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Valorization of Spanish lime (*Melicoccus bijugatus*) shell waste: optimized pectin recovery and preliminary evaluation in edible film formation

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Valorizing agro-industrial by-products into high-value materials aligns with the growing emphasis on circular bioeconomy strategies. In this study, shell waste from Spanish lime (*Melicoccus bijugatus*) was investigated as a novel source of pectin, with subsequent development of an edible film as a potential sustainable packaging application. Response surface methodology was used to optimize the yield and viscosity-average molecular weight of the pectin obtained through hot acid extraction. The pectin was characterized for its physico-chemical and techno-functional properties and then used to develop an edible film. Optimal conditions for maximizing pectin yield (16.37%) were 180 minutes extraction time, a solid–liquid ratio of 1 : 50, and a solvent pH of 1.5 while maximum molecular weight (37 445.18 Da) was achieved at 60 minutes extraction time, a solid–liquid ratio of 1 : 30, and a solvent pH of 3.5. The extracted pectin had an equivalent weight of 1744.66 g mol^{-1} and was classified as high methoxyl pectin, with a degree of esterification of 80.4%. Notably, the pectin exhibited significantly higher antioxidant activity than commercial citrus pectin, with a 20% greater DPPH radical scavenging capacity and a 6.5-fold higher FRAP value. Preliminary testing of the film made from the extracted pectin showed that the film had a DPPH scavenging rate of 25.6%, a FRAP value of 224.65 and had significantly lower oxygen permeability than both PET and HDPE controls. These results demonstrate that the *Melicoccus bijugatus* shell wastes are a promising source of antioxidant-rich pectin suitable for edible film applications.

Sustainability spotlight

This study contributes to sustainable food systems by converting *Melicoccus bijugatus* (Spanish lime) shell waste, an underutilized by-product in the Caribbean and Latin America, into a high-value biopolymer with multifunctional applications. By optimizing an eco-friendly extraction process using citric acid, the research promotes resource efficiency and waste valorization, directly supporting SDG 12 (Responsible Consumption and Production). Furthermore, the development of biodegradable pectin-based films as alternatives to petroleum-derived plastics further aligns with SDG 13 (Climate Action), offering a scalable solution to reduce environmental pollution and support the transition toward a circular bioeconomy.

Introduction

Food waste remains a critical global challenge, with the Food and Agriculture Organisation (FAO) estimating that approximately 1.3 billion tonnes of food are lost annually accounting for economic losses of around €800 billion.[1] The fruit and vegetable sector contributes the most to this waste, accounting for 25–30%, primarily through by-products such as peels, rinds and cores.[2] Re-purposing and valorization of these residues offers a compelling opportunity to reduce economic and environmental burdens while also supporting the move towards a more sustainable and circular food system. The valuable biological compounds found in these wastes, which include biopolymers such as proteins, lipids, cellulose and other polysaccharides, can be processed into value-added products such as biofuels, fertilizers, food additives and bioactive compounds which have found use across multiple sectors.[3–5]

Among the various biopolymers recovered from fruit and vegetable waste, pectin is particularly valued for its functional versatility. Found in the middle lamella and cell walls of plants, pectin has been traditionally used in food systems for its gelling, stabilizing, and emulsifying properties.[6] More recently however, its film forming capacity, biodegradability and renewable origin have made it a biopolymer of substantial prominence in sustainable food packaging applications.[6–8] Pectin can be used to produce biodegradable films with effective barrier properties against moisture, gases, oils, and odors, and has also been incorporated with bioactive compounds to further enhance the shelf life of packaged foods.[9,10] Despite this growing interest in the polymer, over 99% of commercial pectin is still derived from citrus fruits (85%), apples (14%) and sugar

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beets (0.5%).[11] This narrow pectin source poses a sustainability concern, especially as the global demand for pectin, driven in part by its emergent role in sustainable food packaging, is expected to grow at a compound annual rate of 5.1%.[7] In response to this increasing demand and to reduce the dependency on conventional sources, recent research has focused on the evaluation of pectin from alternative sources.[12]

In keeping with this, one such alternative source that warrants evaluation is the Spanish lime fruit (*Melicoccus bijugatus*). Commonly known as chennet in the Caribbean as well as guinep or mamoncillo across Latin America and Mexico, *Melicoccus bijugatus* is an ovoid drupe characterized by a thick, leathery shell surrounding a juicy, peach-colored pulp and seed.[13] The edible pulp is noted for its therapeutic potential in managing cardiovascular conditions, respiratory ailments and gastrointestinal disorders.[14–16] However, despite these medicinal attributes, the outer shell and seed which account for approximately 65% of the total fruit mass, are typically discarded as waste.[17] While Moo-Huchin *et al.*[18] have explored the valorization of the seeds for their starch content and potential in seed flour development, comparable investigations into the shell remain absent. This gap highlights the underexplored potential of the *Melicoccus bijugatus* shells for upcycling into value-added products, particularly as a novel source of pectin for high-value applications.

This study therefore aimed to quantify the pectin content of the *Melicoccus bijugatus* shell waste for the first time, optimize its extraction conditions, and characterize the resulting pectin's key physicochemical and functional properties, including its suitability for edible film formation.

Materials and methods

Chemicals

All chemical reagents used in the study were of analytical grade and sourced from Sigma Aldrich (St. Louis, Missouri, USA).

Preparation of plant material

Freshly harvested fruits were obtained from a local farmer situated in the southern part of Trinidad. The fruits were first washed and sorted for uniformity and defects, after which the seeds and pulp were removed from the shells. The fresh shells were washed to remove any remnants of fruit pulp and then placed in a cabinet dryer (LTE Scientific, Oldham, UK) for 24 h at 55 °C. The dried shells were then ground into a fine powder using a Staub Grinding Mill (Model 4E, PA, USA) and stored in vacuum-sealed plastic pouches (35 cm × 25 cm) until further use.

Optimization of pectin extraction

A three factor Box–Behnken Design (BBD) was used for the optimization of the pectin extraction process. The three independent variables investigated were solvent pH (X_1: 1.5–3.5), extraction time (X_2: 60–180 min) and solid–liquid ratio (X_3: 1 : 30–1 : 50) each at three levels as shown in Table 1. The responding variables were both the pectin yield (%) as well as the viscosity-average molecular weight (M_v) of the extracted pectin. Minitab v. 21.4.10 (Minitab Software, State College, PA, USA) was used to design the BBD and analyse the data obtained. The software and the derived regression models were used to optimize the extraction conditions for yield and molecular weight individually, as well as to perform a simultaneous multi-response optimization that balanced both responses equally. Validation extractions were subsequently conducted under the predicted optimal conditions to verify model accuracy. All physicochemical and functional characterizations were conducted using pectin extracted under yield-optimized conditions to reflect practical relevance for industrial scalability.

