



Article Film Properties of Pectin Obtained from Various Fruits' (Lemon, Pomelo, Pitaya) Peels

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Abstract: Food by-products, as sources of high-value compounds, have shown great promise for formulating novel food packaging and edible films for food preservation. This work focused on extracting pectin from lemon, pomelo, and pitaya peels using an ethanol precipitation method. The degree of esterification and the monosaccharide composition of the extracted pectin were determined, and the antioxidant activity of the pectin was assessed. Subsequently, pectin-based films were prepared using the casting method, employing pectin from various sources. These films' physical and mechanical properties (tensile strength and elongation at break) were assessed, and their microstructure and intermolecular interactions were examined using scanning electron microscopy and Fourier transform infrared spectroscopy analysis. The results of this study demonstrate variations in the degree of esterification among the three pectin samples, with pomelo peel and lemon peel pectins exhibiting higher degrees of methylation compared to pectin from pitaya peel, which had a lower degree of methylation. However, no significant differences were observed regarding the films' moisture content and water vapor permeability. Notably, the films made from pitaya peel pectin exhibited superior tensile strength and elongation at break to those derived from pomelo peel and lemon peel pectins. The pectin recovered from these fruit peels had great film properties and thus could be utilized for food packaging and edible films.

Keywords: pectin; packaging film; mechanical properties; film characterization

1. Introduction

The rapid growth of the global population is driving the expansion of the food processing industry, resulting in a substantial generation of by-products, particularly in the beverage, dairy, and ice cream sectors. Unfortunately, these by-products are often discarded as food waste, diminishing the overall value of raw materials and causing some adverse environmental issues [1]. Raw materials of significance in food processing include fresh fruits and vegetables, which contain numerous high-value substances, such as polyphenols, essential oils, pigments, proteins, enzymes, pectins, and dietary fiber. Pectin, a polysaccharide in plant cell walls, finds extensive applications in the food industry, particularly in confectionery and beverage production, owing to its emulsifying properties [2]. Moreover, pectin can be utilized to develop edible films to facilitate food storage and preservation [3], thereby highlighting the significance of pectin recycling.

Recent studies have demonstrated the potential of food processing by-products as sources of high-value compounds for formulating novel food packaging and edible films.



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). For instance, researchers have successfully extracted starch from loquat seeds, which possess desirable film-forming properties [4]. The loquat seed starch was combined with oregano essential oil to develop a composite film exhibiting remarkable antioxidant and antibacterial characteristics [4]. Additionally, edible films based on mango kernel starch or pineapple seed starch have been reported to effectively reduce the quality deterioration of mangoes during storage [5]. Furthermore, mango kernel starch has shown promise as a coating material for tomato fruits, reducing their respiration rate and extending their storage life [6].

Pectin, a widely utilized biomacromolecular polymer, has also garnered attention for its utility in producing edible films using fruit peel by-products. Recent studies have explored using pectin derived from mandarin peels combined with sage leaf extracts, resulting in pectin films with favorable antioxidant properties. Films based on mandarin peel pectin showed a tensile strength and elongation at break of 13.90–22.54 MPa and 15.82–18.82%, respectively [7]. Another study highlighted the effectiveness of pectin films derived from lime peel pectin, lime peel extract, and coconut water in inhibiting the oxidation of soybean oil [8]. In this study, the films based on lime peel pectin presented a tensile strength and elongation at break of 3.63–20.61 MPa and 1.77–4.78%, respectively [7]. Therefore, certain peel by-products can serve as valuable sources of pectin for developing edible or packaging films for food storage and preservation.

In this study, pectin was prepared from pomelo peel, lemon peel, and white-fleshed pitaya peel using citric acid with the assistance of hydrothermal extraction methods. Subsequently, three edible films were prepared using the pectins extracted from pomelo peel, lemon peel, and white-fleshed pitaya peel, and their physical, thermomechanical, and structural properties were comprehensively characterized.

2. Materials and Methods

2.1. Materials

Pomelos (Guanxi pomelo, Zhangzhou, China) and lemons (Anyue lemon, Ziyang, China) were purchased from a local supermarket (Beijing, China). Pitaya (*Hylocereus undatus*) peels were collected from a local store (Beijing, China) providing fresh-cut fruits. Citric acid and ethanol were purchased from Modern Oriental Technology Development CO., Ltd. (Beijing, China). DPPH (2,2-Diphenyl-1-picrylhydrazyl), ABTS (2,2'-azinobis-(3-ethyl-benzothiazo-line-6-sulfonic acid)), and glycerol were purchased from Macklin Biochemical Co., Ltd. (Shanghai, China). All chemical reagents used in this study were of analytical grade.

2.2. Preparation of Pectin

The peels of pomelo and lemon fruits were separately isolated. Subsequently, the fruit peels were dried in an oven at 60 °C for 12 h to obtain dried peel samples, which were then ground into powder and stored at -18 °C. For pectin extraction, 10 g of dried peel powder was mixed with 200 mL of a 1% (w/v) citric acid solution. The mixture was sonicated for 10 min and then stirred at 92 °C for 1 h. After centrifugation and vacuum filtration, the extracted pectin solutions were collected. Pectin precipitation was achieved by mixing the pectin extracts with 95% ethanol at a ratio of 2:1 (v/v). The mixture was left at 4 °C for 12 h, and the precipitated pectin was washed three times using 95% ethanol solution. The pectin was then dried in an oven at 50 °C for 8 h. Finally, the dried pectin was powdered and stored at -18 °C for further use. The methods of peel pectin extraction are shown in Figure 1.

