Formulation and Characterization of Edible Film from Sago Starch and Pectin from Mango Peel

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***Abstract*— Fruit waste are potential economical source for development of edible film for food packaging. In mango processing industries, mango peels are considered as waste and usually discarded. However, mango peels consist of numerous valuable component which can be used to develop edible film such as pectin. This study was carried out to extract pectin from mango peels, develop edible film from sago starch with incorporation of mango peels pectin and evaluate the effect on films mechanical, physical and barrier properties. Films were prepared using casting method with three different concentrations of extracted mango peels pectin (5 – 15% w/w starch). Pectin yield obtained in the extraction of pH 2.0 at 100 °C for 60 minutes was 19.5%. The finding in this study showed that the addition of pectin in sago starch solution results to increase in film tensile strength from 3.95 to 5.09 MPa. However, decrease in elongation was observed with increasing pectin content from 80.50% to 53.67%. On the other hands, increasing pectin content enhance water vapor permeability varying from 5.00×10^{-9} to 6.82×10^{-9} g s^{-1} m^{-1} Pa^{-1}. The solubility also increased with higher pectin content and temperature. Generally, the results shows that the film have low solubility in water. In addition, film with higher pectin content are more prone to yellowish in color. This study reveals the advantage of mango peels pectin incorporation into sago starch based films as a potential source for the production and development edible film.**

***Keywords*— sago starch, mango peels, pectin, edible film.**

I. INTRODUCTION

Significant researches have been conducted to develop a biodegradable, safe and environmental friendly packaging to substitute of plastic for food packaging. Edible film have been developed as an alternative packaging to partially replace synthetic plastic. Edible film can be consumed with the product and even if it is not consumed it can contribute to the reduction of environmental pollution. They are expected to degrade easily as it is made from edible ingredients [7]. The utmost benefits of edible films are their biodegradability as well as their edibility. Fruit waste and by-product including mango peels extract [14], banana peels pectin [2], papaya peels pectin [19] and *durian* peels pectin [4] have been studied for the development of edible film due to the presence of valuable properties. Fruits waste offers advantageous such as reduction of manufacturing cost and enhanced functional properties of the films.

Natural component that presence in fruit waste can be alternative sources for the formulation of edible film. For instance, industrial processing and consumption of mango produce around 20 – 25 % of mango peels which considered as waste [15]. However, mango peels consist of several valuable compound including polysaccharides, pectin, dietary fibers, and phenolic compound [15], [21], [23]. Pectin from mango peel is one of promising sources that can be used for the formulation of edible film. Serna *et al.* (2016) reported that the major component of soluble dietary fiber in mango peels is pectin [21]. Pectin is suitable material for edible film formulation due to its gelling and emulsifying properties. Girma and Worku, (2016) found that pectin from mango peels are comparable to apple pomace and citrus peel which are used for commercial pectin production in terms of yield and quality [11]. Previous studies utilized mango peels for edible film formulation [14], [23]. The studies highlighted that the naturally existing pectin in mango peels have good effect towards edible film properties due to its thickening, emulsifying and gelling properties. The study also reported that edible film from mango residues have good permeability, antioxidants, and color [23]. The films rigidity also improved with the incorporation of mango peels extract [14].

Several studies have been conducted regrading pectin-starch mixtures to form edible film from various starch including cassava and corn starch [4], [8[, [9], [19]. However, sago starch-pectin edible film has not been reported for the formulation of edible film. Sago starch film shows a superior mechanical strength, barrier properties and have low solubility in water [17]. In addition, high content of amylose in sago starch is expected to contribute to enhance mechanical properties of the film. Therefore, the combination of starch and mango peel pectin expected to produce a good film properties and characteristic. Hence, the purpose of this study is to: (a) extract pectin from mango peels, (b) develop edible films from sago starch with addition of mango peel pectin, and (c) characterize the effect of pectin addition on the mechanical, physical, barrier and optical properties of the film.