Pectin extraction

Pectin extraction was performed using the conventional hot acid method according to Duwee *et al.* with minor modifications.[19] For each experimental run outlined by the Box–Behnken design in Table 1, 5 g of shell powder was added to the extraction solvent (distilled water acidified with citric acid). The solid-to-liquid ratio and pH of the extraction solvent used were adjusted based on the conditions specified for each run by the BBD. The mixture was heated to 80 °C for the specified extraction time and then vacuum filtered to separate the plant material from the pectin extract solution. A double volume of 95% ethanol was then introduced to the solution to precipitate the pectin for 24 h. The mixture was then centrifuged at 16 500×*g* for 15 min to obtain the precipitated pectin, which was then washed with 70% ethanol and left to dry in a vacuum oven at 40 °C for 24 h. The dried chennet shell pectin (CSP) was then milled into a fine powder and stored in vacuum sealed bags until further analysis. The pectin yield was calculated according to eqn (1).

$$\text{Yield}(\%) = \frac{\text{Mass of extracted pectin}}{\text{Mass of shell powder}} \times 100 \quad (1)$$

Physicochemical characterisation of pectin

Viscosity-average molecular weight and equivalent weight. The viscosity-average molecular weight of the extracted pectin was determined through intrinsic viscosity measurements of the pectin solution using an Ubbelohde capillary viscometer as described by Xu *et al.*[20] A 1 mg per mL pectin solution was prepared using 0.1 M NaCl solvent after which approximately 15 mL was used to fill a 0B Cannon Ubbelohde Viscometer (PA, USA). The viscometer was then placed in a Cannon CT 600 Constant Temperature Bath (PA, USA) to equilibrate to 25 °C before measuring the efflux times of the pectin solution. The intrinsic viscosity was then determined using eqn (2)–(4) and the Mark–Houwink eqn (5) was used to calculate the viscosity-average molecular weight. The equivalent weight was determined according to the method outlined by Ebrahim *et al.*[21]

$$[\eta] = [\sqrt{2}(\eta_{sp} - \ln \eta_r)]/c \quad (2)$$

$$\eta_r = t_s/t_o \quad (3)$$

Table 1 Box–Behnken design for the optimization of yield (%) and molecular weight (M_v) of CSP

	Extraction parameters			Responding variables			
Run number	X_1: pH	X_2: time (min)	X_3: solid–liquid ratio (g mL^{-1})	Experimental yield (%)	Predicted yield (%)	Experimental M_v (Da)	Predicted M_v (Da)
1	1.5	60	40	10.46	10.77	13 892.60	13 980.30
2	1.5	180	40	14.40	14.81	4780.50	4870.30
3	3.5	120	30	3.56	4.03	34 908.90	34 414.80
4	2.5	60	30	2.58	2.49	26 854.10	27 433.80
5	1.5	120	50	15.06	14.56	10 160.00	10 615.80
6	2.5	120	40	4.18	4.15	23 501.70	23 319.30
7	3.5	120	50	3.40	3.63	33 058.20	33 733.80
8	2.5	120	40	4.38	4.15	23 423.70	23 319.30
9	1.5	120	30	13.26	13.00	9054.50	8340.80
10	2.5	180	30	4.64	4.47	19 930.50	20 501.80
11	3.5	180	40	3.38	3.05	32 276.80	32 150.30
12	3.5	60	40	3.06	2.63	36 020.60	35 892.30
13	2.5	60	50	2.68	2.82	28 335.80	27 724.80
14	2.5	180	50	5.24	5.30	22 423.60	21 804.80
15	2.5	120	40	3.94	4.15	23 089.70	23 319.30
Optimized conditions							
Max. yield	1.5	180	50	16.37 ± 0.39	16.02	—	—
Max. M_v	3.5	60	30	—	—	37 445.18 ± 741.05	37 047.5
Co-optimization	1.5	60	50	12.03 ± 0.23	11.75	15 816.67 ± 500.83	15 439.2

$$\eta_{sp} = \eta_r - 1 \quad (4)$$

$$[\eta] = kM_v^{\alpha} \quad (5)$$

where: $[\eta]$ is the intrinsic viscosity, η_{sp} is the incremental viscosity, η_r is the relative viscosity, c is the concentration of the pectin solution, t_s is the efflux time of the solvent and t_o is the efflux time of the pectin solution. M_v is the viscosity-average molecular weight and k (2.34×10^{-5}), and α (0.8224) are constants.[22]

Determination of galacturonic acid content. The galacturonic acid content (GalA%) of the extracted pectin was determined according to the method outlined by Blumenkrantz and Asboe-Hansen[23] with minor modifications. Briefly, 1 mL of a prepared pectin solution (0.2 g mL^{-1}) was mixed with 6 mL of 0.0125 M sodium tetraborate solution (in conc. H_2SO_4) in an ice bath. The mixture was then heated in a boiling water bath for 5 min and then cooled in an ice bath to room temperature. Following this, 0.1 mL of 3-phenylphenol in 0.5% NaOH was added and stirred until a pink colour was observed. The absorbance was read at 520 nm and a calibration curve using standard galacturonic acid solutions (0–250 μg mL^{-1}) was used to determine the GalA% in the sample.

Proximate analysis. The moisture content (%), and ash content (%) of the extracted pectin were conducted according to the AOAC methods (Association of Official Analytical Chemists).[24] The protein content (%) was determined by the Lowry method and the carbohydrate content (%) was determined using the phenol-sulfuric acid method using D-glucose as the reference standard.[25]

Morphology and FT-IR analysis of pectin

Scanning electron microscopy. The morphological analysis of the extracted pectin was conducted based on the method by Jong *et al.*[26] The pectin powder was mounted onto circular metal sample holders covered with double sided carbon tape. The pectin powders were then sputter coated with gold and the SEM images were taken using a scanning electron microscope (SEM 515, Philips, Holland) under an accelerating voltage of 20 kV and a magnification of 400×.

FT-IR spectra and degree of esterification. Fourier transform infrared (FTIR) spectroscopy of the extracted pectin was determined using a Shimadzu IRAffinity-1S spectrophotometer with a wavenumber range of 450–4000 cm^{-1} at a resolution of 4 cm^{-1}. The spectrum obtained was also used to determine the degree of esterification (DE) of the pectin, which was calculated using eqn (6).[26] The peak areas for the regions of interest (esterified carboxyl groups = 1730–1700 cm^{-1} and non-esterified carboxyl groups = 1630–1600 cm^{-1}) in the spectrum were determined using OriginLab 2023 graphing and analysis software.

$$\text{Degree of esterification}(\%) = \frac{\text{Peak area of esterified carboxyl group}}{\text{Peak area of esterified carboxyl group} + \text{Peak area of unesterified carboxyl group}} \quad (6)$$

Antioxidant properties

DPPH analysis. The antioxidant properties of the extracted pectin were determined by the DPPH assay method as described by Wang *et al.*[27] A 1 mL volume of a prepared DPPH solution (0.1 mM) was mixed with the same volume of pectin solutions at various concentrations (0.4–2.0 g L^{-1}). The mixtures were then incubated at 30 °C for 30 min in the dark after which the absorbance of each at 517 nm was measured. The DPPH radical scavenging rate was determined using eqn (7).

$$\text{DPPH scavenging rate}(\%) = \left[1 - \frac{A_0 - A_1}{A_2}\right] \times 100 \quad (7)$$

where: A_0 is the absorbance of the pectin/DPPH solution, A_1 is the absorbance of the pectin alone and A_2 is the absorbance of the DPPH alone.

FRAP analysis. The reducing power of the extracted pectin was determined using the Ferric Reducing Power Assay (FRAP) as similarly outlined by Kumar *et al.*[28] The FRAP reagent was freshly made by adding 300 mM acetate buffer of pH 3.6 (adjusted by using NaOH and HCl), 10 mM 2,4,6-tri(2-pyridyl)-*s*-trizine in 40 mM HCl and 20 mM $FeCl_3$ in a 10 : 1 : 1 ratio. A 3 mL volume of the FRAP reagent was added into 1 mL of pectin sample (1 mg mL^{-1}) and kept at 37 °C for 30 min. The absorbance at 593 nm was taken (Genesys 10S UV-VIS spectrophotometer, Thermo Scientific) and the reducing power was determined using the regression equation from the calibration curve obtained from prepared ferric(II) sulphate heptahydrate standards.

