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Production and Characterization of Edible Film from A Mixture of Tapioca Flour, Yellow Pump *(Cucurbita moschatal D)*, Chitosan, and Glycerine for Application as A Layer Cake Packing.

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Abstract. The experiment of production and characterization of an edible film by mixing tapioca starch, pumpkin *(Curcubita moschatal D.)*, chitosan, and glycerine with applications as the wrapping of layer cake has been done. The edible film was produced by mixing 2.5 g of tapioca starch, 3.5 g of pumpkin, and 40 mL of aquadest, then adding 4 % of chitosan, and 2 mL of glycerine. After homogenous, it was poured into an acrylic pan and dried in an oven at 35-45°C degrees for 2 days. Characterization of an edible film physically obtained thickness was 0.25 mm, tensile strength was 0.06 kgF/mm^2 and elongation was 22.77 %. Morphology test of SEM (Scanning Electron Microscopy) produces edible film have pores that were tight, flat, and a bit compatible surface. And the FT-IR spectrum analysis showed the spectrum at 2939.52 cm^{-1} for the Alkana group (CH) and 3402.43 cm^{-1} for the hydroxyl group (OH). The result of the nutrition analysis of edible film was 28.2 % of carbohydrate content, 8.23 % protein content, 1.49 % of fat content, 25.6 % of water content, and 0.36% of ash content. An edible film can also be used as a wrapping for a layer cake.

Keyword: Chitosan. Edible Film, Glycerine, Pumpkin, Wrapping.

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1 Introduction

Food product packaging is a packaging process that uses proper packaging materials to keep food fresh and safe until it reaches the hands of consumers (Hui, Y. H. 2006). Nowadays, the use of synthetic polymers such as plastics, which is a commonly used packaging material, plays an essential part in the economy of modern industrial civilization. However, the use of synthetic polymers harms the environment because they contain dangerous chemicals and contribute a lot of waste that is difficult to decompose because synthetic polymers are difficult to degrade naturally both by biotic components such as decomposing microorganisms and abiotic components such as sunlight. This is a major issue for the environment, hence it is critical to

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study and master the science of creating new materials that can and are easily degraded organically.

With rising public awareness of health and environmental issues fueling an increase in packaging demand, one environmentally friendly (biodegradable) packaging option is an edible film, which can ensure food product safety (Wahyono, 2009). The edible film is a thin layer that protects edible food and can be naturally destroyed by nature (Robertsons, L. G. 1993.). However, apart from being biodegradable, the edible film can be combined with certain components to increase the functional value of the packaging itself, such as being able to be consumed directly with the packaged product, not polluting the environment, improving the organoleptic properties of the packaged product, functioning as a nutritional supplement, flavors, dyes, antimicrobial substances, and antioxidants (Murdianto, 2005).

Starch is a polymer that can be utilized to make edible films. Because it is inexpensive, renewable, and has acceptable physical properties, starch is frequently utilized in the food business as a biodegradable film to replace plastic polymers (Bourtoom, T. 2007).

A thin layer used to coat food (coating) or placed between components that function as a barrier against mass transfer such as water, oxygen, fat, and light content or function as a carrier for food additives is referred to as edible film (Krochta,1997). The benefit of edible film is that it can protect food goods, maintains the original appearance of the product, maybe consumed instantly, and is environmentally safe (Kinzel, 1992). Sumariah, 2014 has carried out the research on the manufacture and characterization of Edible Film from mangosteen rind extract *(Garcinia mangostana L.)* with the addition of Tapioca Flour, Chitosan, Glycerin as Plasticizers" resulting in the nutritional content produced from edible film, the best is a variation of 10 ml of mangosteen rind extract 30 ml of water, 7g of tapioca flour, 2% chitosan and 2 ml of glycerin showed better results because they contain more nutrients with carbohydrates content of 69.69%, the protein content of 3.45%, a fat content of 3.4%, ash content of 3.96%, water content 19.5% and -carotene content 76.0225 ppm.

The distribution of pumpkins in Indonesia has great development potential, both in terms of land potential, species variety, and farmer and technology perspectives. Based on this, the researchers sought to use pumpkin in the production of edible films by using steamed pumpkin to boost the nutritional value of the finished product, glycerin as a plasticizer since it has higher mechanical qualities and chitosan as an antibacterial in edible films. According to the researcher's description, the final edible film will not only have good food packing capabilities but also contain nutrients that can benefit health.

