PHYSICOCHEMICAL CHARACTERISTICS OF *BABY JAVA* ORANGE PEEL PECTIN *(Citrus sinensis)* AND CORN STARCH-BASED EDIBLE FILM WITH GLYCEROL PLASTICIZER

Lilis Suryani Karlan, Safinta Nurindra Rahmadhia *

Program Studi Teknologi Pangan - Fakultas Teknologi Industri - Universitas Ahmad Dahlan Yogyakarta
Jl. Ringroad Selatan, Kragilan, Tamanan, Kec. Banguntapan, Bantul, Daerah Istimewa Yogyakarta 55191
* Corresponding author, email : safinta.rahmadhia@tp.uad.ac.id

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ABSTRACT

Baby Java orange is one sweet orange variant with a high pectin content on its skin. Pectin and starch are examples of the main ingredients in making edible films. Besides its economical price, corn starch is also commonly used in preparing edible films as the biopolymer material that can produce a matrix used for the edible film preparation process. The addition of glycerol plasticizers can improve the structure of the film. This study aimed to determine the effect of pectin concentration of *Baby Java* orange peel and glycerol on the physicochemical characteristics of the resulting edible film and determine the formulation with the highest value in the manufacture of the edible film according to the Japanese Industrial Standard (JIS). This study used a Completely Randomized Design (CRD) with two factors. The first factor is *Baby Java* skin pectin concentration (15%, 25%, and 35% %b b/b starch), while the second factor is glycerol concentration (10%, 20%, and 30% v/v). The edible films were then analyzed for their moisture content, thickness, solubility, color, tensile strength, and water vapor permeability. The results show that the concentration of pectin and glycerol with the addition of corn starch significantly affects the parameters of moisture content, solubility, thickness, tensile strength, color, and water vapor permeability tests. The optimum formulation in the preparation of edible films according to JIS was the formulation with the addition of 15% pectin and 20% glycerol, which resulted in 6.24% of moisture content, 76.57% solubility, 0.25 mm thickness, 0.50 MPa tensile strength, 54.98 lightness, 3.66 blueness, -1.48 appearances and 1.42 g.mm/m^2.h.kPa water vapor permeability

Keywords: Baby Java Orange Peel; Corn Starch; Edible Film; Glycerol; Pectin

INTRODUCTION

Baby Java orange has a round shape and thick skin. There is a reasonably high proportion of pectin content in orange peel. Sweet orange is one type of orange peel containing large amounts of pectin. *Baby Java* orange is one of the sweet orange variants. According to Hashmi *et al.* (2012), the percentage of sweet orange peel pectin content is 20.12%. Pectin is a natural substance found in food crops. It also acts as adhesives and maintains the stability of tissues and cells. Pectin includes hydrocolloid ingredients, a carbohydrate modification, such as starch, alginate, Arabic gum, etc. Therefore, pectin can be used for making edible films (Rachmawati, 2009; Espitia *et al.*, 2014; Chen *et al.*, 2020). Pectin can be found in orange peel, one of which is *Baby Java* orange. The pectin can be taken by extracting it from *Baby Java* orange peel.

Edible films are made of materials that can be consumed and formed into thin layers, which are used to coat food and inhibit the transfer of aroma, carbon dioxide, oxygen, moisture, and various dissolved substances in food (Baldwin *et al.,* 1994). One of the essential ingredients of edible films is starch and pectin. The edible film made of pectin can extend the shelf life of packaged food products because pectin is a polysaccharide derivative that functions as a permeability membrane (Mirzayanti, 2013). In addition to its economical price, corn starch is also commonly used to prepare edible films as the biopolymer material that can produce a matrix used for the edible film making process. According to Sandhu and Singh (2007), among the various types of starch, the corn starch that can be used in making film matrices is the one that has a hydrocolloid component. The reason for using corn starch is due to its 25% of amylose level, which is higher than other starches. This percentage of amylose content has a high potential to form a film, and the film produced will be stronger than starches with lower amylose content. Thus, the more starch is used, the more compact structure the film matrix is formed (Murni *et al.,* 2013). Edible films derived from starch generally have brittle properties. However, adding glycerol, which serves as a plasticizer, will improve the film structure.

