JOBIMB, 2025, Vol 13, No 2, 103-110
https://doi.org/10.54987/jobimb.v13i2.1144

JOURNAL OF BIOCHEMISTRY, MICROBIOLOGY AND BIOTECHNOLOGY
Website: http://journal.hibiscuspublisher.com/index.php/JOBIMB/index
JOBIMB VOL 13 NO 2 2025
AI and Biochemistry

Starch-based Films Incorporated with Pectin Extracted from Papaya Peel Using Acid Hydrolysis

Athirah Syadiyah Sulaiman[1], Nurdiyana Husin[2] and Azlin Shafrina Hasim[1]*

[1]Faculty of Fisheries and Food Science, Universiti Malaysia Terengganu, 21310 Kuala Nerus, Terengganu, Malaysia.
[2]Section of Food Engineering Technology, Universiti Kuala Lumpur Malaysian Institute of Chemical and Bioengineering Technology (UniKL MICET), Lot 1988 Bandar Vendor, 78000 Alor Gajah, Melaka, Malaysia.

*Corresponding author:
Azlin Shafrina binti Hasim,
Faculty of Fisheries and Food Science,
Universiti Malaysia Terengganu,
21300 Kuala Nerus,
Terengganu,
Malaysia.
Email: azlin.hasim@umt.edu.my

History

Received: 19th June 2025
Received in revised form: 21st July 2025
Accepted: 18th Aug 2025

Keywords

Acid hydrolysis
Degree of esterification
Papaya Peels
Pectin
Starch-based Film

SDG Keywords

SDG 2 Zero Hunger
SDG 9 Industry, Innovation and Infrastructure
SDG 12 Responsible Consumption and Production

Abstract

Papaya (*Carica papaya* L.) peel contains bioactive compounds like pectin, widely used in making edible films due to its gel-forming ability, offering a sustainable way to repurpose fruit waste. The reliance on synthetic plastics for packaging and the insufficient management of papaya peel waste highlights the need for a sustainable alternative. Limited research exists on how different acids used for pectin extraction affect film properties. This study focused on extracting pectin from papaya peel using hydrochloric acid (HCl) and citric acid (CA) and incorporating it into starch-based films at 0.5% and 1% concentrations. Pectin extraction efficiency was influenced by acid type, with HCl yielding 9.1%, significantly higher than CA 3.99%. The extracted pectin was characterized as high methoxyl pectin with degrees of esterification (DE) ranging from 51.82% to 52.26%. A higher acid percentage enhanced tensile strength and reduced elongation at break, improving mechanical stability. FTIR analysis confirmed pectin structures and degradation, showing a higher OH peak. Color analysis and water vapor permeability tests revealed that films with 1% pectin extracted using CA had the lowest water vapor permeability, improving moisture barrier properties. UV-Vis spectroscopy analyzed optical properties, UV-Vis spectroscopy showed that films incorporating 1% pectin extracted using HCl exhibited greater UV absorbance due to the presence of phenolic compounds, highlighting their potential as effective natural UV-blocking packaging materials while biodegradability tests showed that films incorporated with 0.5% pectin extracted using CA degraded more efficiently than the control and film incorporated with pectin extracted with HCl. In conclusion, pectin extracted using HCI more effectively than CA. Pectin extraction using HCl proved more efficient, resulting in a higher yield compared to CA. Additionally, incorporating 1% pectin extracted using CA preserves pectin's molecular weight and functional properties, enhancing film quality in starch-based films by improving its physical and mechanical characteristics.

INTRODUCTION

Edible films are defined as a continuous and thin layer of edible material used as a coating or as a film placed between food components, which is non-toxic and naturally biodegradable, over synthetic films. The edible films have been used as an alternative to plastic food packaging with the ability to prevent the transfer of oxygen, aromas, and moisture. Source for edible film production can be found in polysaccharides such as cellulose or chitosan due to their functional properties that can be used as film-forming materials [1]. Edible packaging makes handling and transportation more convenient. Edible packaging can enhance mechanical properties by incorporating biopolymers such as pectin and starch, which form strong intermolecular bonds during film formation. These bonds improve tensile strength and flexibility, resulting in more durable and resilient packaging materials. As a result, the reduced risk of tearing or deformation enhances the convenience. Its functional performance largely depends on the composition, structure, and nature of its components. The selection of film-forming materials and active additives is based on the intended purpose, the characteristics of the food product, and the application method [2]. Starch can be used as a natural polymer (carbohydrate) in the production of composite edible films due to its composition (20% to 25% amylose and 70% to 80% amylopectin). It is readily available, biocompatible, non-toxic, and possesses excellent film-forming properties [3]. Corn starch is a widely used starch polymer due to its high biodegradability, making it suitable for

the development of edible films with strong structural integrity [4]. Papaya (*Carica papaya* L.) is widely consumed in Southeast Asia and subtropical countries. It consists of water and carbohydrates and is rich in natural vitamins and minerals [5]. Papaya peels are usually considered a waste, especially in food industries, contributing approximately 20% - 25% of the papaya weight [6], but the by-products of peel may contain many valuable compounds such as carotenoids, flavonoids, and have strong antioxidant same as papaya fruit [7]. Papaya peel also contains bioactive compounds such as pectin [8].

Pectin is a complex heteropolysaccharide that is found in the cell walls and middle lamella of plants. Pectin classification can be defined by the degree of esterification (DE) and is expressed as the percentage of methyl-esterified carboxyl groups to the total number of galacturonic acid units ratio. High methoxyl (HM) pectin is defined as pectin with DE higher than 50%, and pectin with DE less than 50% is known as low methoxyl (LM) pectin. Papaya peel pectin contains approximately 77.57% galacturonic acid units, which are components of pectic polymers capable of binding to other polysaccharides such as cellulose [9]. In the food industry, pectin can act as a gelling agent, a thickener, a stabilizer for jam, marmalade, fruit juice, and jellies production, and as the main polymer used in biomedical due to its gelling and thickening capabilities [10].

