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Characteristics of Edible Film Made from Pectin of Papaya Peel

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Abstract

Papaya (Carica papaya L.) peel contains a considerable amount of pectin, high molecular weight polysaccharides that can be used in edible film making due to its ability to form gels. However, edible film from pectin usually has poor moisture barrier properties. Therefore, pectin is generally combined with glycerol as plasticizer and starch. This research aimed to utilize pectin from papaya peel with the addition of corn starch in edible film making to determine the characteristics of pectin from papaya peel and the effect of pectin and corn starch concentration on edible film characteristics. Pectin extracted from papaya peel was classified as low methoxyl pectin (LMP). The pectin was then utilized in edible films making together with corn starch addition. Two factors were used in this research, which included pectin amount (0.75 g, 1.0 g, 1.25 g) and corn starch concentration (40%, 50%, 60%, based on pectin weight). The selected edible films formulation was an edible film made from a pectin amount of 1 g with 50% corn starch (based on pectin weight). This formulation showed low water vapor transmission rate (WVTR) of 3.447±0.270 g.mm/m^2/hour, a moderate tensile strength of 1.3121±0.0720 MPa, a moderate elongation percentage of 9.42±0.08%, and a thickness of 0.11±0.01 mm.

Keywords: *corn starch; edible films; papaya peel; pectin*

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INTRODUCTION

Edible films and coatings have been developed extensively nowadays because they offer several advantages over synthetic films, such as non-toxic and naturally biodegradable (Zhang *et al.*, 2016). Edible films can be prepared using hydrocolloids, such as proteins, polysaccharides, and alginate; lipids, such as waxes, fatty acids; and composites (Umaraw and Verma, 2017). However, the polysaccharides-based edible film is one of the main raw materials to obtain edible films due to its renewable characteristics and abundance in nature, with pectin as one of the most significant sources (Valdez *et al.,* 2015).

Pectin is a colloidal carbohydrate with a high molecular weight that naturally occurs in ripe fruits (Valdez *et al.,* 2015). Pectin is often used as a thickening agent in the preparation of jams, jellies, marmalades, and edible coatings due to its ability to form gels (Normah and Hasnah, 2000).

Papaya (*Carica papaya* L.) is often found in many tropical areas, including Indonesia. Production of papaya in Indonesia itself is up to 851,531 tons during 2015 (BPS, 2015). Ripe papaya is most commonly consumed as fresh fruit, whereas green papaya is as vegetable usually after boiling or cooking (Anuar *et al.*, 2008). The by-products of papaya fruit, such as papaya peels, which are approximately 20-25% of the fruit, are often discarded into the environment and can cause organic pollution (Patidar *et al.*, 2016). Meanwhile, papaya peels contain 4-19% of pectin (Yadav *et al.*, 2015), which can serve as the raw material for making edible films. The use of papaya peel for edible film making is still limited despite the high potential and availability of papaya peel.

Moreover, edible films made from polysaccharides are usually low efficient barriers against water transfer because of their hydrophilic nature (Valdez *et al.*, 2015). Still, they have better permeability to gases compared to plastic films. Pectin specifically can form clear and homogenous film, but it has poor moisture barrier properties (Umaraw and Verma, 2017). To overcome this, starch and plasticizer are often added to making edible films. The use of starches as one of the raw materials for films and coatings showed improvement on films' barrier to gasses and mechanical properties such as increase in elongation (Embuscado and Huber, 2009). In this research, corn starch was used because it contains ±25% amylose (Walker and Rapley, 2009), which is higher compared to tapioca starch (±17%) and sago starch (±21%) (Rolland-Sabate *et al.*, 2012). Amylose is associated with forming films and coatings due to its predominant linear nature (Embuscado and Huber, 2009). Previous research also mentioned that corn starch addition could increase the mechanical properties of films (Xu *et al.*, 2005), increase the elongation, however decrease the tensile strength of edible film formed (Wahyuningtyas and Dinata, 2018).

