

## Edible Film Application of Soybean Extract with the Addition of Tapioca Flour and Glycerol as A Green Grape Packer (*Vitis vinifera* L.)

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**Abstract.** This research has been carried out on the manufacture and characterization of edible films made from soybean extract with tapioca flour and glycerol applied in packaging for green grapes. This study aims to determine the effect of glycerol addition on the physical properties of the resulting packaging and the effect of the repetition of grapes immersion in the packaging solution on the grapes' quality. This research's stages were the manufacture of soy milk, film formation, film characterization, water vapor transmission rate (WVTR), and application test of packaging solutions to the quality of green grapes using weight and color loss tests. A variety of glycerol (0.5; 1; 1.5; 2; 3; 5%) and the optimum yield is used to coat green grapes. The results showed that the optimum composition for making an edible film was obtained in the edible film with the addition of 2% glycerol concentration. The addition of 2% glycerol concentration can affect the physical properties of the edible film with a thickness of 0.1235mm, tensile strength of 2.4768 MPa, elongation of 66.8786%, Modulus young 0.03702 and water vapor transmission rate of 2.5306 g/m<sup>2</sup>.hours and optimum immersion variations on grape quality green found at six times immersion.

**Keywords:** Edible film, glycerol, green grapes, soybean extract, and tapioca flour

### INTRODUCTION

Grapes are known to have many benefits, such as being anti-cancer and containing vitamins A, B, C, and E. Grapes are seasonal plants with harvests 2-3 times per year. The abundant availability of grapes during the harvest season actually causes farmers' problems, namely grapes that rot easily (Tawali and Zainal, 2004). This is because the wine's shelf life only reaches 3-6 days at room temperature, so it needs special handling to make it more durable both physically and minimize weight loss (Hilma et al., 2018).

One way to extend an agricultural product's shelf life is by efficient packaging such as plastics because it has barrier properties against oxygen, carbon dioxide, and water vapor (Apandi, 1984). However, this plastic is non-biodegradable, so that plastic waste can pollute the environment and is not safe for consumption (Ningsih, 2015).

Innovations regarding the manufacture of biodegradable packaging need to be made. This biodegradable packaging can be made from natural materials so that it is safe for the environment. The use of edible films as packaging can slow down quality degradation because the function of edible films is as an inhibitor of water vapor transfer, inhibits gas exchange, prevents loss of aroma, prevents fat transfer, improves physical characteristics, and as a carrier for additives (Krochta and Mulder, 1997).

According to Galieta et al. (1998), there are three components of natural polymers that are often used in making edible films, namely hydrocolloids, lipids, and composites. One of the sources of hydrocolloid is soy. Soybean (*Glycine max*) is an agricultural crop that contains high protein. Soybeans with superior varieties have a protein content of 35% and can even reach 40-43% (Natalia, 2017). Soy protein has an essential amino acid composition close to that of milk protein amino acids (Smith and Circle, 1972).

Another source of hydrocolloids is starch. Starch is often used as a biodegradable film because it is economical, renewable, and has good physical properties (Bourtoom, 2007). Tapioca starch or sweet potato starch has properties that are very potential for use in food industry as a thickener filler, binder and stabilizer. Starch also can be used as a basic ingredient in making edible film (Muljoharjo, 1997).

One of the edible films' weaknesses is fragile (Kester and Fennema, 1986). The property is easily brittle because the material for the edible film comes from natural polymers. To reduce the fragile nature, it is necessary to add a plasticizer (Andarini and Marseno, 2014). The most commonly used plasticizers are glycerol and sorbitol. Glycerol is a hydrophilic plasticizer, making it suitable for hydrophobic film-forming materials such as starch. Glycerol can increase the absorption of polar molecules such as water. The role of glycerol as a plasticizer, and its concentration increases the flexibility of the film (Krochta and De Mulder, 1997).