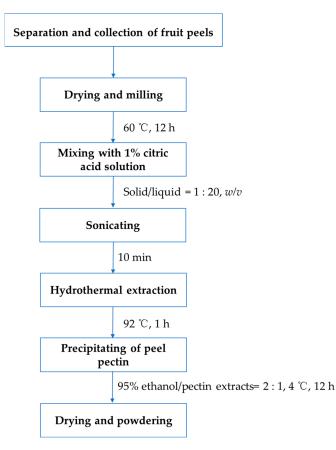


Figure 1. Process flow chart for the extraction of pectin from fruit peels.

2.3. The Determination of the Degree of Esterification (DE) of Pectin

The DE of prepared peel pectin was measured according to a published report [9]. DE is defined as the ratio of the number of methyl-esterified carboxyl groups to the number of total carboxyl groups. The infrared spectra of pectin were measured with a Perkin Elmer Spectrum 1000 (Perkin-Elmer Corporation, Waltham, MA, USA). The wave number range was 4000–500 cm⁻¹ with a resolution of 4 cm⁻¹ and 32 scans. The bands at 1745 and 1630 cm⁻¹ were assigned to methyl-esterified and esterified carboxyl groups, respectively. DE was calculated according to Equation (1):

$$DE(\%) = \frac{A_{1745}}{A_{1745} + A_{1630}} \times 100$$
(1)

Herein, A_{1745} was the absorbance intensity at 1745 cm⁻¹ for methyl-esterified carboxyl groups, and A_{1630} was the absorbance intensity at 1630 cm⁻¹ for esterified carboxyl groups.

2.4. The Monosaccharide Composition of Peel Pectin

The monosaccharide fraction of the pectin extracted from the peel was determined using high-performance liquid chromatography (HPLC). Before the determination, the extracted peel pectin was hydrolyzed using trifluoroacetic acid. Standard curves were constructed using standard solutions of fucose, rhamnose, arabinose, galactose, glucose, mannose, galacturonic acid, and glucuronic acid.

2.5. The Antioxidant Activities of Pectin

The antioxidant activities of peel pectin were evaluated according to the DPPH and radical scavenging capacities [10]. To determine the DPPH radical scavenging capacity, 50 μ L of 0.5 mM DPPH-methanol solution was mixed with 100 μ L of sample solution. The mixture was incubated at room temperature in a light-free environment for 10 min.

The absorbance under 517 nm was recorded. The ABTS radical scavenging capacity was measured using the following method [11]. The solution consisting of 7 mM ABTS and 2.45 mM potassium persulfate was prepared and placed in a dark place for 16 h. The solution was diluted to a concentration corresponding to an absorbance ranging from 0.7 to 0.9 under 734 nm to obtain an ABTS work solution. The reaction system consisted of 50 μ L of sample solution and 100 μ L of ABTS work solution. The mixture was incubated at room temperature in a light-free place for 10 min. The absorbance under 734 nm was recorded. To eliminate the influence of pigment color, a system including 50 μ L of sample solution and 100 μ L of distilled water was prepared as the control.

2.6. The Preparation of the Films Based on Prepared Pectin

A total of 2 g of pectin powder was mixed with 100 mL of distilled water. The mixture was then heated to 90 °C and stirred at 500 rpm for 30 min to ensure complete dissolution of the pectin. After cooling the film solution to room temperature, 25 wt% glycerol (based on the mass of pectin) was added and stirred for 30 min. The film solution was then subjected to negative pressure to remove any air bubbles generated during stirring. Subsequently, 17.5 mL of the film-forming solution was poured into a 9 cm diameter polystyrene Petri dish and allowed to dry at room temperature for 48 h, forming a film. The dried films were carefully peeled off from the Petri dishes and placed in a container maintained at room temperature and 55% relative humidity for further storage.

2.7. The Thickness and Color of Pectin Films

The thickness of the films was determined using a digital microscope (Three Measure Technology Co., Ltd., Guangzhou, China) with a resolution of 0.001 mm.

2.8. The Moisture Contents of Pectin Films

The films were dried in an electric blast dryer at 105 $^{\circ}$ C to bring the samples to a constant weight. The moisture content (MC) of the films was calculated according to Equation (2):

MC (%) =
$$\frac{W_{\rm I} - W_{\rm D}}{W_{\rm I}} \times 100$$
 (2)

W_I and W_D are the initial film mass and post-drying mass, respectively.

2.9. The Opacity of Pectin Films

The absorbance values of the film samples at 600 nm were determined using a UV-Vis spectrophotometer (UV-2550, Shimadzu, Tokyo, Japan). The opacity of the films was calculated according to Equation (3):

$$Opacity = \frac{Abs_{600}}{T}$$
(3)

 Abs_{600} refers to the absorbance value of the film at 600 nm, and T refers to the thickness of the film (mm).

2.10. Water Vapor Permeability (WVP) of Films

The WVP of pectin films was determined according to the methods in a recent report [12]. The film sample was closely attached to the opening of a tube containing 6 mL of distilled water. The sealed tube was placed in a chamber at a controlled temperature of 25 ± 2 °C and a relative humidity of $50 \pm 5\%$. After 48 h, the sealed tube was weighed, and the value was recorded as the initial weight. Then, the sealed tube was weighed again after 72 h. The WVP of the film was calculated using Equation (4):

$$WVP = \frac{\Delta W \times T}{\Delta P \times t \times A}$$
(4)

where ΔW denotes the weight loss after 72 h, T denotes the film thickness (m), ΔP denotes the partial vapor pressure difference between films (Pa), t denotes the test time (s), and A denotes the permeability area (m²).

