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Pectin extracted from *Dillenia serrata* fruit peels using ammonium oxalate and microwave-assisted extraction methods and its usage in film packaging

Adiansyah Syarifuddin [a,b,*], Ahmad Azhari Arif [a,b], Serli Hatul Hidayat [a], Zainal [a,b], Andi Dirpan [a,b], Intan Syafinaz Mohamed Amin Tawakkal [c]

[a] Department of Food Science and Technology, Faculty of Agricultural Technology, Hasanuddin University, Makassar, 90245, Indonesia
[b] Bio-Based Food Packaging Research, Makassar, 90245, Indonesia
[c] Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia

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ABSTRACT

This research focused on extracting and characterizing pectin from the peel of *Buah Jongi* or *Dengen* (*Dillenia serrata*) using two methods: ammonium oxalate and microwave-assisted ammonium oxalate. The study utilized two types of peels, namely peel puree and dried peel powder, to assess their impact on the physical, mechanical, optical, and barrier properties of pectin-gelatin film. Results showed that the type of peel significantly influenced the yield, methoxyl content, and degree of esterification of the pectin. Among the peels, the dried peel powder extracted with ammonium oxalate yielded the highest results, with a yield of 21.32% and a methoxyl content of 2.18%. Pectin obtained from dried peel powder through both extraction methods was then evaluated as a film packaging material at varying pectin concentrations (3%, 4%, and 5%). Regarding film properties, a 3% pectin concentration produced the thinnest films (0.07 mm), while the ammonium oxalate extraction method resulted in films with the highest L value. In contrast, the microwave-assisted ammonium oxalate method showed lower water solubility (59.84%) and water vapor transmission rate (2.76 g m^{-2} day^{-1}) but exhibited higher tensile strength (1.08 MPa). An increased methoxyl content and degree of esterification were found to enhance the quality of pectin-gelatin film properties.

1. Introduction

The use of petroleum-derived polymers, particularly plastics, has been steadily rising in numerous sectors, such as food packaging. In Indonesia, plastic waste constitutes a significant portion of household refuse, accounting for 19.12% of the 30 million tons produced each year (Zuhdi et al., 2025). To address the growing environmental concerns associated with plastic usage in various food products, several approaches have been adopted, including the incorporation of biodegradable biopolymers like pectin, which is considered a promising option for eco-friendly food packaging.

Pectin, a natural polysaccharide, holds potential as a key material for developing food packaging films. It is composed of a linear sequence of poly-α-(1–4)-D-galacturonic acid with varying degrees of methyl esterification and comprises three polysaccharide domains: homogalacturonan, rhamnogalacturonan I, and rhamnogalacturonan II (Jiang et al., 2024). The global pectin market size to be valued at USD 1.9 billion by 2025 and is expected to grow at a compound annual growth rate (CAGR) of 7.1% during the forecast period (Grand View Research Inc., 2026). This growth is fueled by its beneficial characteristics, including excellent gel-forming capability, biocompatibility, and biodegradability. Additionally, the Food and Drug Administration has designated pectin as generally recognized as safe (GRAS) for use in the food industry (Roy et al., 2023). This trend is mirrored by the increasing scientific interest in pectin, as evidenced by a substantial increase in related publications over the past decade—from 229 in 2013 to 5356 in 2023—focusing on its diverse functions like gelling, film formation, emulsification, extraction methods, and molecular interactions (Assifaoui et al., 2024).

Dillenia serrata, a member of the Dillenia genus found in Sulawesi, is known for its seasonal fruit with a notably sour taste. In South Sulawesi, it is called *Dengen*, while in Central Sulawesi, it is known as *Jongi* (Wibawa et al., 2021). This fruit is recognized for its significant antioxidant properties, which can inhibit the growth of bacteria like *Pseudomonas aeruginosa, Staphylococcus aureus*, and *Streptococcus* mutans (Wibawa et al., 2021). Additionally, it contains bioactive substances

* Corresponding author at: Department of Food Science and Technology, Faculty of Agricultural Technology, Hasanuddin University, Makassar, 90245, Indonesia.
E-mail address: adiansyah@agri.unhas.ac.id (A. Syarifuddin).

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such as koetjapic acid, betulinic acid, and 3-oxoolean-12-en-30-oic acid, which have shown potential in reducing prostaglandin E2 production (Jalil et al., 2015). A variety of studies indicated that the pectin content of *Dillenia indica* fruit ranges from 10.3% to 20%, depending on the extraction method and pH applied (Choudhury et al., 2025; Kamal et al., 2020; Silva et al., 2024). However, to date, no studies have reported the pectin content of the peel and pulp of *Dillenia serrata*.

Pectin extraction is the foundational step in creating films based on pectin. Various extraction methods have been utilized, including traditional techniques like the use of organic (Chen et al., 2022) and inorganic acids (Biratu et al., 2024), as well as innovative methods such as deep eutectic solvent-based extraction (Della Posta et al., 2022), microwave-assisted extraction (Mahmud et al., 2021), ultrasound-assisted extraction (Panwar et al., 2023), ohmic heating (Sharifi et al., 2022), enzyme-assisted extraction (Ke et al., 2023), and subcritical water extraction (SWE) (Tristanto et al., 2024). The process of extracting pectin from fruit peels is affected by numerous factors, including the plant source, the specific part of the plant used, the extraction method chosen, and various process conditions like temperature, solvent type, and extraction duration (Alara et al., 2021). Research has been conducted on extracting pectin from the *Dillenia genus* and its impact on the characteristics of edible films. For example, some authors found that pectin from *Dillenia indica* fruit, extracted using ultrasound-assisted extraction and conventional mechanical stirring, shows promise for use in food ingredients (Silva et al., 2024). Huda et al. (2025) also discovered that combining pectin extracted from *Dillenia serrata* with fish oil enhanced the physical, mechanical, and barrier properties of edible films. Selecting the right extraction technique is crucial, as it significantly affects the quality, yield, and functional properties of the pectin obtained. Pectin exhibits a notable limitation in its mechanical performance (Sani & Alizadeh, 2022), attributed to restricted intermolecular chain mobility and hydrophilic backbones. To address this, gelatin is frequently incorporated into pectin-based films. The interaction between gelatin and pectin significantly affects film formation by facilitating the development of ionic and hydrogen-bonded complexes (Lin et al., 2021).

Numerous studies have explored the use of ammonium oxalate in pectin extraction from plants. In the cell walls of plants, pectin is frequently bound with calcium ions (Ca^{2+}), creating structures such as calcium pectate, which complicates its extraction. Ammonium oxalate can be used to address this issue, as the oxalate ion ($C_2O_4^{2-}$) has a strong affinity for calcium, forming insoluble calcium oxalate. This process effectively removes Ca^{2+} from the pectin matrix, making the pectin soluble and easier to extract without significant degradation. Ma et al. (2021) reported that pectin extracted from sunflowers using ammonium oxalate and freeze drying had a lower molecular weight (316 ± 3 kDa), galacturonic acid content (76.2%), and was easier to form into a gel. Han et al. (2024) examined pectin extraction from sweet potatoes using ammonium oxalate, achieving a degree of esterification of 12.76% and a degree of methoxy of 1.92%, indicating it is naturally low-ester pectin. New methods, including microwave-assisted extraction, are being used to extract pectin from various food-derived waste products. Han et al. (2021) reported that polysaccharides extracted using microwave-assisted ammonium oxalate yielded the highest polysaccharide content (9.3 ± 0.5%), with lower molecular weight distributions and higher total reducing power and scavenging ability. According to the previous study, the extraction time is varied from 30 min to 3 h to find the point where yield improves before microwave exposure starts degrading the pectin Liew et al. (2018). There is a notable deficiency in data regarding the extraction of pectin from *Dillenia serrata* using ammonium oxalate and microwave-assisted ammonium oxalate methods, as well as analyses of the structural properties and the impact on pectin-based film packaging. Consequently, this study not only explores the use of *Dillenia serrata* peel as a novel source of pectin but also examines the effects of conventional ammonium oxalate extraction and microwave-assisted ammonium oxalate extraction on pectin functionality and the performance of pectin-based films. Therefore, this study contributes to a better understanding of the relationship between extraction method, pectin functionality, and film performance.

