.

The information presented in Table 1 shows that the percentage of the peel of the Ataulfo and Haden cultivars is slightly higher than the percentage of the seed, which means that the industry can (theoretically) process more than 50 % of its waste. However, after pectin extraction, approximately 80 % would remain (of the 50 % mentioned above). Considering the above, it would be interesting to perform a second and perhaps a third extraction of pectin from the waste to determine if a cumulative yield of pectin can be obtained and further reduce the final waste of the process.

3.2. Proximate analysis of mango peel and mineral composition

3.2.1. Moisture

The moisture content (Table 2) of the peel of the analyzed cultivars remained between 67 and 75 %. These results coincide with those of Puligundla, Mok, Oh, & Obulam [38], who showed 70 % moisture in an unspecified Pakistani cultivar, and with those of Serna-Cock, García-Gonzales, & Torres-León [39], in Badami and Raspuri cultivars from India, both with 72 % moisture. In mango Ataulfo, Rojas et al. [36] reported 80 % moisture. While the Chaunsa, Anwar Ratol, Langra, Dusehri, and Desi cultivars presented 68 and 71 % moisture [40].

3.2.2. Ash

Regarding the ash content, the results remained in a range of 0.66–1.61 % (Table 2), similar to that reported by Serna-Cock et al. [39], with 1.1 % of ash for the Raspuri cultivar and 1.3 % in the Badami cultivar. In comparison, the Desi cultivar contains 1.8 % [40]. In contrast, Rojas et al. [36] determined that the peel of the Chiapas′ Ataulfo mango has 2.1 % ash, higher than the 1.6 % of this study for the same cultivar.

3.2.3. Lipids

The peels of the studied cultivars presented a lipid percentage between 0.05 and 1.5 % (Table 2), where the Panameño cultivar statistically showed ($p < 0.05$) the lowest percentage. This information is relevant since the defatting stage before using this raw material could be omitted, for example, in extracting pectins. The results of the other cultivars analyzed are similar to those reported in the peel of Tommy Atkins (1 %) and Badami (1.7 %). While Rojas et al. [36], presented higher lipid values in the Ataulfo mango (2.35 %) and Raspuri (2 %) [39].

Table 1
Anatomical composition of mango fruits in the ripening stage.

Cultivar	Weight (g)	Peel (%)	Pulp (%)	Seed (%)
Ataulfo	157.3 ± 17.8	13.9 ± 0.9	73.3 ± 1.4	11.2 ± 0.5
Panameño	137.1 ± 9.7	15.5 ± 0.1	65.7 ± 0.8	17.9 ± 0.9
Manila	248.7 ± 11.7	15.1 ± 3.9	68.4 ± 4.1	15.6 ± 2.2
Haden	436.9 ± 17.8	16.9 ± 0.9	69.7 ± 8.1	11.4 ± 2.2

Mean ± standard deviation ($n = 5$).

Table 2
Proximate analysis of fresh mango peel from Mexican cultivars.

Parameter (%)	Ataulfo	Panameño	Manila	Haden
Moisture	70.73 ± 0.52 [a]	72.21 ± 0.20 [b]	67.43 ± 0.30 [c]	74.55 ± 0.76 [d]
Ash	1.61 ± 0.01 [a]	1.11 ± 0.02 [b]	1.33 ± 0.04 [c]	0.66 ± 0.03 [d]
Lipids	1.20 ± 0.07 [a]	0.05 ± 0.01 [b]	1.50 ± 0.04 [c]	1.20 ± 0.14 [a]
Protein	2.27 ± 0.23 [a]	0.81 ± 0.01 [b]	2.28 ± 0.14 [a]	2.61 ± 0.31 [a]
Soluble fiber	4.94 ± 0.39 [a]	6.19 ± 0.68 [a]	5.53 ± 1.03 [a]	6.89 ± 0.40 [a]
	*16.88 ± 1.33	*22.27 ± 2.45	*16.98 ± 3.16	*27.07 ± 1.57
Insoluble fiber	8.15 ± 0.69 [a]	4.04 ± 0.47 [b]	6.52 ± 0.83 [c]	3.29 ± 0.27 [b]
	*27.84 ± 2.36	*14.54 ± 1.69	*20.02 ± 2.55	*12.93 ± 1.06
Total fiber	13.10 ± 0.84 [a]	10.23 ± 1.10 [a]	12.05 ± 1.71 [a]	10.18 ± 0.39 [a]
	*44.76 ± 2.87	*36.81 ± 3.96	*37.00 ± 5.25	*40.00 ± 1.53

Mean ± standard deviation (n = 3). In each row for each parameter, values with different letters indicate significant differences ($p \leq 0.05$). *dry basis.