II. METHODOLOGY

A. Materials

Mangoes (*Mangifera indica* L.) were purchased from a local market in Gombak, Selangor in April 2019. Sago starch was purchased from EzyMix Ingredient House (Kepong, Selangor) whereas ethanol 95% were obtained from Jana Supplier, (Skudai, Johor). Sulfuric acid 99% and silica gel were obtained from chemistry laboratory UiTM Shah Alam, Selangor. All chemicals used were analytical grade.

B. Pectin Extraction

Mango peels were manually separated and immediately washed with tap water. Then, the peels were rinsed using distilled water.

The cleaned peels were dried in the oven (Memmert, UFE500) at 60°C for 24 hours and subsequently grounded to mill (Panasonic, MX800S). Dried mango peels powder were kept at room temperature in an airtight and moisture-proof container until used.

Pectin was extracted from dried mango peels according to Patel (2017) methodology with slight modification [15]. Mango peels were mixed with water at pH 2.0 with substrate to water ratio 1:40 (w/v). The desired pH for water were adjusted with 99% sulfuric acid and measured using pH meter (Metler Toledo, EMP-FE20). Thereafter, the solution was heated at 100°C for 60 minutes with frequent stirring. The mixture was then filtered using filter cloth and subsequently precipitated with equal volume ethanol 95% for 3 h. The obtained pectin is filtered and dried in oven at 40°C overnight. Yield of pectin was determined using the formula as follows.

$$Y_{pec}\,(\%) = (P / P_i) \times 100\%$$

Where, Y_{pec} is the yield of extracted pectin in percentage (%); P represents the amount of dry pectin obtained (g); and P_i is the initial amount of dried mango peels used (g).

C. *Film Formulation*

Film formulation was prepared according to the method of Poeloengasih and Anggraeni (2014) and Fishman *et al*. (1996) with some modification [8], [17]. Starch solution was prepared by dissolving sago starch (6% w/v solution) in distilled water and heated to 75°C with frequent stirring. When the temperature reach 75°C, glycerol (25% w/w starch) was added into the starch solution and the heating continued for 30 minutes at maintained temperature of 75°C. Pectin solution were prepared by dissolving different percentage of mango peel pectin (5, 10 and 15% w/w sago starch) in 20 mL distilled water at room temperature with frequent stirring. Control films were prepared using the same method without addition of mango peel pectin

After gelatinization of starch is achieved, pectin solution was added into starch solution and subsequently heated at 70°C for 5 minutes with frequent stirring. The mixture was then cooled to room temperature and subsequently poured into petri dishes (diameter 9 cm). To control film thickness, the amount of film forming solution distributed in petri dishes were fixed at 20 mL. Lastly, the solutions were dried at 30 °C for 96 hours. Prior to analysis, the film is conditioned at a temperature of 25 ± 2°C with a relative humidity of 70 ± 5% for 48 hours.

D. *Film Characterization*

Thickness

A hand-held digital micrometer (Mitutoyo, No. 293-340-30) was used for the measurement of film thickness to the nearest 0.001 mm. Ten random positions were measured on each specimen and a mean value of thickness was calculated.

Color Measurement

Color measurement of film were evaluated using chromameter (Model A60-1012-402). The measurement was taken at three random positons on film sample and a mean value of parameter L*, a*- and b*- value which indicated film lightness, redness/greenness and yellowness/blueness were recorded.