The objectives of this study were to examine the effects of two distinct extraction techniques, ammonium oxalate (AO) and microwave-assisted ammonium oxalate (MAO), on two types of peels, namely peel puree and dried peel powder, and to evaluate their effect on the physical, mechanical, optical, and barrier properties of film packaging. Ammonium oxalate was selected in this study because of its considerably lower environmental impact than that of strong acids and bases. In addition, it causes significantly less equipment corrosion in practical production compared with conventional acid-based methods. Additionally, 0.6% ammonium oxalate was selected because it had maximized pectin hydrolysis (Ma et al., 2021). To achieve these aims, four treatments were conducted, following a full factorial design that incorporated the two extraction methods and the two peel types. Specifically, film was produced using dried peel powder obtained from both ammonium oxalate and microwave-assisted ammonium oxalate extractions with different pectin concentrations to determine the efficiency of each extraction method and pectin concentrations.

2. Materials and methods

2.1. Raw materials and reagents

Dillenia serrata fruits were collected from East Luwu regency, South Sulawesi Province, Indonesia. Additional analytical-grade reagents, including ammonium oxalate, ethanol 96%, and citric acid from Merck, were purchased from a local chemical supplier and used without further purification. Food-grade gelatin (Essenli, Indonesia) was purchased from a commercial supplier.

2.2. Dillenia serrata fruit peel preparation

The fruits of *Dillenia serrata* were washed under running water to eliminate impurities and non-edible parts before being peeled. The peels were then sliced into small pieces, and a puree was made by blending the peel with water using a food juicer, resulting in a moisture content of 94.50%. To create dried peel powder, the *Dillenia serrata* fruit peels were again washed under running water. These peels were sliced into smaller pieces and oven-dried at 80 °C for 8 h, achieving a moisture content of 7.20%. The dried peels were subsequently ground to obtain a particle size of 80 mesh.

2.3. Pectin extraction

2.3.1. Ammonium oxalate (AO)

A total of 5 g of dried peel powder and 20 g of peel puree were mixed with 100 mL of ammonium oxalate solution, and the pH was adjusted to 2.5, which is acidic enough to hydrolyze protopectin and release pectin (Leão et al., 2018; Sengkhamparn et al., 2019). The mixture was then stirred at 90 °C for one hour. Subsequently, ethanol was added in a 1:2 (w/v) ratio to induce pectin precipitation, and the mixture was left to equilibrate for 24 h. Following this, the supernatant was separated by centrifuging at 8000 × *g* force for 10 min, rinsed three times with ethanol, and the resulting wet pectin was dried in an oven at 40 °C for 12 h.

2.3.2. Microwave-assisted ammonium oxalate (MAO)

A total of 5 g of dried peel powder and 20 g of peel puree were added to 100 mL of ammonium oxalate solution. The mixture's pH was adjusted to reach a level of 2.5. Subsequently, the solution was placed in a domestic microwave (maximum power: 900 W, frequency: 2450 MHz) (Sharp R-725DA BK). The extraction process was conducted under constant conditions for 30 min. The final extraction temperature, measured immediately following the treatment, reached approximately

90 °C. Once completed, the sample was rapidly cooled to room temperature. Ethanol was then introduced to the mixture in a 1:2 (w/v) ratio to precipitate pectin, and the mixture was allowed to equilibrate for 24 h. Afterward, the supernatant was removed by centrifugation at 8000 × *g* force for 10 min. The precipitate was then washed three times with ethanol, and the resulting wet pectin was dried in an oven at 40 °C for 12 h.

2.4. Pectin properties

2.4.1. Yield of pectin

The pectin yield obtained from dried peel powder was determined according to Eq. (1):

$$\text{Yield}(\%) = \frac{\text{the dry weight of pectin extracted}}{\text{the dry weight of } \textit{Dillenia serrata} \text{ fruit peel before extraction}} \text{x}100\% \quad (1)$$

2.4.2. Equivalent weight

The equivalent weight of pectin was determined following a slightly modified procedure from by Rahman et al. (2026). 250 mL conical flask was used to hold 0.5 g of pectin, to which 5 mL of 96% ethanol was added. The mixture was then stirred with a magnetic stirrer for 10 min. Subsequently, 1 g of NaCl, 100 mL of deionized water, and three drops of phenol red indicator were introduced. Stirring was maintained until the pectin was fully dissolved, after which the solution was titrated with 0.1 mol/L sodium hydroxide to a pink endpoint. The neutralized solution was then reserved for methoxyl content analysis. The EW of pectin was calculated using Eq. (2):

$$\text{Equivalent weight}\left(\frac{\text{g}}{\text{mol}}\right) = \frac{\text{Weight of sample (g) x 1000}}{\text{Alkali used (mL)x Molarity of alkali}\left(\frac{\text{mol}}{\text{L}}\right)} \quad (2)$$

2.4.3. Methoxyl content

The methoxyl pectin content was determined following the methodology outlined in a previous study (Dambuza et al., 2024). To the neutral solution derived from the equivalent weight, 25 mL of 0.25 mol/L NaOH was added and stirred using a magnetic stirrer for 30 min. Subsequently, 25 mL of 0.25 mol/L HCl was added, and the solution was titrated to the pink endpoint. The methoxyl content was then calculated using Eq. (3):

$$\text{Methoxyl content (\%)} = \frac{\text{mL of alkali x Normality of alkali x 31}}{\text{Weight of sample (g)x 1000}} \text{x}100\% \quad (3)$$

2.4.4. Degree of esterification (DE)

The DE was calculated using the formula previously described by Khamsucharit et al. (2018) using Eq. (4). In this study, anhydrouronic acid (AUA) was determined according to previous study (Dambuza et al., 2024) using Eq. (5). z is the milliliters (titer) of sodium hydroxide from the equivalent weight determination while y is the milliliters (titer) of NaOH from the methoxyl content determination, and w is the weight of the sample.

$$\text{Degree of esterification (\%)} = \frac{\text{176 x Methyl content}}{\text{31 x AUA}} \text{ x 100\%} \quad (4)$$

$$\text{AUA}(\%) = \frac{176 \;\; 0.1z \, x \, 100}{w \, x \, 1000} + \frac{176 \;\; 0.1y \, x100}{w \, x \, 1000} \quad (5)$$

