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Optimization and Characterization of Physical–Mechanical Properties of Biodegradable Edible Films Based on Pectin from Breadfruit Peel for Food Packaging

Andi Hidayatullah Mappamadeng[1*)] and Rizka Amalia[1)]

[1)] Industrial Chemical Engineering, Faculty of Vocational School, Diponegoro University
Jl. Prof. Soedarto, SH, Tembalang, Semarang

[*)] Corresponding author: oelinplas90@gmail.com

Abstract *– This study aims to determine the characteristics and optimization of biodegradable films based on pectin from breadfruit peel. The study was employed using a factorial design with multiple variables: pectin (2 and 4 grams), sorbitol concentration (10% and 20%) and drying temperature (120^{0}C and 140^{0}C) were studied. The results obtained from eight samples showed that the water vapor transmission rate was in the range of 3.525 - 6.952 g/m^2.day. The best-achieved water vapor transmission rate (3,525 g/m^2.day) was obtained at specific operating conditions, namely 4 grams of pectin, 20% of sorbitol concentration and drying temperature of 140^{0}C. In this study, the most influential factor for water vapor transmission rate is pectin weight with the value effect of -1.238. The highest tensile strength test and percentage elongation were 116.55 kgf/cm^2 and 10%, respectively. The FTIR analysis showed that the pectin from breadfruit peel was according to commercial pectin standard, by the presence of OH and ester (COOH) groups. The SEM Analysis showed that the molecular structure of edible films did not look porous, dense, but not flat on its surface, thus less permeable to air.*

Keywords: *Bioedible film, Breadfruit peel, Pectin, Food packaging*

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INTRODUCTION

The accumulation of plastic waste in the world generated in Indonesia continues to increase, particularly in more urban areas. This over consumption makes Indonesia become second-largest plastic polluter in the world after China (Kementerian Lingkungan Hidup dan Kehutanan RI, 2020). It has potential to become materials that harm all life on Earth, because it acknowledges various environmental issues such as cannot be recycled and cannot be decomposed naturally by microorganisms in the soil. Thereby causing environmental pollution.

Synthetic plastic is widely used, because it is not easily brittle, strong and stable. Because of its stability, it cannot be broken down naturally. As a result, it will pile up as it takes a long time for it to break down. Another problem is that plastics are made up from petroleum, where its presence is dwindling and cannot be renewed. The current effort to reduce plastic consumption is to develop new biodegradable packaging materials from natural polymers.

The packaging technology that is safe and does not pollute the environment is edible film. Edible film is a biodegradable packaging that acts as a barrier in controlling the transfer of water vapor, oxygen, volatile components and lipids from and into food. It may be eaten together with the product (Widyaningsih et al., 2012). The edible films are classified into three categories taking into account the nature of their components:, namely hydrocolloids (proteins, polysaccharides, alginates), lipids (fatty acids, acyl glycerol or waxes) and composites (a mixture of hydrocolloids and lipids) (Fennema et al., 1994). Pectin as a hydrocolloid has the potential to be used as raw materials to obtain edible films to replace plastic polymers by its renewable and low-cost character.

In 2017, breadfruit production in Indonesia reached 104.966 tons (Badan Pusat Statistika, 2017). Breadfruit (*Artocarpus altilis*) is one of the natural resources in Indonesia that can be emlpoyed in several aspects, including its frit and waste. The abundance of breadfruit peel contains pectin has the potential to be used as a raw materials of biodegradable edible film. The reserach findings from (Madjaga et al., 2017) found that to produce optimal pectin in breadfruit, the best-achieved citric acid concentration was 7%, temperature at 90ºC, and time at 180 minutes. The results of the composition of pectin were methoxyl content of 8.091%, galacturonic content of 89.76%, and molecular weight of 73,673.997 g/mol.