Previously, research on edible film characteristics from soybean extracts was carried out with tapioca flour and glycerol as food packaging materials, and obtained the best edible film was the addition of glycerol 4 mL / 100 mL soy milk (Sinaga et al., 2013). Furthermore, in making the edible film, it is tested with various parameters to determine its characteristics. Tests were carried out on mechanical properties and chemical properties. The mechanical characteristic tests carried out on the edible film were thickness, tensile strength, elongation, modulus of elasticity, and water vapor transmission (WVTR). Testing chemical properties using the FTIR (Fourier Transmission Infra-Red) instrument.

## MATERIALS AND METHODS

### Tools and materials

The tools used in this study include a set of the beaker, spatula, thermometer, 20x20 cm mica plastic, baking sheet, magnetic stirrer, porcelain cup, jar, filter, retort stand, oven, blender, hot plate, analytical balance, hairdryer, hotplate, screw micrometer, Texture and Tensile Strength Test Instrument (Universal Testing Machine), FTIR (Fourier Transform Infrared Spectroscopy) and Konica Minolta CR-400 Chromameter.

The materials used in this research include soybeans, commercially modified tapioca starch, p.a. glycerol, green grapes, distilled water, NaCl, and silica gel.

### Making soy milk

Soybeans are selected and weighed as much as 75 grams, then cleaned and washed. The soybeans are then soaked using boiling water for 60 seconds while stirring using a wooden stirrer. Soaked soybeans are then added with hot water with a temperature of 90°C as much as 450 ml, then crushed using a blender. The soybean slurry was then reheated at a temperature of 95-98°C while stirring using a glass stirrer for 10 minutes. The soy slurry is then filtered and squeezed using filter paper to get soy milk. Soy milk is cooled to room temperature (Ariani et al., 2017).

### Edible film making

A total of 100 ml of soy milk is put in a beaker glass and added with 10 grams of flour while stirring for 10 minutes until it is homogeneous. The mixture is then heated on a hotplate while stirring using a stirrer until the solution temperature reaches 60°C, and then glycerol is poured in variations (0.5; 1; 1.5; 2; 3 and 5%). The mixture is then stirred until all the mixture is homogeneous and reaches a temperature of 65-70°C. The heating was continued until the solution was gelatinized, then the solution was poured into an acrylic glass mold with a size of 20 x 20 cm. The mold is then dried in an oven at 50°C for 3 hours, and the dry edible film is left to stand overnight so that it can be removed from the mold and ready to be tested (Cicilia and Kurniati, 2017).

### Edible film characterization

#### Thickness test

The thickness is measured using a micrometer by placing the film between the micrometer jaws. For each film sample to be tested, the thickness is measured at five different points. The film thickness value is the average of the measurement results at these five points (Sari et al., 2013).

#### Tensile strength test

The tensile strength is measured with a length of 10 cm and a width of 1.5 cm. After that, what is done is to do the test using a universal tensile machine auto strain brand Yasuda Seiki. The results of the tensile strength test are measured based on the following formula:

$$\Sigma = \frac{F}{A} \dots \dots \dots (1)$$

#### Elongation test

The edible film was cut to a length of 10 cm and a width of 1.5 cm and then tested using a universal tensile m22 machine auto strain brand Yasuda Seiki. The result of elongation is measured based on the formulation:

$$\% \epsilon = \frac{\Delta l}{l} \times 100\% \dots \dots \dots (2)$$

#### Young's modulus

The young modulus test was obtained from the comparison of tensile strength and elongation (Setiani et al., 2013). The results of modulus young are measured based on the formulation:

$$\text{Young's modulus} = \frac{\text{tensile strength (MPa)}}{\text{elongation}} \dots \dots \dots (3)$$

#### Fourier transmission infra-red (FTIR) test

Fourier Transmission Infra-Red (FTIR) analysis through the functional groups contained therein. The sample in the form of a film is placed into a set holder on an infrared spectrophotometer with a wavenumber of 4000-400 cm<sup>-1</sup> to obtain the functional groups (Widyastuti, 2017).