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2 Materials and Methods

2.1 Equipments

The following instruments were used in this study: hotplate, oven, analytical balance, beaker glass (pyrex), measuring flask, measuring cup (Pyrex), thermometer, magnetic stirrer, tensile test equipment, SEM (scanning electron microscopy), FT-IR spectrophotometer, acrylic plate, soxhlet apparatus, distillation apparatus (Gerhard born), furnace (m desiccator, porcelain cup, Whatman no.41 filter paper, glass plate, fisher scientific incubator, counter, Yamato sn20 autoclave, condenser.

2.2 Materials

The materials used in this study include pumpkin, chitosan, tapioca flour, glycerin (E-Merk), aquadest$_{(l)}$, $CH_3COOH_{(aq)}$ (E-Merk), $HCl_{(l)}$, Selenium (E-Merk), H_2S0_4 (E-Merk), H_3BO_4 (E-Merk), NaOH (E-Merk), Boric Acid (E-Merk), Tashiro indicator, *Escherichia coli*, $NaOH_{(aq)}$ (E-Merk), $H_2SO_{4(aq)}$ (E-Merk), *Staphylococcus aureus*, a recipe for the layer cake.

2.3 Sample Preparation

The pumpkin fruit sample was bought from the Sei Market in Sekambing. The Latin name for pumpkin fruit is Cucurbita Moschatal D. A 250 g pumpkins were cleaned, steamed for about 10 minutes, and then crushed/blended until smooth, and pumpkin pulp was obtained.

2.4 Production of Edible Film

2.4.1 Variations of Chitosan

A glass beaker with 43 mL of distilled water was filled with 2.5 g of tapioca flour. Stir until completely smooth. Heat on a hotplate until thickened, between 68^{o}C - 70^{o}C. 2 g pumpkin pulp was added and stirred until smooth. After that, 50 mL of 1% chitosan solution and 2 mL glycerin were added. Allow thickening after stirring until homogenous. The liquid was poured and smoothed onto an acrylic plate. for 2 days in the oven at a temperature of 35 to 40^{o}C. For variation 2%, 3%, 4%, and 5% chitosan underwent the same process.

2.4.2 Variations of Pumpkins

2.5 g tapioca flour was placed in a beaker glass that had been filled with 41.5 mL distilled water. Stir until completely smooth. Heat on a hotplate until thickened, between 68 and 70^{o}C. 2 g pumpkin pulp was added and stirred until smooth. After that, 50 mL of chitosan solution (4%) and 2 mL of glycerin were added. Allow thickening after stirring until homogenous. The liquid was poured and smoothed onto an acrylic plate. Dry for 2 days at a temperature of 35^{o}C-40^{o}C in the oven. The same procedure was carried out for pumpkin with variations of 2.5 g, 3 g, 3.5 g, and 4 g and distilled water with variations of 41 mL, 40.5 mL, 40 mL, and 39.5 mL.

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2.5 Edible Mechanical Properties Testing Film

2.5.1 Thickness Test

The thickness of the film used in the packaging of the product is an important factor to consider. When the edible film breaks, the thickness can affect the rate of transmission of steam, gas, and volatile substances, as well as other physical attributes including tensile strength and elongation (Sagala, 2013).

2.5.2 Tensile Strength (MPa)

Tensile strength is one of the most important basic properties of polymer materials and is often used to characterize a polymeric material. The tensile strength of a material is defined as the maximum load (F_{max}) used to break the specimen divided by the initial cross-sectional area (A_0).

$$\sigma = \frac{Fmaks}{A_0}$$

Where,

σ = tensile strength of the material (kgf/ mm^2)

F = maximum stress (kgf)

A_0 = cross-sectional area (mm^2)

2.5.3 Elongation Test

The elongation (σ) is used to determine the mechanical properties of the material in addition to the tensile strength (ε). The percent elongation of the edible film was calculated using the results of the product's tensile strength test, yielding two data points: the edible film's initial length (before the tensile strength test) and final length (after the tensile strength test), which were calculated using the formula:

$$\varepsilon\ (\%) = \frac{I_t - I_0}{I_0} \times 100\%$$

Where,

ε = Elongation (%)

I_0 = The initial length of the specimen (mm)

I_t = The length of the specimen after being drawn (mm) (Wirjosentono, 1996).