2.11. Mechanical Properties of Films

The film sample strip (8 mm \times 60 mm) was prepared for testing. The samples were conditioned at 50 \pm 5% relative humidity for 2 days before analysis. The mechanical properties of the film strips were evaluated using a physical property analyzer (CT3, Brookfield Ltd., Wisconsin, USA). The effective tensile length and tensile rate were 28 mm and 0.5 mm/s, respectively. The tensile strength and elongation at break of the films were calculated according to the following equations:

$$TS (MPa) = \frac{F}{A}$$
(5)

$$EB(\%) = \frac{\Delta L}{L_{I}} \times 100 \tag{6}$$

TS is the tensile strength of the film, F is the maximum tensile force at film rupture (N), A is the cross-sectional area of the film (m²), EB is the elongation at break of the film, ΔL is the stretch length at film rupture (mm), and L_I is the initial effective length before stretching (mm).

2.12. Morphological Analysis of Films using Scanning Electron Microscopy (SEM)

To observe the cross-section of the film, the film was snapped off with tweezers after being snap-frozen in liquid nitrogen. A scanning electron microscope (JSM-6700F, Jeol Ltd., Tokyo, Japan) was used to observe the microscopic morphology of the cross-section of the thin-film sheets.

2.13. The Fourier Transform Infrared (FTIR) Spectroscopy of Films

The infrared spectra of thin-film samples were measured using Perkin Elmer spectrum 1000 (Perkin-Elmer Corporation, Waltham, MA, USA). The wave number range was 4000–500 cm⁻¹ with a resolution of 4 cm⁻¹ and 32 scans.

2.14. Statistical Analysis

SPSS 22.0 for Windows and one-way analysis of variance (ANOVA) were used for statistical analysis. Duncan's multiple range tests were conducted for comparison at a 95% confidence level.

3. Results

3.1. The Yield and DE of Pectin

The results of the yield and esterification of pectin from fruit peels are presented in Table 1. The pectin yields were $13.70 \pm 0.51\%$, $9.35 \pm 0.44\%$, and $20.88 \pm 0.74\%$ for pomelo peel, lemon peel, and white-fleshed pitaya peel, respectively. It can be observed that the pectin yield from white-fleshed pitaya peels was significantly higher compared to that from pomelo and lemon peels. Various extraction methods have been explored for pectin extraction from fruit peels, including microwave-assisted extraction and ultrasoundassisted extraction. For example, Tongkham et al. (2017) achieved a maximum pectin yield of 23.11% from red heart pitaya peel using a microwave-assisted method with specific power and time conditions [13]. Similarly, using an ultrasound-assisted method, Zaid et al. (2020) reported a pectin yield of 30.11% from red heart pitaya peel. These findings demonstrate the potential of using alternative extraction techniques to improve pectin yield from fruit peels [14].

	РРР	LPP	WPPP
Yield (%)	13.70 ± 0.51 ^b	9.35 ± 0.44 ^c	$20.88\pm0.74~^{\rm a}$
DE (%)	55.87 ± 0.34 a	55.10 ± 0.58 a	$46.06 \pm 0.37 \ ^{\rm b}$
Monosaccharide			
composition (%)			
Fucose	0.03 ± 0.01 ^b	0.03 ± 0.01 ^b	0.12 ± 0.04 ^a
Rhamnose	2.40 ± 0.00 ^b	2.09 ± 0.05 ^b	21.11 ± 0.37 a
Arabinose	19.35 ± 0.89 a	19.77 ± 2.33 a	5.36 ± 0.19 ^b
Galactose	25.86 ± 0.94 a	11.40 ± 0.75 ^c	14.78 ± 0.34 ^b
Glucose	15.09 ± 0.29 ^b	18.8 ± 0.89 a	7.52 ± 0.11 c
Mannose	2.14 ± 0.19 a	2.52 ± 0.52 a	2.8 ± 0.35 a
Galacturonic acid	$33.12 \pm 1.82^{\ b}$	44.27 ± 4.28 a	47.10 ± 0.69 a
Glucuronic acid	1.35 ± 0.01 $^{\rm a}$	1.12 ± 0.15 a	1.22 ± 0.04 a

Table 1. The yield, degree of esterification, and monosaccharide composition of pomelo, lemon, and white-fleshed pitaya peel pectin.

DE: degree of esterification, LPP: lemon peel pectin, PPP: pomelo peel pectin, and WPPP: white-fleshed pitaya peel pectin. Values with different letters in each row indicate statistically significant differences at a 5% confidence level.

Regarding the degree of esterification, pectins can be classified as highly methylated pectins (>50% methyl esterification) and low methylated pectins (<50% methyl esterification) [15]. The degree of esterification for pomelo peel, lemon peel, and white-fleshed pitaya peel pectins was determined to be $55.87 \pm 0.34\%$, $55.10 \pm 0.58\%$, and $46.06 \pm 0.37\%$, respectively. Hence, pomelo and lemon peel pectins were identified as highly methyl-esterified pectins, while white-fleshed pitaya peel pectin was classified as low methyl-esterified pectin. These findings are consistent with previous studies on pomelo peel pectin [9] and sweet lime peel pectin [16], demonstrating similar esterification levels. Additionally, pitaya peel pectin was reported to have an esterification of $41.96 \pm 0.81\%$ [17], aligning with the esterification of the pectins from red pitaya peels with esterification degrees ranging from $57.50 \pm 1.33\%$ to $73.60 \pm 1.04\%$ [14].

