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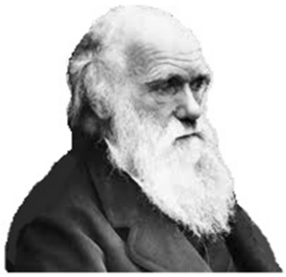

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Effect of Addition Glycerol Plasticizer to the Characteristics of Catfish (*Pangasius* sp.) Surimi Protein Edible Film

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ABSTRACT

Catfish (*Pangasius* sp.) is a freshwater fish commodity that is processed into filet. The development of the catfish fillet industry has generated a lot of waste. One of the uses is making surimi, but not all surimi produced is of high quality. Surimi is an intermediate product that has undergone a process of washing, pressing, and freezing. Surimi with low quality can be improved as raw material for making edible films. Edible film is a thin layer made of material that is safe for consumption and biodegradable. This research aims to determine the best concentration of glycerol as a plasticizer in the catfish surimi protein edible film and to determine the characteristics of the edible film produced. The method that was used in this research is the experimental method with Completely Randomized Design (CRD) with three treatments and three repeats of addition glycerol plasticizer concentrations are 0.6%, 0.8%, and 1.0%. The parameters observed were thickness, tensile strength, elongation, transparency, and solubility. The resulting data were analyzed using ANOVA and continued with the Duncan test ($\alpha = 5\%$). The results showed that edible film with the addition of 1.0% glycerol plasticizer concentration has the best characteristics based on Japanese Industrial Standard (JIS) and statistic analysis with a thickness of 0.068 mm, tensile strength of 402.02 kgf / cm^2, elongation of 33.3%, clarity of 98.06% and transparency of 1.94.

***Keywords*:** waste, intermediate product, biodegradable, japanese industrial standard, Catfish, *Pangasius*

1. INTRODUCTION

Technological developments in food packaging function to maintain quality and extend shelf life in packaged products. The packaging that is currently still widely used is plastic which

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can cause environmental pollution because it is difficult to experience biodegradation and has a negative impact on health. Research on material science, especially food packaging materials, is needed to reduce the increasing use of plastics so that there is a need for environmentally friendly plastics that can maintain edible foodstuffs such as edible film.

The edible film is a food packaging material that can biodegrade naturally (biodegradable) so that it is environmentally friendly, safe for health and can be consumed together with coated food products. The main components making up edible film can be grouped into three categories, namely hydrocolloids, lipids, and composites (a mixture of hydrocolloids and lipids). Myofibril protein is a type of hydrocolloid that can be used as a component of edible film. Edible films made from proteins have good characteristics and mechanical inhibition is superior to edible films made from polysaccharides. This is because proteins contain 20 different types of amino acids and have special characteristics that make functional characteristics more varied compared to polysaccharides which are mostly homopolymers.

Myofibril protein used as a raw material for making edible films is surimi. Surimi is an intermediate product in the form of meat that has been washed, pressed, and frozen. Surimi contains a very high concentration of myofibrils because it can form elastic and rubbery products. One type of fish meat that can be made surimi is catfish meat from the fillet processing waste.

Catfish (*Pangasius* sp.) is one of the freshwater fish commodities that is determined as the national flagship commodity in the acceleration of the industrialization program. Catfish is a fish that is easy to be cultivated, has a high nutritional content that is favored by the community and has a high production volume. The production of catfish in 2015-2016 showed an increasing pattern of 339,069 tons in 2015 to 437,111 tons in 2016, but the production of catfish in 2017 decreased to 319,967 tons.

Catfish can be used as an industrial material by processing it into the fillet. This is because catfish has the advantage that it is easy to make a good fillet. After all, it has reddish-white flesh and relatively little thorns. The development of the catfish fillet industry raises new problems, namely the presence of waste generated by the industry. Waste from the processing of catfish fillet in the form of head, skin, tail-bone, belly flap meat, and stomach contents that have not been used optimally.

Utilization of fish meat waste into a product can produce sales value as it is processed into edible film from surimi raw materials. The results of research on making edible films made from trash fish surimi have a weakness that is the magnitude of porosity (pores) on the resulting edible film. This problem can be overcome by using plasticizer as additional material in making edible films to improve the characteristics of edible films and increase the elasticity and flexibility of the resulting edible films. One of the plasticizers used in making edible films is glycerol.