#### Water vapor transmission test (WVTR)

The water vapor transmission test (WVTR) of the film was measured using the gravimetric method. The edible film tested was placed in the plate's mouth with a film size of 7 x 7 cm<sup>2</sup>, which contained 10 g of silica gel. The edges of the cup and the film layer are covered with wax or insulation. The cup is then put into a jar containing a 40% (w / v) NaCl solution. The water vapor that diffuses through the film will be absorbed by the silica gel and increase its weight. The equilibrium water vapor transmission rate condition is reached within 7-8 hours (steady-state conditions) and is weighed periodically every hour (starting from the 0th to the 7th hour). The increase in weight shows the diffusion rate of water vapor through the film. The data obtained is made a linear regression equation, and the WVTR value can be determined by the equation:

$$\text{WVTR} = \frac{\text{slope of incremental plate weight } (\frac{g}{\text{hour}})}{\text{surface area of sample (m}^2\text{)}} \dots \dots \dots (4)$$

#### Weight loss test

The application technique for the green grape edible film uses a coating method. Green grape samples were washed and dried and then divided into 3 groups, namely green grape samples without immersion as a control, green grape samples for 6 times immersion, and green grape samples for 10 times immersion. Green wine samples were immersed in an edible film solution with optimum glycerol concentration for 60 seconds, then removed and then dipped again. The repetition step of this immersion is repeated for 6 and 10 times of immersion. After being immersed in the edible film solution, the green wine samples were dried using a hairdryer and stored at room temperature around 25-27°C. Observations were made every day for weight loss.

Measurement of weight loss was carried out to compare the difference in weight before storage and after storage. The following is the weight loss formula:

$$\% \text{ weight Loss} = \frac{W_o - W_t}{W_o} \times 100\% \dots \dots \dots (5)$$

Note:

W<sub>o</sub> = Weight of the initial sample (grams)

W<sub>t</sub> = Weight of the final sample (grams)

## RESULTS AND DISCUSSION

The basic principle used in the manufacture of hydrocolloid-based edible films uses the gelatinization principle. Gelatinization causes the amylose bonds to be close together due to the presence of hydrogen bonds, and the drying process at the edible film manufacturing stage will result in shrinkage due to the release of water so that the gel will form a stable film.

### Thickness

One of the important parameters and influences the use of edible film as a food coating. The thicker the edible film, the smaller the permeability to water vapor and gas, and the more protected the packaged food (Rachmawati, 2009). The following are the results of measuring the thickness of the edible film of soybean starch extract and glycerol, as shown in Figure 1.

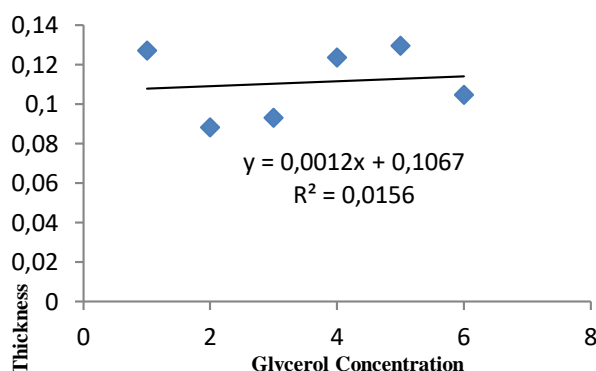


Figure 1. Diagram of the results of the thickness of the edible film extract of soybean starch and glycerol

Based on Figure 1, the best mechanical properties are at 2% glycerol concentration, with a thickness of 0.1235 mm. Good interaction can occur because the resulting edible film can be homogeneous between the matrix and the filler. According to (Bourtoom, 2007), the edible film's thickness can experience differences due to several factors, namely the difference in the volume of the edible film poured and the drying temperature. The edible film's thickness can be adjusted by adjusting the amount of solution poured into the mold and the area of the mold used. The more volume of the edible film solution is poured, the thicker the edible film is obtained (Widyastuti, 2017).