Functional characteristics

Water/oil holding capacity. The water holding capacity (WHC) and oil holding capacity (OHC) of the extracted pectin was determined according to the method outlined by El Fihry *et al.*[29] with minor modifications. Vegetable oil or distilled water (10 mL) was mixed with 0.2 g of extracted pectin and then vortexed for 1 min. Following this, the mixtures were centrifuged at 3000×*g* for 30 min and the remaining volume of oil or water was measured and recorded. Experiments were conducted in triplicate and reported as grams of water or oil retained per gram of pectin.

Emulsion properties. The emulsion capacity (EC) and stability (ES) of the extracted pectin were determined by the method outlined by Muñoz-Almagro *et al.*[30] with minor modifications. A 10 mL of a 1% pectin solution was mixed with 5 mL of vegetable oil and thoroughly homogenized for 3 min after which, the emulsions formed were centrifuged at 3000×*g* for 5 min. The EC% was determined by eqn (8).

$$\text{EC}\ (\%) = V_s/V_e \times 100 \quad (8)$$

where: V_s is the total volume of the system and V_e is the volume of the emulsion phase.

The emulsion was then incubated at 80 °C in a water bath and then centrifuged again at 3000×*g* for 5 min. The ES% was determined by eqn (9).

$$\text{ES}\ (\%) = F_e/I_e \times 100 \quad (9)$$

where: I_e is the initial volume of the emulsified layer and F_e is the volume of the final emulsified phase after incubation and centrifugation.

Film application

Optimization of pectin for film formation. For the application of the extracted CSP in preliminary film formation, a separate multi-response optimization was performed using Derringer's desirability function in which molecular weight was weighted twice as much as yield.[31] This was done to reflect the critical role of molecular weight in determining the film-forming properties of pectin. Edible films were subsequently prepared using CSP extracted under these optimized conditions to prioritize functionality in the film formation process.

Film formation. The pectin-based film was prepared according to the method outlined by Sood and Saini[32] with minor modifications. Briefly, a 5% pectin solution was prepared by mixing 2 g of pectin in 40 mL of distilled water and stirring for 2 h at 800 rpm. Glycerol was then added (30% v/w of pectin) and the mixture stirred for a further 30 min prior to degassing. The film forming solution (FFS) was then casted onto poly methyl methacrylate plates using a film application machine (Elcometer 4340 Automatic Film Applicator, USA) and an Elcometer 3580 casting knife at a height of 2 mm. Films were left to dry overnight at 30 °C and then conditioned at 50% RH for three days prior to testing.

Mechanical properties. A universal testing machine (Tinius Olsen H25KT, Tinius Olsen Ltd UK) was used for preliminary analysis of the mechanical properties of the film in accordance with the ASTM D882-18 standard.[33] Rectangular film samples (150 mm × 20 mm) were prepared and conditioned at 25 °C and 50% RH for 48 h prior to analysis. The samples were then loaded into the machine with an initial grip separation of 100 mm and then stretched at a cross-head speed of 10 mm min^{-1} until rupture. The tensile strength and elongation at break values were determined using eqn (10) and (11) respectively.

$$\text{Tensile strength}(\text{MPa}) = \frac{\text{Peak force}(\text{N})}{\text{Cross sectional area of film}(\text{m}^2)} \quad (10)$$

$$\text{Elongation at break}(\%) = \frac{\text{Film length after stretching} - \text{initial film length}}{\text{Original film length}} \times 100 \quad (11)$$

Oxygen permeability. The oxygen permeability of the prepared film was determined in accordance with the ASTM D3985-02 standard (2004).[34] A 6 cm diameter portion of the pectin film was prepared and loaded into an oxygen permeation cell (PreSens, Germany) and sealed with vacuum grease. Nitrogen gas was used to purge the lower and upper chambers of the cell before loading the bottom chamber with ultrapure oxygen gas. The amount of oxygen that permeated the film was recorded and the oxygen permeability (OP) was calculated using eqn (12).

$$\mathrm{OP}\left(\mathrm{cm^3\ \mu m\ per\ m^2\ per\ day\ per\ bar}\right) = \left(\frac{P_{O_2} \times V \times l}{\text{Standard pressure} \times A \times \Delta P_{O_2}}\right) \times \left(\frac{\text{Standard temperature}}{\text{Measured temperature}}\right) \quad (12)$$

where: P_{O_2} is the increase of oxygen partial pressure in the upper chamber (hPa per day), V is the volume of the upper chamber (116 cm^3), l is the thickness of the film, the standard pressure is taken as 1013 hPa, A is the permeation area of the film (0.0068 m^2), ΔP_{O_2} is the change in oxygen partial pressure between the upper and lower chambers (0.96 bar) and standard temperature is taken as 273 K.

Water vapor permeability. The water vapour permeability was determined in accordance with the E96/E96M-10 standard.[35] Test cups (68-3000 EZ-Cup Vapometer Cup, Thwing-Albert Instrument Company USA) were filled with 20 mL of water and films were placed to the top of the cups and sealed. The initial mass of the cup was taken before being placed in a desiccator (0% RH) for 5 h. The mass of the cup was then taken in 30 min intervals and used to create a graph of the mass change of water against time. The slope of the graph (S_L) was calculated and eqn (13)–(15) were then used to determine the water vapor permeability.

$$\text{Water vapor transmission} = \frac{S_L}{A} \quad (13)$$

$$\text{Permeance} = \frac{\text{Water vapor tramsnission}}{S(R_1 - R_2)} \quad (14)$$

$$\text{Water vapor permeability} = \text{Permeance} \times \text{average thickness} \quad (15)$$

where: S_L is the slope of the water mass change in the cup *vs.* time graph and A is the test area of the cup (0.0032 m^2), S denotes the saturation vapor pressure at the test temperature of 22 °C (20.941 mmHg), R_1 represents the relative humidity at the vapor source, (100%), while R_2 denotes the relative humidity at the vapor sink, (0%).

Antioxidant properties. Film extracts were prepared by completely dissolving the films in distilled water. These extracts were then analysed for antioxidant properties using the DPPH method and FRAP assay in accordance with the methods outlined by Chaichi *et al.*[36] and Homthawornchoo *et al.*[37] respectively.

Statistical analysis

All experiments were conducted in triplicate and the results were presented as average value ± standard deviation (SD). Minitab (version 21.4.1) was used to configure and analyse the Box–Behnken design. Significant differences were determined using an ANOVA test followed by Tukey's multiple range test, with a significant level set at ($p < 0.05$).

Results and discussion

Model evaluation and statistical analysis

A total of 15 pectin extraction runs were conducted using a Box–Behnken design to investigate the effect of the extraction parameters on both the pectin yield and viscosity-average molecular weight (M_v) from *Melicoccus bijugatus* shells. Table 1 shows the observed and predicted values for both responses based on the developed regression models. Both quadratic regression models were statistically significant, with p-values < 0.05 and high F-values of 121.96 for yield and 246.94 for M_v, indicating strong model fit. The lack-of-fit was also not significant for the yield model (p = 0.11) nor the M_v model (p = 0.05), suggesting acceptable model adequacy.