3.2.4. Protein

The protein content in the mango peel was statistically the same ($p < 0.05$) among the Ataulfo, Manila, and Haden cultivars (2.27–2.62 %) (Table 2), which was relatively close to 3.04 % in Ataulfo [36] but less than 5.2 % in Totapuri mango [41], and 3.5 % in an unspecified cultivar [38]. In ripe mango peel flour from the Chokanan cultivar is reported to be 0.18 g kg^{-1} (0.018 %) [42], which is even lower than the protein content in the Panameño cultivar (0.81 %), which was the lowest in this study. The differences in the protein content of the different cultivars highlight the importance of characterizing each cultivar.

3.2.5. Dietary fiber (total, soluble, and insoluble)

The content of soluble dietary fiber in the fresh peel of Ataulfo, Panameño, Manila, and Haden mangoes can be seen in Table 2, showing no statistical differences between samples ($p < 0.05$), remaining in a range of 4.9–6.9 %. On the other hand, the insoluble dietary fiber was significantly higher ($p < 0.05$) in the Ataulfo mango peel (8.15 %) compared to the other cultivars, followed by the Manila mango (6.52 %).

Sommano et al. [14], reported different values of total fiber in the fresh peels of mangoes Chok Anan (8.6 %), Rad (10.4 %), Keaw (11.4 %), Nam Dok Mai (11.5 %), Mahachanok (12.6 %), Tar Lub Nak (15.5 %) and Sam Pee (16.7 %), similar to our results (except for the last two cultivars). In contrast, Rojas et al. [36] reported only 5.6 % of crude fiber in ripe Ataulfo mango peel, less than the 13 % found in this study.

The amount of total and insoluble dietary fiber in the four cultivars was lower than that reported by Serna-Cock et al. [39] in the dried peel of Tommy Atkins, Raspuri, Haden, and Badami mangoes (total: 28–78.3 %, soluble: 13.8–28.1 %, insoluble: 14.2–50.3 %), but similar in soluble fiber content. This difference may be because the authors analyzed peel with pulp remains at commercial maturity, while in the present study, only the peel of mango ripe for consumption was used. This assertion is supported by Abdul, Wong, Bhat, & Cheng [42], who reported a significant decrease (indicated by literals) of total dietary fiber in peel (green: 5.94a, ripe: 5.68b) and pulp (green: 4.77c, ripe: 3.20d) of Chokanan mango as the maturation progresses. While in mango peel powder, Ataulfo, Keitt, and Tommy Atkins, it was reported to be 39, 29 and 40 % of total dietary fiber, respectively [43], which is comparable to Ataulfo cultivars (45 %), Panameño (37 %), Manila (37 %) and Haden (40 %) of the present study, both studies in fruits with ripe for consumption.

Other studies indicate that the mango peel has around 29 % soluble fiber and 27 % insoluble fiber [10,11] and between 28.7 and 42.4 % total fiber, 15.9–21.7 % soluble fiber, and 12.8–20.7 % insoluble fiber in the peel of Tommy Atkins, Kent, Palmer, and Nam Dok Mai mangoes in different stages of maturity [44]. The above is similar to what was observed in this study in Mexican cultivars (Table 2) and higher than the total dietary fiber content in 100 g of mango pulp with 1.6 g [45]. Also, a higher dietary fiber (40.5 g/100 g) and a lower amount of starch (21 g/100 g) have been reported in the peel flour of immature stenospermocarpic mangoes compared to the pulp flour thereof (11.7 g/100 g and 41 g/100 g) [46].