Pectin extraction is an important physical-chemical process to obtain pectin from plant tissue, and their solubilization is influenced by different factors, mainly temperature, pH, and time [11]. The use of acid hydrolysis for pectin degradation may disrupt the bonds between side-chain sugars and the cell wall, leading to the release of pectin in a water-soluble form [12]. The application of organic acids like citric acid and inorganic acids like hydrochloric acid (HCl) are commonly used in extract pectin. Yadav et. al. [13] reported that pectin contained in papaya peel consists of 4% to 19.1% extracted by hydrochloric acid while using citric acid extracted by 2.0% to 16.2%. The type of acid used influences the extraction efficiency, the structural integrity of the extracted pectin, and its physicochemical properties due to differences in acid strength, pH levels, and chemical interactions during the extraction process [12].

MATERIALS AND METHODS

Materials

The materials used include 0.5 M Hydrochloric acid (HCl) (LabChem), 0.5 M Citric Acid (CA) (ChemPure), Glycerol (ChemPure), starch, and distilled water. Papaya peel was obtained from a local fruit stall in Kuala Terengganu.

Preparation of Papaya peel powder

The papaya peel was washed to remove any remaining dirt, chopped into smaller pieces, and dried at 80 °C for 7 hours in a dryer. Then, ground dried peel used a blender and sieved to obtain smooth powder then stored at dry place [14].

Extraction of Papaya Peel pectin

The pectin compound in papaya peel was extracted with two different acids which were hydrochloric acid and citric acid. A total of 25 g of papaya peel powder was added to 100 ml of distilled water and 3 ml of 0.5 M HCl and 0.5 M CA concentration was added to the mixture. Then the mixture heated for 60 minutes at 90 °C. After extraction, the mixture was filtered with a filter cloth to separate the liquid from the solid pectin. The solids were discarded. The filtrate (pectin extract) was cooled, and 95% ethanol was added to precipitate the pectin, which was kept for an hour. Afterward, filtration was performed again to separate the coagulated pectin. The coagulated pectin was then dried in an oven at 50°C for 24 hours [14]. The pectin yields were calculated using the Equation 1:

$$yield\ (\%) = \frac{weight\ of\ dried\ \ pectin\ (g)}{weight\ of\ peel\ powder\ (g)}\ x\ 100 \qquad \text{(Equation 1)}$$

Degree of Esterification (DE)

The degree of esterification was calculated using a Thermo Scientific Nicolet iS10 FTIR Spectrometer, (United States) with 64 scans accumulated (wavenumber range of 600–4000 cm^{-1}). The FT-IR data was used to determine the DE value based on the areas corresponding to the ester carbonyl groups (1745–1760 cm^{-1}) and free carboxylate groups (1620–1640 cm^{-1}). The DE values were calculated and interpreted using Origin 9.0 software. The DE values were calculated using Equation 2:

$$DE = \frac{area\ of\ esterified\ carboxyl}{area\ of\ esterified\ carbonyl\ groups + area\ of\ nonesterified\ carboxyl\ groups}\ x\ 100$$
(Equation 2)

Basic formulation

Production of bio-composite films

Different percentages of papaya peel pectin extract from HCl and citric acid (1% and 0.5%) were dissolved in 100 mL of distilled water (**Table 1**). The mixture was stirred using a magnetic stir hot plate with continuous stirring until fully dissolved. Then, 5 g of corn starch was added to the mixture, which was continuously stirred and heated until the temperature reached 80°C. Next, 2 g of glycerol was added to the mixture, and it was continuously heated and stirred for 15 minutes, or until gelatinization occurred. The solution was then poured onto a petri dish and dried in an oven at 50°C for 24 hours. The dried samples were removed from the plate and stored in a desiccator for 48 hours at 25°C and 50% relative humidity for further analysis [14].

Colour profile

The color measurement was determined using Konica Minolta CR-400 chroma meter, (Tokyo, Japan). The color parameters, represented as L*, a*, and b* values, were recorded. The L* (0 = black, 100 = black) represents lightness, the a* value (positive value = red, negative value = green), and the b* value (positive values = yellow, negative value = blue) components of the colour [15].

Table 1. Formulation for production of papaya peel pectin and corn starch-based film.

Raw materials	In gram (g)				
	Formulation 1 (COM)	Formulation 2 PHCl	Formulation 3 PHCl	Formulation 4 PCA	Formulation 5 PCA
Papaya peel pectin	0	1	0.5	1	0.5
Corn starch	5	5	5	5	5
Glycerol	2	2	2	2	2
Distilled water (mL)	100	100	100	100	100

Notes: COM; Commercial pectin, PHCl: Papaya peel pectin extract with HCl, PCA; Papaya peel pectin extract with Citric Acid.

Mechanical properties
Each film's tensile strength (TS) and elongation at break (EAB) were clamped between the grips and cross directions were measured using a Texture Analyzer Stable Microsystem, Surrey. TA.XTPlus, (England) testing machine. The samples were prepared following the ASTM D822_02/TG standards, with an initial grasp separation of 50 mm and a crosshead speed of 5 mm/min. The films were cut into strips, 10 mm wide by 70 mm long. The mechanical properties of the samples were determined using a 5N load cell. Each film's stated results were the average of at least three measurements [9]. The total TS and EAB were calculated by the equations 3 and 4:

$$TS = \frac{F}{S} \qquad \text{(Equation 3)}$$

where F represents the maximum load, and S represents the initial cross-sectional area.

$$EAB\% = \frac{L-L0}{L0} X\ 100\% \qquad \text{(Equation 4)}$$

While L0 is the film's initial length, and L is the film's length at the point of rupture.

Fourier Transform Infrared Spectroscopy (FTIR)
The functional group of the edible composite film was evaluated by using a Thermo Scientific Nicolet iS10 FTIR Spectrometer, (United States). FT-IR device PerkinElmer with spectroscopy wavelength range from 600 cm^{-1} to 4000 cm^{-1}. In preparing samples, the edible composite film was measured 2.5 cm × 3 cm and directly positioned on the ray-exposing stage. Spectra were obtained using 64 scan summations at 4 cm^{-1} resolutions [9].

Light Transmittance analysis
A UV-Vis spectrophotometer (Shimadzu UV1900i, Tokyo, Japan) was utilized to evaluate light transmission across a wavelength of 400 nm. This analysis was conducted to determine the film's barrier properties against UV and visible light, as well as its opacity. Film strips measuring 1 × 4 cm were placed directly into the measuring cell, while a blank cuvette served as the baseline reference [16].