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2.5.4 SEM (Scanning Electron Microscopy) Analysis

Scanning Electron Microscopy is a method to generate a macroscopic image of a specimen's surface. The specimen is exposed to an electron beam with a diameter of 5 to 10 nm. Backscattering of the electron beam, x-rays, secondary electrons, and electron absorption are all caused by the electron beam's interaction with the object. Based on the optimal mechanical properties of the edible film, the surface of a mixture of starch, wheat flour, pumpkin pulp, chitosan, and glycerin was observed in this case.

2.5.5 FT-IR (Fourier Transform Infra Red) Analysis

The FT-IR analysis is an analysis of the interaction of the compounds contained in the edible film in the form of stretching or indentation of functional groups displayed in the form of a wave spectrum. In this case, the functional group interaction spectrum of the edible film mixed with starch, wheat flour with chitosan, pumpkin pulp, and glycerin was observed based on the optimal mechanical properties of the edible film.

2.6 Determination of Nutrient Levels

2.6.1 Water Content

The edible film was weighed as much as 1-2 g in a weighing dish with a known weight. Dry in the oven at a temperature of 105 °C for 3 hours. Cool in a desiccator. Then weighed until a constant weight is obtained.

$$Water\ content = \ final\ sample\ weight\ /\ initial\ sample\ weight\ X\ 100\%$$

2.6.2 Ash Content

The edible film weighs between 2 and 3 g. In the oven, it was dried. It was ashed for 3 hours in an ashing furnace at a maximum temperature of 550 °C. In a desiccator, it was cooled. Then it was weighed until it reached a steady weight.

$$Ash\ content = \ ash\ weight\ /\ \ sample\ weight\ X\ 100\%$$

2.6.3 Fat Content

The edible film was weighed as much as 1-2 g and put into a paper sleeve which was lined with paper. Dry in the oven at a temperature of not more than 80 °C for about 1 hour. Then put into a Soxhlet tool that has been connected to a bottom flask that already contains boiling stones. Extracted with hexane or other fat solvents for about 6 hours. Hexane was distilled and the fat extract was dried in an oven at 105ºC, cooled, and weighed to a constant weight.

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$$Fat\ content = \ fat\ weight\ /\ sample\ weight\ X\ 100\%$$

2.6.4 Protein Content

1 g of the edible film was weighed and put into a 100 ml Kjeldahl flask, added 2 g of selenium and 25 ml of H_2SO_4(p), heated over an electric heater or a fire burner until it boiled and the solution became clear greenish (about 2 hours). Allowed to cool, then put into a 100 ml volumetric flask and diluted with distilled water to the marking line. Pipette 5 ml of 40% NaOH(aq) and 1-2 drops of mixed indicator. Distill for about 10 minutes. $NH_{3(g)}$ was accommodated in an Erlenmeyer glass containing 10 ml of a 2% borate solution that had been mixed with indicators. Rinse the tip of the cooler with distilled water. Titrate with 0.1 N HCl solution.

$$Protein\ content = ((V1 - V2)\ x\ N\ x\ 0.014\ x\ f.k\ x\ f.p)/(W\ )\ x\ 100\ \%$$

2.6.5 Carbohydrate Content (by difference)

Carbohydrates (including fiber content) are calculated by deducting the contents of water, ash, protein, and fat from 100 percent (Winarno, 1992).

$$Carbohydrate\ Content = 100\ \% - \%\ (protein + fat + ash + water)$$

2.7 Production of Layer Cake

Put the coconut milk, pandan leaves, and salt to a boil. Remove and cool after stirring until boiling. In a separate bowl, combine rice flour, tapioca flour, sugar, and vanilla powder. Blend the steaming yellow pumpkin, eggs, and coconut milk until smooth, then gradually stir in the flour mixture. Divide the dough in half and add chocolate powder to one half. Preheat the pan and oil it with margarine. Fill the pan with the yellow mixture. Steam for 10 minutes, then pour in the chocolate mixture and continue until the dough was gone. Finally, after about 30 minutes of steaming, remove and chill.