2.5. Pectin-gelatin film (PF) preparation

The choice to use pectin derived from dried peel powder for pectin-based film was made because the dried peel powder exhibited a higher degree of esterification compared to that of puree peel. Additionally, the methoxyl content of the dried peel powder is higher, which is good for making film packaging. They provide important information regarding the chemical structure of pectin and its potential gel-forming and film-forming behavior. In this experiment, six films were created using a full factorial design that considered two variables: pectin concentration levels (3%, 4%, 5%) and the extraction methods (ammonium oxalate, microwave-assisted ammonium oxalate). The chosen pectin concentrations were based on a prior study (Tristanto et al., 2024). 3–5 g of pectin was dissolved in 100 mL of distilled water. Next, 1 g of gelatin powder and glycerol 30% (w/w based on pectin) were successively added to the mixture and stirred on a hotplate stirrer (DLAB MS-H280-Pro) at 60ºC, followed by homogenization with an ultra-turrax (Heidolph RZR 2021) until a homogeneous solution was obtained. The solution intended for film formation was uniformly poured into a Petri dish and subsequently dried in an oven (Faithful GP-30BE) at 40 °C for 16 h. Afterward, it was removed and kept in an incubator. Six films were labeled as pectin-gelatin film with ammonium oxalate (PF-AO) dan pectin-gelatin film with microwave ammonium oxalate (PF-MAO): (i) PF-AO 3%, (ii) PF-AO 4%, (iii) PF-AO 5%, (iv) PF-MAO 3%, (v) PF-MAO 4%, (vi) PF-MAO.

2.6. Film characterization

2.6.1. Thickness

In this study, the film's thickness was assessed by measuring at five randomly selected locations using a digital caliper (TDT25) with an accuracy of 0.01 mm, followed by averaging these measurements.

2.6.2. Colour

The films' color properties were evaluated using a colorimeter (CHN Spec CS-10). The color measurement parameters included Lightness (L*), which spans from 0 (dark) to 100 (light); redness (a*), where positive values represent red and negative values represent green; and yellowness (b*), with positive values signifying yellow and negative values signifying blue (Ghoshal & Kaur, 2023). The testing process involved placing sensors on the surfaces of the films. Each measurement was conducted three times, and the results were averaged.

2.6.3. Transparency

Film transparency was measured using a previously performed method (Ríos-De-benito et al., 2021). A 1 × 3 cm piece of film was prepared, then attached to the top and bottom edges of the cuvette, and its transmittance at 600 nm was measured using a spectrophotometer (SHIMADZU, UV-1280). The transparency was then calculated with the following Eq. (6):

$$T = \frac{A600}{\delta} \quad (6)$$

where T is the transparency, A is the absorbance, and δ is the thickness.

2.6.4. Water solubility (WS)

First, a 2 × 2 cm film was dried using a drying oven (Huanghua, GP-

30BE) at 105 °C for 24 h to ensure complete drying. The initial weight (W1) of the sample was recorded with a precision of 0.001 g. The dried film was then immersed in 50 ml of water for 24 h and gently stirred at room temperature. After the soaking process, the film was dried again at 105 °C for 24 h to ensure the final dry weight (W2), which was found to be insoluble in water. The solubility of the films was then calculated using the following Eq. (7):

$$WS\ (\%) = \frac{W1 - W2}{W1}\ x\ 100\% \tag{7}$$

2.6.5. Water vapor transmission rate (WVTR)

WVTR of film was measured according to previous study (Silva et al., 2019) with slight modification. First, the film was cut into a circle larger than the mouth of the test cup. 10 mL of distilled water was added to the 3 cm diameter cup, then covered with film and sealed with liquid paraffin. The weight of the entire device (test cup, water, and film) was recorded every 24 h over a 6-day period while stored in a desiccator with silica gel. The weight change, Δ_W, represents the amount of water absorbed by the silica gel over time. The slope of each line, Δ_W/Δ_t, was calculated using linear regression, and *A* represents the area of the film in square meters. The WVTR was expressed using the following Eq. (8):

$$WVTR = \frac{\Delta W}{\Delta t.A} \tag{8}$$

2.6.6. Tensile strength

The tensile strength of the film was calculated using a Testometric material testing machine, with slight adjustments to the method described in the previous study (Sariningsih et al., 2019). First, the room temperature and relative humidity were set at 25 ± 1 °C and 51%. Next, the film was cut into a rectangle measuring 20 × 50 mm. The film was then clamped between the machine's clamps and stretched at a speed of 100 mm/min. The tensile strength of the films at the breaking point was determined using the following equation. τ represented tensile strength (MPa), Fmax was the force (N), and A indicated the cross-sectional area (mm^2). The TS was calculated according to Eq. (9):

$$\tau = \frac{Fmax}{A} \tag{9}$$

2.6.7. FTIR

The films were characterized using FTIR analysis (QATR-S, Shimadzu). The analysis covered a spectral range from 4000 cm^{-1} to 500 cm^{-1}, with 32 scans performed at a resolution of 2 cm^{-1}.

2.7. Statistical analysis

Data analysis was performed using R software (version 4.3.2; R Core Team, 2023). A two-way analysis of variance (ANOVA) was applied to the pectin data, with extraction methods and types of peels designated as fixed factors. Similarly, for the pectin-gelatin films data, a two-way ANOVA was conducted, considering the extraction method and pectin concentrations as fixed factors. All experiments were executed in triplicate, and results were reported as mean ± standard deviation. Error bars in figures show the standard deviation. Statistical significance was determined at $P < 0.05$ for all data analyses. Post hoc comparisons were conducted using the Bonferroni test. Additionally, a Principal Component Analysis (PCA) was performed employing the PCA function from the *FactoMineR* package Figs. 1–3.

3. Results and discussion

3.1. Pectin characterization

3.1.1. Yield of pectin

The yields of pectin extracted from dried peel powder with ammonium oxalate were 21.32 ± 3.34% and 24.90 ± 3.66% for dried peel powder extracted with microwave-ammonium oxalate, while those from peel puree extracted with ammonium oxalate were 5.35 ± 0.95% and 6.75 ± 1.43% for peel puree extracted with microwave-ammonium oxalate, as shown in Table 1. ANOVA was conducted on pectin yield, considering the extraction method and peel type as fixed factors, with the findings presented in Table 2. The findings indicated that the type of pectin peel significantly affected the yield of pectin. Nonetheless, neither the extraction method nor the interaction between the extraction process and the type of pectin peel had a notable impact. The yields of *Dillenia serrata* fruit peel extracted from dried peel powder are significantly higher than those extracted from peel puree. The yield of dried peel powder in this study is highly competitive with the extraction yields using acid conditions (24.91%) (Dirpan et al., 2025) and microwave-assisted extraction method (26.63%)(Hossain et al., 2024), banana-papaya mixed peels (23.74%) (Mada et al., 2022) and significantly greater than the yield obtained from banana peel under microwave-assisted extraction (14.34%) (Aklilu, 2021), pineapple peel with ultrasound-assisted extraction (16.24%) (Shivamathi et al., 2022),