Water vapor permeability (WVP)
The water vapor permeability (WVP) was measured using the ASTM E96 (2024) standard. Plastic cups with a diameter of 5.5cm and length of 3cm were used and 10 g of sodium chloride (NaCl) was placed in each cup. Next, the cups were covered with the prepared film samples and sealed using Parafilm. The cups were weighed along with their content and placed in a desiccator. The relative humidity was maintained by silica gel at the bottom of the desiccator to provide 75% RH at 25 ± 1°C. Each cup was weighed every 24 hours for 7 days.

Biodegradability of the films
A biodegradability test on edible film was carried out by composting soil. The initial properties of edible film such as thickness and weight were measured and recorded before running a test. The samples are cut into 20 mm diameter and buried (10 mm) in composting soil. The containers used to place soil are evenly distributed and exposed to the environmental conditions and water is sprayed twice a day to sustain the moisture. The biodegradation process was evaluated every seven days [17]. The amount of degradation was calculated using Equation 5.

$$Soil\ biodegradation\ (\%) = \left[\frac{Wi - Wd}{Wi}\right] x\ 100 \qquad \text{(Equation 5)}$$

where W_i is the initial weight of the sample and W_d is the final weight of the sample.

Statistical Analysis
Data were presented as mean ± standard deviation (SD) from three times observations. Anova and Tukey mean comparison test ($p<0.05$) was used to evaluate data obtained from the analysis and this statistical test was carried out by using Minitab Statistical Software version 21.

RESULTS AND DISCUSSION

Characterization of papaya peel pectin with different extraction

Extraction yield of papaya peel pectin.
Table 2 shows the extraction yield of papaya peel pectin. The extraction of pectin from papaya peel powder using hydrochloric acid (HCl) and citric acid (CA) revealed that HCl produced a higher yield of pectin (9.1% ± 10.06) compared to CA (3.99% ± 1.62). This difference is likely due to the stronger acidic nature of HCl, which more effectively breaks down polysaccharides to release pectin [18]. According to Altaf et. al. [2], the pectin yield extracted using HCl for 60 minutes was 9.4%, while citric acid (CA) yielded 4.5%. The results of this study show a comparable yield for CA and are generally consistent with the trends observed in their research. The use of CA, being an organic and environmentally friendlier acid, presents a sustainable alternative, but with reduced efficiency. Factors such as the natural variability of papaya peel composition and the optimization of extraction parameters play an important role in determining pectin yield.

Table 2. The extraction yield of papaya peel pectin.

	Mean (±) SD
Weight of papaya peel (PP) powder (g)	25.078 ± 0.05
Weight of papaya peel pectin (PPP) HCl (g)	2.97 ± 2.06
Yield (%)	9.1 ± 10.06
Weight of papaya peel (PP) powder (g)	25.00 ± 0.00
Weight of papaya peel pectin (PPP) CA (g)	1.00 ± 0.40
Yield (%)	3.99 ± 1.62

Note: Values are expressed in mean ± standard deviation for three replications (n=3)

Fourier-transform infrared (FTIR) spectra analysis of papaya peel pectin
Fig. 1 shows the FTIR spectra of papaya peel pectin extract with HCl and CA. Similar functional groups characteristic of pectin, such as strong O-H stretching in the range 3200 cm^{-1}-3600 cm^{-1}, C-H stretching near 2900 cm^{-1}. The band at approximately 1730-1744 cm^{-1} was attributed to C=O easter starching vibration. Then, C=O stretching of carboxylic groups around 1600–1620 cm^{-1} is ascribed to the stretching vibration of the methyl ester in the carboxyl group (COO-R). However, subtle differences are observed, particularly in the 1600–1400 cm^{-1} region, where the citric acid (1620 cm^{-1}) extract exhibits sharper peaks, potentially indicating a higher degree of esterification or structural variations caused by the extraction process compared to HCl (1618.07 cm^{-1}).

Additionally, asymmetric COO^- stretching vibrations for pectin samples extracted with HCl and CA appeared in 1412.08 cm^{-1} and 1409.15 cm^{-1}, respectively. The fingerprint region (400–1200 cm^{-1}) also shows more defined peaks in the pectin extracted using the CA spectrum, suggesting differences in glycosidic bond integrity and interactions with the extraction agent. This data was supported by Erika et. al. [10] and Bonrood et. al., [19] who also obtained similar spectra for both HCl and CA extraction where pectin characterization can be determined

from the areas of the esterified carbonyl (1745–1760 cm^{-1}) and free carboxylate groups (1620-1640 cm^{-1}). These results indicated that even though both methods effectively extract pectin, CA may yield pectin with a marginally different structural profile, possibly influencing its functional properties.

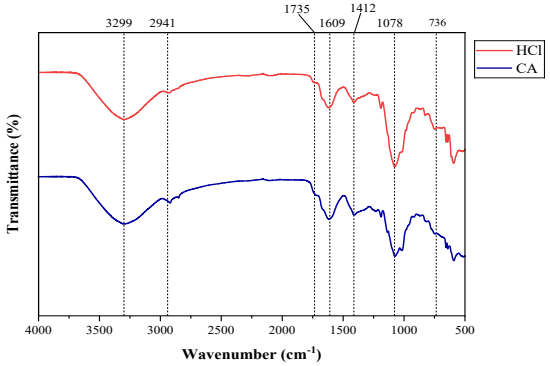


Fig. 1. FTIR spectra of papaya peel pectin extract with HCl and CA.

The Degree of Esterification (DE) refers to the ratio of esterified galacturonic acid groups to the total galacturonic acid groups present in pectin. It is an important characteristic that determines the gelling nature of pectin. **Table 3** shows the characteristics of papaya peel pectin based on FTIR. The DE values extracted using CA were higher compared to those extracted using HCl. According to the Erika et. al., [10] and Altaf et. al., [20] the degree of esterification for HCl is 53.4% while for CA 57.54% shows both are high methoxyl pectin. Moreover, the degree of esterification of papaya peel pectin depends on factors such as fruit ripeness, the part of the fruit used, botanical origin, and the extraction method. Unripe papaya tends to have a higher degree of esterification [19].

Table 3. The characteristics of papaya peel pectin based on FTIR.