The addition of plasticizers is often done in edible film making because they are compatible with the polysaccharides to improve the film networks (Zhang *et al.*, 2016). The addition of plasticizers is known to enhance the flexibility of edible films (Lu *et al.*, 2009). Glycerol is the most used plasticizer as it is non-toxic, compatible with amylose, and could interfere with amylose chain packing. Glycerol gives a more plastic-like structure on edible films, specifically starch films. By increasing chain mobility in the starch network, a decrease in tensile strength and an increase in elongation are noticed (Bertuzzi *et al.*, 2012, Rachtanapun, 2009). Glycerol can produce clearer films. Therefore, the addition of glycerol to starch films can improve their optical properties and reduce their yellowness (Mohanty *et al.*, 2000).

This research aimed to utilize pectin from papaya peel with the addition of corn starch in edible film making. The characteristics of pectin from papaya peel and the effect of both pectin and corn starch concentration on edible film characteristics were also investigated.

RESEARCH METHOD

Materials and Equipment

Materials used in this research were papaya peel (*Carica papaya* L.) obtained from local market at Jakarta, corn starch, glycerol, $CaCl_2$, HCl (37%), NaOH, ethanol (97%), silica gel, and distilled water. Equipment used in the research were cabinet dryer, milling machine, filter cloth, texture analyzer "Lloyd" instrument (LR 50K), vacuum pump "Buchner", heater "Cimerec", thermometer, edible film applicator, drying oven "Memmert UNB 500", cooler "Sanden Intercool", sieve (35 mesh), Vernier calliper, and glasswares.

Extraction of Pectin from Papaya Peel

Extraction started with papaya peel powder making. Peel of papaya was washed, chopped, and dried at 60^{o}C for 6 hours in a cabinet dryer. Then, the dried peel was ground using blender and sieved with 35 mesh to obtain the powder with uniform degree of smoothness. (Rosida *et al.*, 2017). Papaya peel powder obtained was then analyzed for its moisture content (AOAC, 2005) and yield.

Furthermore, method for pectin extraction was done based on previous research conducted by Yadav *et al.* (2015) and Altaf *et al.* (2015) with modification. 50 g of papaya peel powder that was added with 1000 ml of distilled water. Then, HCl with 0.5 N concentration was added until the pH reached 1.5. The extraction was done by heating the mixture for 90 minutes at 80^{o}C. After extraction, the mixture was filtered using filter cloth to separate between the filtrate and the solids. The solids were then discarded. The remaining filtrate (pectin extract) was cooled, added with 96% ethanol, and kept for an hour. Filtration was done again to separate the coagulated pectin. The coagulated pectin was dried in oven at 50^{o}C for 6 hours. Dried pectin powder was obtained and characterized. The analyses included yield (%), moisture content (AOAC, 2005), ash content (AOAC, 2005), equivalent weight (Akili *et al.*, 2012), methoxyl content (Akili *et al.*, 2012), galacturonic acid content (Akili *et al.*, 2012), and degree of esterification (Akili *et al.*, 2012).

Preparation of Edible Film

The making of edible film was done according to Rosida *et al.* (2017) with modification. Formulation for edible film making can be observed on Table 1.

Table 1. Formulation for edible film making

Raw Materials	Unit	Amount
Pectin	gram	0.75, 1, and 1.25
$CaCl_2$*	%	0.064
Corn Starch *	%	40, 50, and 60
Plasticizer(glycerol) *	%	20
Distilled water	ml	50

Note: * based on weight of pectin
Source: Rosida *et al*. (2017) with modification

About 0.75g, 1 g, or 1.25 g of pectin was mixed with 25 ml of distilled water and added with 0.064% of $CaCl_2$. On a separate beaker glass, 40%, 50%, or 60% of corn starch (based on the weight of pectin) was dissolved in 25 ml of distilled water with heating. Corn starch is added to improve the mechanical properties of the edible film, such as tensile strength (Rachtanapun, 2009). Afterward, both mixtures were mixed and added with 20% of glycerol (based on the-weight of pectin). Glycerol is used as a plasticizer to improve the flexibility of edible film (Wahyuningtyas and Dinata, 2018). The mixture was heated at 80ºC for 15 minutes. The mixture was poured into a film applicator to shape the film. The film was then dried using the oven at 60ºC overnight. Some analyses were done on an edible film obtained, i.e., thickness (Guo *et al.*, 2012), water vapor transmission rate (WVTR) (ASTM, 1995), tensile strength (Dashipour, 2014), and elongation (Dashipour, 2014).