### Tensile Strength

Tensile strength is the maximum tensile strength that a film can withstand (Khotimah, 2006). Following are the results of the measurement of the tensile strength of edible film extracts of soybean-tapioca starch and glycerol are shown in Figure 4.3

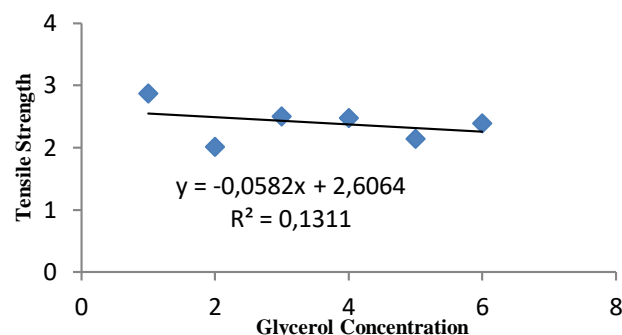


Figure 2. Diagram of the results of the tensile strength of edible film extract of soybean-tapioca starch and the addition of glycerol to check its x-axis

Based on the tensile strength test results presented in Figure 2, the resulting values ranged from 2.0189-2.8642 MPa with the highest value at 0.5% glycerol concentration and the lowest value at 1% glycerol concentration. This shows that the edible film's tensile strength tends to decrease, indicating that the higher the glycerol concentration, the lower the tensile strength. This is because the plasticizer will decrease the tensile force between the polymers when there is water evaporation, resulting in decreased resistance to the film's mechanical treatment.

### Elongation

Elongation is a condition where the edible film breaks after experiencing a change in length from its actual size when it is stretched. The following are the edible film elongation measurement results of soybean-tapioca starch and glycerol extracts shown in Figure 3.

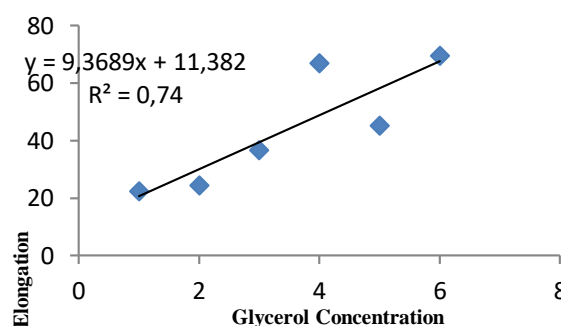


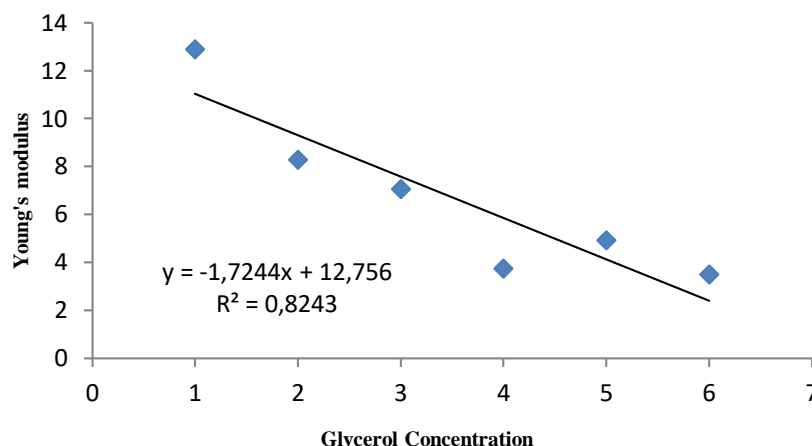
Figure 3. Diagram of the results of the edible film elongation of soybean-tapioca starch extract and the addition of glycerol to check its x-axis

Based on the elongation test (percent elongation), which is presented in Figure 3, the elongation value of edible film tends to increase, indicating that the higher the glycerol concentration, the greater the elongation produced. The increase in glycerol will decrease the cohesion bond between the polymers, which form an elastic film.

# Young's Modulus

Young's modulus is the stiffness elasticity of the resulting edible film. The following are the measurement

results of the modulus of young edible film extract of soybean-tapioca starch and glycerol shown in Figure 4.