Furthermore, for the yield model, the coefficient of determination (R^2 = 0.99) and predicted R^2 (0.93) demonstrated a strong correlation between the independent variables and the response, as well as high predictive reliability. Similarly, the M_v model showed excellent fit and predictive power, with R^2 = 0.99 and predicted R^2 = 0.96. The close agreement between experimental and predicted values (Table 1) further supports the validity of both models. The developed regression models are shown in the following eqn (16) and (17).

$$\text{Yield (\%)} = 34.10 - 22.94X_1 + 0.0938X_2 - 0.119X_3 + 4.347{X_1}^2 - 0.00019{X_2}^2 + 0.00307{X_3}^2 - 0.01508X_1X_2 - 0.0490X_1X_3 + 0.000208X_2X_3 \quad (16)$$

$$M_v\ \text{(Da)} = -3543 + 23035X_1 - 159.5X_2 - 266X_3 - 2093{X_1}^2 + 0.138{X_2}^2 + 5.50{X_3}^2 + 22.37X_1X_2 - 73.9X_1X_3 + 0.421X_2X_3 \quad (17)$$

where X_1 = pH, X_2 = time and X_3 = S/L ratio.

Optimization of pectin yield and M_v and model validation

The optimum conditions for maximizing pectin yield and viscosity-average molecular weight (M_v) were determined using Minitab v.21.4.10 based on the regression models (eqn (16) and (17)). For a predicted maximum yield of 16.02%, the optimal conditions were pH 1.5, 180 min extraction time, and a solid–liquid ratio of 1 : 50. In contrast, for the highest predicted M_v (37 047.5 Da) the optimal conditions were pH 3.5, 60 min extraction time, and a solid–liquid ratio of 1 : 30. Validation experiments conducted in triplicate under these respective conditions yielded a pectin recovery of 16.37% and an M_v of 36 924.51 Da, with prediction errors of 2.14% and 0.33%, respectively. Given that the shell waste constitutes approximately 48.6% of the total fruit waste, this validated optimized yield of 16.37% corresponds to approximately 79.5 g of pectin recovered per kilogram of fresh fruit.[38] For simultaneous optimization of both responses, the multi-response analysis identified the co-optimal conditions as pH 1.5, 60 min extraction time and a solid–liquid ratio of 1 : 50. Under these parameters, the predicted values were a yield of 11.75% and an M_v of 15 439.2 Da. Validation extractions resulted in yields of 12.03% and 15 816.7 Da, with corresponding prediction errors of 2.33% and 2.39% respectively. As all prediction errors were below 10%, the models demonstrated strong predictive accuracy and reliability for optimizing the extraction of pectin from the *Melicoccus bijugatus* shells.[26]

Physicochemical properties

Galacturonic acid (GalA%) and equivalent weight. The galacturonic content of pectin is a key indicator of its purity and functional capabilities.[39] Higher GalA% content indicates a higher level of pectin purity and unbranched homogalacturonan regions which enhance the gelling, thickening and stabilizing capacity of the pectin polymer.[40] According to the Food and Agriculture Organization (FAO), a minimum GalA% content of 65% is required for commercial-grade pectin to ensure adequate purity and structural integrity for industrial use.[41] In this study, the GalA% content of the extracted CSP was 77.80%, which surpassed the FAO standard, and thereby suggested minimal contamination and preservation of the native CSP structure, which supports its potential for various functional applications.

As equivalent weight indicates the mass of pectin that contains one mole of free carboxyl groups, it also serves as an indicator of the degree of esterification of the polymer. A high equivalent weight indicates a lower proportion of un-esterified galacturonic acid units which is characteristic of high-methoxyl pectins. Conversely, low methoxyl pectin, which contain more free carboxyl groups, typically exhibit lower equivalent weights.[42] Moreover, according to the International Pectin Producers Association (IPPA), equivalent weights that fall within the 600–800 g mol^{-1} range reflect pectin with high gel-forming capabilities.[43] As seen in Table 2, the CSP was shown to have a very high value of 1744.66 g mol^{-1}, which was more than twice the upper threshold suggested by the IPPA. This value was determined to be much higher than reported values for other emerging pectin sources such as pineapple peel pectin (803.30 g mol^{-1}) and watermelon rind pectin (983.90 g mol^{-1}).[41,44] This elevated equivalent weight would have typically been attributed to the presence of a large amount of non-pectin co-extractants (*e.g.* proteins, polyphenols and other macromolecules). These compounds would have interfered with the titrimetric determination of the equivalent weight by contributing to sample mass without adding titratable carboxyl groups, thereby resulting in an overestimation of the value. However, the high GalA% (77.80%) of the CSP indicates minimal contaminants from such impurities and therefore suggests that it possesses a structurally distinctive pectin framework compared to other fruit waste pectin. This high equivalent weight of the extracted CSP also suggested that it was a high methoxyl pectin and highlighted its potential for use in high-quality gel formation within food and pharmaceutical formulations.

Proximate composition. The moisture content (MC) of the extracted CSP was determined to be 15%, which slightly exceeded the 12% maximum recommended by the International Pectin Producers Association (IPPA) for commercial pectin.[42] Moisture content is a critical factor in determining shelf stability, as elevated levels can promote microbial growth and enzymatic degradation. Regarding pectin, high moisture levels may facilitate pectinase activity, leading to structural breakdown and reduced quality over time. The higher MC observed in this study is likely attributed to insufficient drying during the post-extraction process, resulting in greater moisture retention in the final product.

Ash content reflects the concentration of inorganic residues, such as potassium, magnesium, and sodium, that remain after ignition and oxidation of organic matter. In pectin, high ash content is generally indicative of lower purity and is strongly influenced by the extraction method employed.[45] Mineral acid extraction typically yields pectin with higher ash levels compared to organic acid methods. In support of this, Altaf *et al.*[46] reported ash contents of 7.2% and 4.8% for pectin extracted from papaya peel using hydrochloric and citric acid respectively. In this study, the use of citric acid for CSP extraction resulted in a relatively low ash content of 5%, which complies with the International Pectin Producers Association (IPPA) limit of 10% for commercial-grade pectin.[40]

The protein content of pectin plays a critical role in emulsion formation and stability, as the amphipathic nature of protein molecules facilitates stable interactions at the oil-water interface.[47,48] In previous studies, El Fihry *et al.*[29] reported protein contents ranging from 1.07–4.65% for Moroccan citrus peel pectin, Muñoz-Almagro *et al.*[30] reported 1.58–2.49% for strawberry pectin, and Madhuvanthi *et al.*[49] recorded 2.2% for pectin derived from papaya seeds. In this study, the CSP exhibited a protein content of 3.18%, which falls on the higher end of values reported in the literature and therefore indicates its potential for emulsifying applications.

Although the CSP exhibited a high galacturonic acid content (77.8%), its total carbohydrate content, determined *via* the phenol-sulphuric acid method, was relatively low at 23.79% (Table 2). Since glucose was used as the reference standard, this value represents glucose equivalents rather than the absolute carbohydrate content and therefore would not have accounted for uronic acids such as galacturonic acid. Previous studies that quantified carbohydrate content in a similar manner also reported low values for pectin from sour cherry (26.43%), red chilto (22.01%), and cocoa pod husks (18.5%).[47,50,51] However, higher values were observed for medlar (87.25%) and okra pectins (66–87%), likely due to their extraction from more

Table 2 Summary of CSP properties[a]

Property	Value
Physicochemical properties	
Equivalent weight (g mol^{-1})	1744.66 ± 35.55
Galacturonic acid (%)	77.80 ± 0.02
Moisture (%)	15.34 ± 0.01
Ash (%)	5.00 ± 0.02
Protein (%)	3.18 ± 0.15
Carbohydrate (%)	23.79 ± 0.21
DE (%)	80.40
Techno-functional properties	
WHC (mL g^{-1})	4.20 ± 0.90
OHC (mL g^{-1})	2.10 ± 0.79
EC (%)	46.89 ± 0.10
ES (%)	81.33 ± 0.05

[a] DE, degree of esterification; WHC, water holding capacity; OHC, oil holding capacity; EC, emulsion capacity; ES, emulsion stability.

carbohydrate-rich tissues.[52,53] The relatively low carbohydrate content of CSP in this study can therefore be attributed to its origin from the tough, inedible outer shell of *Melicoccus bijugatus*, rather than the carbohydrate-dense fruit pulp.