Experimental Design and Data Analysis

The experimental design used in this research was a completely randomized design with two factors and three replications. The first factor was the addition of pectin with 0.75 g, 1 g, and 1.25 g, and the second factor was the addition of corn starch with 40%, 50%, and 60% (w/w) based on pectin weight. All data obtained were analyzed using Univariate analysis and Post Hoc Duncan with SPSS version 22 software.

RESULTS AND DISCUSSION

Characteristics of Pectin Extracted from Papaya Peel Powder

The papaya cultivar utilized in this research was IPB-9 or more commonly known as "*Pepaya California*" which was grown in Bogor, Indonesia. The yield of papaya peel powder obtained was 10.77 ± 0.74%, and the moisture content was 13.26 ± 0.17%. The moisture content of papaya peel powder obtained in this research was higher than Didier *et al.* (2017), i.e., 9.82%. However, papaya peel powder can still be stored for a longer time because it is resistant to mold growth, as it has a low moisture content (5-15%) (Werthera *et al.,* 2000). Furthermore, pectin was then extracted from papaya peel powder. The yield and characteristics of pectin extracted from papaya peel powder are presented in Table 2.

According to Valdéz *et al.* (2015), yield of pectin extracted from fruit peel can be various, depending on the raw material, the extraction methods, and further chemical purification treatments. Based on Table 2, yield of pectin obtained in this research (19.32 ±2.08%) is higher compared to previous research by Sofiana *et al.* (2012), which was 2.835%-3.495% and Altaf *et al.* (2015), which was 2.8% -16%. Moreover, compared to other fruit peels, the pectin from papaya peel is higher, for example, when compared to dragon fruit peel, i.e., 10.40-16.76% (Tang *et al.,* 2011), mango peel (8.8%), banana peel (2.8%), apple pomace (12.5%) (Panchami and Gunasekaran, 2017) or squash (6.7%) (Daryono, 2012). However, the pectin yield of papaya peel is lower compared to pectin from grapefruit peel (26.74%) (Xu *et al.,* 2014) and orange peel (25.5%) (Panchami and Gunasekaran, 2017).

The moisture content of pectin obtained in this research was 7.33±0.31% which fulfilled the requirement by IPPA. Based on this research, the ash content of pectin extracted from papaya peel powder was 3.12±0.26%. The IPPA standard for ash content of pectin is at maximum 1%, meaning that the ash content did not meet with the standard. This could be caused by use of high temperature and heating in extraction process that can cause hydrolysis of protopectin (Budiyanto and Yulianingsih, 2008). Hydrolysis of protopectin resulted in an increasing amount of calcium and magnesium as protopectin can exist as calcium pectate or other salts (Reese, 2013). Increase in calcium and magnesium content, then increase the ash content.

Table 2. Yield and characteristics of pectin extracted from papaya peel powder

Parameter	Amount	IPPA standard
Yield (%)	19.32 ±2.08	-
Moisture content (%)	7.33±0.31	Max. 12%
Ash content (%)	3.12±0.26	Max. 1%
Equivalent weight (mg/mol)	1038.15±18.94	600-800 mg/mol
Methoxyl content (%)	2.28±0.12	2.0-7.1 % (low methoxyl)
Galacturonic acid content (%)	29.89± 0.83	Min. 35%
Degree of esterification (%)	43.15±1.40	< 50% (low methoxyl)

Notes: IPPA = International Pectin Producers Association

Equivalent weight measures free galacturonic acid content (not esterified) in the pectin molecular chain (Williams and Phillips, 2000). Based on this research, the equivalent weight of pectin extracted from papaya peel powder was 1038.15±18.94 mg, higher than IPPA standard. However, equivalent weight does not directly determine the purity or quality of pectin because it is further used for calculating and determining the galacturonic acid content and degree of esterification (DE). Those two parameters would determine the purity and gelling ability of pectin solution (Azad *et al.,* 2014).