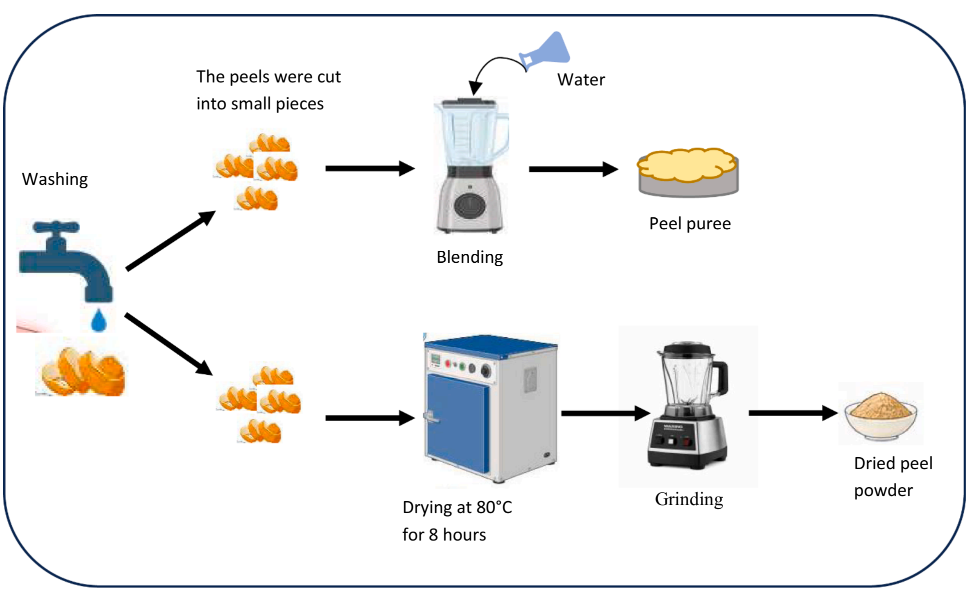

Fig. 1. *Dillenia serrata* fruit peel preparation.

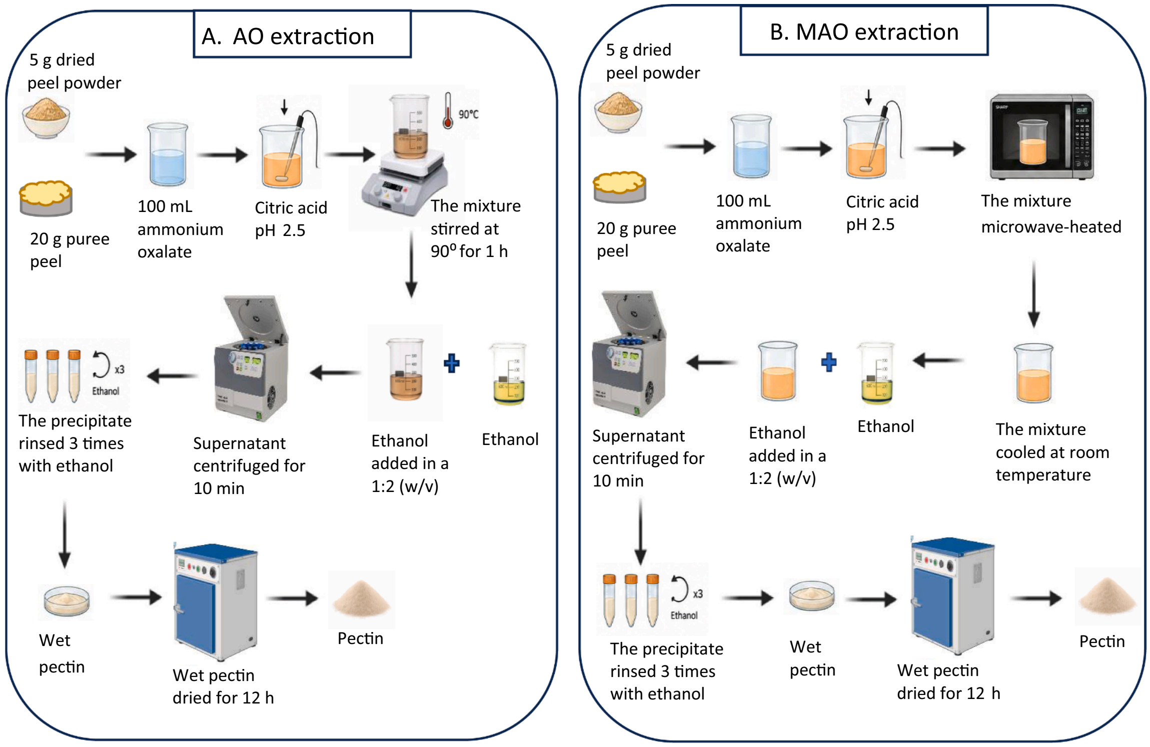

Fig. 2. A diagrammatic representation of pectin extraction using the AO and MAO methods.

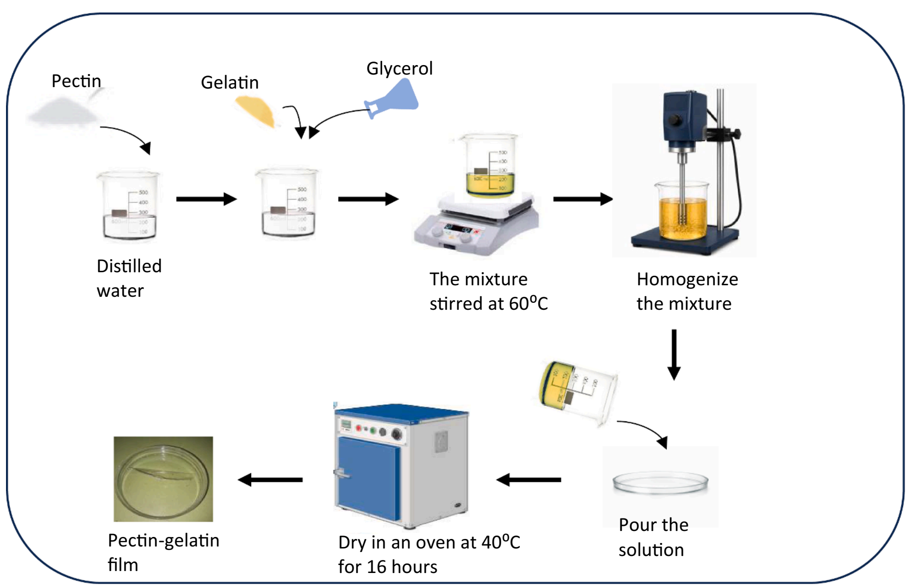

Fig. 3. Flow chart of pectin-gelatin film.

fresh dragon fruit peel puree (13.40%) and dried peel powder (15.28%) extracted using subcritical water extraction and conventional acid extraction methods (Tristanto et al., 2024) as well as yield obtained from *Dillenia indica* fruits (Silva et al., 2024). These findings indicate that drying can compromise the integrity of the cell wall, thereby facilitating the solvent's ability to extract pectin. The movement of pectin molecules increases when exposed to high temperatures, leading to the entanglement of molecular chains. This results in more junction zones being accessible to free calcium ions, thereby significantly increasing pectin content (Nguyen & Pirak, 2019; Tristanto et al., 2024). The reported variability in pectin yield is affected by several factors, including pH levels, temperature, duration of extraction, the type of acid employed, and the characteristics of the pectin sources (Mamiru & Gonfa, 2023).

3.1.2. *Equivalent weight*

The equivalent weight of pectin extracted from dried peel powder using ammonium oxalate was found to be 714.67 ± 16.68 g/mol, while for dried peel powder extracted with microwave-ammonium oxalate, it was 608.21 ± 23.90 g/mol. In addition, pectin from peel puree extracted with ammonium oxalate had an equivalent weight of 793.89 ± 76.18 g/mol and for peel puree extracted with microwave-ammonium oxalate was 535.04 ± 12.20%. These values, as presented in Table 1, are consistent with the equivalent weight range of pectin derived from Citrus maxima peel (525–717 g/mol) (Hossain et al., 2024) and from

Table 1
Characteristics of *Dillenia serrata* peel pectin obtained through ammonium oxalate (AO) and microwave-assisted extraction (MAO) methods from different types of peels.