Structural and morphological analysis

FT-IR spectra and degree of esterification. The degree of esterification (DE) of pectin is an important physicochemical property that directly influences its gelling mechanism and application.[54] High-methoxyl (HM) pectin (>50% esterification), is widely used in the food industry as thickeners, gelling agents in jams and jellies, and stabilizers due to its ability to gel in the presence of high sugar concentrations.[50] Conversely, low-methoxyl (LM) pectin require divalent cations such as Ca^{2+} to form gels and is suitable for low-sugar formulations.[55] In this study, FT-IR spectroscopy was employed to determine the DE of the extracted CSP. The spectra (Fig. 1) revealed characteristic peaks in the 1750–1500 cm^{-1} region, inclusive of a peak at 1712 cm^{-1} which corresponds to the ester carbonyl (C═O) stretch of esterified carboxyl groups, and a peak at 1617 cm^{-1} attributed to the stretching vibration of un-esterified carboxylate anions (COO^-).[26] The areas under these peaks were used to calculate a DE of 80.4% (eqn (6)), thereby confirming the CSP as a HM pectin, which was consistent with the equivalent weight findings.

This high DE likely reflects both the maturity stage of the harvested fruit, and the acid-based extraction method employed. As fruits ripen, the activity of pectin methylesterase (PME) increases, which catalyzes the de-esterification of pectin thus progressively converting high-methoxyl (HM) pectin into low-methoxyl (LM) pectin.[56] The high DE observed in this study therefore suggests that the *Melicoccus bijugatus* shells were not sourced from very ripe or overripe fruit. Additionally, Wang *et al.*[27] showed that while alkali extraction generally yields lower DE values (around 40%), acid extraction generally leads to higher values (around 70%). Together, these factors explain the elevated DE of the CSP, reinforcing its potential for application in the food industry as an effective gelling and thickening agent.

Scanning electron microscopy (SEM). The surface microstructures of the CSP and commercial citrus pectin were comparatively analysed using SEM with the images shown in Fig. 2. The CSP exhibited a smoother, flatter, slab-like morphology, whereas the commercial pectin showed a more irregular, wrinkled surface. This slab-like structure of CSP is consistent with observations reported by Jong *et al.*[26] and Kazemi *et al.*[57] where similar acid extraction methods were employed. Previous studies suggest that pectin with small, amorphous, and porous structures exhibit higher water uptake compared to those with smoother surfaces.[58,59] Since the binding of water to pectin occurs not only through hydrophilic functional groups but also *via* physical adsorption on the surface, the relatively smooth morphology of CSP implies a potentially lower, more moderate capacity for water binding.[26]

Antioxidant properties

The antioxidant capacity of the extracted CSP was evaluated by both the DPPH radical scavenging assay and the FRAP assay, with the results compared to commercial citrus pectin and ascorbic acid. As shown in Fig. 3, ascorbic acid exhibited the highest antioxidant activity as expected, with a maximum DPPH scavenging rate of 97% and a FRAP value of 0.204. Notably, the CSP demonstrated significantly greater ($p < 0.05$) antioxidant capacity than the commercial pectin. The FRAP value for CSP (0.052) was 6.5 times greater than that of the commercial pectin (0.008), indicating a markedly enhanced electron-donating ability (Fig. 3a). Moreover, at a concentration of 2.0 g L^{-1}, CSP exhibited a maximum DPPH scavenging rate that was 20% higher than the commercial pectin at the same concentration

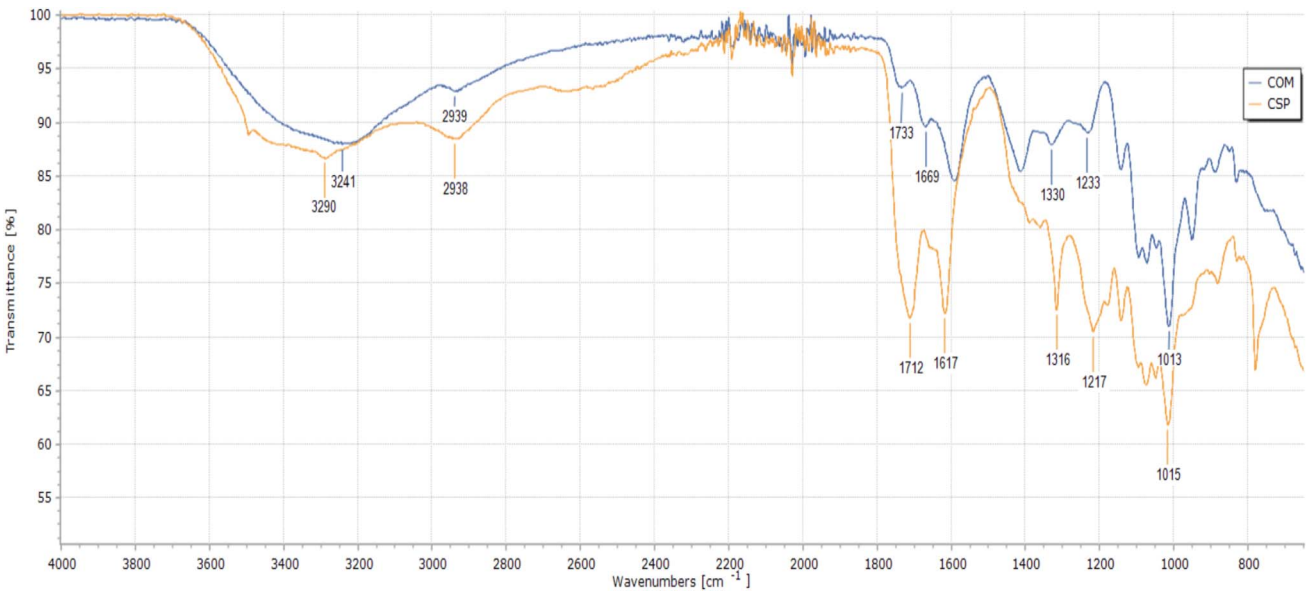

Fig. 1 FT-IR spectra of extracted *Melicoccus bijugatus* shell pectin (CSP) and commercial citrus pectin (COM).