Methoxyl content of pectin extracted from papaya peel powder in this research was 2.28±0.12%, with degree of esterification of 43.15±1.40%. Based on its methoxyl content and degree of esterification, pectin obtained in this research is classified as low methoxyl pectin (LMP). Low methoxyl pectin could form gels with lower concentrations of sugar in the presence of polyvalent cations for example calcium (Featherstone, 2016; Walter, 2012). Therefore, in this research, $CaCl_2$ is used in the formulation to assist gel formation from the pection obtained.

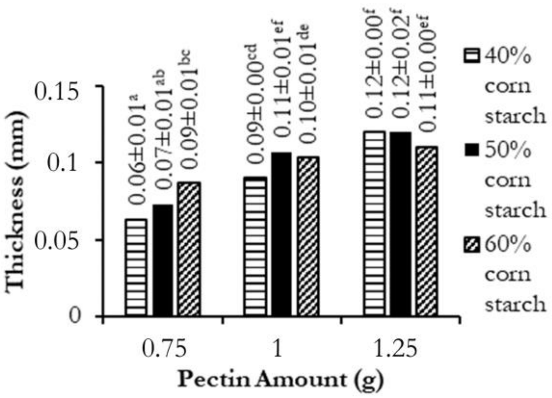
Pectin consists of a chain of galacturonic acid units as the structure backbone. Galacturonic acid content has an important role in determining the properties of pectin solution. Higher galacturonic acid content shows higher quality of pectin (Febriyanti *et al.,* 2018). It also affects the structure and texture of the gel formed by pectin (Constenla and Lozano, 2003). Based on this research, the galacturonic acid content of pectin extracted from papaya peel powder was 29.89± 0.83 %, lower than the standard for commercial pectin (min. 35%). The difference in galacturonic acid content is affected by extraction time, as longer extraction time results in higher galacturonic acid content (Maryati *et al.*, 2014). The difference could also be affected by the presence of sugars. Sugars such as D-galactose, L-arabinose, and L-rhamnose could be coagulated together with pectin when the coagulation process was done using ethanol (Akili *et al.*, 2012).

Edible Film Characteristics

Thickness

The thickness of the edible film is related with the permeability of the film or coating towards water vapour and gasses. It is also related with the ability of the film or coating to evenly protect the food product without altering its texture (Embuscado and Huber, 2009). Statistical analysis using Univariate analysis showed a significant effect ($p \leq 0.05$) of different pectin amounts, corn starch concentration, and interaction between pectin amount and starch concentration towards the thickness of the edible film. The results for the thickness of the films made of different formulations are presented in Figure 1.

Figure 1 shows that higher pectin and starch concentration resulted in a thicker edible film. According to Dhanapal *et al.* (2012), the total dissolved solids affected the thickness of edible film. Higher pectin amount and corn starch concentration increase total dissolved solid, leading to the production of thicker films.

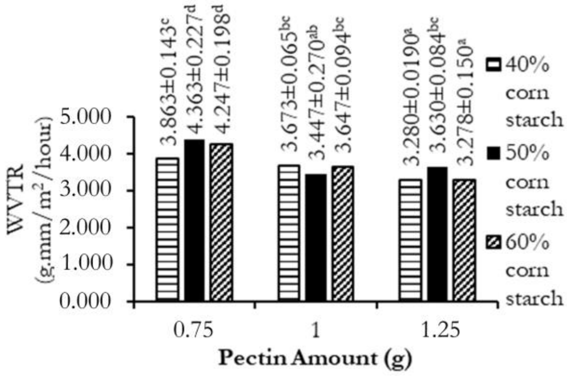


Note: Different superscripts indicate there is a significant difference ($p \leq 0.05$)

Figure 1. Effect of pectin amount and corn starch concentration on thickness of edible films

Water Vapor Transmission Rate (WVTR)

Water vapor transmission rate (WVTR) describes the transmission rate of moisture through a polymeric substrate at a controlled external temperature and humidity (Barbosa, 2008). Statistical analysis using Univariate analysis showed a significant effect ($p \leq 0.05$) of different pectin amounts, corn starch concentration, and interaction between pectin amount and starch concentration towards the WVTR of edible film. The results for WVTR of the films made of different formulations are presented in Figure 2.