**Figure 4.** Diagram of the modulus of young edible film extract of soybean starch and tapioca starch and the addition of glycerol.

The results presented in Figure 4 show that the modulus of young edible film extract of soybean-tapioca starch with the addition of glycerol ranges from 0.00345 - 0.1279. The addition of variations in the concentration of glycerol affects the modulus of young from the resulting edible film. The increasing concentration of the added variation of glycerol will decrease the edible film's

modulus young value.

## Spearman Correlation Statistical Test

The results of the Spearman correlation test on mechanical properties are presented in Table 1.

**Table 1.** Spearman Correlation Test Results

			Persen Gliserol	Ketebalan	Kuat Tarik	Elongasi	Modulus Elastisitas
Spearman's rho	Persen Gliserol	Correlation Coefficient	1.000	.135	-.368	.862**	-.891**
		Sig. (2-tailed)		.676	.240	.000	.000
		N	12	12	12	12	12
	Ketebalan	Correlation Coefficient	.135	1.000	.098	.032	-.053
		Sig. (2-tailed)	.676		.761	.922	.871
		N	12	12	12	12	12
	Kuat Tarik	Correlation Coefficient	-.368	.098	1.000	-.042	.238
		Sig. (2-tailed)	.240	.761		.897	.457
		N	12	12	12	12	12
	Elongasi	Correlation Coefficient	.862**	.032	-.042	1.000	-.951**
		Sig. (2-tailed)	.000	.922	.897		.000
		N	12	12	12	12	12
	Modulus Elastisitas	Correlation Coefficient	-.891**	-.053	.238	-.951**	1.000
		Sig. (2-tailed)	.000	.871	.457	.000	
		N	12	12	12	12	12

\*\* . Correlation is significant at the 0.01 level (2-tailed).

Based on the data shown in Table 1, it appears that the addition of percent glycerol to thickness has a correlation number of 0.135 and a significance value of 0.676. This shows that the resulting correlation is very weak. Meanwhile, the resulting spearman correlation value for tensile strength is negative 0.368, with a significance value of 0.240. This shows that the variation in the percentage of glycerol has a strong enough correlation with tensile strength. The spearman correlation value between the percent variation of glycerol and elongation is 0.862 and has a significance of 0.000. Based on these data, it can be seen that the variation of glycerol has a very strong correlation to the elongation value of the edible film.

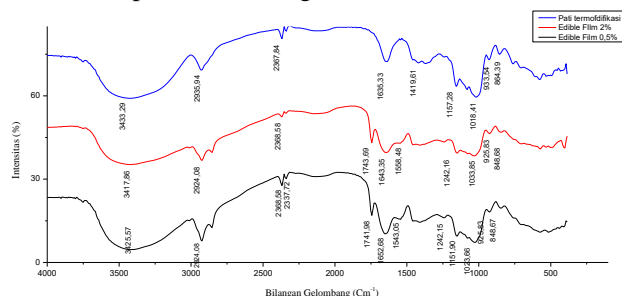
Besides, calculations using statistics show that the addition of the percentage variation of glycerol has a very strong relationship to the modulus of young with a negative correlation value. This suggests that the addition of percent glycerol tends to decrease the modulus of young. The

resulting correlation value between the two variables is - 0.891 and a significance value of 0.000. Overall, it can be concluded that the addition of percent glycerol has a very strong and significant relationship to elongation and modulus of young whereas, it has a sufficiently strong correlation with tensile strength and very weak correlation with thickness.

## Fourier Transform-Infrared Spectroscopy (FT-IR)

Characterization using FT-IR was carried out to determine the chemical properties of the edible film (Sastrohamidjojo, 2007). Identification of functional groups of soybean-tapioca starch extract edible film with 2% glycerol variation compared to soybean-tapioca starch extract edible film with 0.5% glycerol variation and modified tapioca starch as a control that appeared in IR spectra was carried out at a wavelength of 4000-400 cm<sup>-1</sup>. The results of the characterization of edible films using

FT-IR are presented in Figure 5.