Glycerol is a polyhydric alcohol compound that has water-soluble properties (hydrophilic), can increase the viscosity of the solution, and bind water. The use of glycerol plasticizer is better than sorbitol because glycerol is easily mixed in a film and water solution, the appearance does not change during storage, decreases the stiffness of the polymer and increases the flexibility of edible film, whereas sorbitol is difficult to mix and easily crystallizes at room temperature. The making of the edible film from catfish surimi protein raw material with the addition of glycerol plasticizer has not been done much. Therefore, more specific research needs to be done on the use of catfish surimi myofibril protein in the manufacture of edible films with the addition of glycerol as a plasticizer to get the characteristics of good edible films with different concentrations.

2. RESEARCH METHODS

2. 1. Materials

The tools needed for making catfish surimi protein edible films are analytical balance, thermometer, spatula, grinder, washing container, calico cloth, freezer, knife, cutting board, polyethylene bag, measuring cup, beaker glass, hot plate, magnetic stirrer, pH meter, dropper pipettes, oven, molds (13 cm × 22 cm × 2.5 cm), digital micrometer, UV spectrophotometer, and orbital shaker. The ingredients used are catfish meat, ice, water, salt, catfish surimi, aquades, glycerol, and 1 M NaOH.

2. 2. Research Methods

The research method used is an experimental method using a Completely Randomized Design (CRD). The treatment given is the addition of glycerol as a plasticizer with different concentrations for edible film for 3 treatments (0.6%, 0.8%, and 1.0%) and 3 repeats.

2. 2. 1. Producing Catfish Surimi

Producing of surimi from catfish waste fillet meat is using the modified methods. The process of making surimi is performed by washing raw materials, in fillets to separate meat from bones and skin. Finely ground meat facilitates the washing process (leaching). For meat wash, water of temperature of 1-5 ºC is used, with a ratio of water and meat 3 : 1, twice washing for 10 minutes (adding 0.3% salt in the second washing). Filtering and pressing are done twice using calico cloth. Surimi is packaged and stored in the freezer (–25 ºC).

2. 2. 2. Producing Edible Film

Producing of edible films in this research uses the method of which was modified. The way it works is thawing surimi frozen for 30 minutes, then weighing 10 g of the total aquades used. Aquades were added as much as 100 mL and 1 M NaOH to pH 10, then stirring and heating at 55 ºC for 30 minutes. The obtained solution was reheated at 60 ºC, then glycerol was added each treatment (0.6%, 0.8%, and 1.0%). The solution is homogenized and heated for 25 minutes. The solution is poured into a mold and carried out the process of drying with an oven at 50 ºC for 16 hours, to obtain a film layer. The film layer of catfish surimi protein is cooled to room temperature. Edible film is separated from the mold and ready to be analyzed for its characteristics.

2. 3. Observed Parameters

2. 3. 1. Thickness

The thickness of the edible film is measured using a digital micrometer thickness gauge with an accuracy of 1 μm. Edible film thickness values are determined from an average of five measurement points.

2. 3. 2. Tensile Strength

Tensile strength is the maximum tensile force that a film can withstand until it breaks. Tensile strength is determined based on the maximum load when the film is torn. The tool used for testing is Universal Testing Machine (UTM) made by Orientec Co. Ltd with the UCT-5T model. Edible film samples 50 × 5 mm were placed between the grips with an initial distance of 50 mm / min. Tensile strength is calculated by dividing the maximum force exerted on the film until it is torn (N) with the area of film (m). The tensile strength can be calculated by the formula:

$$\text{Tensile strength (kgf/cm}^2\text{)} = \frac{\text{Fmax}}{\text{A}}$$

2. 3. 3. Elongation

Elongation is calculated by dividing the incremental length of the film when it is torn and the initial length of the film before being pulled. The elongation test was measured using the MPY Testing Machine. Before measuring, an edible film with a size of 25 × 150 mm is prepared and conditioned in a laboratory with 50% humidity (RH) for 48 hours. Instron is set to the initial grip separation 50 mm, crosshead speed 50 mm / minute and loadcell 50 kg. The elongation is calculated when the film is torn. Before drawing, the length of the film is measured to the limit of the handle called the initial length (b), while the length of the film after withdrawal is called the length after breaking up (a). The elongation is calculated using a formula:

$$\text{Elongation (\%)} = \frac{\text{b} - \text{a}}{\text{a}} \times 100\%$$

2. 3. 4. Transparency

Transparency is a picture of the clarity of edible film produced by material to transmit light. Transparency of edible film is measured using a UV spectrophotometer at a wavelength (λ) of 550 nm, then divided by the thickness of the film (x). Transparency is calculated using a formula:

$$T = \frac{A_{550}}{x}$$

2. 3. 5. Solubility

The solubility of the edible film is the percentage of film that is dissolved in water after soaking for 24 hours. Samples were cut 30 × 30 mm in size, placed in an aluminum cup which had been dried and weighed.

The film sample is put into an oven at 100 °C, for 30 minutes. The dry sample is weighed as the initial dry weight (W_0), then sample soaked for 24 hours. Samples that are not dissolved in the solution are removed and dried in an oven for 2 hours at 100 °C, stored in a desiccator for 10 minutes. Then the weight of the dry sample was re-weighed as the sample weight after immersion (W_1). Solubility is calculated using a formula:

$$\text{Solubility (\%)} = \frac{W_0 - W_1}{W_0} \times 100\%$$

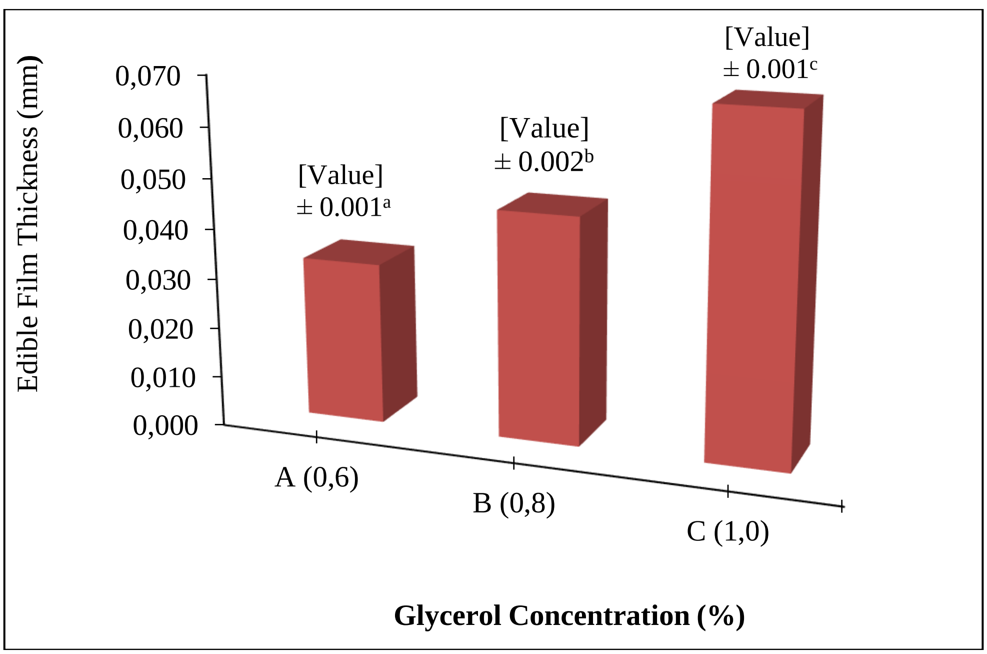
2. 4. Data Analysis

Observation data were analyzed descriptively and statistically using the F test or the Analysis of Variance (ANOVA) test with a confidence level of 95%. If the treatment has a significant effect (F_{count}> Ft_{able}) then it is followed by Duncan's multiple range test (α = 5%) to find out the best treatment.

3. RESULTS AND DISCUSSION

3. 1. Thickness

Thickness is an important parameter that influences the intended use as a product packaging or coating. The thickness will affect the physical and mechanical properties of edible films such as tensile strength, elongation, solubility, and water vapor permeability. The average value of edible film thickness is presented in **Figure 1**.



Note: The average value followed by the same letter shows no significant difference according to Duncan's Multiple Range Test at the 5% test level.