3.2. The Monosaccharide Composition of Peel Pectin

The monosaccharide fractions of the peel pectin are presented in Table 1. Highperformance liquid chromatography (HPLC) was employed to analyze the monosaccharide fractions using rockulose, rhamnose, arabinose, galactose, glucose, mannose, galacturonic acid, and glucuronic acid as standard curves. The pectins obtained from pomelo and lemon peels were primarily composed of galacturonic acid, galactose, arabinose, and glucose. In contrast, the pectin derived from white-fleshed pitaya peels consisted mainly of galacturonic acid, rhamnose, and galactose. Among the three types of pectin, white-fleshed pitaya peel pectin exhibited the highest galacturonic acid content $(47.10 \pm 0.69\%)$. Pomelo and lemon peel pectins had relatively higher arabinose content $(19.35 \pm 0.89\%$ and $19.77 \pm 2.33\%$, respectively), whereas white-fleshed pitaya peel pectin had a lower arabinose content (5.36 \pm 0.19%). Previous studies have reported that the major monosaccharide fractions of pomelo peel and lemon peel pectins are galacturonic acid and galactose [18,19]. Similarly, the major monosaccharide fractions of red heart pitaya pericarp pectin are galacturonic acid, galactose, mannose, and rhamnose [20,21]. The monosaccharide composition of lemon peel pectin was reported to be galacturonic acid (60.0%), galactose (27.2%), arabinose (6.4%), rhamnose (4.3%), xylose (1.4%), and glucose (0.6%) [18]. Additionally, the monosaccharide composition of pomelo peel pectin was measured as follows: galacturonic acid (84.9–86.2%), galactose (5.4–6.0%), arabinose (5.7–6.8%), rhamnose (1.2–1.6%), and glucose (0.7–1.4%) [19]. Further, the monosaccharide composition of red pitaya peel pectin was determined to be galacturonic acid (44.34%), galactose (27.32%), mannose (17.93%), rhamnose (7.62%), and glucose (2.80%) [21].

3.3. The Antioxidant Capacities of Peel Pectin

The antioxidant activities of peel pectin were assessed by measuring the DPPH and ABTS radical scavenging capacities, and the results are shown in Figure 2. The antioxidant capacities of peel pectin were associated with the concentration of peel pectin. The antioxidant properties of the peel pectin may be due to the hydroxyl groups of the pectin. In addition, the phenolic hydroxyl groups of the residual phenolic compounds present in the extracted peel pectin might also, to some extent, provide antioxidant capacities. The DPPH and ABTS radical scavenging activities of PPP and LPP were stronger than those of WPPP, which might be due to the rich flavonoids with great antioxidant activities in the peels of citrus fruits.

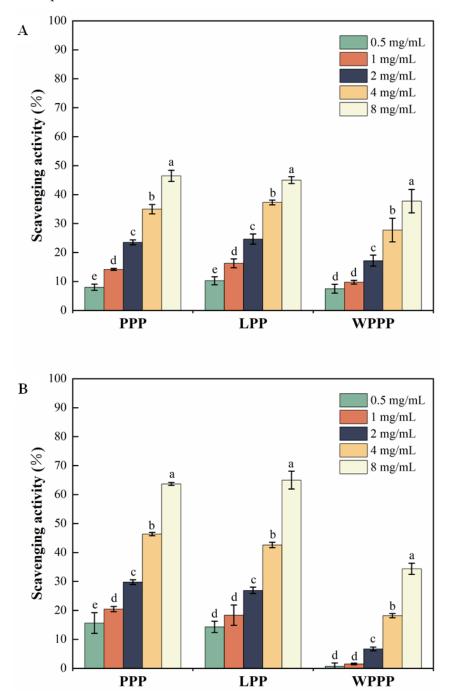


Figure 2. The radical scavenging activity of pomelo peel pectin (PPP), lemon peel pectin (LPP), and white-fleshed pitaya peel pectin (WPPP) toward DPPH (**A**) and ABTS radicals (**B**).

3.4. The Color and Opacity of Peel Pectin Films

Figure 3 shows photographs of the peel pectin films, while Table 2 presents the color and transparency characteristics. All three pectins were yellowish, as indicated by larger b^* values. A recent study on watermelon rind pectin films also reported a yellowish color $(a^* = -1.85 \pm 0.15, b^* = 14.16 \pm 0.26)$ [22]. Lemon peel and pomelo peel pectin films appeared brighter and more transparent, whereas white-fleshed pitaya peel pectin films exhibited lower transparency. This discrepancy may be attributed to the higher content of pigment-like substances in the white-fleshed pitaya peel, leading to the retention of more pigments during the pectin extraction process. Additionally, the white-fleshed pitaya peel pectin film might exhibit some crystallization in the cross-sections, as shown by the SEM analysis (Figure 4), which might be related to their higher opacity.

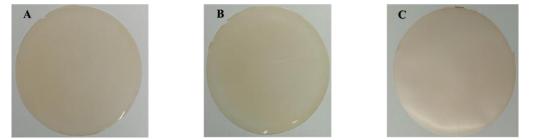


Figure 3. Images of PPP films (**A**), LPP films (**B**), and WPPP films (**C**). LPP: lemon peel pectin, PPP: pomelo peel pectin, and WPPP: white-fleshed pitaya peel pectin.