2.8 Activity Test with Standard Plate Count Metode Method

Prepared 5 test tubes, each containing 9 mL of sterile distilled water. Then weighed as much as 1 g of the test sample to be inserted into the first test tube. From the results of homogenization between 9 ml of sterile distilled water and 1 g of the test sample, a dilution factor with a concentration of 10-1 was obtained. From the results of the 10-1 dilution, 1 ml was taken to be put into the second tube. The results of homogenization in the second tube will obtain a dilution factor with a concentration of 10-2 and so on until a dilution factor of 10-5 was obtained. Take 0.1 ml each from the 10-4 and 10-5 dilutions to be inoculated into 2 different Petri dishes. PCA (Plate Count Agar) media was poured at a temperature range of ±36 °C into a petri dish that already contained 0.1 ml of the solution from the dilution factors of 10-4 and 10-5. The TPC

Production of Edible Film		Parameters		
		Tensile Strength (KgF/mm^2)	**Elongation (%)**	**Thickness (mm)**
Variation of Chitosan (%)	E1	0.0285	13.498	0.20
	E2	0.0396	16.438	0.18
	E3	0.0486	23.886	0.18
	E4	0.0703	29.052	0.16
	E5	0.0347	17.52	0.18
Pumpkin Variations (g)	E600228		16.74	0.25
	E7	0.0238	16.76	0.21
	E8	0.0595	22.36	0.21
	E9	0.06	22.77	0.25
	E10	0.0059	17.44	0.21

results were incubated with the pour cup method at 34 °C for 1 x 24 hours. Count the number of colonies that grow after the incubation period.

3 RESULT AND DISCUSSION

3.1 Mechanical Properties Analysis of Edible Film

Results of analysis of tensile strength and elongation of edible film from mixed variations of chitosan, tapioca flour, glycerin, and smooth yellow pumpkin.

Table 1. Edible Film Characterization Analysis

Information :

E1: Chitosan 1%; E6: Pumpkin 2 g

E2: Chitosan 2%; E7: Pumpkin 2.5 g

E3: Chitosan 3%; E8: Pumpkin 3 g

E4: Chitosan 4%; E9: Pumpkin 3.5 g

E5: Chitosan 5%; E10: Pumpkin 4 g

Based on the findings of tensile strength testing, edible films containing different amounts of chitosan had a stronger tensile strength in the variable 1% chitosan, 2.5 g tapioca flour, 2 g pumpkin, and 2 mL glycerin, namely 0.0703 kgF/mm^2 and elongation of 29,052%. This was due to the reaction that occurs so that the edible film produced was stronger and not easily broken when pulled. The texture and viscosity of chitosan are affected by its concentration. The

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edible film with the highest tensile strength was made with 3.5 g pumpkin, 4% chitosan, 2.5 g tapioca flour, 2 mL glycerin, 0.06 kgF/mm^2, and elongation of 22.77% in the pumpkin variety.

3.2 Nutritional Analysis

The nutritional content of edible film made from a combination of tapioca flour, glycerin, chitosan, and smooth yellow pumpkin yielded the greatest results.

Table 2. Nutritional Analysis

No .	Parameters	Addition of Pumpkin
1.	Water content	25.6 %
2.	Ash content	0.36 %
3.	Fat content	1.49 %
4.	Protein content	8.23%
5.	Carbohydrate content	64.32 %

3.3 SEM (Scanning Electron Microscopy)

The results obtained from the SEM examination showed the surface shape of the edible film of pumpkin *(Cucurbita moschatal D.)* with the addition of 3.5 g pumpkin, 2.5 grams of tapioca flour, 2 mL glycerin, 4% chitosan as antibacterial with 500x magnification. surface results in morphological forms that are too dense and close together. This was influenced by the ingredients of the edible film being evenly mixed, both filler and plasticizer are added, judging from the highest mechanical test, SEM analysis was carried out on the edible film which can be seen in figure 1.