Characteristic	HCl	CA
Degree of esterification (DE) (%)	51.82%	52.26%
Methoxyl Content	> 50% (High-methoxyl pectin)	> 50% (High-methoxyl pectin)

Characterization of the starch-based film

Table 4 illustrates the visual appearance of starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations. Starch-based films incorporated with PHCl 1% show the darkest and most intense coloration, with a noticeable brownish hue. This range with the significantly higher a* (redness) and b* (yellowness) values observed. The darker appearance could be attributed to residual pigments or the impact of the HCl extraction process on the papaya peel pectin, leading to more prominent colour development in the film. The films with PHCl 0.5% and PCA (both 0.5% and 1%) exhibit lighter shades, with PCA-based films showing more yellow tones than red. This difference may result from the milder extraction effect of citric acid compared to hydrochloric acid, which could retain more natural pigments or reduce the formation of browning compounds during the extraction process [21].

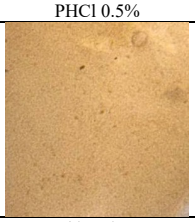
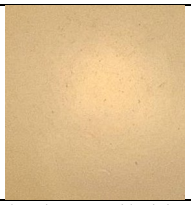
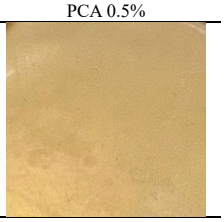
Colour Profile

Fig. 2(a-c) shows the values of L*, a*, and b* of starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations. **Fig. 2(a)** illustrates a significant difference ($p<0.05$) in lightness (L*) between the starch-based films incorporated with pectin extracted from papaya peel via acid hydrolysis. The starch-based films with commercial pectin (COM) exhibited the highest lightness L* value compared to film with (PHCl) 0.5%, 1%, and PCA 0.5%, 1%. However, film with papaya peel pectin extracted using HCl (PHCl) and citric acid (PCA) showed reduced lightness, particularly for the higher concentration (1%), suggesting that acid-extracted pectin interacts with corn starch in a way that alters the light-reflecting properties of the film and commercial pectin produces lighter films. The result aligns with what was reported by Crizel et. al. [22] in which the increase in the concentration of acid resulted in the reduction of the lightness of the film.

In **Fig. 2(b)**, the a* value was significantly higher ($p<0.05$) in the film with PHCl 1% compared to all other samples. This indicates a stronger reddish hue in this sample, which may be due to the interaction of HCl-extracted pectin with starch, likely influencing the colour attributes. Other samples, such as PHCl 0.5% and PCA (0.5% and 1%), exhibited minimal redness, suggesting a more neutral colour. Research by Rodríguez et. al. [23] stated that the positive value of (a*) indicated a greater preference of the films toward redness due to the non-enzymatic browning resulting from elevated drying temperatures.

In **Fig. 2(c),** b* values across the samples. The film with PHCL 1% exhibited the highest yellowness, followed by PHCl 0.5%. This trend suggests that higher concentrations of HCl-extracted pectin intensify yellow tones, possibly due to the residual pigments or the nature of the pectin extracted. However, the film with commercial pectin (COM) showed significant yellowness compared to films incorporated with papaya peel pectin. A study by Rodríguez et. al. [23] stated that positive results for yellowness (b*) indicate the colour towards yellow and it agrees with the natural colour of papaya peel. However, the highest b* with a strong tendency towards yellowness, might be also attributed to the nonenzymatic browning.

Table 4. Starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations.

Sample	COM	PHCl 1%	PHCl 0.5%	PCA 1%	PCA 0.5%
Film					

Notes: COM; Commercial pectin, PHCl: Papaya peel pectin extract with HCl, PCA; Papaya peel pectin extract with Citric Acid.

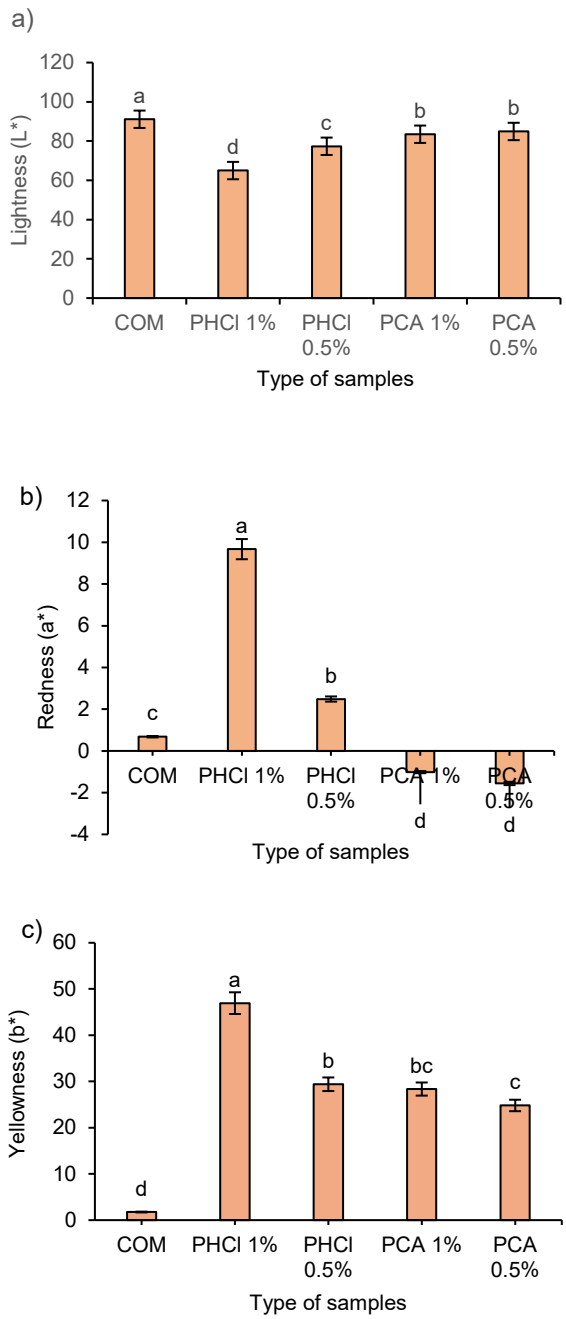


Fig. 2. The values of (a) L*, (b) a*, and (c) b* of starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations. Note: Values are expressed in mean ± standard deviation for three replications (n=3). Values with different superscript letters are significantly different at ($p<0.05$).