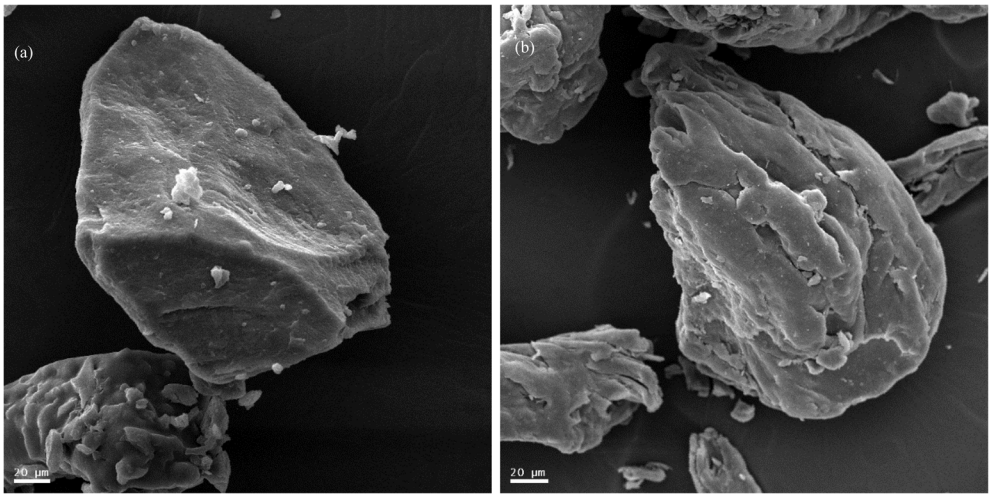

Fig. 2 Scanning electron micrograph (SEM) of extracted CSP at 400× (a) and commercial citrus pectin at 400× (b).

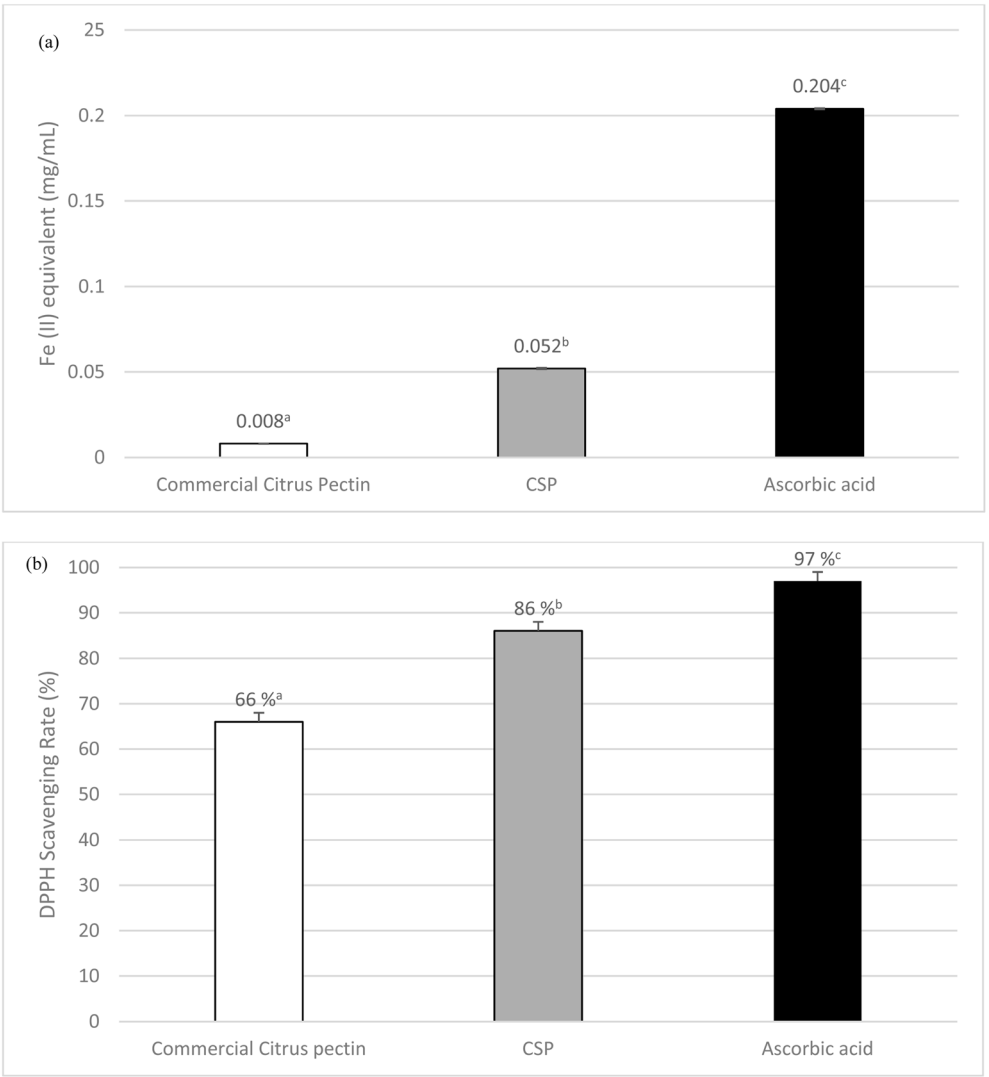

Fig. 3 Comparison of antioxidant properties of *Melicoccus bijugatus* shell pectin, commercial citrus pectin and ascorbic acid. (a) FRAP reducing assay, (b) DPPH scavenging rate. Different superscript letters indicate a significant difference ($p < 0.05$).

(Fig. 3b). While the DPPH scavenging rate of the CSP was significantly lower than that of ascorbic acid ($p < 0.05$), it was only 9% less, underscoring its considerable radical scavenging potential in comparison to commercial pectin. Furthermore, these antioxidant properties of the CSP distinctly set it apart from other tropical fruit waste pectin as similar studies reported DPPH scavenging rates of only 51.58% for dragon fruit pectin and 25.29% for jackfruit pectin.[60,61] These findings suggest that the CSP may have retained co-extracted phenolic and flavonoid compounds from the *Melicoccus bijugatus* shell matrix, which would have contributed to its elevated antioxidant performance. This co-extraction of bioactive compounds during pectin isolation has been previously reported in other fruit waste sources and is known to enhance radical scavenging and reducing capacity through synergistic effects between pectin and associated phytochemicals.[62] This enhanced bioactivity therefore supports its application as a natural antioxidant agent in food and pharmaceutical formulations aimed at reducing oxidative stress.

Functional characterisation of pectin

Water holding and oil holding capacity. Water holding capacity (WHC) and oil holding capacity (OHC) are key functional properties that influence the practical applications of pectin. They refer to the amount of water and oil retained per gram of pectin respectively.[29] High WHC allows pectin to contribute to improved food texture and viscosity through the physical entrapment of water molecules, thereby enhancing sensory and organoleptic qualities.[63] Moreover, a high WHC can help reduce syneresis in products such as jams and yogurts.[29] The CSP exhibited a WHC of 4.20 mL g^{-1}, exceeding values reported for sour orange (3.10 mL g^{-1}) and strawberry pectin (2.8 mL g^{-1}) but was lower than mixed banana–papaya (8.23 mL g^{-1}) and redcurrant pectin (5.6 mL g^{-1}).[30,39,42] In addition to the surface morphology of pectin, WHC is also strongly influenced by the presence and abundance of hydrophilic functional groups such as carboxyl and hydroxyl moieties which can contribute to water retention. Therefore, although the SEM images revealed a smooth, flat CSP surface morphology which would have indicated limited water absorption, the observed moderate WHC of the CSP suggests a high density of hydrophilic groups within the pectin polymer structure. OHC reflects the ability of pectin to bind oil, a key feature for stabilizing emulsions by preventing droplet coalescence at the oil–water interface. The CSP showed a OHC of 2.10 mL g^{-1}, which was below values for strawberry (5.0 mL g^{-1}) and blackberry pectin (6.6 mL g^{-1}) but higher than Moroccan orange (1.55 mL g^{-1}) and lemon peel pectins (1.39 mL g^{-1}).[29,30] This intermediate OHC suggests a moderate affinity for oil, potentially influenced by the CSP's smooth surface morphology and protein content. While not particularly high, this level of oil retention indicated that the CSP may still possess sufficient interfacial activity to contribute to emulsion stabilization.