The addition of glycerol plasticizer can produce a firmer and smoother edible film. Moreover, glycerol strongly influences changes in the film's physical properties (Ariska and Suyatno, 2015). Glycerol also takes a part in increasing the flexibility of the film. Glycerol is suitable to be used as an ingredient in making hydrophilic films such as starch since glycerol is a hydrophilic plasticizer (Damar and Krisna, 2011). According to Coniwanti *et al. (*2014), edible films added with glycerol significantly affect the use of raw materials such as starch. Glycerol is more advantageous than sorbitol since it is more soluble in water (hydrophilic) and miscible in film solutions. Based on the existing background, this study was conducted to prepare edible films with pectin ingredients derived from *Baby Java* orange peel with the addition of corn starch. In order to improve its physical properties, glycerol, which serves as a plasticizer was added.

METHOD

The equipment and instruments used in this study were an oven (Memmert NU 50), cabinet dryer, blender (Philips), 60 mesh sieve, desiccator, micrometer, chromameter (NH 310) and universal testing machine (Zwick).

The main ingredients in this study were *Baby Java* orange peel obtained from Gamping Yogyakarta regional supermarket, corn starch (Tereos), and chemicals, namely glycerol 10%, glycerol 20%, glycerol 30%, NaCl, ethanol 95%, ethanol 70%, aquadest, filter paper and silica gel.

The Preparation of *Baby Java* Orange Peel Powder

The preparation of *Baby Java* orange peel powder used the modified method of Syarifuddin and Yunianta (2015). *Baby Java* orange peel was cleaned with running water and cut into small pieces (2x2 cm). Then, steam blanching was carried out at 85 ºC for 10 minutes, followed by draining. The *Baby Java* orange peel was then put into the dryer cabinet for 10 hours at 55 ºC. The dried *Baby Java* orange peel was grounded using a blender for 3 minutes, and sieving was carried out using a 60-mesh sieve and then stored in a container for the subsequent treatment.

Baby Java Orange Peel Pectin Extraction

Baby Java orange peel pectin was extracted using the method adapted from Syarifuddin and Yunianta (2015) with modifications. Initially, 25 g of *Baby Java* orange peel powder was dissolved in a 5% citric acid solvent at a ratio of 1:20 (ingredient:solvent) and extraction was carried out with a hotplate stirrer at 90 °C for 60 minutes. The extraction results were filtered with an eightfold filter cloth. Then 96% ethanol was added to as much as 500 ml and the precipitation was carried out for 2 hours. The pectin precipitate was filtered off and the washing was carried out 3 times with 50 ml of 96% ethanol and 100 ml of

70% ethanol. The pectin precipitate was dried at 55 ºC for 10 hours until a pectin powder was obtained.

Edible Film Making

Edible films were prepared using modified methods adapted from Syarifuddin and Yunianta (2015). The experimental design used was a Completely Randomized Design (CRD) with two replications. Corn starch as much as 8% (b/v) was added with *Baby Java* orange peel pectin according to the treatments of 15%, 25%, and 35% (b/v). Glycerol 10%, 20%, and 30% (v/v) were added and made into suspension by adding 150 ml of aquadest. Three replications were carried out. Then the stirring and heating were carried out at 85 °C for 30 minutes. Subsequently, the prepared suspension was cooled to room temperature and stirred again. The suspension was filtered using a filter cloth to obtain a clear filtrate and then poured 40 ml into a 10 cm diameter petri dish. The film suspension was dried using an oven at 60 °C for 10 hours. The film was removed from the oven to be cooled at room temperature for 1 hour to make the edible film easily removed. Edible films were then analyzed.

Moisture Content Analysis

Moisture content analysis was done using the method from AOAC (2012). The analysis was repeated three times.

Thickness Analysis

Thickness analysis was done using a micrometer with a precision of 0.01 mm at five different points. The measurement results were averaged as a result of the film thickness, which is expressed in mm.