These findings suggest that the type of pectin used during extraction significantly influenced the optical properties of the starch-based films. The commercial pectin provided lighter and more neutral colour films, while papaya peel pectin, particularly at higher amounts, resulted in darker films with more noticeable yellowness and redness. These variations could be due to differences in chemical composition, residual pigments, or interactions between pectin and corn starch [22].

UV-Vis Spectroscopy

Table 5 shows the UV-Vis spectra of the starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations. The control sample (COM), shows the lowest absorbance, indicating limited UV absorption. In contrast, the sample PHCL 1% exhibits the highest absorbance, demonstrating a significant improvement in UV-blocking capability. This enhanced performance is attributed to the bioactive compounds, such as phenolics and flavonoids, present in the papaya peel pectin, which are known for their UV-absorbing properties. This emphasizes pectin's UV-blocking abilities as pectin's effective absorption in 400 nm [16].

The PHCL 0.5% sample shows a moderate decrease in absorbance, suggesting a concentration-dependent effect of the pectin on UV absorption. Similarly, the samples extracted using citric acid PCA 1% and PCA 0.5% exhibit lower absorbance compared to their hydrochloric acid counterparts, indicating that the extraction method influences the composition and functionality of the pectin. This could be due to the higher efficiency of HCl in extracting phenolic compounds in papaya peel, which are known for their UV-absorbing [6].

Overall, the incorporation of papaya peel pectin into starch-based films enhances their UV-blocking properties, with higher pectin concentrations and HCl extraction yielding better results. Papaya peel pectin-enhanced films have UV-blocking properties because papaya has the properties of a natural UV barrier [9]. These films hold potential as biodegradable and sustainable packaging materials, offering UV protection for sensitive products.

Table 5. UV-Vis spectra of the starch-based films incorporated with pectin extracted from papaya peel using different acid hydrolysis at different concentrations.

Sample	Absorbance (400 nm)
PHCl 1%	1.502 ± 0.057^{a}
PHCl 0.5%	0.847 ± 0.013^{b}
PCA 1%	0.624 ± 0.035^{c}
PCA 0.5%	0.555 ± 0.007^{c}
COM	0.235 ± 0.022^{d}

Note: Values are expressed in mean ± standard deviation for three replications (n=3)
Values with the different superscript letters are significantly different at ($p<0.05$).

Tensile Strength

Fig. 3 shows the tensile strength (MPa) and elongation at break (EAB) of corn starch-based film incorporated with papaya peel pectin extracted using different acid hydrolysis at different concentrations. The control sample (COM), exhibits low tensile strength same as PCA 0.5% but high flexibility, as indicated by its high EAB%. The addition of papaya peel pectin enhances tensile strength, particularly in the sample with 1% pectin extracted using citric acid which shows the highest TS. This improvement is attributed to better intermolecular bonding between the pectin and starch matrix. However, as the pectin concentration decreases (PHC 0.5% and PCA 0.5%), the TS reduces, emphasizing the concentration-dependent effect of pectin [16]. Conversely, the flexibility of the films, represented by EAB%, decreases with the incorporation of pectin. The samples with hydrochloric acid-extracted pectin (PHCl 1% and PHCl 0.5%) exhibit the lowest EAB%, indicating increased rigidity due to the denser matrix formed.

In contrast, citric acid-extracted pectin samples (PCA 1% and PCA 0.5%) demonstrate higher EAB%, with PCA 0.5% showing flexibility comparable to the control. This suggests that citric acid extraction yields pectin that forms a more elastic matrix, albeit at the cost of lower tensile strength compared to hydrochloric acid-extracted pectin. Films with higher tensile strength have a lower elongation percentage, particularly with the presence of cross-linking agents, such as calcium in the pectin network [24].

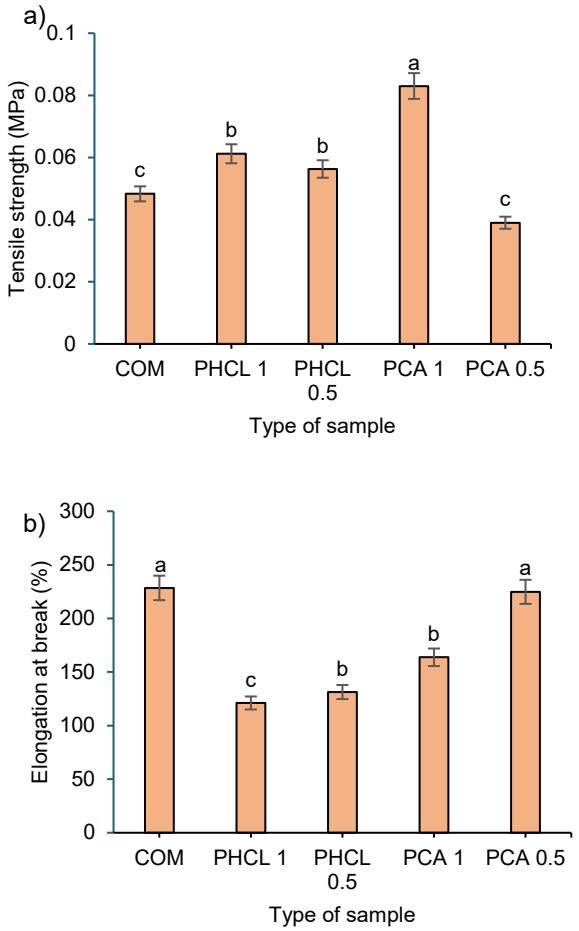


Fig. 3. (a) Tensile strength (MPa) and (b) elongation at break (EAB) of corn starch-based film incorporated with papaya peel pectin extracted using different acid hydrolysis at different concentrations. Note: Values are expressed in mean ± standard deviation for three replications (n=3). Values with different superscript letters are significantly different at ($p<0.05$).

Overall, the mechanical properties of the films depend on the type and concentration of pectin, as well as the extraction method. Hydrochloric acid-extracted pectin improves tensile strength, making the films more rigid and suitable for applications requiring structural integrity. On the other hand, citric acid-extracted pectin enhances flexibility, which may be desirable for applications requiring elasticity.