Note: Different superscripts indicate there is a significant difference ($p \leq 0.05$)

Figure 2. Effect of pectin amount and corn starch concentration on water vapour transmission rate of edible films

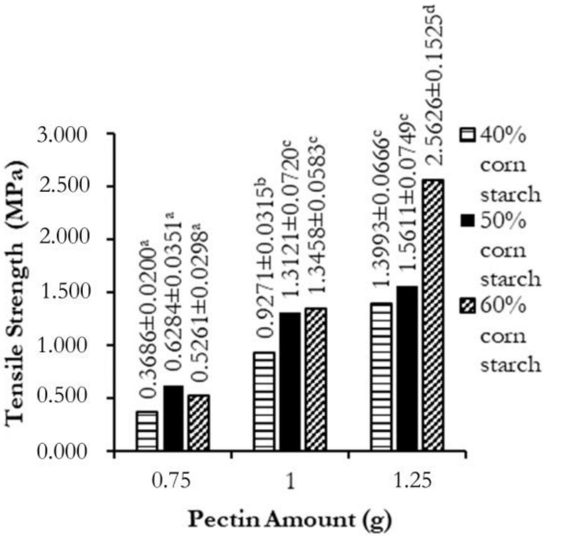
Figure 2 shows that a higher amount of pectin incorporated into the film resulted in a lower WVTR, with the lowest WVTR being 3.278±0.150 g.mm/m^2/hour, obtained from films made from 1.25 g pectin and 60% corn starch formulation. This result follows the research conducted by Darni *et al.* (2017), stating that generally, polysaccharides films are a poor moisture barrier because they contain hydrophilic polar groups that interact with water molecules. However, the water vapor permeability can be reduced by increasing the amount of filler in edible films. Higher concentration of pectin and calcium content incorporated into the film results in a lower WVTR because calcium will form a tissue matrix that makes it difficult for the water molecule to pass.

Moreover, in general, a higher concentration of corn starch tends to increase WVTR. The similar result was found by Rosida *et al.* (2017). This can be explained that starch and glycerol as plasticizers are hydrophilic; therefore, they can increase the WVTR. However, the WVTR of edible films obtained in this research is better than edible films made from papaya peel's pectin added with 5% cassava starch and 10% glycerol, which had WVTR of 116.963 g/m^2/24 hours or about 4.873 g/m^2/hour (Rosida *et al.*, 2017).

Tensile Strength

The tensile strength of the edible film is one of the mechanical examinations to determine the maximum load that can be applied to it before rupturing or tearing (Han, 2005). Statistical analysis using Univariate analysis showed a significant effect ($p \leq 0.05$) of different pectin amounts, corn starch concentration, and interaction between pectin amount and starch concentration towards the tensile strength of edible film. The results for tensile strength of the films made of different formulation are presented at Figure 3.

Figure 3 shows that increases in pectin and starch concentration resulted in higher tensile strength of the edible film. Crosslink structure from starch polymers and pectin-calcium complex would make -



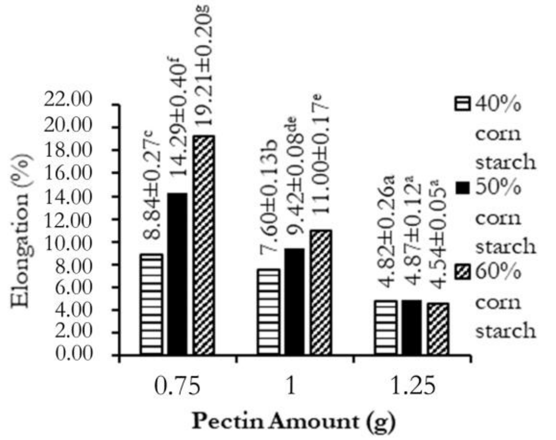
Note: Different superscripts indicate there is a significant difference ($p \leq 0.05$)

Figure 3. Effect of pectin amount and corn starch concentration on tensile strength of edible films

harder and stronger edible films (Seixas *et al*., 2013). Previous research showed that edible films made of corn starch without adding other compounds such as pectin resulted in low and poor tensile strength, ranging from 0.28-0.87 MPa (Godbillot *et al*., 2006). Tensile strength obtained in this research (0.3686 - 2.5626 MPa) is also higher than the edible film made from corn starch-CMC on previous research by Wahyuningtyas and Dinata (2018), i.e., about 0.125-0.5473 MPa.