Water Vapor Permeability

Water vapor permeability of film was determined according to Al Hassan *et al.* (2012) using modified ASTM E-96-00 method [3], [6]. Glass cup with diameter 6.4 cm was used. The cups were filled with 20 g silica gel (0% RH), covered and sealed with test films using a rubber gasket. The cells were placed in a desiccator containing distilled water (100% RH) and kept at temperature 25 ± 2°C. The weight of test cup were recorded at 1 hour interval over 9-hours period. The cups weight were recorded to the nearest 0.0001 g and plotted as a function of time. The slope was determined by linear regression. The water vapor permeability of the film was calculated as follows.

$$WVP = (WVTR\ x) / \Delta p$$

Where, WVP is water vapor permeability (g s^{-1} m^{-1} Pa^{-1}); WVTR is the water vapor transmission rate (g m^{-2} s^{-1}) through the film, calculated from the slope of the straight line per exposed film area (m^2); x represents the mean thickness of the film (m); and Δp is the partial water vapor pressure difference (Pa) across two sides of the films.

Film Solubility

Film solubility was determined according to Perez-Gago and Krotcha (2001) methodology with slight modification [16]. Film samples of 20 × 20 mm were prepared, weighed and immersed in 50 mL of distilled water for 5 minutes at 25, 37 and 90 °C. The undissolved portion of the films was filtered and dried for 24 h at 40°C. Experiments were carried out in triplicate. The solubility of the films was determined as follows.

$$\text{Solubility}\,(\%) = \frac{W_i \times W_f}{W_i} \times 100\%$$

Where, W_i is the initial weight of film samples and W_f is the final weight of dried film sample.

Mechanical Properties

Tensile strength and elongation of films were determined according to Saberi *et al.* (2016) using modified ASTM D882-00 method [5], [20]. Film samples of 60 × 20 mm were prepared and conditioned in a desiccator at room temperature and 75% RH for 72 h prior to testing. The test were performed using Instron Universal Testing Machine (Model 55669) with load cells of 5 kN. The crosshead speed and initial grip were set at 10 mm/min and 40 mm respectively. Measurements were carried out in triplicate.

III. RESULTS AND DISCUSSION

A. *Yield of Pectin*

Pectin was extracted from mango peels at 100ºC for 60 minutes with sulfuric acid at pH 2.0. The acidity of the solution are essential parameter as it affects pectin yield. Sulfuric acid results to better extraction of pectin due to the presence of sulfate ions in soaking solution [15]. The optimum condition for pectin extraction from mango peels is at pH 2.0 [11], [15]. At higher pH, pectin yield significantly decreased due to pectic substance breakdown. Meanwhile, high temperature could attribute to pectin molecule breakdown [18]. The optimum condition as reported from previous studies were applied in this studies for pectin extraction from mango peels. The average pectin yield obtained are 19.5 % as shown in Table 1.

The extracted pectin from mango peels are brownish in color. Similar results was reported by Patel (2017) and Amaliyah (2014). Drying process of mango peels prior extraction might change the natural color of mango peels which could attributes to the color properties of the extracted pectin [4]. Oxidation might occur during drying process due to presence of oxygen which lead to browning. Mango peels can be soaked in sodium metabisulfide solution to prevent oxidation [1]. Sodium metabisulfide is often used as antioxidant and preservative agent. Other studies stated that extracted pectin can be rinsed using ethanol for purification process until the brownish color disappear to obtain white color [4]. However, ethanol can lighten the extract color lead to reduction in the quality [4]. The color of extracted pectin might affect color parameter of the films produced.

Table 1: Experimental results of pectin extraction

Parameter	Values
Amount of mango peels used (g)	10
Amount of dried pectin obtained(g)	1.95
Pectin yield (%)	19.5

B. *Film Characterization*

From the macroscopic point of view, films were highly detachable from the plates and flexible. The flexibility of films are decent due to presence of glycerol as plasticizer in the film. Film formed without plasticizer are brittle and easily broken during the peel off process. The addition of plasticizers reduced intermolecular forces along the polymer chain which subsequently enhance chain mobility and flexibility [10].