Emulsion properties. The ability of pectin to act as an emulsifier by enabling uniform dispersion of two otherwise immiscible liquids is a key functional property widely exploited in both food and pharmaceutical industries. In this study, the emulsification potential of the extracted CSP was evaluated by assessing its emulsion capacity (EC%) and emulsion stability (ES%). The CSP exhibited an EC% of 46.89%, which was comparable to commercial citrus pectin (50%) and higher than pectin from other tropical fruits such as mango peel pectin (34.2%) and pineapple peel pectin (45%).[43,64] Furthermore, the CSP was shown to have an ES% of 81.33% which, again, while slightly lower than the ES% of commercial pectin (94%), exceeded that of other tropical fruit waste pectin such as dragon fruit (71.71%) and mango peel pectin (65.1%).[42,43,62,65] These emulsion properties align with the intermediate oil holding capacity (2.10 mL g^{-1}) and above average protein content observed for CSP, which supports its ability to form stable oil-water interfaces thereby contributing to enhanced emulsion stability. Overall, these findings indicate that CSP exhibits satisfactory emulsifying properties, that outperform many novel pectin sources and approach those of commercial pectin.

Preliminary film assessment

Co-optimized pectin extraction. Derringer's desirability function was performed to simultaneously optimize the pectin yield and viscosity-average molecular weight with the goal of identifying the optimal extraction conditions that would provide both high yield and high viscosity-average molecular weight as higher molecular weight pectin is associated with improved film-forming properties. Individual desirability values for each response were combined into a composite desirability index (D) with the molecular weight response assigned twice the importance of yield to prioritize film-forming quality. The optimal conditions were selected based on the highest D value which corresponded to Run 3 of the Box–Behnken design (D value of 0.42). This gave a co-optimized pectin yield of 3.6% and viscosity-average M_w of 34 908.9 Da, using a solvent pH of 3.5, S/L ratio of 1 : 30 and an extraction time of 2 h.

Mechanical properties. As shown in Fig. 4, the CSP film exhibited a tensile strength (TS) of 0.13 MPa and an elongation at break (EAB) of 11%, compared to the 0.18 MPa TS and 7% EAB for the commercial pectin film. Since both films were fabricated under identical conditions, the lower TS of the CSP film suggests a slightly less cohesive polymer network relative to the commercial counterpart. This difference likely reflected intrinsic variations in the pectin themselves, such as molecular weight, purity and degree of chain branching, all of which have been shown to affect the mechanical performance of pectin-based films.[7] However, the higher EAB of the CSP film indicates that the film was more flexible than the commercial film and suggests that although the CSP formed a slightly weaker film network, it was less brittle and able to undergo more deformation before rupture.

Barrier properties. The pectin-based film produced from the extracted CSP (co-optimized for both yield and molecular weight), exhibited significantly lower ($p < 0.05$) oxygen permeability compared to conventional plastic packaging materials PET and HDPE as shown in Fig. 5a. This notable difference can be attributed to the dense, hydrogen-bonded polysaccharide

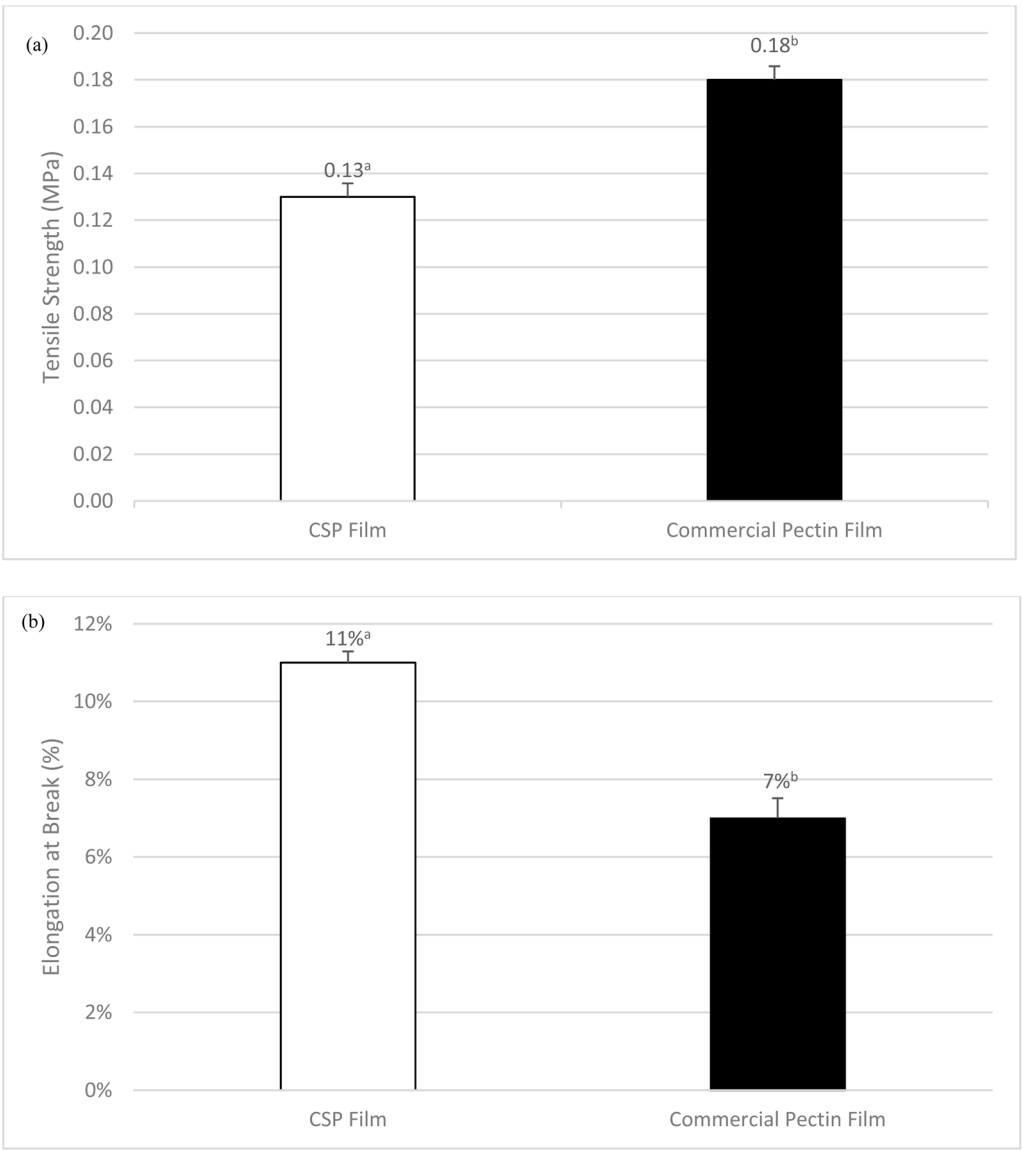

Fig. 4 Comparison of the preliminary mechanical properties of CSP film and commercial pectin film. (a) Tensile strength, (b) elongation at break. Different superscript letters indicate a significant difference ($p < 0.05$).

network formed by pectin, which effectively restricts the diffusion of oxygen through its compact and polar nature.[66] This characteristic is particularly advantageous in packaging applications involving oxygen-sensitive products such as high fat foods or cooked meats where oxidative degeneration leads to rancidity, discoloration and a loss of organoleptic quality. Conversely, the inherent hydrophilic nature of the polymer resulted in a substantially higher water vapor permeability of the film compared to both HDPE and PET (Fig. 5b). As shown in the functional characterisation section, the CSP had a moderate water holding capacity, indicating its affinity for water molecules. This property likely extended to the film enabling it to readily absorb and transmit moisture, thereby facilitating much higher water vapor transmission than the more hydrophobic HDPE and PET controls. These findings highlight the promise of the CSP-derived films as oxygen barriers while indicating the need for further modification to improve moisture resistance to expand potential packaging applications.