The aforementioned may be of interest to the food industry, whose trend is towards the production of functional foods and nutraceuticals such as: mango-based drinks with high fiber content and low glycemic index (45.99), which indicates their potential as functional food [47]; bakery products made with a mango dietary fiber concentrate, where digestibility tests of these products showed a low glycemic index [48]; yogurt formulated with 2 % Manila mango peel flour (Dietary Fiber: 35.41 %), without affecting its sensory attributes and with a prebiotic activity score of 0.25 [49], which is the capacity of a substrate of promoting the growth of a beneficial organism relative to a non-prebiotic substrate such as glucose [50]; and yogurt enriched with 2 % Kensington-pride mango peel powder and 1 % each of *L. casei, L. rhamnosus*, and *B. lactis* probiotic cultures, which inhibited α-glucosidase activity by 8.47 % after intestinal digestion, with a total of 108 phenolic metabolites identified in all extracts (undigested and digested) [51]. Additionally, it has been shown that adding mango peel powder to food improves its fiber content, phenolic compounds, and carotenoids [52]. In pigs fed with an extract of apple pectin or with mango pulp (with pectin as the main component), it was shown that both can significantly reduce total cholesterol in plasma by 18 and 9.5 %, respectively; in addition, the pectin extract reduced 17.2 % LDL-cholesterol in plasma [53].

3.2.6. Minerals

Table 3 shows the mineral content in the peels of the four cultivars analyzed. The Ataulfo cultivar generally showed the highest

mineral content, followed by Manila and Panameño. Moreover, the Haden cultivar had less mineral content (42–53 %) than the other mango cultivars. For Ataulfo mango, the total mineral content on a dry basis is 32 and 14 % higher than that reported by San Martín-Hernández et al. [37] and Mellado-Vázquez et al. [54] in Ataulfo mango peel powder (Table 3). This difference may be due to the fertilization management of each cultivar, as reported by Mellado-Vázquez et al. [54], who observed significant differences in the mineral content in the peel, mesocarp, endocarp, and seed of Ataulfo, Kent, and Tommy Atkins mangoes in a state of physiological maturity grown in 17 orchards in Chiapas, Nayarit, Oaxaca, Campeche, and Sinaloa, fertilized with different types and amounts of fertilizer.

Calcium is especially important because it can form insoluble complexes by binding with the free carboxyl groups and the oxygen atoms of pectin hydroxyl groups, forming an egg box in plant cell walls [55]. The content of Ca ions is similar in fresh (13.24 μmol) and cooked (11.78 μmol) snap-bean pods [56].

3.3. Determination of total phenols, total flavonoids, and antioxidant capacity in mango peel

The total phenolic content in the peel of the four cultivars analyzed showed significant differences ($p < 0.05$), where the Ataulfo mango had a higher concentration in the total phenolic content (4850.89 mg GAE/100 g of peel) (Table 4). Even higher than the concentration of phenols in Ubá mango pulp with 208.7 mg GAE/100 g (fresh weight) [57] and that in Ataulfo mango puree with 109.3 mg GAE/100 g (fresh weight) [58]. In this last data, the difference may be because the puree has higher moisture than the peel (lyophilized) used in this study; however, when making the corresponding adjustment eliminating moisture (≈70 %), the difference remains numerically significant at 364.33 mg GAE/100 g puree (dry weight). Palafox-Carlos [59] reported that the content of phenolic compounds in Ataulfo mango increases during ripening, so this difference may be due to factors other than the maturity stage since both authors used ripened fruits, the same as in the present study.

On the other hand, the Chok Anan cultivar from Malaysia showed more total phenolics in peel flour (102.41) and pulp (54.36 mg g^{-1}) of immature mango than in peel flour (70.20) and pulp (14.57 mg g^{-1}) of ripe mango, that is, in this case, the content of total phenols decreases with maturation [42]. However, in both stages of maturity, the peel presented more phenolic compounds than the pulp, comparing our results of the Ataulfo mango peel with those of a mango puree of the same cultivar, both at the ripening maturity stage [58].

In another study, Zahid et al. [51] reported 2257 mg GAE/100 g DW in ripe Kensington pride mango peel, which is lower than our results in the Ataulfo, Panameño, and Manila cultivars and similar to the Haden cultivar. The phenol content in the Ataulfo, Keitt, and Tommy Atkins mango pericarp powder is 7578, 5228, and 3857 mg GAE/100 g, respectively [43], of which the Ataulfo cultivar presented a higher concentration than the cultivars of the present study. In Manila mango peel flour, Perez-Chabela et al. [49] showed 3705 mg of polyphenols/100 g of sample, similar to the Manila mango peel from this study with 3803.80 mg GAE/100 g dry weight (Table 4). The peel of the Haden cultivar presented a higher content of total phenols (2123.06 mg GAE/100 g DW) than that reported by Rocha-Ribeiro et al. [57] in the pulp of the same cultivar (48.40 mg GAE/100 g FW), equivalent to 161.33 mg GAE/100 g DW (considering that the pulp has 70 % humidity). The results of this study highlight the potential of the mango peel as a source of antioxidants, as it is larger than in the pulp itself due to its role as a secondary metabolite in the plant's frontline defense mechanisms.