Extraction method	Type of peels	Yield (%)	Equivalent weight (g/mol)	Methoxyl content (%)	Degree of esterification (%)
Ammonium oxalate (AO)	Dried peel powder	21.32 ± 3.34[a]	714.67 ± 16.68[a]	2.18 ± 0.08[a]	22.27 ± 0.60[a]
Ammonium oxalate (AO)	Peel puree	5.35 ± 0.95[b]	793.89 ± 76.18[a]	1.19 ± 0.51[b]	17.25 ± 2.51[b]
Microwave-assisted extraction (MAO)	Dried peel powder	24.90 ± 3.66[a]	608.21 ± 23.90[b]	2.31 ± 0.10[a]	21.22 ± 0.37[a]
Microwave-assisted extraction (MAO)	Peel puree	6.75 ± 1.43[b]	535.04 ± 12.20[b]	1.39 ± 0.09[b]	14.97 ± 0.41[b]

Notes: Data are presented as mean ± standard deviation from three replicates. Different superscript letters within the same column reveal significant differences ($p \leq 0.05$).

Citrus macroptera peel (514.03–667.58 g/mol)(Sarkar et al., 2025). The equivalent weight of pectin obtained in this study falls within the IPPA standard of <800 g/mol (Akhter et al., 2024). ANOVA was conducted to evaluate the equivalent weight of pectin, considering the extraction method and type of peel as fixed factors, with the findings presented in Table 2. The findings indicated that the extraction methods significantly affected the equivalent weight of pectin. However, the type of pectin peel did not have a significant impact on the equivalent weight. There was also a significant interaction between the extraction method and the type of pectin peel concerning equivalent weight. Pectin extracted from the peel of *Dillenia serrata* using microwave-assisted ammonium oxalate has a lower equivalent weight compared to that extracted using ammonium oxalate. Utilizing elevated temperatures, reduced extraction durations, and increased power levels may have minimized the degradation of pectin molecules, leading to a decreased equivalent weight. Rahman et al. (2026) reported similar findings. They propose that the lower equivalent weight is due to microwave irradiation, which facilitates depolymerization and demethoxylation processes. This process accelerates proton diffusion and aids in the decomposition of glycosidic bonds within the protopectin structure. The equivalent weight of the pectin derived from this study indicates that pectin extracted from *Dillenia serrata* peels holds significant promise for creating robust and flexible films, especially when combined with other biopolymers like gelatin. Blending pectin with gelatin enhances the barrier, mechanical, and thermal stability properties of the films (Li et al., 2024; Ma et al., 2026).

3.1.3. Methoxyl content

This study also assessed the methoxyl content of pectin, as it affects various film characteristics, including flexibility and elongation, mechanical strength, water solubility, hydrophilicity, and barrier properties. As shown in Table 1, the methoxyl content exhibited significant variation based on the type of pectin peel. The highest methoxyl content was found in dried peel powder extracted using microwave-assisted ammonium oxalate, with a value of 2.31 ± 0.10%. This was followed by dried peel powder extracted with ammonium oxalate, which had a value of 2.18 ± 0.08%. On the other hand, peel puree extracted with microwave-assisted ammonium oxalate showed a lower methoxyl content of 1.39 ± 0.09%, and peel puree extracted with ammonium oxalate had an even lower value of 1.19 ± 0.51%. ANOVA was performed on the methoxyl content of pectin with the extraction method and type of peel as the fixed factor and the results are summarised in Table 2, revealing that both the extraction method and the type of pectin peel significantly influenced the methoxyl content. However, their interaction did not have a significant effect, as indicated in Table 2. The methoxyl content in this study is categorized as low methoxyl as its methoxyl content < 7.12% (Tafa et al., 2025). Similar values were also reported by Muniasamy et al. (2025). They found low methoxyl content extracted from bilimbi and pomelo peels in the range 2.79 ± 0.25% and 1.55 ± 0.18%. The reduced methoxyl content enhances properties such as stronger gel formation, improved moisture resistance, and favorable mechanical strength, making low methoxyl pectin (LMP) well-suited for use in food packaging, edible films, and controlled-release applications (Ma et al., 2021).

3.1.4. Degree of esterification

The degree of esterification is another crucial factor affecting the quality and uses of pectin, as it determines gelling behavior, emulsification, solubility, thermal stability, and interactions with cations. Table 1 shows that the degree of esterification for dried peel powder extracted with ammonium oxalate and microwave-assisted ammonium oxalate is slightly higher at 22.27 ± 0.60% and 21.22 ± 0.37%, respectively. In contrast, the peel puree extracted with ammonium oxalate and the peel puree extracted with microwave-assisted ammonium oxalate have lower esterification levels of 17.25 ± 2.51% and 14.97 ± 0.41%. ANOVA was performed on the degree of esterification with the extraction method and type of peel as the fixed factor and the results are summarised in Table 2. The results showed that the extraction method and the type of pectin peels had a significant impact on the degree of esterification. Nonetheless, neither the extraction method alone nor its interaction with the peel type had a significant effect on the degree of esterification. The degree of esterification values of extracted pectin are consistent with previous research findings, such as 14.8–18.1% for persimmon pectin (Piltz et al., 2022), 13.9% for the passion fruit rind pectin using mild acid extraction (Pereira et al., 2024) and ~20% for carrot jam (Muniasamy et al., 2025). The low degree of esterification value observed during microwave treatment is due to improved proton diffusion and the increased breakdown of glycosidic and ester bonds, resulting in demethylation and depolymerization processes. Similar degradation and demethylation under microwave-assisted extraction were observed in carambola peel (Rahman et al., 2026) and pomelo peel

Table 2
ANOVA conducted on pectin's yield, equivalent weight, methoxyl content, and degree of esterification are presented. Green cells denote significance at $p < 0.001$, grey cells show significance at $p < 0.05$, and empty cells represent no significant effect ($p > 0.05$).

	Yield (%)	Equivalent weight (g/mol)	Methoxyl content (%)	Degree of esterification (%)
Extraction methods	p=0.216	p=0.0004	p=0.02	p=0.018
Type of peels	p<0.0001	p=0.927	p<0.0001	p<0.0001
Extraction methods*Type of peels	p=0.573	p=0.043	p=0.537	p=0.311

pectin (Hossain et al., 2024). This notable difference in esterification between dried peel powder and peel puree can be attributed to the high water content in the puree, which leads to greater degradation of pectin and damage to its main chain. This result aligns with earlier studies indicating that pectins dissolve more readily in water, which allows them to undergo de-esterification and depolymerization through enzymatic processes (Sharma et al., 2024).