Thickness

The thickness of the films is one of the important parameter that affect the properties of the film [14]. Table 2 shows that addition of 5% of pectin increase film thickness compared to control film but not significantly. However, addition of 10-15% of pectin showed significant increase in film thickness compared to control film. The thickness of control film increased almost 20% with the incorporation of 15% mango peel pectin. The increase in film thickness could be due to increase in compactness of film with increasing concentration of pectin incorporated. Furthermore, the addition of pectin resulting to higher dissolved solid in the solution which subsequently increase the thickness. Thicker films theoretically influence the mechanical properties and water vapor permeability due to the interaction in the film matrix [14].

Color Measurement

The optical properties are an essential characteristic of edible film, particularly for the film that used as the packaging of food that sensitive to degradation by light [23]. Color parameter of the films are presented in Table 2. Addition of mango peel pectin from 5 to 15% showed decrease in L* and –a* value. Meanwhile, the addition of mango peel pectin has increased the b* value showed that the films were prone to yellowish color and increase toward lighter chroma [13]. The increase value of b* are due to the presence of natural yellow coloration pigment present within the extract of mango peels pectin [23]. The dominant carotenoid present in ripe mango is β-carotene which resulting the peels exhibited yellowish color [14].

Although colorless and transparent materials are usually used for food packaging, colored film are preferred for the prevention of ultraviolet and visible rays exposure which may lead to food deterioration [14].

Water Vapor Permeability

The WVP of films incorporated with various concentrations of mango peel pectin are tabulated in Figure 1. Water Vapor Permeability represents the moisture ability to penetrate a material. This is one of the essential properties that have to be measured in determining the quality of food packaging. Food packaging with decent water barrier properties can prevent loss of moisture from food and prevent moisture from atmosphere from migrating into the food, which speed up microorganism growth and reduce shelf-life of the food. For this reasons, lower WVP values considered ideal. The trends from results shows that WVP increased with addition of pectin.

Pectin itself are naturally hydrophilic, therefore, this characteristic probably responsible for the increase in WVP of film with higher addition of pectin. Pectin Similar results was reported by Amaliyah (2014) on *Durian* and *Cempedak* pectin film [4]. They observed that the increase of pectin up to 15% did not give significant change toward film WVP.

Film Solubility

The solubility of sago starch film with addition of various concentrations of mango peels pectin are tabulated in Figure 2. The increase in temperature resulting to higher solubility for all films. Film incorporated with 5% of pectin showed a significant increase in solubility compared to control film. Meanwhile, the incorporation of 10 – 15 % of mango peels pectin also improve the

Table 2: Physical and mechanical properties of films

Films	**Thickness (mm)**	**Color**			**Elongation at break (%)**	**Tensile Strength (MPa)**
		L*	**a***	**b***		
Control	0.1465 ± 0.0138	33.40 ± 0.39	-0.25 ± 0.03	0.12 ±0.04	80.50 ± 5.08	3.95 ± 0.11
Pectin 5%	0.1575 ±0.0073	32.77 ±0.48	-0.49 ± 0.03	2.46 ±0.15	74.42 ± 4.30	4.38 ± 0.15
Pectin 10%	0.1732 ±0.0163	32.26 ± 0.67	-0.69 ± 0.04	4.48 ± 0.58	58.58 ± 2.54	4.66 ± 0.15
Pectin 15%	0.1936 ± 0.0060	31.40 ± 0.46	-0.75 ± 0.04	6.70 ±0.34	53.67 ±4.64	5.09 ± 0.23

solubility of the film, however the value are not significant. The increase in solubility probably due to presence of pectin which have high solubility [12]. Besides, the addition of pectin results to increase of dry matter that solubilized in water which subsequently improves the solubility of the film.

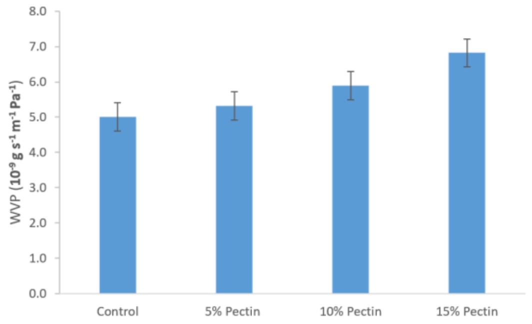

Fig. 1: Water vapor permeability of films of sago starch film with addition of 5. 10 and 15% mango peel pectin. Control: no pectin added.