Solubility Analysis

Solubility analysis was done using Saleha *et al.* (2017) method with modifications. The film samples were dried in an oven at 40 °C for 24 hours, then cooled in a desiccator and weighed. The film was soaked with aquadest for 6 hours. The film was dried with tissue paper and weighed. To calculate the weight loss, the film was dried in an oven for 24 hours. Once it dried, the sample was placed in a desiccator and weighed. The weight difference from the initial weight is a decrease in mass or solubility of the edible film.

Tensile Strength Analysis

The analysis of the tensile strength of the edible film was carried out using the Universal Testing Machine (UTM) (Zwick) by following the manual procedure, and the data for the tensile strength of the edible film was obtained. From this tool, the data on the force needed to tear the edible films were generated.

Color Analysis

The color analysis was done using a chromameter. The color scale was then read, in which the parameters L stands for brightness and a, and b stands for chromaticity values. Parameter a is for red and parameter b is for yellow.

Water Vapour Permeability Analysis

The analysis of water vapor permeability was done using the method from López De Dicastillo *et al.* (2016) with modifications applied. The film sample to be tested was cut into 3x3 cm. Containers filled with silica gel were covered with the film to be tested. The container was placed in a desiccator at 25 °C and 60% RH with saturated NaCl solution. Containers in the desiccator were weighed every 12 hours for five days.

RESULTS AND DISCUSSION

Edible Film Moisture Content

Based on the results in Figure 1, it can be seen that the moisture content of edible film ranges from 3.83% to 9.28%. The highest moisture content was obtained at a pectin concentration of 35% and a glycerol concentration of 30%, then the lowest moisture content was found at a pectin concentration of 15% and a glycerol concentration of 10%. According to Junianto *et al.* (2013), fungi and mold generally grow organically in situations where the moisture content of the product is between 20% to 60%. Hence, the moisture content aspect is one of the critical parameters that need to be fulfilled to make edible films. Films with high moisture content are more elastic and flexible, which can be used in various food applications (Hanani *et al*., 2018).

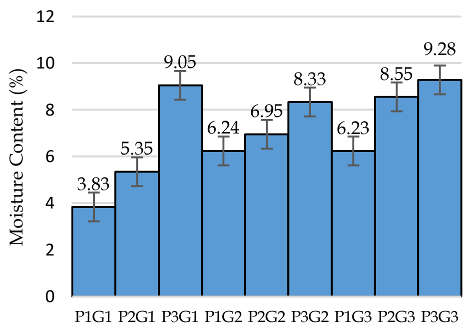

Figure 1. The moisture content of the edible film made of *Baby Java* orange peel pectin
*P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

In this study, the moisture content increased as the pectin and glycerol concentrations increased in each treatment. It is because pectin and glycerol are hydrophilic. The moisture content in the edible film will be trapped by the polymer bonds formed. In consequence, this causes the water difficult to evaporate and the moisture content increases during the drying process (Amaliya *et al*., 2014).

The value of moisture content in this study was lower than the value in the previous research conducted by Syarifuddin and Yunianta (2015), in which the edible film made of pectin albedo grapefruit produced moisture content ranging from 10.89-26.72%, while in the research by Amaliya *et al*. (2014) showed moisture content ranging from 11.801-14.306%.

Edible Film Thickness

In Figure 2, it can be seen that the thickness value of the edible film has increased with the addition of pectin concentration and glycerol concentration. The concentrations of pectin and glycerol have significantly affected the manufacture of edible films. The increase in the concentration of *Baby Java* orange peel pectin was caused by the increase in the thickness value of the edible film, and glycerol caused the increase in the viscosity of the film solution. The addition of corn starch can also increase the thickness of the resulting edible film (Amaliya *et al*., 2014). It is in accordance with Baldwin *et al*. (1994) statement that the higher the concentration of the material used, the thicker the edible film produced. In addition to the increase in pectin concentration and glycerol concentration, the increasing thickness of the edible film is also influenced by the area of the mold, the volume of the solution, and the number of total solids in the film solution (Jacoeb *et al*., 2014).