3.2. Film characterization

3.2.1. Thickness

Thickness is a fundamental parameter in film packaging because it governs physical, mechanical, barrier and optical properties and is influenced by film composition and processing conditions. ANOVA (extraction method and pectin concentration as fixed factors) was also conducted on the thickness of films. The pectin concentrations revealed a significant effect on thickness ($F = 16.97$; $p = 0.0003$), while extraction methods and their interaction showed no significant effect on thickness ($p > 0.05$). Films with the addition of 3% pectin concentration showed the lowest thickness (0.07 ± 0.01 mm) compared to those with higher concentrations (0.12 ± 0.01 mm), as shown in Fig. 4a. When the concentration of pectin in the film-forming solution is increased, both the increase in total polymer solids and the interactions between pectin and gelatin contribute to the formation of a more compact and dense polymer network. Therefore, these results may be explained by the differences in the total solid content. Future studies should employ formulations with constant total solid content. This observation aligns with findings from other researchers, who have noted that a higher pectin concentration elevates the level of dissolved solids, consequently resulting in the creation of thicker films (Tong et al., 2023). Biratu et al. (2024) also found that film thickness increases with higher pectin concentrations in coffee pulp pectin-based edible films. The thickness of

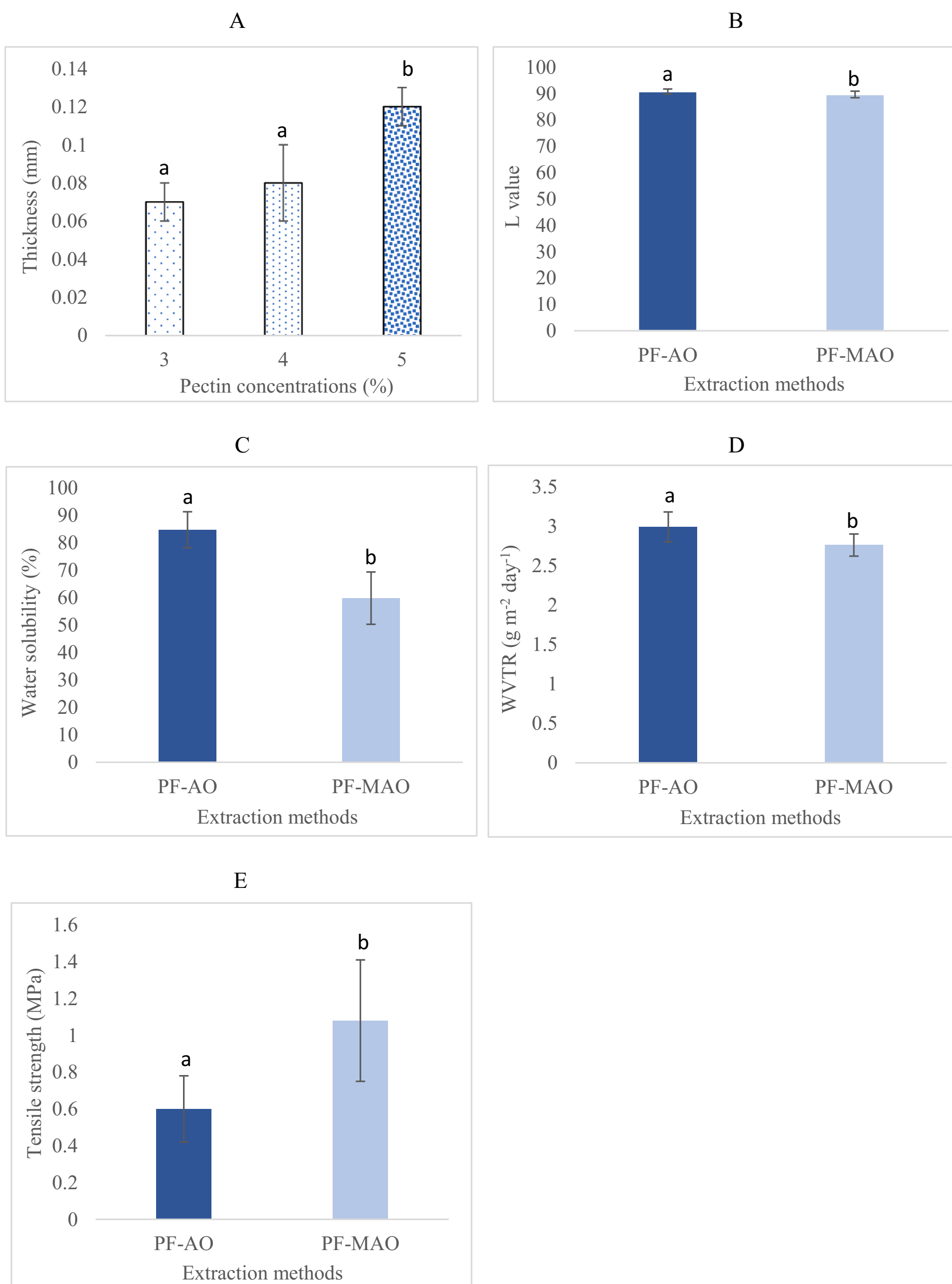

Fig. 4. Characterizations of film influenced by extraction methods and pectin concentration.

films in this study is comparable with pectin-starch blended films (do Nascimento et al., 2024), coffee pulp pectin films (Biratu et al., 2024), and red dragon fruit pectin films (Tristanto et al., 2024). However, it was higher compared to pectin films combined with pullulan (0.047–0.055 mm) (Santamaría et al., 2025) and pectin and lignin-containing cellulose nanofiber film (0.05–0.086 mm) (Bello & Peresin., 2024). Future studies should utilize formulations with a consistent total solid content to more effectively isolate the specific contribution of pectin concentration.

3.2.2. Color properties

ANOVA was performed on the *L* value of films, considering the extraction method and pectin concentration as fixed factors. The analysis showed that the extraction method significantly influenced the *L* value ($F = 4.913$; $p = 0045$), whereas pectin concentrations and their interaction did not have a notable impact ($p > 0.05$). Fig. 4b demonstrates that PF-AO exhibited a higher lightness value compared to PF-MAO. Despite both samples showing a significant effect, their L^* values suggest a high degree of lightness, indicating that the film retained its brightness level regardless of the extraction method used. The presence of ammonium oxalate might cause the bonds between pectin and water-soluble colorants to weaken, resulting in a lighter color in pectin. However, the extraction method, pectin concentration, and their interactions did not significantly affect the a^* value. Regarding the b^* value, the extraction method ($F = 11.565$, $p = 0.005$) and its interaction ($F = 8.909$, $p = 0.004$) had a significant effect. The b^* value increased from 4.14 ± 1.58 for PF-AO to 6.32 ± 2.29 for PF-MAO, which indicated a yellowish shift in the film color. This yellowish color of the film is caused by some of the water-soluble pigments that dissolve and precipitate during the microwave-assisted extraction process. This finding was consistent with the previous study by Tristanto et al. (2024) who found that the higher solubility and precipitation of water-soluble pigments tend to increase the yellowness value of the resulting pectin-gelatin film. In addition, pectin extracted with microwave assistance tends to increase the non-crystalline regions, making the pectin more susceptible to pigment entrapment or browning reactions during film drying (Yang et al., 2024).

3.2.3. Transparency

Transparency plays a crucial role in the design of film packaging, as it affects both light protection and the visual appeal of the food inside. Typically, food packaging films that are transparent have an opacity measurement below five (Tristanto et al., 2024). ANOVA (extraction method and pectin concentration as fixed factors) was also conducted on the transparency of films. The extraction methods ($F = 10.906$, $p = 0.006$) and pectin concentrations ($F = 4.387$, $p = 0.028$) revealed a significant effect on the transparency, while their interaction showed no significant effect on the transparency ($p > 0.05$). The pectin films in this research exhibit transparency ranging from 1.65 ± 0.57 for PF-AO to 2.52 ± 0.73 for PF-MAO, demonstrating relatively good transparency. The findings indicate that microwave radiation may have contributed to a reduction in the molecular weight and disruption of the pectin structure, resulting in a significant increase in the transparency value of the film. Moreover, the higher concentration of pectin used in this study results in an increased transparency of the film because pectin blocked light from passing through the film. This transparency aligns with the previous study for Citroen peel pectin (Asfaw et al., 2023). In film formulation, higher pectin concentration tends to compact the film network reduces scattering.