Usually, pectin-based edible films are stiff and brittle, so it is necessary to add a hydrophilic plasticizer that can reduce water loss and increase the amount of bound water (Gennadios, 2002).Glycerol and sorbitol are commonly used as plasticizer for the polysaccharide films. In this study, sorbitol was used as a plasticizer because compared to glycerol, sorbitol is a more effective plasticizer, which has the advantage of reducing the internal hydrogen bonding in intermolecular bonding so that it can be used to reduce water evaporation from the product, can dissolve in each polymer chain thus, it can facilitate polymer chain mobility, lower oxygen permeability, available in large quantities, low cost, and non-toxicity (Astuti, 2010). The addition of excess sorbitol can reduce the tensile strength of the biodegradable edible, so it is necessary to add CMC (Carboxymethyl Cellulose) which is used to increase the tensile strength and improve the surface structure of the biodegradable edible film (Zuwanna & Meilina, 2017). Based on the explanation above, the researchers conducted a study to determine the best operating conditions for the optimization of biodegradable edible films based on pectin from breadfruit peel by adding sorbitol and to characterize the physical and mechanical properties of the product.

MATERIALS AND TOOLS

The tools used in this research were divided into two groups: equipment for pectin purification and producing edible film. Pectin purification equipment, namely blender, winnow, glass beaker, plastic bowl, stirrer, sieve. Equipment for producing edible films, namely hot plate magnetic stirrer, blender, filter, measuring cup, oven, desiccator, beaker glass, evaporating dish, stirrer, water bath. The materials used for pectin purification were breadfruit peel, 7% citric acid and 96% ethanol. The materials used for the producing edible films were pectin from breadfruit peel, aquadest, plasticizer sorbitol, and CMC.

Methods

Dependent Variable

Pectin extraction for beadfruit peel

Beadfruit starch ratio : Citric acid = 1:50 (g/ml)
Filtrate : Rastio Etanol 96% = 1 : 1,5 (ml/ml)
Extraction temperature = 90ºC
Extraction time = 180 minutes

Producing edibele film based on pectin from breadfruit peel :

CMC : Aquadest = 3 : 250 (g/ml)
Water temperature = 70ºC
Solution temperature = 90ºC

Independent variable

Pectin = 2g and 4 g
Sorbitol = 10% and 20%
Drying temperature =120ºCand140ºC

Preparation of Tools and Materials

Pectin Purification

The starch was weighed at 10 grams, then added 500 ml of citric acid with a concentration of 7%. Pectin extraction was carried out by heating at a temperature of 90ºC for 180 minutes. The hot extracts were filtered with a filter cloth. Cooled and precipated, furthermore add 96% ethanol with a ratio of 1:1,5, cooled for 24 hours and stirred until precipitate formed. The precipitate was separated from the solution through the filter paper. Poured the precipitate into container with a flat bottom, then dried at 50ºC for 24 hours in an oven. Then ground it with a blender, and got pectin

Producing edible film

Prepared a beaker containing 250 ml of distilled water then heated to a temperature of 70ºC, then dissolved 3 grams of CMC or 1.2% (g/ml) in distilled water, added the pectin from breadfruit peel (2 and 4 grams). The solution was heated at a temperature of 90ºC for 30 minutes using a water bath, then add the plasticizer sorbitol (10% and 20%) of the pectin weight. stirred and heated until homogeneous at a temperature of 90ºC. Poured the solution into the mold and then dried in an oven at a temperature of 120ºC and 140ºC for 6 hours.

Analysis and Characteristics of edible films

The results of edible film was analyzed and characterized to determine the mechanical and physical properties of the edible film. For testing mechanical properties includes tensile strength, percent elongation, and water vapor transmission rate

(WVTR), while for testing physical properties includes FTIR (Fourier Transform Infrared Spectroscopy) and SEM (Scanning Electron Microscopy) analysis.

Factorial Design Analysis

Factorial design is regression model provides a function that describes the relationship between a response variable and one or more independent variables. The model obtained from the analysis is in the form of a mathematical equation. Two-level factorial design means that there are two factors, each of which is tested at two different levels, namely low and high levels. A factorial design allows an experiment can be designed to determine the dominant factor significantly in a response (Bolton, 1997).