Regarding the content of total flavonoids in mango peels, the Ataulfo cultivar had a significantly ($p < 0.05$) higher concentration (Table 4). In ripened mangoes of the Chok Anan cultivar, 29.24 mg g^{-1} in mango peel flour is reported [42], higher than our results. At the same time, the authors showed an antioxidant capacity of 65.92 ($TEAC_{FRAP}$) and 43.30 ($TEAC_{DPPH}$), both results expressed in mg g^{-1}, so the antioxidant capacity of the mango peel of the Mexican cultivars analyzed in this study is lower than what Abdul et al. [42] reported in the cultivar Chok Anan from Malaysia.

In ripe mango peel powder, Quintana-Obregón et al. [43], reported an antioxidant capacity of 34811, 27256, and 23270 μmol TE 100 g^{-1} in the Ataulfo, Keitt, and Tommy Atkins cultivars, respectively, which is less than the antioxidant capacity per 100 g (Table 4 x 100) of the four cultivars in this stud and have a higher antioxidant capacity (μmol TE 100 g^{-1}) than guava (1 518), watermelon (264), peach (238) and papaya (183) [60]. What stands out is that the fiber of the peel of the Mexican cultivars contains compounds with potential antioxidant and reducing capacity with possible use in the industry for its health benefits. On the other hand, in Manila

Table 3
Mineral content in mango peel in ripe for consumption (mg/100 g).

Mineral	*Ataulfo (DW)	†Ataulfo (DW)	Ataulfo (FW)	Panameño (FW)	Manila (FW)	Haden (FW)
Na	15.71 ± 1.08	NR	7.52 ± 2.24	9.36 ± 2.57	10.86 ± 2.87	9.15 ± 0.59
K	754.55 ± 24.99	1370	451.17 ± 30.68	383.06 ± 15.24	438.56 ± 28.66	193.85 ± 11.84
Ca	614.85 ± 3 5.17	300	92.44 ± 5.22	20.95 ± 0.98	86.68 ± 5.56	17.44 ± 1.03
Mg	168.47 ± 3.45	190	70.60 ± 4.39	32.93 ± 4.21	44.49 ± 3.33	32.12 ± 1.39
Fe	5.08 ± 0.28	27.69	1.78 ± 0.36	1.13 ± 0.16	1.38 ± 0.38	1.16 ± 0.22
Zn	0.50 ± 0.00	0.89	0.48 ± 0.14	0.26 ± 0.06	0.30 ± 0.04	0.26 ± 0.02
Cu	NR	0.53	0.42 ± 0.03	0.32 ± 0.04	0.26 ± 0.06	0.21 ± 0.03
Mn	2.62 ± 0.13	1.57	0.77 ± 0.03	0.11 ± 0.02	1.30 ± 0.09	0.23 ± 0.02
Total	1561.78	1890.68	625.18	448.12	583.84	254.42
Total (DW)	1561.78	1890.68	2155.79	1600.43	1769.21	1017.68

Mean ± standard deviation (n = 3). *Ataulfo = mineral content reported by Ref. [37]. †Ataulfo = mineral content reported by Ref. [54]. NR = not reported. DW = dry weight. FW = fresh weight.

Table 4
Content of total phenols and flavonoids and antioxidant capacity in mango peel.

Cultivar	Total phenols (mg GAE/100 g DW)	Total flavonoids (mg QE/g DW)	TEAC (μM TE/g DW)
Ataulfo	4850.89 ± 323.02 [a]	2.70 ± 0.17 [a]	937.49 ± 125.31 [a]
Panameño	2986.92 ± 157.57 [b]	0.74 ± 0.08 [b]	538.64 ± 23.16 [b]
Manila	3803.80 ± 294.02 [c]	1.06 ± 0.20 [b]	837.79 ± 63.01 [a]
Haden	2123.06 ± 121.84 [d]	1.02 ± 0.08 [b]	374.79 ± 59.41 [b]

Mean ± standard deviation (n = 3). Values with different letters in each response variable indicate significant differences ($p \leq 0.05$). GAE = gallic acid equivalents. QE = quercetin equivalents. TE = Trolox equivalents. TEAC = antioxidant capacity in Trolox equivalents. DW = dry weight.