3.2.4. Moisture content and water solubility

In film packaging, one commonly assessed parameter is the moisture content and water solubility, which are crucial for determining the packaging's suitability for products with varying moisture levels. To evaluate the impact of extraction methods and pectin concentrations on the films' moisture content and water solubility, we conducted two-way ANOVAs, using extraction method and pectin concentrations as fixed factors. The results showed that all treatments showed no significant effect on the moisture content of the film. The moisture content of films were $19.22 \pm 5.24\%$ for PF-AO and $18.48 \pm 4.97\%$ for PF-MAO. For water solubility, the results revealed that extraction methods showed a significant effect, whereas pectin concentration and its interaction were not significant. The water solubility of films prepared with PF-AO was higher ($84.77 \pm 6.56\%$) compared to those prepared with PF-MAO ($59.84 \pm 9.54\%$), as shown in Fig. 4c. The water solubility in this study is comparable with the pectin film from dragon fruit peel (*Hylocereus polyrhizus*) (Tristanto et al., 2024) and pectin film from the byproducts of *Hibiscus sabdariffa* L. (Estrada-Girón et al., 2020). The findings are consistent with moisture content, indicating that microwave irradiation could promote stronger pectin-gelatin complexation, creating an insoluble surface film, which effectively lowers the water solubility of the film. In other words, films prepared with PF-AO could exhibit more fragmentation and porous characteristics, leading to higher water solubility.

3.2.5. Water vapor transmission rate (WVTR)

WVTR is utilized to assess the rate at which water vapor permeates a packaging film under specific environmental conditions. Lower WVTR values signify a more effective moisture barrier. To explore the impact of the extraction method and pectin concentrations on the WVTR of film, we conducted ANOVA on WVTR. The results showed that extraction methods significantly influenced the WVTR of the film ($F = 7.384$, $p = 0.001$). However, pectin concentrations and the interaction had no changes in the WVTR of films ($p > 0.05$). The WVTR value of the films measured decreased from 2.99 ± 0.19 g m^{-2} day^{-1} for PF-AO to 2.76 ± 0.14 g m^{-2} day^{-1} for PF-MAO, as shown in Fig. 4d PF-MAO demonstrated the most effective water vapor transmission rate, as evidenced by the lowest WVTR value (2.76 ± 0.14 g m^{-2} day^{-1}). The findings indicate that the reduced water vapor transmission rates are linked to microwave-induced alterations in pectin structure, along with the presence of gelatin. These factors likely contributed to the development of a more compact and cohesive polymer matrix. This effect is associated with microwave-assisted extraction, which yields pectin with a lower equivalent weight and degree of esterification, thereby promoting stronger ionic crosslinking. As a result, a more compact and well-ordered network is formed, contributing to decreased water vapor permeability (Çavdaroğlu et al., 2023; Nguyen et al., 2024).

3.2.6. Tensile strength

Measuring the tensile strength of a film determines the highest load it can withstand before breaking, which is a commonly assessed characteristic of materials used in packaging applications. In this study, mechanical characterization was limited to tensile strength without measuring the stretchability/flexibility of the films. Two-way ANOVA (extraction method and pectin concentration as fixed factors) was also conducted on the tensile strength of films. The extraction methods revealed a significant effect on tensile strength, while pectin concentrations and their interaction showed no significant effect on tensile strength. Films extracted with microwave showed the highest tensile strength (1.08 ± 0.33 MPa) compared to those extracted without microwave (0.602 ± 0.18 MPa), as shown in Fig. 4e. The effects arise mainly through changes in the degree of esterification as observed also in this study. In the presence of gelatin, microwave-assisted extraction may have enhanced the compatibility between the extracted pectin and the gelatin matrix, promoting the formation of a denser composite network that could contribute to the higher tensile strength observed. This was achieved through improved interfacial adhesion between the two polymer matrices, thanks to the microwave's ability to provide uniform heating (Singh & Zafar., 2019; Ullah et al., 2021). Further experiments are required to test this hypothesis. The obtained values in this study were still higher than those regenerated from dragon fruit peel pectin + potato starch (0.05–0.14 MPa) (Tien et al., 2023) but still lower than those reported from banana peel (2.36 MPa) (Marichelvam et al., 2025). Therefore, the developed films could be more suitable for applications such as coatings or biodegradable protective layers, where moderate mechanical strength is acceptable.

3.2.7. FTIR

FTIR is utilized to analyze the internal composition of pectin molecules and to detect the chemical bonds present within them. The comparison of FTIR spectra for PF-AO and PF-MAO, depicted in Fig. 5, highlighted the importance of the extraction method in this research.

The FTIR spectrum in Fig. 5 displays characteristic absorption bands of pectin polysaccharides, including the O–H stretching vibration in the range of 3733–3298 cm^{-1}, the C═O stretching vibration near 1646 cm^{-1}, and the C–O and C–O–C vibrations around 1024 cm^{-1}, which are linked to glycosidic bonds in the pectin backbone. The presence of these bands suggests that the core structure of pectin remained intact following microwave treatment. Variations in the intensity and sharpness of the absorption bands were noted between the PF-AO and PF-MAO samples. In the O–H region (3733–3298 cm^{-1}), the PF-MAO samples exhibited changes in intensity compared to PF-AO. Alterations were also observed in the band around 1646 cm^{-1}, associated with the carboxylate (C═O) groups of galacturonic acid units. In the microwave-treated samples (PF-MAO), this band showed changes in intensity. With the addition of gelatin, the strong intense peak of methylated carboxyl groups of pectin was still observed in all microwave-treated samples (PF-MAO), which are explicitly linked to hydrogen-bond interactions between pectin and gelatin. In addition, amide I around 1646 cm^{-1} showed strong intermolecular interaction between gelatin's protein groups and pectin's carboxyl groups, which is induced by microwave extraction. The bands at 1322 cm^{-1} and 1024 cm^{-1}, corresponding to C–H bending and C–O–C stretching vibrations, respectively, displayed slight intensity changes in the PF-MAO samples. The FTIR analysis indicates that microwave treatment does not modify the essential chemical functional groups that characterize the pectin structure, although it does affect band intensity and intermolecular interactions. These observations are consistent with earlier research, which found no alteration in the fundamental chemical structure of pomelo peel pectin obtained through acid extraction, acid–ultrasound extraction, alkaline extraction, and alkaline–ultrasound extraction (Lin et al., 2026). Similarly, the extraction method does not change the basic chemical structure of pectin derived from hot acid and ultrasound-microwave assistance (Chandrasekar et al., 2026), and there is no significant difference in pectin extracted via hot acid extraction, microwave-assisted extraction, ultrasound-assisted extraction, and enzymatic-assisted extraction (Yu et al., 2024). The findings suggest that microwave-assisted extraction potentially yielded pectin with molecular properties conducive to enhanced interactions with gelatin, thereby forming a more cohesive and compatible film matrix.