Figure 1. The average value of the thickness of the edible film at various concentrations of glycerol plasticizer

The thickness of the catfish surimi protein edible film ranged from 0.032 mm to 0.068 mm (Figure 1). The highest thickness of the edible film is with the addition of 1.0% glycerol plasticizer by 0.068 mm. The lowest thickness resulted from the addition of 0.6% glycerol plasticizer by 0.032 mm. The thickness value of the edible film obtained is quite good because it meets the standards according to the Japanese Industrial Standard (JIS) which is less than

0.25 mm. Thick coatings (> 0.25 mm) are not good because they can limit the exchange of gas produced by respiration so that the product can be easily damaged.

Based on Figure 1 one may see that the addition of glycerol plasticizer concentration will increase the thickness of the edible protein film of catfish surimi produced. This is in accordance with the opinion, the higher the concentration of glycerol plasticizer added will increase the solids in the solution so that the thickness increases and the polymers that make up the matrix of edible film more and more.

Thickness is formed due to the expansion or development of denatured protein molecules in surimi, thereby opening up the reactive groups present in the polypeptide chain. The bond between the reactive groups of proteins will hold all the liquid, then the gel will form. If the liquid is separated from the coagulated protein, the protein will precipitate and produce a film sheet.

The thickness of the edible film is affected by the area of the mold, the volume of the solution, and the amount of total solids in the film solution. The amount of volume of water in the edible film will affect the thickness of the film, where the greater the volume of water in the material will increase the thickness of the edible film with the same surface area. The difference in thickness value is also caused by the nature of the glycerol which is hydrophilic so that it binds more water which will evaporate after the curing process.

3. 2. Tensile Strength

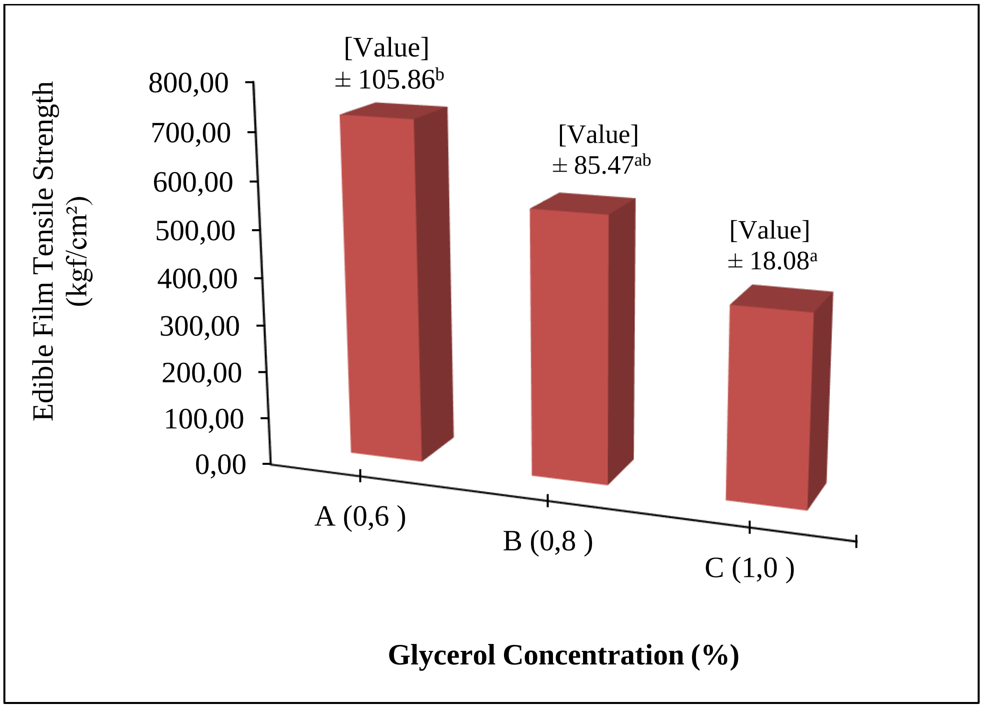
Tensile strength is the maximum pull that can be achieved until the edible film persists before tearing. Tensile strength determines the strength of edible film. The greater the tensile strength, the better edible film can withstand mechanical damage. Tensile strength that is too low indicates that the resulting film cannot be used as packaging, because the physical character is less strong and easily torn. The average value of edible film tensile strength is presented in **Figure 2**.