Table 5
Pectin extraction techniques in Ataulfo mango peel.

Technique	Mass/solvent ratio	Ethanol volume	Yield
Güzel & Akpinar, 2019	5 g/100 mL water (pH = 1)	100 mL	16.30 ± 1.0%[a]
Guo et al., 2012	2 g/100 mL water (pH = 1.5)	200 mL	16.18 ± 0.4%[a]
Azad et al., 2014	2 g/60 mL water (pH = 7)	120 mL	10.32 ± 1.7%[b]

Mean ± standard deviation (n = 3). Values with different literals indicate significant differences ($p \leq 0.05$).

mango peel flour, an antioxidant activity (TEAC) of 1525.75 μmol TE g^{-1} is reported [49], higher than our results in the four cultivars.

3.4. Pectin extraction in mango peel

The pectin extraction techniques reported by Güzel & Akpinar [27] and Guo et al. [25] managed to extract the highest pectin content with a yield of 16 %. However, the Güzel & Akpinar methodology implies a lower cost since it requires less use of reagents (requiring 400 mL less ethanol for every 5 g of hydrolyzed peel). On the other hand, with the method of Azad et al. [26], only 10 % of pectin was extracted (Table 5).

The Güzel & Akpinar [27], methodology was chosen to provide a higher pectin extraction yield with a lower process cost, which yields are shown in Table 6.

Similar extraction yields to this study were reported by Berardini et al. [19] in the peel of 14 mango cultivars (12–21 %) from different countries, while Sommano et al. [14], reported from 0.8 to 22.4 % pectin in seven Thai cultivars. Kumar et al. [41], reported the following values for Indian mango peel of the cultivars Totapuri (18.2 %), Banginapalli (15.6 %), Neelam (14.1 %), Sindhoora (10.5 %) and Rumani (6 %); which demonstrates that the pectin content is influenced by factors such as the cultivar, the maturity stage, the country of origin, the climate and even by pre- and post-harvest factors and by the extraction conditions [10,61]. In addition to these factors, the bleaching, the type of drying, the particle size, and the irradiation influence the pectin quality characteristics as reported in the peel of Tommy Atkins, Kent, Palmer, Nam Dokmai, and Kaew in Germany [44].

3.5. Partial characterization of mango peel pectin

3.5.1. Analysis of particles by dynamic light scattering (DLS)

The molecular weight (Mv), the intrinsic viscosity ([η]n), the polydispersity (P), the hydrodynamic radius (Rh(v)w), and the constants K and α of the peel pectin of Ataulfo, Panameño, Manila and Haden mangoes are shown in Table 7. The pectin's [η]n (414.02) of Panameño mango peel was slightly higher than the [η]n Ataulfo, Manila, and Haden (281.86, 304.73, and 293.38 mL/g, respectively), which were similar to the viscosity of the chickpea peel pectin with 296 mL/g and an Mv of 105 kDa [62]. On the other hand, San Martín-Hernández et al. [37] reported a molecular weight of 94 kDa for Ataulfo mango peel pectin, which differs from the viscometric molecular weights of the cultivars analyzed in this study with higher values (957–4859 kDa). This difference may be because San Martín-Hernández et al. [37] hydrolyzed pectin for a longer time, decreasing its ramifications and molecular weight and viscosity. It's worth noting that the technique used in this study to measure viscosity is more precise than the one used by the authors, further validating our research methodology. In contrast, the molecular weights of the pectin from the peel of the Ataulfo, Manila, and Haden mangoes were similar to the molecular weight (2000 kDa) of the pectin extracted from the citrus peel [63].

Table 6
Pectin extraction yield in mango peel.

Cultivar	Yield
Ataulfo	17.34 ± 0.26%[a]
Panameño	22.85 ± 0.07%[b]
Manila	18.43 ± 0.27%[a]
Haden	17.61 ± 0.10%[a]

Mean ± standard deviation (n = 3). Different literals indicate significant differences ($p \leq 0.05$).

Table 7
Molecular properties of mango peel pectin.