Film	<i>a</i> *	b^*	ΔE^*	WI	Opacity (A. mm ⁻¹)
PPP	-0.90 ± 0.05 ^b	$10.92\pm0.38~^{\rm a}$	$8.11\pm0.36^{\text{ b}}$	$84.11\pm0.32^{\text{ b}}$	$2.44\pm0.16~^{\rm c}$
LPP	-1.3 ± 0.17 ^b	9.23 ± 0.40 ^b	$6.46\pm0.39\ ^{\rm c}$	$85.57\pm0.39~^{\rm a}$	3.21 ± 0.03 ^b
WPPP	$2.63\pm0.66~^{a}$	11.24 ± 0.74 $^{\rm a}$	9.79 ± 0.72 $^{\rm a}$	$81.13\pm0.70~^{\rm c}$	11.66 ± 0.34 $^{\rm a}$

Table 2. Film color parameters based on PPP, LPP, and WPPP.

LPP: lemon peel pectin, PPP: pomelo peel pectin, WI: whitish index, and WPPP: white-fleshed pitaya peel pectin. Values with different letters in each column indicate statistically significant differences at 5% confidence level.

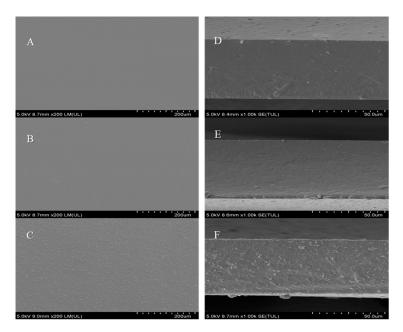


Figure 4. SEM of the surface $(200 \times)$ and cross-section $(1000 \times)$ of PPP films (**A**,**D**), LPP films (**B**,**E**), and WPPP films (**C**,**F**). LPP: lemon peel pectin, PPP: pomelo peel pectin, SEM: scanning electron microscopy, and WPPP: white-fleshed pitaya peel pectin.

Consequently, the white-fleshed pitaya peel pectin film might slightly mask the color of the food itself but possesses better light absorption properties. Numerous studies have aimed to enhance film activity by incorporating natural plant extracts with antioxidant and antimicrobial properties. However, such additions may also reduce the films' brightness (L^*) and transparency.

3.5. The Thickness, Moisture Contents, and WVP of Peel Pectin Films

Table 3 provides information on the pectin films' thickness, moisture content, and water vapor permeability. The thickness of white-fleshed pitaya peel pectin and pomelo peel pectin films is generally greater than that of lemon peel pectin films. The film thickness is typically associated with the amount of dry matter in the film solution for a constant film volume and forming area. No significant differences were observed in water content and water vapor permeability among the pectin films derived from pomelo peel, lemon peel, and white-fleshed pitaya peel.

Table 3. Thickness, moisture content, water vapor permeability, and mechanical properties of films based on PPP, LPPP, and WPPP.

Film	Thickness (µm)	MC (%)	WVP (10 ⁻⁸ g·m ⁻¹ ·s ⁻¹ ·Pa ⁻¹)	TS) (MPa)	EB (%)
PPP	$52.26\pm1.34~^{\rm a}$	$20.81\pm0.36~^{a}$	16.65 ± 0.30 $^{\rm a}$	$22.25\pm1.52^{\text{ b}}$	6.48 ± 0.82 ^b
LPP	49.57 ± 1.52 ^b	18.53 ± 0.68 ^b	16.26 ± 1.35 a	$31.26\pm2.3~^{a}$	$5.71 \pm 0.62^{\ \mathrm{b}}$
WPPP	$53.76\pm1.71~^{a}$	$18.50\pm0.22^{\text{ b}}$	16.47 ± 1.06 $^{\rm a}$	$32.4\pm1.65~^{a}$	10.95 ± 1.51 $^{\rm a}$

LPP: lemon peel pectin, PPP: pomelo peel pectin, and WPPP: white-fleshed pitaya peel pectin. Values with different letters in each column indicate statistically significant differences at a 5% confidence level.

Pectin is known to be highly hydrophilic and contains numerous hydroxyl groups that facilitate water molecule binding, resulting in high water vapor permeability. In this study, the water vapor transmission rates of pectin films from pomelo peel, lemon peel, and white-fleshed pitaya peel were 1.67 ± 0.30 , 1.63 ± 1.35 , and $1.65 \pm 1.06 \times 10^{-9} \text{ g} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$, respectively. A previous study reported a water vapor transmission rate of $4.00 \pm 0.15 \times 10^{-9} \text{ g} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ for watermelon peel pectin films [22] and $3.62 \pm 0.50 \times 10^{-9} \text{ g} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ for mandarin peel pectin films [7], indicating a higher permeability than found for the pectin films in this study. In contrast, the water vapor transmission rate of sweet lime peel pectin film was reported to be $1.86 \pm 0.12 \times 10^{-10} \text{ g} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ [8], suggesting it had a lower permeability than the pectin films in the present study. Various factors, including film thickness, air humidity within the measuring dish, and ambient air humidity, influence a film's water vapor transmission rate.

3.6. The Mechanical Properties of Peel Pectin Films

Table 3 provides information on the tensile strength (MPa) and elongation at break (%) of the peel pectin films. The tensile strength of pomelo peel, lemon peel, and white-fleshed pitaya peel pectin films was 22.25 ± 1.52 , 31.26 ± 2.30 , and 32.40 ± 1.65 MPa, respectively. The tensile strength of the lemon peel and white-fleshed pitaya peel pectin films was higher than that of pomelo peel pectin films. Among the three films, white-fleshed pitaya peel pectin films demonstrated better mechanical properties than pomelo peel and lemon peel pectin films.