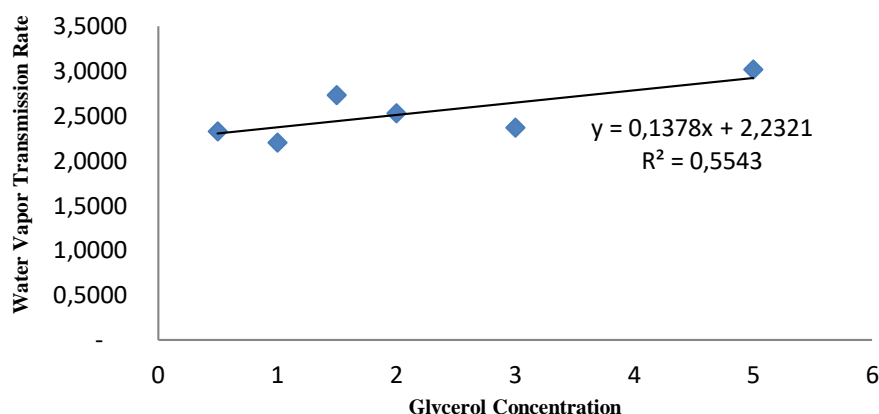
**Figure 5.** FT-IR Spectra (a) Modified Starch (b) 2% Glycerol Edible Film (c) 0.5% Glycerol Edible Film

Based on Figure 4.6, it can be observed that the results of the infrared spectrum on the modified starch have a C = O group in the 1635.64 absorption region. Then there is a C-H stretch bond at 2931.80 absorptions. Meanwhile, the 3425.62 absorption indicates O-H vibration and carbohydrates are strong at the absorption 1018.41-1157.28, indicating the presence of C-O. The

absorption rate of 933,54-884,39 shows the characteristics of different types of carbohydrates. Besides, for spectra (b) and (c), it can be seen that the resulting spectra are almost the same, namely spectra (b), there is C = O at 1643.35-1743.65 absorption while in spectra (c) at 1651.07-1743 absorption, 65. The broad bands at 3417.86 (c) and 3425.58 (b) arise from the N-H range, and the carbonyl group is part of the amide. The bands at 2924.09 in both spectra show a C-H bond that is characteristic of the aldehyde group.

### Water Vapor Transmission Rate

A packaged edible film's permeability is the ability to pass gas particles and water vapor over a unit area of material under certain conditions. According to (McHught and Sanesi, 2000), the value of WVTR represents the ability of an edible film or packaging to hold moisture that will enter the package. The following are measuring the water vapor transmission rate of the edible film extract of soybean-tapioca starch and glycerol shown in Figure 6.



**Figure 6.** Diagram of the results of the water vapor transmission rate of edible film soybean-tapioca starch extract and the addition of glycerol

Figure 6 shows that the value of the water vapor transmission rate of soybean extract and tapioca starch with the addition of glycerol is 2.2041-3.0204 g / m²h. The lowest water vapor transmission rate was when the glycerol concentration variation was 1%, while the highest water vapor transmission rate was when the glycerol concentration variation was 5%. The resulting graph shows

an increasing trend, which means that the increasing concentration of the added variation of glycerol will increase the water vapor transmission rate of the edible film. The edible film water vapor transmission rate test was further analyzed using Kruskal Wallis. Table 2. shows the real difference using statistical analysis of the Kruskal Wallis water vapor transmission rate data.

**Table 2.** Statistical Test of Water Vapor Transmission Rate with Kruskal Wallis.

	Air 1 Jam	Air 2 Jam	Air 3 Jam	Air 4 Jam	Air 5 Jam	Air 6 Jam	Air 7 Jam
Chi-Square	15.271	14.709	15.896	11.499	15.441	12.900	15.201
Df	5	5	5	5	5	5	5
Asymp. Sig.	.009	.012	.007	.042	.009	.024	.010

a. Kruskal Wallis Test

b. Grouping Variable: Konsentrasi

Based on the above calculations, the Kruskal Wallis of glycerol is significantly different from the water vapor value obtained can be concluded that the Asymp. Sig value transmission rate. on the statistical test is <0.05. This shows that the variation