Fourier-transform infrared (FTIR) spectra analysis

The FTIR spectra in **Fig. 4 (a and b)** shows FTIR spectra of (a) papaya peel pectin extract with HCl and CA (b) starch-based film incorporated with papaya peel pectin extracted using different acid hydrolysis at different concentrations. The peak in a range of 1768 cm^{-1} to 1705 cm^{-1} shows a characteristic peak associated with methyl-esterified carboxyl groups (COO-R), which reduced in starch-based film incorporated with papaya peel pectin (**Fig. 4(a)**). The broad peak at 1680 cm^{-1} – 1600 cm^{-1} shows carboxylate group stretching in the pectin reducing its range in starch-based film incorporated with papaya peel pectin. **Fig. 4(b)** shows starch-based films with papaya peel pectin extracted using HCl (PHCl) and CA (PCA) revealing important functional group interactions and showing characteristic peaks of polysaccharides. Similar spectral profiles across all samples show similar chemical compositions between the control and film incorporated with pectin extracted from papaya peel. The spectra also show a strong and broad peak in the range from 3480 cm^{-1} to 3090 cm^{-1} due to hydrogen bonding facilitated by galacturonic acid. The hydroxyl group of pectin has no change after being added to starch-based films.

Furthermore, the prominent peaks in the 1200 to 900 cm^{-1} range, attributed to C-O-C stretching and glycosidic bonds, highlight the polysaccharide backbone's integrity, which increases in starch-based films incorporated with papaya peel pectin. This data was supported by [16] which obtained similar spectra for starch-based incorporated with pectin with peaks ranging from 1630 cm^{-1} to 1600 cm^{-1} stretching in the pectin film that represents carboxylate group [COO^-]. The reduction in peak 1700 cm^{-1} to 1750 cm^{-1} that represents the carbonyl stretching band in starch-based films is due to the formation of hydrogen bonds between the functional group of pectin, including methoxyl groups, hydroxyl (OH) and carboxyl (COOH) [25]. Peaks here often signify polysaccharide structures, such as starch and pectin and citric acid may enhance peaks due to esterification [26]. Overall, the spectra highlight significant chemical interactions but minimal structural changes in the starch-pectin films.

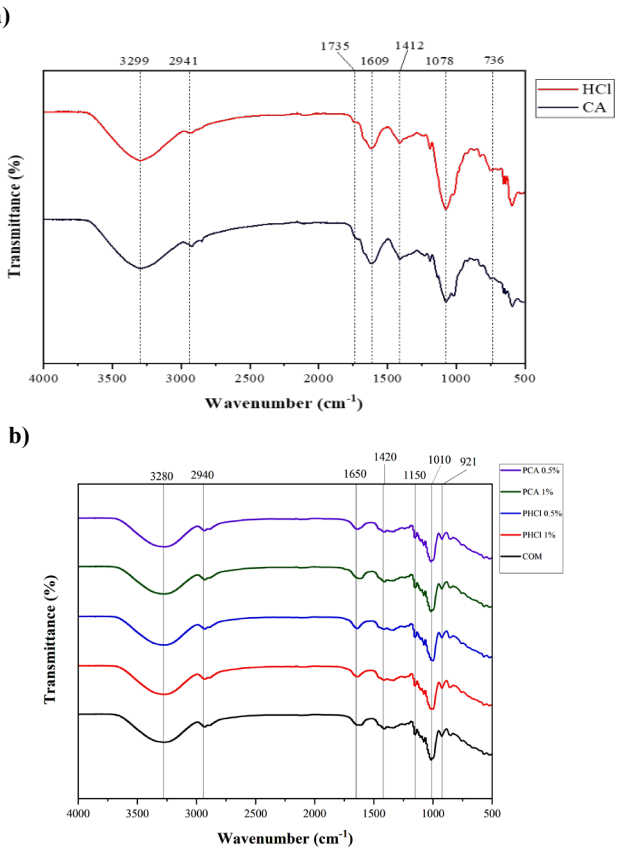


Fig. 4. FTIR spectra of (a) papaya peel pectin extract with HCl and CA (b) starch-based film incorporated with papaya peel pectin extracted using different acid hydrolysis at different concentrations.

Water Vapor Transmission Rate (WVTR)

Table 6 shows the water vapor transmission rate (WVTR) of starch-based films incorporated with pectin extracted from papaya peel via acid hydrolysis. The higher amount of pectin incorporated into the film resulted in a lower WVTR, which is the lowest WVTR from films with PCA 1% (1.778 ± 0.037 g mm/m² h kPa). This result obtained is because polysaccharides film is hydrophilic which are poor moisture barrier and from the polar group that interact with water molecules [27]. Moreover, 1% of pectin extracted with both acids incorporated into the film results in a lower WVTR may be due to calcium forming a tissue matrix that makes it difficult for water molecules to pass through [14].

To compare both films incorporated with pectin extracted using HCl and citric acid (CA), the film with citric acid-extracted pectin (PCA) 0.5% pectin concentration, was 3.446 ± 0.093 g·mm/m²·h·kPa, which is lower than that of the film with hydrochloric acid-extracted pectin (PHCl) at 4.938 ± 0.045 g·mm/m²·h·kPa. Increasing the pectin concentration to 1% further reduced the WVTR, with the PCA film reaching 1.778 ± 0.037 g·mm/m²·h·kPa compared to 2.743 ± 0.019 g·mm/m²·h·kPa for the PHCl film. The composite film (COM), likely incorporating multiple components or methods, exhibited the lowest WVTR of 1.576 ± 0.010 g·mm/m²·h·kPa, similar to the PCA 1% film, indicating enhanced synergy or densification within the film matrix. The result obtained in WTVR can be aligned with tensile strength which the increases of WTVR lead to the decrease of tensile strength due to the incorporation of pectin in films that enhance the permeability but compromise mechanical integrity [14].

Table 6. Water vapor transmission rate (WVTR) of starch-based films incorporated with pectin extracted from papaya peel via acid hydrolysis.