Cultivar	M_v (kDa)	$[\eta]_n$ (mL/g)	P (Mw/Mn)	$Rh(v)_w$ (nm)	K (mL/g)	α
Ataulfo	956.9	281.86	2.96	41.62	0.56	0.49
Panameño	4859	414.02	2.86	72.82	0.12	0.25
Manila	1526	304.73	2.95	48.07	0.78	0.45
Haden	2919	293.38	3.82	57.34	8.68	0.26

Mv = Viscosimetric molecular weight, [η]n = Intrinsic viscosity, P=Polydispersity, Rh(v)w = Hydrodynamic radius, Mw and Mn = data not shown, constants K and α, n = 1.

Studies indicate that a high Mv gives the polymeric molecule a high [η]n and that the change in [η]n occurs only when there are alterations in the main pectin chain [64], such as shown in our results. Furthermore, the present study shows that the Ataulfo and Manila mango pectin presented similar molecular properties, so their polymeric behavior is expected to be similar; this may allow the processing of both biomasses in the same pectin extraction batch. On the other hand, the Haden mango pectin showed one of the highest viscometric molecular weights with 2919 kDa but with an [η]n of less than 20 % on average concerning the other cultivars, indicating that it could have a more compact conformation despite the presence of high molecular weight polysaccharides [65]. In addition, the Haden mango pectin had a lower glucomannan content, which makes it less viscous [66]. It can also be seen that P and Rh (v)w) are high, indicating that the mango pectins were highly polydisperse, which explains why they were not fully soluble in water. This characteristic of being partially soluble in water is important since it reveals that pectin and other cell wall components, such as cellulose and hemicellulose, were probably extracted because the pH used in the hydrolysis was very acid (pH = 1).

3.5.2. Neutral sugars profile of the mango peel pectin

The neutral sugars content in the pectin extracted from the mango peel of the Ataulfo, Panameño, Manila, and Haden cultivars is shown in Table 8. The results showed that the composition of the polysaccharides is mainly formed of glucose, mannose, galactose, arabinose, and rhamnose units, which suggests the presence of galactans, arabinans, glucomannans, and arabinogalactans. Furthermore, the acid fraction consists of chains of homogalacturonans and rhamnogalacturonans I formed by repeated disaccharides of galacturonic acid and rhamnose [62,67].

The presence of glucose and mannose in the Panamanian cultivar pectin may imply the presence of glucomannans, with an approximate ratio of 8:5 in terms of mannose and glucose, respectively, together by bonds ß (1 → 4) [66]. Glucomannans are high molecular weight polysaccharides and are part of the soluble fiber, creating highly viscous solutions, which could be associated with the high intrinsic viscosity of the pectin from this cultivar (Table 7). On the other hand, it is worth noting that the Haden mango pectin may contain arabinoxylans since it was the only pectin with xylose (2.28 %). However, the Haden cultivar pectin may mostly contain arabinogalactans and rhamnogalacturonans due to its high galactose (59.13 %), arabinose content (10.86 %), and rhamnose (10.21 %) content. Also, the pectin of the Panamanian mango contains a higher percentage of arabinose (36 %). It can also be observed that the Ataulfo and Manila mango pectins have a higher rate of glucose, with 25 % each, indicating that long glucose chains may be formed that may negatively affect the solubility of the pectin.

3.5.3. FTIR and degree of methyl esterification of mango peel pectin

The FTIR (Fourier Transform Infrared Spectra) of Fig. 1 can be compared with the frequencies of the bands of the functional groups of the pectins in Table 9. In Fig. 1, a broad area of absorption is observed in the region of the 3600 and 2500 cm^{-1} characteristic of the absorption of the O–H stretch due to hydrogen bonds of the galacturonic acid polymer [31]. In the range of the 3000-2800 cm^{-1} bands (around 2950 cm^{-1}), the C–H stretching and bending vibrations of the CH, CH2, and CH3 groups are detected [31]. However, in pectin, the C–H region is seen as a band overlapping the broader O–H region [31], so this region cannot be used to determine the degree of methoxylation (DM). The wavelength range from 1200 to 950 cm^{-1} is considered the "fingerprint of carbohydrates" and is characteristic of all pectins [33,62]. On the other hand, the degree of esterification (DE), reported by many authors as DM, was determined using the peak area of the esterified groups or carbonyl ester (C=O) at 1760-1745 cm^{-1} (1750 cm^{-1}) and the free carboxyl groups or carboxylate ions (COO-) found at 1640-1620 cm^{-1} (1650 cm^{-1}) [31,32]. It should be noted that since DM and DE are always very similar and that sometimes there is confusion between them, some authors have begun to use the term degree of methyl-esterification (DME). This study observed that the DME was higher when the esterified groups (C=O) presented intensity and high band area, and the free carboxyl groups (COO-) stretching showed intensity and low band area.