Research data from the addition of glycerol plasticizer concentration of 0.6%, 0.8%, and 1.0% obtained tensile strength values were 722.85 kgf/cm^2, 559.75 kgf/cm^2, and 402.02 kgf/cm^2 (Figure 2). Based on the standards of the Japanese Industrial Standard (JIS), the minimum value of tensile strength of edible films is 40 kgf/cm^2. The results showed that the tensile strength of edible protein from catfish surimi film met JIS standard, was of good quality and could be categorized as a food coating.

Based on Figure 2 it can be seen that with the addition of glycerol plasticizer concentration, the value of the tensile strength produced is lower. This is consistent with the opinion of which states that the addition of glycerol concentration has a tensile strength value tending to decrease, in line with the addition of glycerol concentration. Increasing the concentration of glycerol plasticizer causes the tensile strength of edible films to decrease due to decreased interaction between the molecules of the constituent of edible film constituents.

The tensile strength of edible films is influenced by the composition of the material in solution and the thickness of the edible film. The higher tensile strength value indicates that edible film can withstand pressure well and is not easily brittle. Edible film which has a high tensile strength value shows that the strength in holding pressure is getting better.

The high tensile strength value of surimi edible film is due to the influence of base addition which causes denaturation of surimi protein. This denaturation breaks the complex binding of proteins to simpler ones. The addition of surimi concentration will increase the amount of protein present and cause the tensile strength of edible films to increase.



Note: The average value followed by the same letter shows no significant difference according to Duncan's Multiple Range Test at the 5% test level.

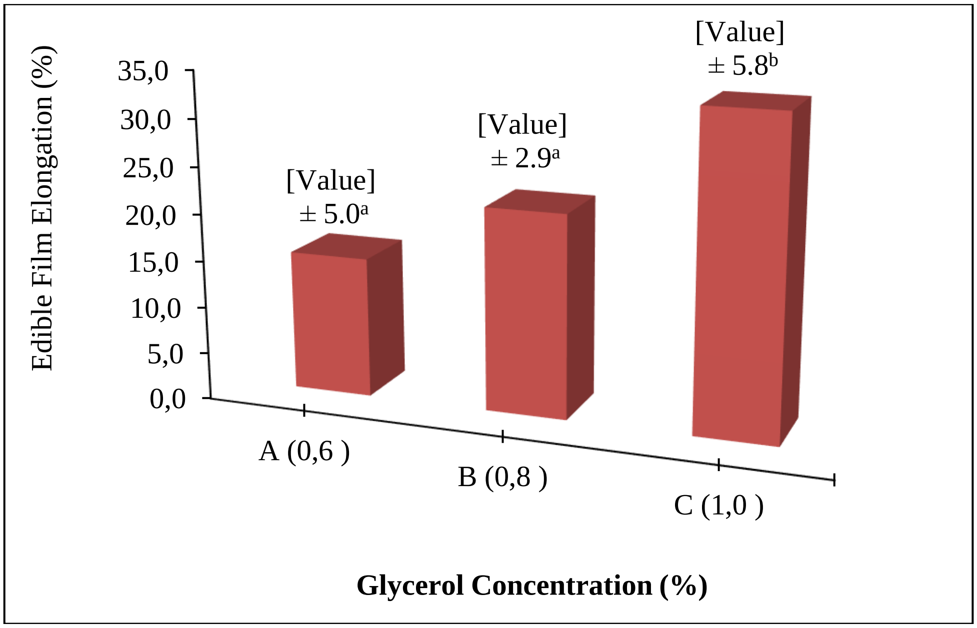
Figure 2. The average value of tensile strength of edible films at various concentrations of glycerol plasticizer

3. 3. Elongation

The elongation is the change in maximum length at the time of stretching until the film sample is torn. The elongation at the time of break shows a change in the maximum film length when obtaining a tensile force until the film is torn compared to the initial length. The elongation shows the plasticity of edible film. Plasticity is generally required by edible films to maintain its integrity when applied to food products. The average value of edible film elongation is presented in **Figure 3**. The elongation of catfish surimi protein edible films ranged from 0.032 mm to 0.068 mm (Figure 3). The addition of glycerol plasticizer concentration of 1.0% has the highest elongation value of 33.3% and the lowest elongation value is at the addition of glycerol plasticizer concentration of 0.6% by 15.0%. Based on the JIS (Japanese Industrial Standard) standard, edible film for packaging is categorized as good which has an elongation value of 10% - 50%. The elongation value of edible film in this research meets the JIS standard which shows that catfish surimi protein edible film has a flexible and elastic quality. Increasing the concentration of glycerol plasticizer causes the elongation of catfish surimi films to increase (Figure 3).