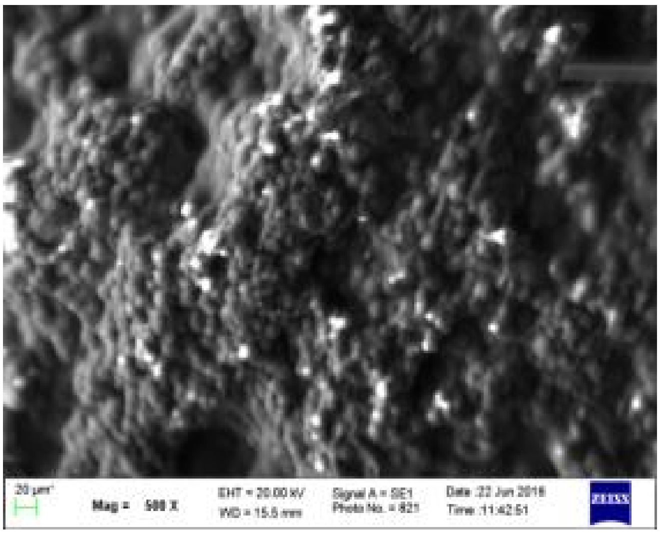

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Figure 1. SEM images of edible film with 500x magnification

3.4 FT-IR (Fourier Transform Infra Red)

From the results of FT-IR on the manufacture of edible films from 2.5 g of tapioca flour, 4% chitosan, and 2 ml of glycerin with the addition of 3.5 g of finely ground pumpkin *(Cucurbita moschatal D.)* the results of the FT-IR analysis provide a spectrum with absorption in the area of 3361.17 cm^{-1} indicates the presence of a hydroxyl group (OH) or -NH group derived from chitosan, in Appendix 3 gives an absorption spectrum with an absorption of 3297.98 cm^{-1} indicating the presence of a hydroxyl group (OH) and in the area of wave number 2931.95 cm^{-1} derived from -glucose, from appendix 4 gives a spectrum with absorption in the region of 3297.00 cm^{-1} indicating the presence of a hydroxyl group (OH) derived from glycerin and absorption in the region of wave number 2880.17 cm^{-1} indicating the presence of CH aliphatic, and edible film with FT-IR test gave an absorption spectrum in the area of 3402.43 cm^{-1} indicating the presence of a hydroxyl group (OH) and 2939.52 cm^{-1} indicating the presence of an alkane group (CH). This shows the occurrence of mixing between tapioca flour, chitosan, and glycerin in the resulting edible film. Where the comparison of the FT-IR spectrum before and after mixing there was a change that was not so sharp. The comparison of the results of the FT-IR test of chitosan, tapioca flour, glycerin, and edible film.

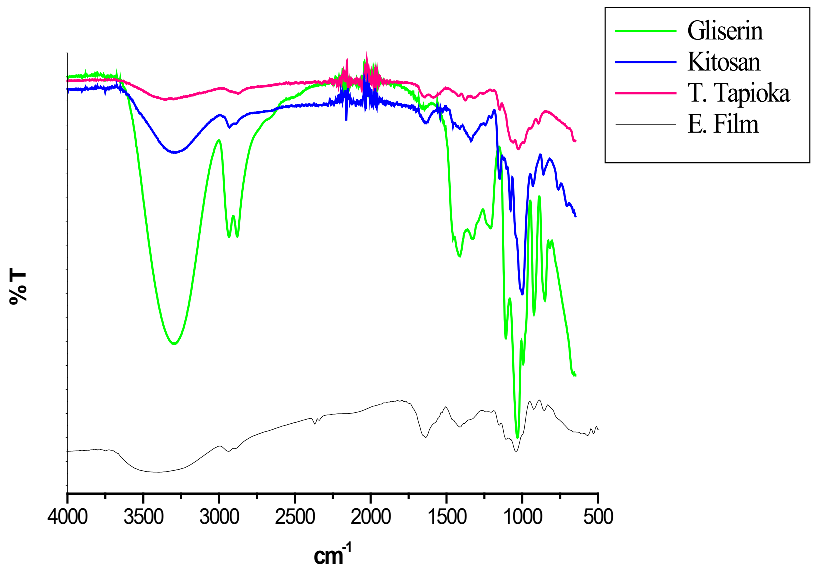

Figure 2. Comparison of FT-IR of Gliserin; Chitosan, Tapioca Flour, and Edible Film

The results of the interpretation of functional groups with FTIR analysis can be seen in Table 3.