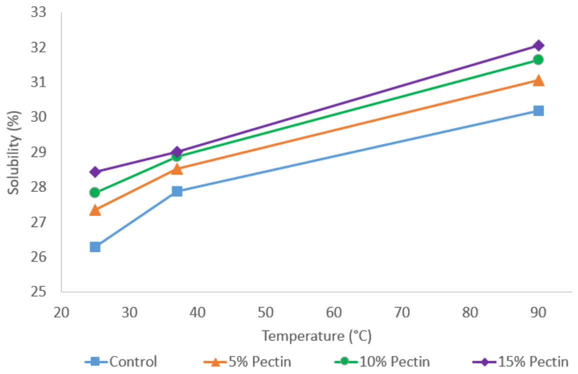

Fig. 2: Solubility of films of sago starch film with addition of 5. 10 and 15% mango peel pectin at temperature 25, 37 and 90 °C. Control: no pectin added.

Mechanical Properties

Tensile strength is one of the important properties of edible films. Results obtained tabulated in Table 2 showed that the tensile strength of control film was 3.95 MPa. The addition of pectin has significantly improved the tensile strength up to 5.09 MPa. The increase in tensile strength could be due to higher polysaccharides content which subsequently improve the formation of film structure. The lower TS value of control film is because the larger molecular spaces [12]. Sothornvit and Pitak (2007) reported that small amount of pectin can enhanced the tensile strength of film significantly [22]. Similar results was also reported by Fishman and Coffin (1995) on pectin-starch film [9].

On the contrary, higher pectin content leads to reduction in elongation of the film. Control film showed the highest value of elongation, 80.50%. The addition of 10% of pectin results to decrease in the elongation value to 58.58% which is around 37% reduction compared to control film. Film with addition of pectin may have denser structure of polymeric matrix [14].

IV. Conclusion

This study revealed that the addition of mango peels pectin in sago starch film resulting to a thicker film. The addition of pectin extract from mango peels contributed to improvement of the film tensile strength compared to the control film. Further research should explore the potential of mango peels pectin as a food packaging and optimize variables for film formulation.

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References

[1] Abdul Aziz, N. A., Wong, L. M., Bhat, R., Cheng, L. H. Evaluation of processed green and ripe mango peel and pulp flours (Mangifera indica var. Chokonan) in terms of chemical composition, antioxidant compound and functional properties. *Journal Science Food Agriculture*, vol. 92, pp. 557 – 563, 2012.

[2] Akili, M. S., Ahmad, U., Suyatma, N. E. Characterization of Edible Film Based on Pectin Extracted from Banana Peel. *Jurnal Keteknikan Pertanian*, vol. 26, no.1, pp. 39-46, 2012.

[3] Al-Hassan, A. A., Norziah, M. H. Starch-gelation edible film: Water Vapor Permeability and Mechanical Properties as Affected by Plasticizers. *Food Hydrocolloids*, vol. 26, pp. 108 – 117, 2012.

[4] Amaliyah D. M. Utilization of Durian and Cempedak Peels Waste as an Edible Film. *Jurnal Riset Industri Hasil Hutan*, vol. 6, no. 1, pp. 27-34, 2014

[5] ASTM. (2000a). *Standard test methods for tensile properties of thin plastic sheeting, method d883-00.* Philadelphia, PA. American Society for Testing and Material.

[6] ASTM. (2000b). *Standard test method for water vapor transmission of materials, method E 96-00.* Philadelphia, PA. American Society for Testing and Material.

[7] Bourtoom, T. Edible Films and Coatings: Characteristics and Properties. *International Food Research Journal*, vol. 15, No. 3, pp. 237-248, 2008.