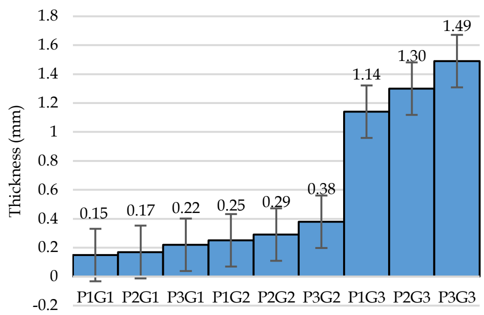

Figure 2. The thickness of the edible film made of *Baby Java* orange peel pectin
*P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

The thickness of the edible film in this study ranges from 0.15-1.49 mm. The values of edible film thickness at 10% glycerol concentration with 15%, 25%, and 35% pectin concentrations and edible film at 15% pectin concentration with 10% glycerol concentration are classified as acceptable. It is since the value is still below the maximum standard thickness of the edible film, which according to the Japanese Industrial Standard, is maximum at 0.25 mm. Thickness values can affect the rate at which water vapor is produced. According to Yulianti and Ginting (2015), the thickness can affect the rate of water vapor, gas, and other volatile compounds. The thicker the edible film produced will increase its ability to inhibit the gas and water vapor rate, so food products' shelf life extends.

The thickness of the edible film produced in this current study is thicker than several previous studies that used different materials. Syarifuddin and Yunianta (2015) research showed that the film made of pectin albedo grapefruit with glycerol plasticizer had a thickness value ranging from 0.11-0.23 mm. Furthermore, the research conducted by Zahra and Munawar (2020) utilized corn starch to make edible films with a thickness value ranging from 0.233-0.317 mm.

Edible Film Solubility

The increase in pectin and glycerol concentrations increased the film's edible solubility level (Figure 3). It is due to the properties of the two components, pectin, and glycerol, that have hydrophilic properties and can dissolve in water. It can cause the solubility percentage to be higher as the film matrix constituent components increase (Siswanti *et al.*, 2017). Glycerol is also a water-soluble component. Thus, the addition of a high concentration of glycerol increases the solubility of the film. According to Coniwanti, *et al.* (2014), increasing the concentration of glycerol will increase the solubility of the edible film.

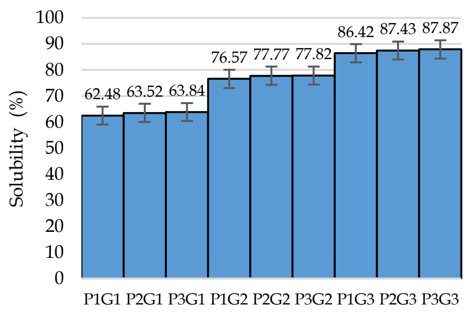

Figure 3. The solubility of edible film made of *Baby Java* orange peel pectin
*P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

The average value of edible film solubility based on *Baby Java* orange peel pectin ranges from 62.48% to 87.87%. The solubility value in this study is higher than in the research of Syarifuddin and Yunianta (2015), which used albedo grapefruit with a solubility value of 46.71-62.35%. In the previous study by Nugroho *et al.* (2013) that used banana peel pectin, the solubility value was 54.48-60.58%. According to Gontard *et al.* (1993), film solubility is strongly determined by the source of the basic ingredients used for making the film. Edible films with high solubility show that it is easy to be consumed.

Edible Film Tensile Strength

The highest tensile strength value in the edible film of *Baby Java* orange peel pectin was 4.79 MPa at a pectin concentration of 35% and a glycerol concentration of 10%. In contrast, the lowest edible film tensile strength is 0.13 MPa at a pectin concentration of 15% and a glycerol concentration of 30%. Figure 4 shows that the higher the glycerol concentration, the lower the tensile strength value. According to Akili *et al.* (2012), glycerol can reduce internal hydrogen bonds on intermolecular bonds, however, glycerol also reduces the tensile strength of the film.