In related studies, the tensile strength and elongation at break of mandarin peel pectin films were reported to be 22.54 ± 1.96 MPa and $17.26 \pm 1.67\%$, respectively [7]. Watermelon peel films exhibited a tensile strength of 42.30 ± 0.82 MPa and an elongation at break of $10.77 \pm 0.70\%$ [22]. Sweet lime peel pectin films had a tensile strength of 13.49 ± 0.77 MPa and an elongation at break of $3.11 \pm 0.20\%$ [8]. These results indicate significant differences in the mechanical properties of films derived from different sources of pectin. Furthermore, the mechanical properties of similar fruit peel pectin films have been reported by other studies. For example, one study showed that the tensile strength and elongation at break of lemon peel pectin films was 24.65 \pm 2.15 MPa and 5.65 \pm 0.84% [11]. Additionally, it was reported that films based on pitaya peel pectin demonstrated a tensile strength and elongation at break of 27.67–33.97 MPa and 14.28–18.21%, respectively [23]. Further, reports showed that red pomelo peel pectin films had a tensile strength and elongation at break of 9.65 \pm 0.19 MPa and 7.15 \pm 0.12%, and white pomelo peel pectin films presented a tensile strength and elongation at break of 15.58 \pm 0.31 MPa and 5.91 \pm 0.16%, respectively [24,25].

The molecular forces within the film matrix generally influence the mechanical properties of films. The concentration and type of plasticizers incorporated into the films can also impact the molecular forces within the film matrix. For example, the tensile strength of sweet lime peel pectin films with 0.3 wt% glycerol was approximately 20.32% lower than that of sweet lime peel pectin films with 0.2 wt% glycerol, while the elongation at break increased by about 75.71% [8]. It is hypothesized that including glycerol between the polymer chains reduces intermolecular attraction, reducing film rigidity but increasing chain mobility and flexibility [22,26].

3.7. SEM Analysis of Peel Pectin Films

The microstructure of the peel pectin films was examined using scanning electron microscopy (SEM), and the results are shown in Figure 4. All three films exhibited a relatively uniform surface structure, indicating good film-forming properties. The intact and continuous microstructure of films based on pomelo peel pectin, lemon peel pectin, and white-fleshed pitaya peel pectin has been observed in other studies [11,23,24]. How-ever, compared to pomelo peel and lemon peel pectin films, the surface of white-fleshed pitaya peel pectin films appeared rougher and contained granular substances. A similar rough surface structure with particle aggregation was observed in watermelon peel pectin films [22]. In contrast, pomelo and lemon peel pectin films in this study had smoother surfaces, while the pectin films from another citrus fruit, orange, displayed a rough and uneven surface morphology [27].

Cross-sections of the peel pectin films were also examined, revealing that the crosssections of pomelo and lemon peel pectin films had a smoother and more homogeneous structure. On the other hand, the cross-sections of white-fleshed pitaya peel pectin films exhibited a rough morphology and the presence of granular material, which correlated with the surface structure observations. A similar roughness was observed in the cross-sections of watermelon rind pectin films [28]. The morphology of the film cross-section is associated with various physical properties of the film. For example, having more pores in the film cross-section can lead to poorer mechanical properties, barrier properties, and thermal stability. These observations highlight the importance of a film's microstructure in determining its properties and performance. A uniform and smooth surface structure is generally desirable for films, as it can contribute to improved mechanical strength, barrier properties, and overall film functionality.

3.8. FITR Analysis of Pectin Films

FITR can be utilized to characterize the structural information of films. Figure 5 displays the infrared spectra of the three peel pectin films. The observed peaks at 3390, 3379, and 3397 cm⁻¹ correspond to O–H stretching vibrations, while the peaks at 2934, 2937, and 2938 cm⁻¹ are attributed to C–H stretching vibrations, mainly CH, CH₂, and CH₃ [29]. The characteristic peaks at 1745 and 1748 cm⁻¹ indicate stretching vibrations of methylesterified C=O, while the peaks at 1615, 1624, and 1638 cm⁻¹ represent stretching vibrations of total C=O [22]. The peaks observed at 1015–1101 cm⁻¹ and 955–970 cm⁻¹ may be associated with C–O and C–C bonds in the pyranose ring [19].

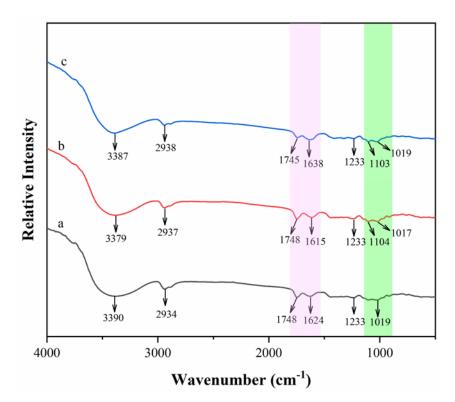


Figure 5. FTIR spectra PPP (**a**), LPP (**b**), and WPPP (**c**) films. FITR: Fourier transform infrared spectroscopy. LPP: lemon peel pectin, PPP: pomelo peel pectin, and WPPP: white-fleshed pitaya peel pectin.