[8] Coffin, D. R., Fishman, M. L. Mechanical Properties of Pectin-Starch Film.ACS Symposium Series; American Chemical Society: Washington, DC, 1994.

[9] Fishman M. L., Coffin D. R., Unruh J. J., T.LY. Pectin/Starch/Glycerol Films: Blends or Composites. *Journal of Macromolecular Science*, vol. 133, No. 5, pp. 639-654, 1995.

[10] Galus, S., Uchanski, P., Lenart, A. Colour, Mechanical Properties and Water Vapor Permeability of Pectin Films. *Acta Agrphysica*, vol. 20, No. 3, pp. 375-384, 2013.

[11] Girma E., Worku T. Extraction and Characterization of Pectin from Selected Fruit Peel Waste. *International Journal of Scientific and Research Publication*, vol. 6, No. 2, pp. 447 – 454, 2016.

[12] Meneguin, A. B., Ferreira Cury, B. S., Evangelista, R. C. Film from resistant starch-pectin dispersions intended for colonic drug delivery. *Carbohydrates Polymer*, vol. 99, pp. 140-149, 2014.

[13] Moalemiyan, M., Ramaswamy, H. S., Maftoonazad, N. Pectin-based Edible Caoting for Shelf-life extention of Ataulfo Mango. *Journal of Food Process Engineering*, vol. 1, pp. 1-18, 2011

[14] Nor Adilah, A., Jamilah B., Noranizan, M. A., Nur Hanani Z. A. Utilization of mango peel extracts on the biodegradable films for active packaging. *Food packaging and Shelf Life*, vol. 16, pp. 1 – 7, 2018.

[15] Patel, P. M. Extraction of Pectin from Mango Peel and Application of Pectin. *Journal of Natural Products and Resource*, vol. 66, pp. 1103-1108, 2017.

[16] Perez-Gago, M. B., Krochta, J. M. (2001). Denaturation Time and Temperature Effects on Solubility, Tensile Properties, and Oxygen Permeability of Whey Protein Edible Films. *Food Engineering and Physical Properties*, vol. 66, No. 5, pp. 705 – 510, 2001.

[17] Poengelasih, C. D., Anggraeni, F. D. Exploring the characteristic of sago starch films for pharmaceutical application. *Starch/Stärke*, vol. 3, No. 1, pp. 102-103, 2014.

[18] Rehman, Z. U., Salariya, A. M., Habib, F., Shah, W. H. Utilization of mango peels as a source of pectin. *Journal Chemical Society of Pakistan*, vol. 26, No. 1, pp. 73 – 76, 2004.

[19] Rosida, Sudaryati, Yahya, A. M. Edible Film from the Pectin of Papaya Skin (The Study of Cassava Starch and Glycerol Addition). Journal of Physics: Conf. Series, 953 012248, 2017

[20] Saberi, B., Vuong, Q., Chockchaisawasdee, S., Golding, J., Scarlett, C., Stathopoulos, C. Mechanical and physical properties of pea starch edible films in the presence of glycerol: Properties of Pea Starch Edible Film. *J. Food Process. Preserve*, vol. 40, No. 6, 2016.

[21] Serna, L., Garcia, E., & Torres, C. Agro-industrial potential of mango peel based in its nutritional and functional properties. *Food Reviews International*, vol. 32, pp. 364-376. 2016.

[22] Sothornvit, R., Pitak, N. Oxygen Permeability and Mechanical Properties of Banana Films, *Food Research International,* vol. 40, No. 3, pp. 365 – 370, 2007.

[23] Torres-León, C., Vicente, A. A., Flores-López, M. L., Rojas, R., Serna-Cock, L., Alvarez-Pérez, O. B., Aguilar, C. N. Edible films and coating based on mango (var. Ataulfo) by-products to improve gas transfer rate of peach. *Food Science and Technology*, vol. 97, pp. 624 -631, 2018.