Elongation

Elongation is one of the mechanical properties that helps to determine the flexibility and stretchability of films. Statistical analysis using Univariate analysis showed a significant effect ($p \leq 0.05$) of different pectin amounts, corn starch concentration, and interaction – between pectin amount and starch concentration towards the elongation of edible film. The results for elongation of the films made of different formulations are presented in Figure 4.



Note: Different superscripts indicate there is a significant difference ($p \leq 0.05$)

Figure 4. Effect of pectin amount and corn starch concentration on elongation of edible films

Figure 4 shows that higher concentration of pectin resulted in a lower elongation of the films. The highest elongation was 19.21±0.20% obtained from films with 0.75g pectin and 60% corn starch formulation. In comparison, the lowest elongation was 4.54±0.05% which was obtained from films with 1.25 g pectin and 60% corn starch formulation.

Increasing pectin concentration forms a strong pectin crosslink structure with calcium ions. It is due to the formation of intermolecular junction zones between pairs of carboxyl groups in the homogalacturonic smooth regions of pectins from different nearby chains (Lara-Espinoza *et al.*, 2018). As a result, edible films produced were stronger, however, they became more brittle and had poor elongation properties. However, this problem can be overcome by modifying the formulation with appropriate starch and plasticizer additions (Jantrawut *et al.*, 2017).

The addition of plasticizers, such as glycerol, can increase elongation because plasticizers can reduce molecules' force and increase polymer chain mobility (Darni *et al.*, 2017). As a result, the films formed will be more flexible (Wiset *et al.*, 2014). This research also shows that higher starch concentration resulted in a higher elongation of the films. Chiumarelli and Hubinger (2014) also stated that higher cassava starch concentrations with the same concentration of glycerol resulted in higher elongation percentage.

Generally, edible films with higher tensile strength have a lower elongation percentage. This follows previous research conducted by Seixas *et al.* (2013), where substituting starch with pectin at making edible film formulation would increase its strength but decrease its flexibility, particularly with the presence of the cross-linking agent, such as calcium in the pectin network. In this research, the formulation with the highest tensile strength (60% corn starch and 1.25 g of pectin) has the lowest elongation, i.e., 4.54±0.05%.

When compared to other research, the elongation of edible film in this research (4.54%-19.21%) is higher compared to the edible film made from konjac flour without glycerol addition, i.e., 2.63-3.57% (Wiset *et al.*, 2014), comparable to Rosida *et al.* (2017), i.e., 14.223% and Darni *et al.* (2017), i.e., 2.419-12.84%. However, it is lower than *Xanthosoma sagittifolium* starch-based edible film, i.e., 27.126-59.846% (Warkoyo *et al.*, 2014).

Selected Edible Film Formulation

The desired properties for the edible film are low water vapor transmission rate to prevent moisture loss from food that causes dehydration over the storage period, high tensile strength to protect the food from physical damage, and high elongation to produce a film with good elasticity and stretchability (Han, 2005). The edible film made from 1.25 g of pectin and 60% corn starch has the highest tensile strength and the lowest WVTR but exhibits the lowest elongation percentage. On the other hand, formulations that gave good elongation were from those with 0.75 g of pectin. However, they show poor tensile strength properties.

Therefore, the selected formulation in this research was the edible film with 1 g of pectin and 50% of corn starch. This edible film has WVTR that is not significantly different from the edible film made from 1.25 g pectin and 60% corn starch formulation, which was 3.447±0.270 g.mm/m^2/hour. This formulation also exhibits a moderate tensile strength of 1.3121±0.0720 MPa and elongation percentage of 9.42±0.08%.

CONCLUSIONS

Pectin extracted from papaya peel powder had yield of 19.32±2.08% and was classified as low methoxyl pectin (LMP) with degree of esterification of 43.35±1.40 %. Higher concentration of pectin and corn starch incorporated into the edible films resulted in a low water vapour transmission rate (WVTR) and high tensile strength, but lower elongation percentage. The concentration of pectin and corn starch that were chosen in edible film making was 1 g pectin with 50% of corn starch.

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