Table 2. Analysis of the results for 2-level factorial design in vapor transmission rate

No	S	P	T	Vapor Transmission Rate	-1	-2	-3	Interaction	Effect	Response	P(%)
1	-	-	-	6,952	12,927	22,547	41,122	8	Average	5,14025	7,14
2	+	-	-	5,975	9,62	18,575	-4,702	4	S	-1,1755	21,43
3	-	+	-	5,89	10,11	-3,137	-4,952	4	P	-1,238	35,71
4	+	+	-	3,73	8,465	-1,565	-2,448	4	SP	-0,612	50,00
5	-	-	+	5,13	-0,977	-3,307	-3,972	4	T	-0,993	64,29
6	+	-	+	4,98	-2,16	-1,645	1,572	4	ST	0,393	78,57
7	-	+	+	4,94	-0,15	-1,183	1,662	4	PT	0,4155	92,86
8	+	+	+	3,525	-1,415	-1,265	-0,082	4	SPT	-0,0205	107,14

RESULTS AND DISCUSSION

The method used for analyzing the data was factorial experimental design with three independent variables, namely sorbitol concentration, pectin weight and drying temperature. The design has been employed to determine the independent variables and optimum operating conditions. By using the factorial design method, the most influential variables on producing edible films based on pectin from breadfruit peel will be obtained.

Tabel 1. Results of Research Design

Run	Results			
	Sorbitol (%)	Pectin (%)	Drying Temperature (ºC)	Water vapor transmission rate (g/m^2.day)
1	10	2	120	6,952
2	20	2	120	5,975
3	10	4	120	5,89
4	20	4	120	3,73
5	10	2	140	5,13
6	20	2	140	4,98
7	10	4	140	4,94
8	20	4	140	3,525

Table 1 shows that the highest to the lowest value of water vapor transmission rate was achieved in the 8th experiment (4 grams of pectin, 20% of sorbitol concentration and 140ºC of drying temperature) reached 3,525 g/m2.day, while the lowest to the highest value of water vapor transmission rate was achieved in the first experiment (4grams of pectin, 20% of sorbitol concentration and 140ºC of drying temperature) reached 6.925 g/m2.day. According to Japanesse Industrial Standard, good vapor transmission rate is less than 10 g/m2.day. The lower the value of the vapor transmission rate, the better the quality of the edible film, because the amount of water remains will be lower and the water resistance is higher and better (McHUGH et al., 1994). Table 1 shows the relationship between sorbitol and pectin and vapor transmission rate is inversely proportional, where the greater amount of pectin, the lower vapor transmission rate due to increasing the concentration of pectin will increase the amount of polymer and viscosity that will compose the film matrix. The larger the polymer that composes the film matrix, the lower the amount of water that will enter the film network, which results in a lower vapor transmission rate (Liu & Han, 2005). According to Hidayati et al in Putra et al. (2017) sorbitol is hydrophilic (capable of binding water) and softens the surface of the film so that the addition of sorbitol concentration can increase water vapor transmission rate but this study Got opposite results, where along with the addition of sorbitol made the thicker the edible film. This is because the more concentration of sorbitol added will increase the total solids in the solution which will affect the thickness of the edible film, where when the substance evaporates, the edible film formed becomes thicker along with the increasing number of total solids that settle as ingredients for the edible film. According to McHaugh et al and Henrique et al in Salsabila et al. (2017) thickness determines the resistance of film to the water vapor transmission rate, the thicker the edible film produced, the higher its ability to inhibit the rate of gas and water, so the vapor transmission rate is lower or better (Yulianti & Ginting, 2012).

Estimation of The Variable Effects on Vapor Transmission Rate

The main and interaction effects on the process of producing edible films on the results of vapor transmission rate can be seen in Table 2 as follows:

The relationship between P and the effect value can be seen in table 2 where the P value is obtained from the formula P=(i 0.5)x100%/7 where i is the order number .