Table 8
Neutral sugars composition of mango peel pectin from Mexican cultivars.

Cultivar	% Neutral sugars						
	Rhamnose	Galactose	Glucose	Arabinose	Xylose	Mannose	Others
Ataulfo	9.51 ± 0.41	31.51 ± 1.17	24.89 ± 0.67	14.94 ± 0.80	ND	4.96 ± 0.17	14.19
Panameño	16.81 ± 0.71	11.65 ± 0.75	10.85 ± 0.90	35.93 ± 2.21	ND	18.33 ± 1.37	6.43
Manila	12.08 ± 0.64	28.94 ± 2.37	25.15 ± 2.65	8.91 ± 0.77	ND	10.72 ± 0.35	14.2
Haden	10.21 ± 0.20	59.13 ± 1.71	9.97 ± 0.51	10.86 ± 0.17	2.28 ± 0.10	2.34 ± 0.06	5.21

ND = No detected.

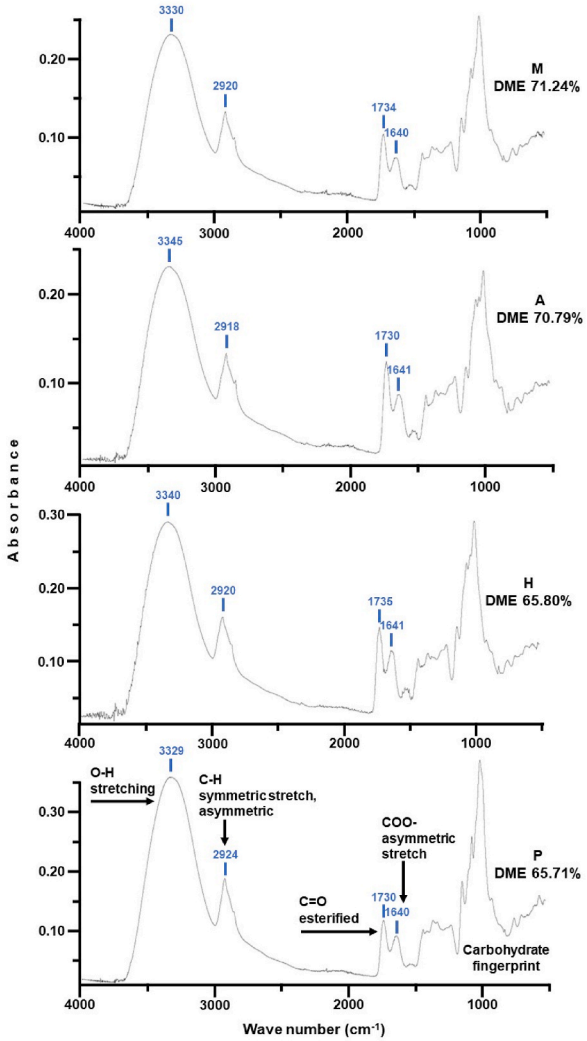

Fig. 1. FTIR of mango peel pectin from Mexican cultivars. Functional groups are marked with arrows. M = Manila, A = Ataulfo, H= Haden, P= Panameño, DME = degree of methyl-esterification.

Table 9
FTIR frequencies and intensities of the mango pectin functional groups.

Frequency (Number of bands, cm^{-1})	Functional groups	Intensity
3600–2500	O–H stretching	broad, strong
3000–2800	C–H symmetric stretch, asymmetric	Acute, occasionally double overlap with O–H
1760–1745	C=O esterified	Strong
1640–1620	COO- asymmetric stretch	Strong
1200–950	Main chemical groups in polysaccharides	Carbohydrate fingerprint