The results of this research are in accordance with that, as an increase in the concentration of glycerol plasticizer will increase the elongation value of the edible film produced. An increase in the amount of plasticizer decreases intermolecular forces, consequently the level of mobility between molecular chains increases.

This results in OH groups in glycerol which will form intermolecular bonds with reduced polymer chains. Increased glycerol will reduce the cohesion bonds between polymers that form a more elastic film. The use of glycerol as a plasticizer is able to reduce internal hydrogen bonds by increasing the free space between molecules to be filled by glycerol, thereby reducing stiffness and increasing film flexibility.



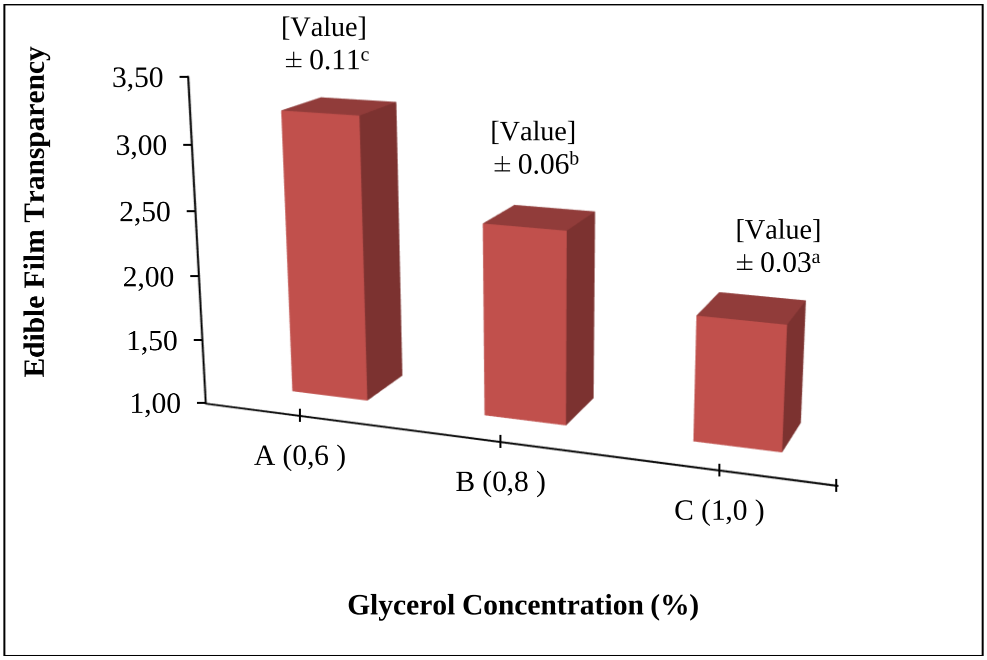
Note: The average value followed by the same letter shows no significant difference according to Duncan's Multiple Range Test at the 5% test level.

Figure 3. The average value of edible film elongation at various concentrations of glycerol plasticizer

The use of surimi as the base material for the edible film will affect the elongation value of an edible film. When addition of surimi concentration is used, the greater the value of elongation is produced. An increase in the concentration of surimi results in an increase in the amount of protein in the solution. When the solvent evaporates, protein aggregation occurs. The more protein, the opportunity for interaction between molecules increases, causing greater edible film flexibility and higher elongation value.

3. 4. Transparency

Transparency is a description of the level of clarity of edible film produced by material to transmit light. Transparency of edible film can increase consumer acceptance as a food packaging material and be one of the aesthetic assessments in the marketing of edible film. High-quality edible film has optical properties in the form of transparent colors so that the coated product can be seen. The average values of transparency and clarity of edible films are presented in **Figures 4** and **5**.



Note: The average value followed by the same letter shows no significant difference according to Duncan's Multiple Range Test at the 5% test level.