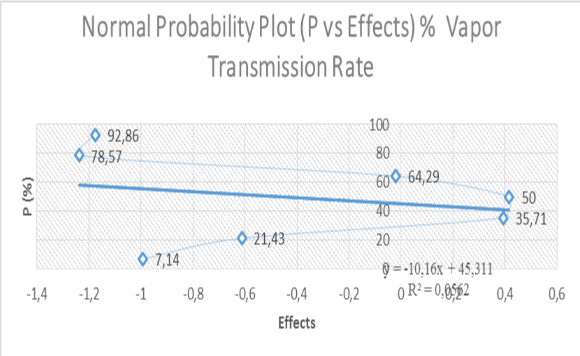


Figure 1. Normal Probability Plot of Effects (Result of Vapor Transmission Rate)

From Figure 1 it can be seen that the P effect (pectin concentration) is the farthest point with a value of -0.1238. Thus, the concentration of Pectin is the most influential variable on the vapor rate transmission. This is in line with the research conducted by Syarifuddin & Yunianta (2015) where the increasing pectin concentration will further reduce the water content of the edible film, because increasing the suspension of pectin concentration will increase the amount of polymer and viscosity that will compose the film matrix. The larger the polymer that composes the film matrix, the lower the amount of water remains, so that the vapor transmission rate is lower or better.

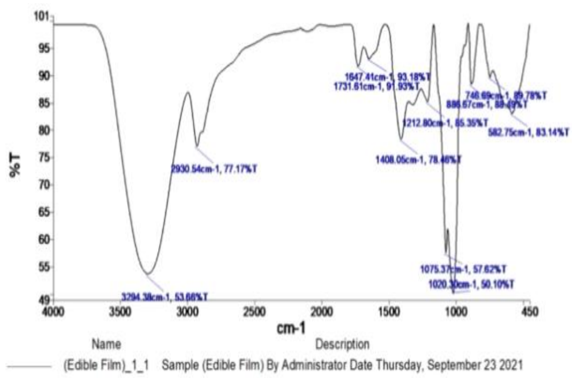
Analysis Result of Tensile strength and elongation

The results of the edible film with the best water vapor transmission rate in the 8th experiment (4 grams of pectin, 20% of sorbitol concentration and 140ºC of drying temperature) were analyzed for tensile strength and elongation using the ASTM D882 method, where tensile strength obtained 116,55 kgf/cm^2 , this is in line with the characteristics of edible films according to Japanese Industrial Standard (JIS), which is more than 40 kgf/cm^2 .

According to Petersson & Stading (2005) the tensile strength of edible films increased with increasing concentration along with the addition of pectin. This happens because the addition of more pectin, the matrix formed is getting better, the matrix structure of the film is getting stronger so that the strength given to support the load from the outside is getting bigger. The greater the tensile strength, the greater the resistance to damage due to stretching and pressure, resulting in improved physical quality.

The elongation value of the pectin from edible film from sample 8 (4 grams of pectin, 20% of sorbitol concentration and 140ºC of drying temperature) got a value of 10%. This is not in line with the characteristics of elongation from Japanese Industrial Standard (JIS) which is more than 70%. According to Su et al in Warkoyo et al. (2014) the increasing addition of pectin was accompanied by an increasing pectin: sorbitol ratio, resulting in a lower percentage of elongation. The greater the addition of plasticizer, the percentage of elongation will increase, but after the addition reached at a certain concentration the value will decrease, because the more concentration of plasticizer the cohesion bonds between polymers will be smaller and the film formed will be softer so that the edible film formed is easily broken (Bourtoom, 2008).