Figure 5a,b depict the IR spectra of the pectin films derived from pomelo peel and lemon peel, respectively. The peak areas generated by the methylated C=O stretching vibrations are greater than those associated with the total C=O stretching vibrations, indicating that pomelo peel and lemon peel pectins are highly methylated. Figure 5c illustrates the infrared spectrum of white-fleshed pitaya peel pectin, where the peak area at 1748 cm⁻¹ is smaller than that of the peak at 1636 cm⁻¹, indicating that white-fleshed pitaya peel pectin is low methylated pectin. The similarity of the characteristic peaks in the FTIR spectra of these three peel pectin films suggests a comparable chemical structure.

4. Conclusions

The present study demonstrated that the pectins extracted from pomelo peel, lemon peel, and white-fleshed pitaya peel were methyl-esterified at levels of 55.87 \pm 0.34%, $55.10 \pm 0.58\%$, and $46.06 \pm 0.37\%$, respectively. Pomelo and lemon peel pectins were highly methyl-esterified, while white-fleshed pitaya peel pectin exhibited lower methylation. Pomelo and lemon peel pectins primarily consisted of galacturonic acid, galactose, arabinose, and glucose, whereas white-fleshed pitaya peel pectin mainly contained galacturonic acid, rhamnose, and galactose. Edible films were prepared using the pectins extracted from these three fruits peel, and their physical properties were assessed. Lemon peel and pomelo peel pectin films exhibited greater transparency and brightness, while whitefleshed pitaya peel pectin films had lower transparency. The thickness of white-fleshed pitaya peel pectin and pomelo peel pectin films was greater than that of lemon peel pectin films. Moisture content and water vapor permeability did not differ significantly among the three peel-based pectin films. The tensile strength of lemon peel and white-fleshed pitaya pectin films was higher than that of pomelo peel pectin films. Among the three peel pectin films, the white-fleshed pitaya peel pectin film displayed the highest elongation at break. Thus, the pitaya peel pectin films generally exhibited superior mechanical properties compared to the pomelo peel and lemon peel pectin films. SEM analysis revealed that the cross-sections of pomelo peel and lemon peel pectin films exhibited a smoother and

more homogeneous structure, while the cross-sections of white-fleshed pitaya peel pectin films displayed a rough morphology. The infrared spectra of these three peel pectin films exhibited similar characteristic peaks, indicating their comparable chemical structures. The pectin from pomelo peel, lemon peel, and white-fleshed pitaya peel demonstrated excellent film-forming properties, which supported their valorization into food packaging. The pectin from these waste fruit peels could be used as a film-forming matrix for food packaging. To improve the physical and functional properties of films based on fruit peel pectin, some reinforcing fillers (metal nanoparticles and nano cellulose, for example) and bioactive agents (such as polyphenols and essential oils) could be incorporated into films. Property-improved fruit peel pectin films might present better preservation effects. Additionally, further studies targeting the possible applications of films based on fruit peel pectin in food preservation could be carried out.

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References

- Mirabella, N.; Castellani, V.; Sala, S. Current options for the valorization of food manufacturing waste: A review. J. Clean. Prod. 2014, 65, 28–41. [CrossRef]
- 2. Alavi, F.; Chen, L. Complexation of nanofibrillated egg white protein and low methoxy pectin improves microstructure, stability, and rheology of oil-in-water emulsions. *Food Hydrocoll.* **2022**, *124*, 107262. [CrossRef]
- Nastasi, J.R.; Kontogiorgos, V.; Daygon, V.D.; Fitzgerald, M.A. Pectin based films and coatings with plant extracts as natural preservatives: A systematic review. *Trends Food Sci. Technol.* 2022, 120, 193–211. [CrossRef]
- Cao, T.L.; Song, K.B. Effects of gum karaya addition on the characteristics of loquat seed starch films containing oregano essential oil. *Food Hydrocoll.* 2019, 97, 105198. [CrossRef]
- Rodrigues, A.A.M.; Santos, L.F.D.; Costa, R.R.D.; Félix, D.T.; Nascimento, J.H.B.; Lima, M.A.C.D. Characterization of starch from different non-traditional sources and its application as coating in 'Palmer' mango fruit. *Cienc. e Agrotecnol.* 2020, 44, e011220. [CrossRef]
- 6. Nawab, A.; Alam, F.; Hasnain, A. Mango kernel starch as a novel edible coating for enhancing shelf-life of tomato (*Solanum lycopersicum*) fruit. *Int. J. Biol. Macromol.* **2017**, *103*, 581–586. [CrossRef] [PubMed]
- Han, H.S.; Song, K.B. Antioxidant activities of mandarin (*Citrus unshiu*) peel pectin films containing sage (*Salvia officinalis*) leaf extract. *Int. J. Biol. Macromol.* 2020, 55, 3173–3181. [CrossRef]
- Rodsamran, P.; Sothornvit, R. Lime peel pectin integrated with coconut water and lime peel extract as a new bioactive film sachet to retard soybean oil oxidation. *Food Hydrocoll.* 2019, 97, 105173. [CrossRef]
- 9. Liew, S.Q.; Ngoh, G.C.; Yusoff, R.; Teoh, W.H. Sequential ultrasound microwave-assisted acid extraction (UMAE) of pectin from pomelo peels. *Int. J. Biol. Macromol.* 2016, 93, 426–435. [CrossRef]
- 10. Jiang, H.; Zhang, W.; Pu, Y.; Chen, L.; Cao, J.; Jiang, W. Development and characterization of a novel active and intelligent film based on pectin and betacyanins from peel waste of pitaya (*Hylocereus undatus*). *Food Chem.* **2023**, 404, 134444. [CrossRef]
- 11. Jiang, H.; Zhang, W.; Cao, J.; Jiang, W. Effect of purple sugarcane peel extracts on properties of films based on lemon peel waste pectin and the application in the visible detection of food freshness. *Food Hydrocoll.* **2022**, *133*, 107982. [CrossRef]
- 12. Jiang, H.; Zhang, W.; Jiang, W. Effects of purple passion fruit peel extracts on characteristics of *Pouteria campechiana* seed starch films and the application in discernible detection of shrimp freshness. *Food Hydrocoll.* **2023**, *138*, 108477. [CrossRef]
- Tongkham, N.; Juntasalay, B.; Lasunon, P.; Sengkhamparn, N. Dragon fruit peel pectin: Microwave-assisted extraction and fuzzy assessment. *Agric. Nat. Resour.* 2017, 51, 262–267. [CrossRef]
- Zaid, R.M.; Mishra, P.; Noredyani, A.S.; Tabassum, S.; Ab Wahid, Z.; Sakinah, A.M. Proximate characteristics and statistical optimization of ultrasound-assisted extraction of high-methoxyl-pectin from *Hylocereus polyrhizus* peels. *Food Bioprod. Process.* 2020, 123, 134–149. [CrossRef]