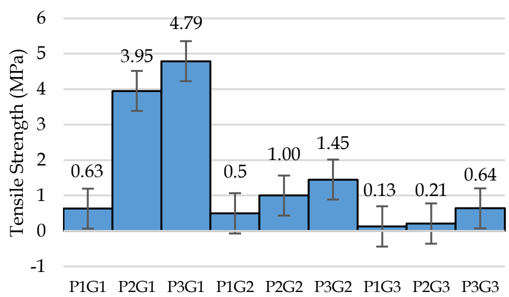

Figure 4. The tensile strength of edible film made of *Baby Java* orange peel pectin
*P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

The tensile strength value in this study decreased as the pectin concentration and glycerol concentration increased. This contrasts the thickness analysis, which increases as the pectin and glycerol concentrations increase. This is because plasticizers decrease the cohesion force between chains (Wijaya, 2013). The higher the concentration of plasticizer added, the lower the resulting tensile strength value. The added glycerol dissolves into each polymer chain, making it easier to move the polymer molecules and resulting in a decrease in the glass transition temperature. If the glass transition temperature decreases, the film formed will become softer and cause its tensile strength to be lower (Jacoeb *et al.,* 2014).

The tensile strength value resulting from this study is smaller than the tensile strength value obtained by Polnaya *et al.* (2016) in films made of natural sago starch, which ranged from 3.05-31.49 MPa. The smallest value in this study is 0.13 MPa which means that the value is less than the minimum value set by the Japanese Industrial Standard, which is 0.39 MPa. According to Marpongahtun (2016), tensile strength is a mechanical property of edible film that will protect the product from damage. Therefore, the smaller the tensile strength value of the edible film produced, the less likely it is to protect the product from damage.

Edible Film Color

The average L, a, and b color values in edible films produced in this study ranged from 54.66 to 56.48, -0.02 to -1.48 and 2.47 to 4.67 as respectively obtained pectin concentration and glycerol concentration treatments (Table 1). Overall, the resulting edible film color is clear or transparent which shows the characteristics of bright color and yellow-green color. This is slightly different from the research obtained by Afrianti *et al.* (2013) showing that the edible film of polyblend of chitosan succulent starch has a color value of L 80.49, a 2.29, b -12.7, with a pale grey edible film color which shows the characteristics of bright color and bluish red color.

In the brightness level (lightness), the addition of glycerol concentration and pectin concentration decreases the brightness value of the resulting edible film. Furthermore, there was also found an increase in the number of polymer constituents of the film matrix. An increase in the number of polymer constituents of the film matrix can cause an increase in thickness which increases the diffusion of light and results in the edible film appearing dull and opaque. This is in accordance with the statement of Bertuzzi *et al.* (2007) which states that low plasticizer content in edible films (<15%) produces high transparency. The plasticizer content that exceeds 15% or even reaches 30% will experience a decrease in intermolecular forces and result in the polymer matrix absorbing a lot of water, therefore, the edible film will lose its transparency.

Table 1. The color of edible film made of *Baby Java* orange peel pectin

Sample	Color L*	Color a*	Color b*
P1G1	54.97 ± 0.50^{a1}	-1.40 ± 0.06^{a1}	3.09 ± 0.12^{b1}
P2G1	54.71 ± 0.57^{a1}	-1.44 ± 0.06^{a1}	2.47 ± 0.28^{a1}
P3G1	55.22 ± 0.54^{a1}	-1.41 ± 0.04^{a1}	3.68 ± 0.13^{c1}
P1G2	54.98 ± 0.38^{a1}	-1.48 ± 0.09^{a1}	3.66 ± 0.16^{b2}
P2G2	54.66 ± 0.62^{a1}	-1.29 ± 0.04^{a1}	2.91 ± 0.09^{a2}
P3G2	55.39 ± 0.21^{a1}	-0.02 ± 1.93^{a1}	3.55 ± 0.08^{c2}
P1G3	56.48 ± 3.25^{a1}	-1.45 ± 0.01^{a1}	3.27 ± 0.19^{b3}
P2G3	54.82 ± 0.64^{a1}	-1.47 ± 0.04^{a1}	3.36 ± 0.07^{a3}
P3G3	54.84 ± 0.22^{a1}	-0.75 ± 0.86^{a1}	4.67 ± 0.10^{c3}

*The letter symbol indicates the difference in significance of the pectin concentration (P) (p<0.05) in the same column. The number symbol shows the difference in significance of glycerol concentration (G) (p<0.05) in the same column. P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

The value of appearances shows approaching the greenish color, as well as the value of blueness approaching the yellowish color. The addition of pectin concentration and glycerol concentration increases the edible film blueness. This is influenced by the pectin produced, which has a greenish-white color. The higher the glycerol concentration will increase the edible film's blueness value. This is thought to be the result of the influence caused by the thickness factor of the edible film.