In this study, too much sorbitol plasticizer was used, namely 20%, this happened in the study conducted by Shara et al in Harumarani & Ma'ruf (2016). where 0.9% sorbitol addition there was a decrease in elongation while the addition of 0.3% - 0.7% sorbitol increased the elongation alue.The study of Irmayatul et al. (Khotimah & Tjahjani, 2020) showed that elongation could be increased by adding 40% sorbitol concentration. However, when the 50% sorbitol concentration was added, the



elongation decreased, therefore the elongation obtained was not in line with the characteristics of the Japanese Industrial Standard (JIS)

Analysis results of functional group using FTIR

Figure 2. Results of FTIR edible film

Table 3. FT-IR of isolated pectin (pectin from breadfruit peel) and commercial pectin

Wave number (cm^{-1})		
Standard pectin	Isolated pectin	Results
2886,33-2973,30	2930,54	C-H
1730,00-1750,41	1731,61	C=O
1626,49-1680,00	1647,41	C=C
1014,40-1246,44	1075,37	C-O

(SDBS, 2019)

The main functional groups of pectin are usually in the region between 1,000 and 2,000 cm-1 of the FTIR spectrum (Ismail et al., 2012). the presence of a carbonyl group (C=C) at 1647.41 indicates that the sample is classified as pectin, and the presence of a C-O group at 1075.37 and 1020.30 followed by a carbonyl group of 1647.41 indicates that the sample contains an ester group.

FTIR test proved the presence of OH groups and also esters (COOH). The presence of these functional groups indicates that the edible film can be degraded well in soil (Ban et al., 2006). In table 3, a comparison of the wave numbers between isolated pectin (pectin from breadfruit peel) and standard pectin (commercial pectin) indicates that the isolated pectin (pectin from breadfruit peel) complies with commercial pectin standarS.

Analysis results of the surface morphology of the edible film with SEM (Scanning Electron Microscope)

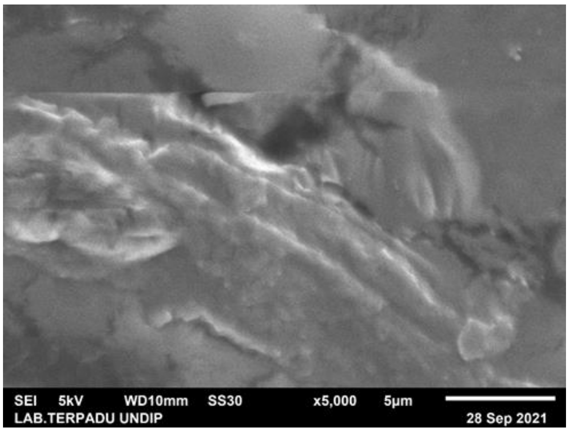


Figure 3. Cross-sectional area of Edible Film from Breadfruit pectin (4gram) sorbitol 20% and drying temperature 140ºC with 5000x Magnification

Based on the results of the SEM test with a variable composition of 4gram of pectin, 20% of sorbitol concentration and 140ºC of drying temperature, it can be seen that the molecular structure of edible films did not look porous, dense, but not flat on its surface, thus less permeable to air. The non-smooth surface indicates that the film is less homogeneous.

CONCLUSION

Based on the research that has been carried out, the vapor transmission rate from eight consecutive experiments has resulted in the results of 6.952 - 3.525 g/m^2.day. The best-achieved optimum operating conditions vapor transmission rate were obtained in the 8th experiment with 4gram of pectin, 20% of sorbitol concentration and 140ºC of drying temperature with vapor transmission rate of 3.525 g/m^2.day.). The best-achieved results of the tensile strength and elongation were 116.55 kgf/cm2 and 10%, respectively. This is not in line with the characteristics of the Japanese Industrial Standard (JIS), which is more than 70%. In the FTIR test, the results of isolated pectin (pectin from breadfruit peel) were in line with commercial pectin standards indicated by the presence of OH and ester (COOH) groups. In the morphological analysis using SEM, it can be seen that the molecular structure of edible films did not look porous, dense, but not flat on its surface, thus less permeable to air.

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