3.2.8. Principal component analysis (PCA)

PCA was conducted to assess the connection between film formulations and their physicochemical characteristics. The first two principal components accounted for 76.22% of the total variance, with PC1 contributing 52.87% and PC2 contributing 23.35%, as shown in Fig. 6.

In the individual map, the score plot showed a distinct separation between PF-AO and PF-MAO along PC1, indicating that the extraction methods used significantly impacted the film properties. Films PF-AO 3%, PF-AO 4%, and PF-AO 5% were clustered in the positive PC1 region, primarily associated with physicochemical properties, contrasting with films PF-MAO 3%, PF-MAO 4%, and PF-MAO 5%, which were positioned in the negative PC1 region, highlighting different properties compared to PF-AO films. In the variable map, WVTR and water solubility were positively correlated and aligned with the positive PC1 axis, suggesting that PF-AO films exhibit higher hydrophilicity and permeability. This implies that ammonium oxalate extraction might enhance the film matrix's affinity for water molecules, thereby increasing water solubility and vapor transmission. On the other hand, the transparency value was aligned with the negative PC1 region, indicating a stronger association with PF-MAO films. This suggests that microwave energy might foster stronger intermolecular interactions within the polymer matrix, leading to enhanced mechanical strength. Along PC2, the sample distribution reflects the impact of pectin concentration. PF-MAO 5% was located in the positive PC2 region, suggesting that higher pectin concentrations might enhance specific structural interactions within the film matrix, thereby increasing thickness. Conversely, PF-MAO 3% appeared in the negative PC2 region and was linked to water content, indicating that lower pectin concentrations might result in films with higher moisture content. These findings suggest that the physicochemical properties of the films are strongly influenced by the extraction method and polymer concentration, ultimately determining the balance between barrier, mechanical, and optical properties needed for food packaging applications.

4. Conclusion

To conclude, we showed that microwave-assisted ammonium oxalate extraction and pectin concentration significantly influenced the properties of films prepared using pectin extracted from dried *Dillenia serrata* peel powder. Films prepared from lower pectin concentration (3%) showed better thickness compared to higher pectin concentrations. Additionally, films prepared using the microwave-assisted ammonium

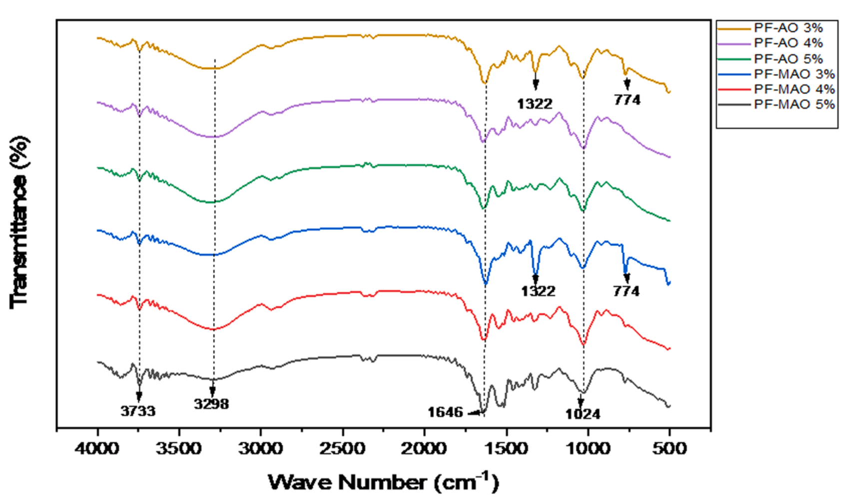

Fig. 5. FT-IR spectra of pectin extracted using different extraction methods and pectin concentrations.

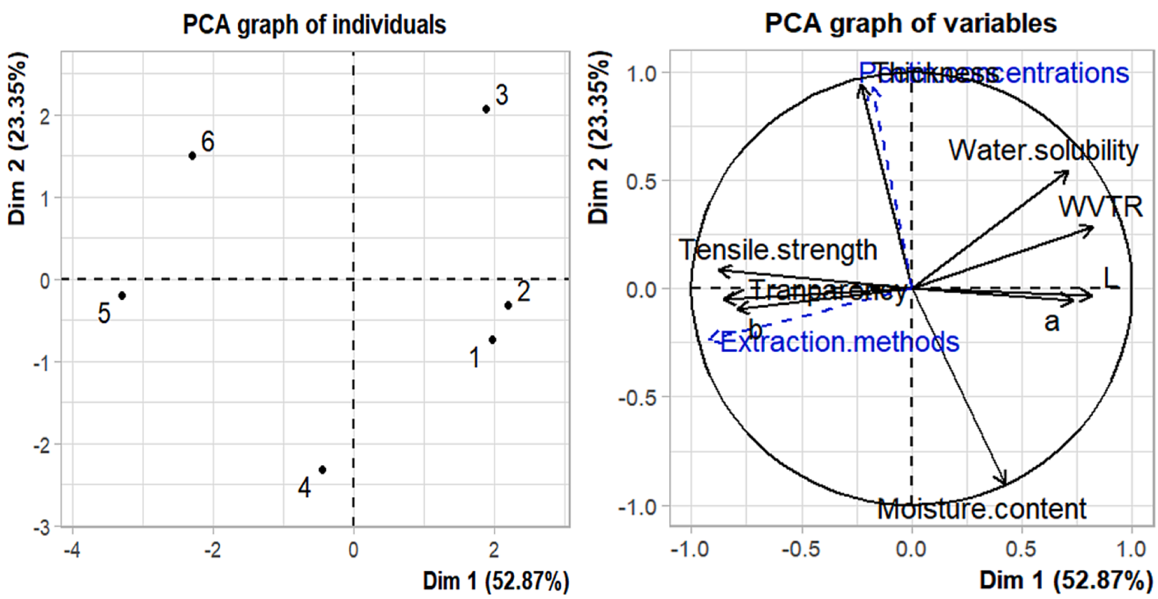

Fig. 6. The biplot visualization is derived from the first two dimensions of a PCA conducted on the physical, mechanical, and barrier properties data collected for the six films. The films themselves served as the individuals, while the variables included thickness, color (*L, a,* and *b*), tensile strength, transparency, moisture content, water solubility, WVTR, and tensile strength characteristics that were observed. 1 = PF-AO 3%, 2 = PF-AO 4%, 3 = PF-AO 5%, 4 = PF-MAO 3%, 5 = PF-MAO 4%, 6 = PF-MAO 5%.

oxalate (PF-MAO) method exhibited lower water solubility and a lower water vapor transmission rate and showed greater tensile strength. A limitation of this study is that films were prepared only from dried peel powder-derived pectin. Further studies should investigate the film-forming performance of puree-pectin to better understand the role of structure on the physical, mechanical, and optical properties of film. Moreover, internal structure, surface morphology, or thermal analysis study would help to evaluate the role of microwave extraction.

CRediT authorship contribution statement

Adiansyah Syarifuddin: Writing – original draft, Validation, Supervision, Conceptualization. **Ahmad Azhari Arif:** Writing – original draft, Methodology, Investigation. **Serli Hatul Hidayat:** Writing – review & editing, Data curation. **Zainal:** Writing – review & editing, Validation. **Andi Dirpan:** Writing – review & editing, Data curation. **Intan Syafinaz Mohamed Amin Tawakkal:** Supervision, Formal analysis.

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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Data availability

Data will be made available on request.

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