- 15. Thakur, B.R.; Singh, R.K.; Handa, A.K.; Rao, M.A. Chemistry and uses of pectin—A review. *Crit. Rev. Food Sci. Nutr.* **1997**, *37*, 47–73. [CrossRef]
- 16. Rahmati, S.; Abdullah, A.; Kang, O.L. Effects of different microwave intensity on the extraction yield and physicochemical properties of pectin from dragon fruit (*Hylocereus polyrhizus*) peels. *Bioact. Carbohydr. Diet. Fibre* **2019**, *18*, 100186. [CrossRef]
- 17. Dao, T.A.T.; Webb, H.K.; Malherbe, F. Optimization of pectin extraction from fruit peels by response surface method: Conventional versus microwave-assisted heating. *Food Hydrocoll.* **2021**, *113*, 106475.
- 18. Rahmani, Z.; Khodaiyan, F.; Kazemi, M.; Sharifan, A. Optimization of microwave-assisted extraction and structural characterization of pectin from sweet lemon peel. *Int. J. Biol. Macromol.* **2020**, *147*, 1107–1115. [CrossRef]
- 19. Wandee, Y.; Uttapap, D.; Mischnick, P. Yield and structural composition of pomelo peel pectins extracted under acidic and alkaline conditions. *Food Hydrocoll.* **2019**, *87*, 237–244. [CrossRef]
- Muhammad, K.; Zahari, N.I.M.; Gannasin, S.P.; Adzahan, N.M.; Bakar, J. High methoxyl pectin from dragon fruit (Hylocereus polyrhizus) peel. Food Hydrocoll. 2014, 42, 289–297. [CrossRef]
- Zaid, R.M.; Mishra, P.; Tabassum, S.; Ab Wahid, Z.; Sakinah, A.M. High methoxyl pectin extracts from *Hylocereus polyrhizus's* peels: Extraction kinetics and thermodynamic studies. *Int. J. Biol. Macromol.* 2019, 141, 1147–1157. [CrossRef]
- 22. Han, H.S.; Song, K.B. Antioxidant properties of watermelon (*Citrullus lanatus*) rind pectin films containing kiwifruit (*Actinidia chinensis*) peel extract and their application as chicken thigh packaging. *Food Packag. Shelf Life* **2021**, *28*, 100636. [CrossRef]
- Jiang, H.; Zhang, W.; Xu, Y.; Cao, J.; Jiang, W. Properties of pectin-based films from white-fleshed pitaya (*Hylocereus undatus*) peel waste as affected by montmorillonite. *Food Packag. Shelf Life* 2021, 34, 100952. [CrossRef]
- 24. Sood, A.; Saini, C.S. Red pomelo peel pectin based edible composite films: Effect of pectin incorporation on mechanical, structural, morphological and thermal properties of composite films. *Food Hydrocoll.* **2022**, *123*, 107135. [CrossRef]
- 25. Sood, A.; Saini, C.S. Utilization of peel of white pomelo for the development of pectin based biodegradable composite films blended with casein and egg albumen. *Food Chem. Adv.* **2022**, *1*, 100054. [CrossRef]
- Rojas-Lema, S.; Nilsson, K.; Trifol, J.; Langton, M.; Gomez-Caturla, J.; Balart, R.; Garcia-Garcia, D.; Moriana, R. Faba bean protein films reinforced with cellulose nanocrystals as edible food packaging material. *Food Hydrocoll.* 2021, 121, 107019. [CrossRef]
- Baron, R.D.; Pérez, L.L.; Salcedo, J.M.; Córdoba, L.P.; do Amaral Sobral, P.J. Production and characterization of films based on blends of chitosan from blue crab (*Callinectes sapidus*) waste and pectin from orange (*Citrus sinensis* Osbeck) peel. *Int. J. Biol. Macromol.* 2017, 98, 676–683. [CrossRef]
- 28. Guo, Z.; Ge, X.; Li, W.; Yang, L.; Han, L.; Yu, Q.L. Active intelligent film based on pectin from watermelon peel containing beetroot extract to monitor the freshness of packaged chilled beef. *Food Hydrocoll.* **2021**, *119*, 106751. [CrossRef]
- 29. Shivangi, S.; Dorairaj, D.; Negi, P.S.; Shetty, N.P. Development and characterization of a pectin-based edible film that contains mulberry leaf extract and its bio-active components. *Food Hydrocoll.* **2021**, *121*, 107046. [CrossRef]

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