Figure 4. The average value of transparency of edible films at various concentrations of glycerol plasticizer

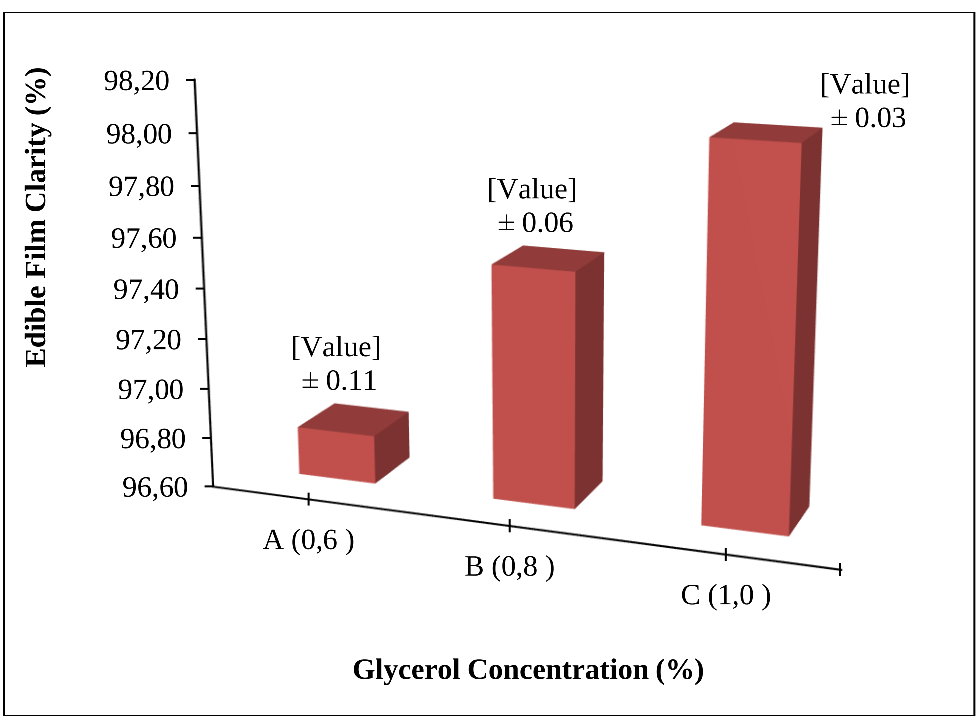


Figure 5. The average value of clarity of edible films at various concentrations of glycerol plasticizer

The addition of glycerol plasticizer concentration of 0.6% results in the highest transparency value of 3.20 ± 0.11 and the lowest transparency value with the addition of 1.0% glycerol plasticizer concentration, which is 1.94 ± 0.03 (Figure 4). Unlike the case with the value of the clarity of the edible film produced shows, that with the addition of a glycerol plasticizer of concentration 1.0% produces the highest clarity value of 98.06% and a clarity value of 96.80% results in the lowest value with the addition of a glycerol plasticizer of concentration 0.6% (Figure 5).

Based on research that has been done one may see that the addition of glycerol plasticizer concentration to catfish surimi edible film has a lower transparency value, but the higher the concentration of glycerol plasticizer added increases the value of clarity. High or low value of transparency shows a different appearance in the resulting edible film. A low transparency value indicates that the edible film produced has a clear appearance, while a high transparency value indicates that the edible film has a blurry appearance. Transparency tends to decrease with increasing plasticizer, meaning that the greater the concentration of glycerol added, the degree of transparency (clarity) tends to increase. This is consistent with the statement of that decreasing the value of transparency will increase the degree of clarity of the edible film.