### Weight Loss Test

The weight loss test was carried out on the weight loss of green grapes without coating (control), grapes with 6x

immersion repetitions, and 10x immersion repetitions for 14 days of storage. The weight loss percentage of green grapes during 14 days of storage is presented in Figure 4.8

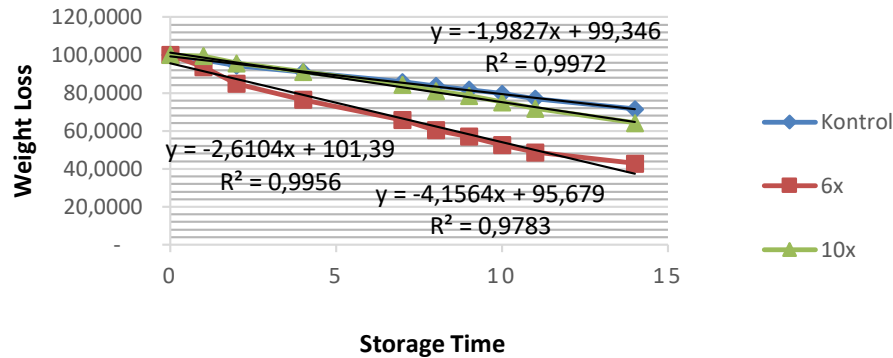


Figure 7. Graph of measurement results of weight loss of green grapes during storage

Based on Figure 7, it can be seen that the percentage of weight loss decreases with the length of the storage of green grapes. This can happen continuously and cannot be prevented because of the physiological and transpiration processes during storage (Widyastuti, 2017). Green grapes without edible film coating during storage experienced a weight loss percentage of 2.52% - 40.95%, the percentage of weight loss of green grapes with 6x immersion repetitions ranged from 13.00% - 66.91%, and the percentage of weight loss of green grapes with repetition of 10x immersion ranged from 0.08% - 51.26%. Based on these data, it can be seen that the largest

percentage in green grapes is 6x immersed, while the lowest percentage of weight loss is in green grapes without dyeing.

Changes in the surface of green grapes during shelf life to wilt and wrinkles. This can occur because the high water vapor transmission rate results in dehydration. Measurement of weight loss is clarified using statistical analysis Kruskal Wallis to see the significant difference from the dyeing treatment variation. The following are the results of weight loss analysis with Kruskal Wallis statistics in Table 3.

Table 3. Results of Weight Loss Analysis with Kruskal Wallis Statistics

	Hari ke 0	Hari ke 1	Hari ke 2	Hari ke 4	Hari ke 7	Hari ke 8	Hari ke 9	Hari ke 10	Hari ke 11	Hari ke 14
Chi-Square	.000	8.573	4.819	5.135	4.292	4.292	4.643	4.713	4.351	4.433
df	2	2	2	2	2	2	2	2	2	2
Asymp. Sig.	1.000	.014	.090	.077	.117	.117	.098	.095	.114	.109

a. Kruskal Wallis Test

b. Grouping Variable: Variasi

Based on the above calculations, it can be seen that the asymp value. Sig <0.05 was found on day 1, which stated no significant difference on day 1. Broadly speaking, there was no significant difference in weight loss for 14 days of storage on immersion reps.

### CONCLUSIONS

Based on the research that has been done, it can be concluded that:

1. The optimum composition for making the edible film is obtained in the edible film with the addition of 2% glycerol. The addition of 2% glycerol to the edible film from soybean extract with the addition of tapioca flour and glycerol can affect the physical and mechanical properties of the edible film with a thickness of 0.1235 mm, tensile strength of 2.4768 MPa, percent elongation of 66.8786%, modulus of elasticity 0.03702 MPa and WVTR 2.5306 g/hr.m<sup>2</sup>.
2. The variation of the repetitions of dyeing the film solution on the quality of green grapes obtained the optimum immersion at 6 times.

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