Sample	WVTR (g mm/m² h kPa) *
PHCl 0.5%	4.938 ± 0.045[a]
PCA 0.5%	3.446 ± 0.093[b]
PHCl 1%	2.743 ± 0.019[c]
PCA 1%	1.778 ± 0.037[d]
COM	1.576 ± 0.010[d]

Note: Values are expressed in mean ± standard deviation for three replications (n=3)

Biodegradable of films

Film biodegradation refers to the breakdown of the physio-mechanical properties of materials, the disintegration of films, and chemical transformations induced by microbial activity and enzymes in the environment [17]. **Fig. 5** shows the biodegradability of corn starch-based film incorporated with pectin extracted from papaya peel via acid hydrolysis. The percentage of weight loss in all films increased over time. After seven days, the rate of degradation increased significantly. The higher percentage of degradation observed in films with PCA 0.5% shows higher biodegradability compared to PHCl. The control sample (COM) exhibited the slowest biodegradability rate among all the films, showing that incorporating papaya peel pectin into the film enhanced the biodegradability. According to Shivangi et. al. [21], the degradation rates of films showed that pectin-based films resulted in higher degradation rates due to the natural and biodegradable nature of pectin which can easily break down.

Based on the FTIR spectra (**Fig. 4**), show broad peaks of hydroxyl groups (-OH), indicating -OH stretching vibrations prevalent in both starch and pectin. These groups enhance the hydrophilicity of starch- and pectin-based materials [28] facilitating water absorption, which is essential for microbial action during biodegradation. The degradation process involves microbial enzymes breaking down polymer chains, and the presence of -OH groups provide accessible sites for enzymatic attack, accelerating the process [29]. Pectin, known for its gel-forming ability, also contributes to the structural integrity of the composite, influencing the degradation rate when blended with starch.

Films containing pectin extracted with citric acid (PCA) showed higher biodegradability than films with HCl-extracted pectin (PHCl) due to differences in chemical properties. Citric acid, being a mild organic acid, preserves more of the natural pectin structure, including neutral sugars and side chains that enhance microbial recognition and degradation. In contrast, HCl a strong inorganic acid, can degrade the pectin structure more severely, reducing its biodegradability. Additionally, PCA-extracted pectin films are typically more hydrophilic and accessible to microbes, while PHCl-extracted pectin films tend to form denser, more hydrophobic films that resist microbial attack. The possible presence of residual citric acid in PCA films may also promote microbial activity by creating a slightly acidic environment. These factors collectively contribute to the higher biodegradability observed in PCA-based films.

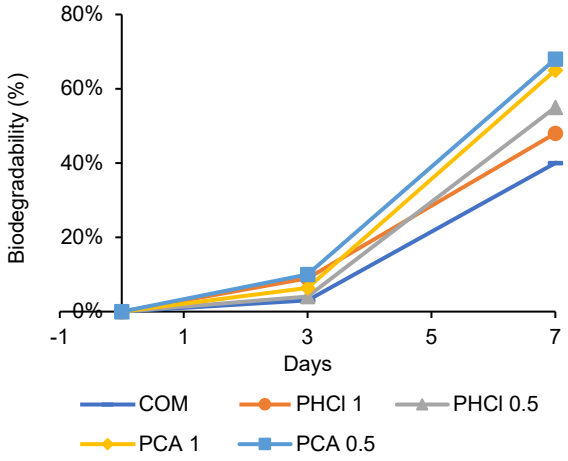


Fig. 5. Biodegradability of corn starch-based film incorporated with pectin extracted from papaya peel via acid hydrolysis.

CONCLUSION

Pectin was successfully extracted from papaya peel using acid hydrolysis (hydrochloric acid and citric acid). The results show that HCl provided a higher extraction yield of 9.1% compared to CA with 3.99%. The incorporation of papaya peel pectin into starch-based films was successfully achieved. The physical, mechanical, and biodegradable properties of the starch-based films were characterized, and it shows that the incorporation of papaya peel pectin into starch-based films improved the mechanical properties (tensile strength and elongation at break), light transmittance, and biodegradability compared to control. Additionally, the colour lightness (L*) increases as the % of pectin increases, especially for starch-based films incorporating papaya peel pectin extracted using HCl. Moreover, starch-based films containing pectin extracted with CA exhibited the optimum results, meaning they exhibited the best overall performance in terms of tensile strength, light transmittance, and biodegradability, which make the films more suitable for biodegradable packaging applications, as they combine strength, flexibility, and environmental sustainability.

ACKNOWLEDGMENT

The author would like to thank the Faculty of Fisheries and Food Science, University Malaysia Terengganu for the facilities provided and support. This work was partly funded by the Talent and Publication Enhancement Research Grant (TAPE-RG) (UMT/TAPE-RG/2022/55405) from Universiti Malaysia Terengganu.