The results of the DME of the four mango pectins are shown in Fig. 1. It can be seen that all are high methoxyl (capable of forming gels in the presence of 65 % total soluble solids and acidic pH: 2–3.5), with a range of 65.71–71.24 %, the same as Tommy Atkins (56.3 %), Kent (61.7 %) and Palmer (58 %) mango peel pectins, although with a lower DME [44]. Therefore, they could be used in the production of jams and gums as an encapsulant for enteric medicines, among other applications. The pectins of the Haden and Panameño cultivars are classified as medium rapid set pectins, while those of the Ataulfo and Manila cultivars are considered rapid set [68]. The pectin yields on a dry basis obtained in this study are the following: Panameño (22.85 %), Manila (18.43 %), Haden (17.61 %), and Ataulfo (17.34 %). These results contrast with what was reported by Davara, Dabhi, Rathod, & Bhatu [69] in the Kesar mango peel and with what was reported by San Martín-Hernández et al. [37], in the Ataulfo mango peel, with yields of 14.78 and 5.4 %, respectively, both low methoxyl pectins. In the Ataulfo cultivar, the difference between our results and those of San Martín-Hernández

et al. [37], is due to the extraction conditions since the authors hydrolyzed for a longer time at a higher pH, factors that affect both the yield and the molecular characteristics of the pectin.

The higher pectin content in the Panamanian cultivar (between 4.4 and 5.5 % more than the other cultivars) could be an incentive to add value to this cultivar, currently only marketed in some regions of Guerrero and Oaxaca. The above may be because most of the peel of the Panamanian cultivar remains green even when ripe, even though the pectin content has been shown to decrease with ripening [44,70,71]. Previous studies observed a correlation between the chlorophyll and pectin content (protopectin and high and low methoxyl pectins) in Tommy Atkins mango peel, where both compounds decreased from day 84–112 after flowering (anthesis) [72]. In Elephant mango, the same behavior is observed between the pectin and chlorophyll content, where the chlorophyll content (a + b) increases rapidly from week 1 (0.083 mg g^{-1} fresh peel) to week 7 (0.302 mg g- 1 fresh peel) and decreases slowly until week 17 (0.098 mg g^{-1} fresh peel), while the pectin content presents a rapid increase from week 3 (4.3 %) to week 9 (7.4 %) and gradually decreases up to week 17 (2.8 %) [73]. On the other hand, in guava, it is reported that the chlorophyll and pectin content increased from weeks 2–10 (after anthesis), and the content of both compounds gradually decreased from weeks 10–15 [74].

Another way to define if a pectin is a high or low methoxyl is to determine its content of methoxyl groups, which must be greater than 7 % for high methoxyl (HM) pectins and less than 7 % for low methoxyl pectins (LM) [75]. For example, in the Kesar mango pericarp, Davara et al. [69], reported a methoxyl group content of 3.91 % and a pectin yield of 14.78 % extracted by acid hydrolysis in two steps of 1 h each, using an exchange resin cationic to increase yield. The extraction conditions of the previous study were reproduced by San Martín-Hernández et al. [37], in Ataulfo mango pericarp with the difference that they carried out a single 2-h extraction without the resin. The authors reported a yield of 5.4 %, which shows that the cation exchange resin does contribute to obtaining a higher yield; the content of methoxyl groups was 6.35 % with a GE of 46.07 %. In both studies, the pectin obtained was LM (with gelling properties in the presence of Ca++ and low concentrations of soluble solids). The authors suggest that the low GE is due to the mango's ripeness since, as ripening progresses, pectinases hydrolyze the ester bonds and depolymerize the pectin [37,44]. However, the authors did not consider that prolonged hydrolysis times affect both the extraction yield and the content of methoxyl groups by eliminating part of the pectin side chains, specifically the ramifications of type I and II rhamnogalacturonans.

4. Conclusions

The results of this study demonstrate that the mango peel of the Ataulfo, Panameño, Manila, and Haden cultivars can be used in the preparation of nutraceutical or functional foods due to its high content of minerals, polyphenols, antioxidant capacity, dietary fiber, and pectin or as a source of these compounds, focused on the consumption trend towards healthier foods. The information generated in this research is important because it can contribute to giving added value to this by-product of the mango industry considered as waste. In addition, the yield, and molecular properties of the mango peel pectin of the Mexican cultivars analyzed are comparable with commercial pectins, so this undervalued by-product could be used as a pectin source, which can contribute to reducing the pollution generated by the mango industry and increasing its profits, by satisfying part of the growing demand for pectin.

Availability of data and materials

All data generated or analyzed during this study are included in this published article.

CRediT authorship contribution statement

Andrés A. Pacheco-Jiménez: Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Conceptualization. **Jaime Lizardi-Mendoza:** Resources, Investigation. **J. Basilio Heredia:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization. **Erick P. Gutiérrez-Grijalva:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **Eber A. Quintana-Obregón:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **María D. Muy-Rangel:** Writing – review & editing, Validation, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

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

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