Antioxidant properties. Given the significantly higher antioxidant properties of the CSP compared to commercial citrus pectin, the antioxidant activity of the derived films was also evaluated. As shown in Fig. 5(c and d), similar results were observed as the CSP-derived films exhibited significantly greater ($p < 0.05$) antioxidant activity than the film made from commercial citrus pectin under identical conditions. Similar findings were reported by Rodsamran and Sothornvit[67] who demonstrated that pectin films formulated with higher concentrations of pineapple peel pectin extract displayed significantly higher antioxidant activity, as evidenced by increased DPPH and ABTS radical scavenging capacities. The DPPH scavenging rate of the CSP film (25.6%) was almost double that of the commercial pectin film (14.17%) while the

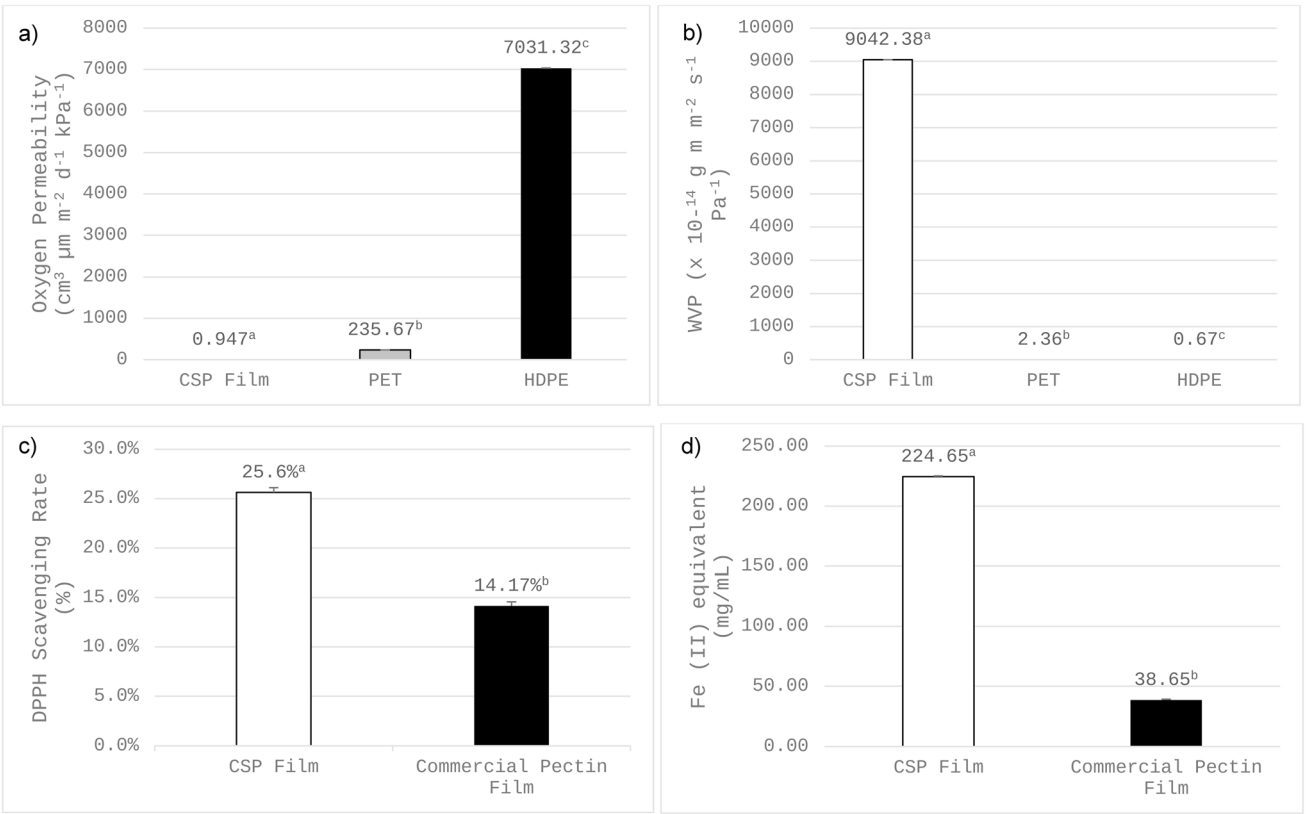

Fig. 5 Summary of barrier and antioxidant properties of CSP films. (a) Oxygen permeability, (b) water vapor permeability, (c) antioxidant activity using DPPH assay and (d) antioxidant activity using FRAP assay. Different superscript letters indicate a significant difference ($p < 0.05$).

FRAP value was nearly six times higher (224.56 and 38.65 Fe^{2+} mg mL^{-1} respectively). This showed that the bioactive compounds retained in the CSP were effectively incorporated into the film matrix, conferring enhanced antioxidant functionality. This added element of bioactivity is especially advantageous for active food packaging applications, particularly where delaying oxidative spoilage in food systems is critical for preserving organoleptic quality and shelf life.

Conclusion

This study demonstrated the potential of *Melicoccus bijugatus* shell waste as a novel and sustainable source of functional pectin (CSP) in light of the growing demand for value added utilization of agro-industrial by-products. The pectin extraction from the shell waste produced an optimized yield of 16.37% and viscosity-average molecular weight of 37 445.18 Da. Characterisation revealed that the CSP exhibited moderate water and oil holding capacities and high emulsion stability. Notably, it demonstrated significantly enhanced antioxidant activities, with DPPH scavenging rates and FRAP values being 20% and 6.5 times higher, respectively, than those of commercial pectin indicating its strong potential as a natural antioxidant. The CSP-based films showed superior oxygen barrier performance compared to conventional plastics (PET and HDPE), supporting their potential in active and sustainable packaging applications. However, the high-water vapor permeability, inherent to the hydrophilic nature of the pectin polymer, indicates the need for further optimization. Strategies such as the incorporation of hydrophobic additives or multilayer configurations would help improve moisture resistance for broader food packaging applications. Preliminary mechanical evaluation indicated that the CSP films formed slightly weaker but more flexible networks compared to commercial pectin films, confirming adequate film integrity but also highlighting the need for future reinforcement strategies. While a thorough evaluation of the mechanical properties of the films is essential for assessing their food packaging potential, such exhaustive testing was outside of the scope of this study. Film formation and evaluation were conducted in a preliminary manner, intended only to screen the suitability of CSP for potential use in edible film applications. Consequently, a more exhaustive mechanical, structural and barrier characterisation of the CSP films would be conducted in a future study specifically as it relates to the optimization of the films in terms of the pectin concentration and plasticizer concentration utilized in film formation. Overall, the findings demonstrate the viability of *Melicoccus bijugatus* shell waste as a high-value source of functional pectin for use in food systems. Its strong antioxidant properties, film-forming potential, and alignment with circular bioeconomy goals support its application in biodegradable packaging and functional food innovations.

Author contributions

Both authors contributed to the conception and design of the study. Material preparation, data collection and analysis were performed by Che John under the supervision of Rohanie Maharaj. The first draft was written by Che John and reviewed and edited by Rohanie Maharaj.

Conflicts of interest

The authors declare that they have no conflict of interest.

Data availability

All the data is presented within the manuscript itself.

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