REFERENCES

1. Mostafavi FS, Zaeim D. Agar-based edible films for food packaging applications: a review. Int J Biol Macromol. 2020;159:1165–76. 10.1016/j.ijbiomac.2020.05.123
2. Elsabee MZ, Morsi RE, Fathy M. Chitosan-oregano essential oil blends use as antimicrobial packaging material. 2016; Editor(s): Jorge Barros-Velázquez, Antimicrobial Food Packaging, Academic Press,. https://doi.org/10.1016/B978-0-12-800723-5.00044-9
3. Roy K, Thory R, Sinhmar A, Pathera AK, Nain V. Development and characterization of nano starch-based composite films from mung bean (*Vigna radiata*). Int J Biol Macromol. 2020;144:242–51. https://doi.org/10.1016/j.ijbiomac.2019.12.113
4. Sucheta, Chaturvedi K, Sharma N, Yadav SK. Composite edible coatings from commercial pectin, corn flour, and beetroot powder minimize post-harvest decay, reduce ripening, and improve the sensory liking of tomatoes. Int J Biol Macromol. 2019;133:284–93. https://doi.org/10.1016/j.ijbiomac.2019.04.132
5. Silva JAT, Rashid Z, Nhut DT, Sivakumar D, Gera A, Souza MT, et al. Papaya (*Carica papaya L.*) biology and biotechnology. Plant Biotechnol Rep. 2007;1(1):47–73. https://api.semanticscholar.org/CorpusID:54196276
6. Salla S, Sunkara R, Ogutu S, Walker LT, Verghese M. Antioxidant activity of papaya seed extracts against H_2O_2-induced oxidative stress in HepG2 cells. LWT - Food Sci Technol. 2016;66:293–7. https://doi.org/10.1016/j.lwt.2015.09.008
7. Gbenga-Fabusiwa FJ, Jeff-Agboola YA, Ololade ZS, Akinrinmade R, Agbaje DO. Waste-to-wealth: nutritional potential of five selected fruit peels and their health benefits: a review. Afr J Food Sci. 2022;16(7):172–83. https://doi.org/10.5897/AJFS2021.2138
8. Koubala BB, Christiaens S, Kansci G, Van Loey AM, Hendrickx ME. Isolation and structural characterization of papaya peel pectin. Food Res Int. 2014;55:215–21. https://doi.org/10.1016/j.foodres.2013.11.009
9. Ashfaq J, Channa IA, Shaikh AA, Chandio AD, Shah AA, Bughio B, et al. Gelatin- and papaya-based biodegradable and edible packaging films to counter plastic waste generation. Polymers (Basel). 2022;15(3):1046. https://doi.org/10.3390/ma15031046
10. Setyajati EF, Prasetyo KV, Husin SA, Ratri CM, Junedi S, Sanjayadi S, et al. Comparative physicochemical properties of isolated pectin from various tropical fruit peel wastes. Trop J Nat Prod Res. 2023;7(2):2408–13. http://.www.doi.org/10.26538/tjnpr/v7i2.17
11. Devi WE, Kumar RS, Mishra AA. Extraction of pectin from citrus fruit peel and its utilization in preparation of jelly. Int J Eng Res. 2014;3(5):1925–32. https://api.semanticscholar.org/CorpusID:87181381
12. Barrera-Chamorro L, Fernandez-Prior A, Rivero-Pino F, Montserrat-de S. A comprehensive review on the functionality and biological relevance of pectin and its use in the food industry. Carbohydr Polym. 2024;322:122794. https://doi.org/10.1016/j.carbpol.2024.122794
13. Yadav PK, Yadav AL, Yadav AS, Yadav HC. Effect of integrated nutrient nourishment on vegetative growth and physico-chemical attributes of papaya (*Carica papaya Linn.*) fruit cv. Pusa Dwarf. Plant Arch. 2011;11(1):327–9. https://www.cabidigitallibrary.org/doi/full/10.5555/20123402027
14. Halim Y, Darmawan CR. Characteristics of edible film made from pectin of papaya peel. Reaktor. 2021;21(3):116–23. https://doi.org/10.14710/reaktor.21.3.116-123
15. Fang Z, Yang Y, Lin S, Xu L, Chen S, Lv W, et al. Development and antimicrobial activity of composite edible films of chitosan and nisin incorporated with perilla essential oil–glycerol monolaurate emulsions. Food Chem. 2024;462:141006. https://doi.org/10.1016/j.foodchem.2024.141006
16. Said NS, Olawuyi IF, Lee WY. Tailoring pectin-PLA bilayer film for optimal properties as a food pouch material. Polymers. 2024;16(5):712. https://doi.org/10.3390/polym16050712
17. Abdillah AA, Charles AL. Characterization of a natural biodegradable edible film obtained from arrowroot starch and iota-carrageenan and application in food packaging. Int J Biol Macromol. 2021;191:618–26. https://doi.org/10.1016/j.ijbiomac.2021.09.141
18. Shakila BM, Bharathi KG, Gayatrri P, Nadezhda H, Nandhini J. Comparative studies of pectin yield from fruits using different acids. J Chem Pharm Res. 2012;4(1):6349–51. https://doi.org/10.15680/IJIRSET.2014.0308078
19. Bonrood D, Kamonrad R, Niamsup H. Extraction and physicochemical characteristics of acid-soluble pectin from raw papaya (*Carica papaya*) peel. Chiang Mai J Sci. 2006;33(1):129–35. https://api.semanticscholar.org/CorpusID:218840670
20. Altaf U, Immanuel G, Iftikhar F. Extraction and characterization of pectin derived from papaya (*Carica papaya Linn.*) peel. Int J Sci Eng Technol. 2015;3(4):2348–4098. https://api.semanticscholar.org/CorpusID:212520536
21. Shivangi S, Dorairaj D, Negi PS, Shetty NP. Development and characterisation of a pectin-based edible film that contains mulberry leaf extract and its bioactive components. Food Hydrocoll. 2021;121:107046. https://doi.org/10.1016/j.foodhyd.2021.107046
22. de Moraes Crizel T, de Oliveira Rios A, Alves VD, Bandarra N, Moldão-Martins M, Hickmann Flôres S. Active food packaging prepared with chitosan and olive pomace. Food Hydrocoll. 2018;74:139–50. https://doi.org/10.1016/j.foodhyd.2017.08.007
23. Rodríguez GM, Sibaja JC, Espitia PJP, Otoni CG. Antioxidant active packaging based on papaya edible films incorporated with *Moringa oleifera* and ascorbic acid for food preservation. Food Hydrocoll. 2020;103:105630. https://doi.org/10.1016/j.foodhyd.2019.105630
24. Seixas FL, Turbiani F, Salomão PG, Souza RP, Gimenes ML. Biofilms composed of alginate and pectin: effect of concentration of crosslinker and plasticizer agents. Chem Eng Trans. 2013;32:1693–8. https://doi.org/10.3303/CET1332283
25. Wang H, Li S, Wang S, Zhou J, Liu C, Chen C, et al. Development of controlled-release antioxidant poly(lactic acid) bilayer active film with different distributions of α-tocopherol and its application in corn oil preservation. Food Chem. 2023;439:138094. https://doi.org/10.1016/j.foodchem.2023.138094
26. Avramescu SM, Butean C, Popa CV, Ortan A, Moraru I, Temocico G. Edible and functionalized films/coatings—performances and perspectives. Coatings. 2020;10(7):687. https://doi.org/10.3390/coatings10070687
27. Darni Y, Dewi FY, Lismeri L. Modification of sorghum starch–cellulose bioplastic with sorghum stalk filler. J Rekayasa Kim Lingkungan. 2017;12(1):22–8. https://doi.org/10.23955/rkl.v12i1.5410
28. Zhang Y, Han JH. Plasticization of pea starch films with monosaccharides and polyols. J Food Sci. 2006;71(6):E253–61. https://doi.org/10.1111/j.1750-3841.2006.00075.x
29. Malekzadeh E, Tatari A, Dehghani Firouzabadi M. Effects of biodegradation of starch–nanocellulose films incorporated with black tea extract on soil quality. Sci Rep. 2024;14(1):16123. https://doi.org/10.1038/s41598-024-69841-2