Table 3. Interpretation of Functional Groups of Compounds from FTIR Analysis

Functional Groups	Results (cm^{-1})	Teory (cm^{-1})
CH	2931.95 (T) 2880.17 (G)	2885 – 3000

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	2939.52 (E)	
OH	3297.00 (G) 3297.98 (T) 3361.17 (K) 3402.43 (E)	3200 – 3500
NH	3361.17 (K)	3100 – 3500

Description: G=Glycerin; T= Tapioca; P=Pectin; E=Edible Film

3.5 Edible Film Application by Observing the Growth of Bacterial Colonies on Layer Cakes Wrapped with Edible Film and Wrapped with Ordinary Plastic with the Standard Plate Count Method

The calculation of the number of bacterial colonies was taken from slices of the layer cake that had been diluted 10^{-1} and then inoculated on PCA media. Table 3.4 shows the results of calculating the number of colonies for 3 days, which shows the difference in colony growth between layer cakes wrapped in edible film and layer cakes wrapped in ordinary plastic. The treatment for the layer cake samples wrapped in the edible film had less visible colony growth compared to the layer cake samples wrapped in ordinary plastic. So that the edible film produced is more effective in reducing the growth of bacteria/microbes in layer cakes. So it is suitable as a food packaging material.

Table 4. Colony growth test results on layer cakes wrapped in edible film and wrapped in plastic

Sample	Colony Growth		
	Day- 1	Day- 2	Day- 3
Layer cake without wrapping	15	3,752	TBUD
Layer cake wrapped in edible film	7	51	381

Note: TBUD = Cannot be calculated

4 Conclusion

Based on the results of the research conducted, the following results were obtained:

1. The best edible film characterization was found in variations of 2.5 g tapioca flour, 3.5 g pumpkin, 4% chitosan, and 2 mL glycerin showing characteristics that include tensile strength

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of 0.06 kgF/mm^2, elongation 22.77%, 0.25 mm thickness and the results of the SEM test which shows the pores were tight and compatible. FT-IR analysis which shows the mixing of hydroxyl groups (OH) and the presence of aliphatic CH groups.

2. The nutritional content produced from the best edible film was 28.2% carbohydrate content, 8.23% protein content, 1.49% fat content, 0.36% ash content, and 25.6% water content.

3. The application of Edible film as a layer cake wrapper which was carried out on days 1, 2, and 3 showed that the layer cake sample wrapped with the edible film had less visible colony growth compared to the layer cake sample wrapped in ordinary plastic. So that the edible film's ability as a layer cake wrapping material can inhibit the growth of good colonies.

References

Bourtoom, T. 2007. *Effect of Some Process Parameters on The Properties of Edible Film Prepared From Starch*. Songkhla: Department of Material Product Technology Prince of Songkla University Hat Yai.

Hui, Y. H. 2006. Handbook Of Food Science, Technology, And, Engineering Volume I. CRC Press. USA

Krochta and De Mulder Johnston. 1997. *Edible and Biodegradable Polymers Film: Changes & Opportunities. Food Technology (51)*

Robertsons, L. G. 1993. *Food Packaging Principles and Practice*. New York: Marcel Dekker. Inc.

Sagala, S.T. 2013. Karakterisasi Pembuatan Edible Film Dari Campuran Tepung, Rumput Laut (Eucheuma sp.), Kitosan dan Gliserin. [Skripsi]. Medan: Universitas Sumatera Utara, Departemen Kimia.

Sumariah., 2014. "Karakterisasi *Edible Film* Dari Ekstrak Kulit Manggis *(Garcina mangostana L.)* dengan penambahan Tepung Tapioka, Kitosan Dan Gliserin Sebagai Pemlastis" [Skripsi]. Medan : Universitas Sumatera Utara, Sarjana

Wahyono, 2009. Karakteristik Edible Film Berbahan Dasar Kulit Dan Pati Biji Durian (Durio Sp) Untuk Pengemasan Buah Strawberry. Surakarta : Universitas Muhammadiyah Surakarta.

Wirjosentono, B. 1995. Perkembangan Polimer di Indonesia Orasi Ilmiah Lustrum 6. Medan: FMIPA USU.