The thicker the edible film formed results in a higher degree of brightness and yellowness of the edible film color. This is in accordance with Baldwin *et al*. (1994) statement that the thicker the edible film will give a non-transparent color and less attractive appearance. In this study, the researcher has produced an edible film with a clear or transparent color. The advantages of transparent color on the packaging are that if applied to a product, the product can be visible, flexible, or easily formed. In contrast, the weakness of the packaging is that it is not resistant to heat.

Edible Film Water Vapor Permeability

Baby Java orange peel pectin film edible has a value ranging from 1.06-9.53 g.mm/m^2.h.kPa. The higher the pectin and glycerol concentration, the higher the permeability value to water vapor (Table 2). The highest value of permeability to water vapor was found in the concentration of glycerol 30% and pectin 35% which is 9.53 g.mm/m^2.h.kPa. This is because glycerol and pectin are hydrophilic, therefore, increasing the number of components that are hydrophilic makes them increase permeability to water vapor. The lower the migration of water vapor produced, the better the properties of the edible film in maintaining shelf life (Nugroho *et al*., 2013).

The water vapor permeability value is also influenced by the thickness of the edible film. A thicker film will increase the rate of water vapor. This is in line with the research of Yulianti and Ginting (2015), thickness can affect the rate of gas vapor, water, and other volatile compounds.

Table 2. The water vapor permeability of edible film made of *Baby Java* orange peel pectin

Sample	WVP (g.mm/m^2.h.kPa)
P1G1	1.06 ± 0.04^{a1}
P2G1	1.08 ± 0.03^{b1}
P3G1	1.41 ± 0.07^{c1}
P1G2	1.42 ± 0.14^{a2}
P2G2	1.86 ± 0.11^{b2}
P3G2	2.58 ± 0.23^{c2}
P1G3	6.75 ± 0.06^{a3}
P2G3	7.55 ± 0.46^{b3}
P3G3	9.53 ± 0.06^{c3}

*The letter symbol indicates the difference in significance of the pectin concentration (P) ($p<0.05$) in the same column. The number symbol shows the difference in significance of glycerol concentration (G) ($p<0.05$) in the same column. P1 = pectin 15%; P2 = pectin 25%; P3 = pectin 35%. G1 = glycerol 10%; G2 = glycerol 20%; G3 = glycerol 30%

The edible film produced that is increasingly thick will increase its potential to slow the rate of gas or water vapor, otherwise, it will extend the shelf life of food products. Compared to the research conducted by Siswanti *et al*. (2017) with a permeability value of water vapor ranging from 0.687–1.270 gH2O.mm/jam.m^2, the edible water vapor permeability of *Baby Java* orange peel pectin film has a smaller value. This is because the inhibitory characteristics of the film were influenced by the hydrophilic/hydrophobic properties and the type of level and suitability of the plasticizer used (Nemet *et al*., 2010).

CONCLUSIONS

Based on the results, it can be concluded that the concentration of *Baby Java* orange peel pectin and glycerol with the addition of corn starch significantly affected the physical characteristics of the edible film produced on the parameters of solubility, thickness, tensile strength, color, and water vapor permeability. The concentration of Orange *Baby Java* skin pectin and glycerol with the addition of corn starch has a significant effect on the chemical characteristics of the resulting edible film. The highest formulation in the preparation of edible films according to the Japanese Industrial Standard is the addition of 15% pectin concentration and 20% glycerol concentration which has a water content value of 6.24%, solubility 76.57%, thickness 0.25 mm, tensile strength 0.50 MPa, lightness 54.98, blueness 3.66, appearances -1.48, and water vapor permeability 1.42 g.mm/m2.h.kPa.

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