3. 5. Solubility

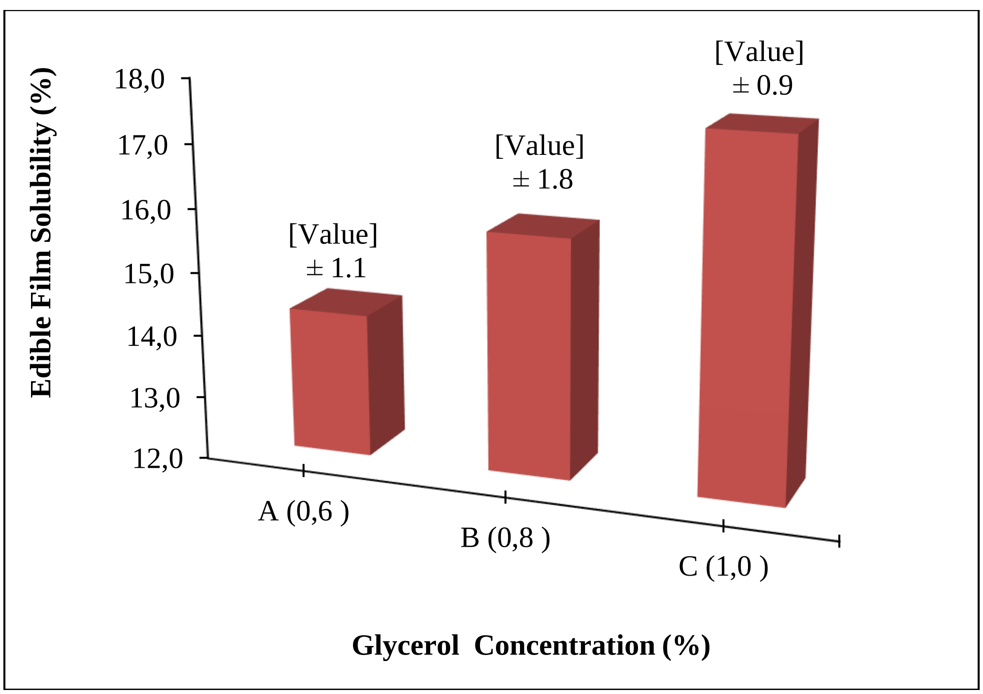


Figure 6. The average value of solubility of edible films at various concentrations of glycerol plasticizer

Solubility is the percentage of dry weight dissolved after being immersed in water for 24 hours, indicating the ability of the film in a liquid environment. The solubility of edible films is a factor that determines the film's biodegradability and determines the likelihood that an edible film can be applied. The solubility of the edible film in water is determined by the composition of the material in the formation of edible film. Edible films with high solubility show the film is easy to consume. The average values of solubility edible film are presented in **Figures 6**.

The addition of glycerol plasticizer of concentration 1.0% has the highest solubility value of 17.5% and the lowest solubility value of 14.3% with the addition of glycerol plasticizer of concentration 0.6% (Figure 6). Based on the test results obtained, the solubility value of the edible film is directly proportional to the addition of different glycerol plasticizer concentrations. Increasing the concentration of glycerol plasticizer results in higher solubility values.

The presence of plasticizer also affects the solubility of edible film. The greater the addition of glycerol, the greater the solubility of edible film in water. The higher the use of hydrophilic plasticizers (the ability to dissolve in water) such as glycerol, the easier it will be to form strong interactions with water and then form hydrogen bonds. This causes the percentage of solubility to increase with increasing hydrophilic components making up the edible film matrix.

If the application of a film is desired as a suitable packaging material, high solubility is desired, and vice versa, if the application of edible film in food as high in water content is used, then edible film is not soluble in water. The solubility of films is largely determined by the source of the film's base material. Edible films with a high solubility show the film is easy to consume.

3. 6. Recapitulation of Research Result

Based on the research results of the characteristics of catfish surimi protein edible films with the addition of different glycerol plasticizer concentrations on several parameters including thickness, tensile strength, elongation, transparency, and solubility, a recapitulation was made to see the treatment that produced edible films with the best characteristics. The recapitulation of the research results is presented in **Table 1**.

Table 1. Recapitulation of the Results Catfish Surimi Protein Edible Film with Addition of Glycerol Plasticizer

Parameter	**Glycerol Plasticizer Concentration**			**Japanese Industrial Standard (JIS)**
	0.6%	**0.8%**	**1.0%**	
Thickness (mm)	0.032[a]	0.045[b]	0.068[c]	Max. 0.25
Tensile Strength (kgf/cm^2)	722.85[b]	559.75[ab]	402.02[a]	Min. 40
Elongation (%)	15.0[a]	21.7[a]	33.3[b]	10 – 50
Transparency	3.20[c]	2.47[b]	1.94[a]	-
Solubility (%)	14.3	15.8	17.5	-

Note: The average value followed by the same letter shows no significant difference according to Duncan's Multiple Range Test at the 5% test level.

Success in making edible films is determined by the characteristics of the resulting film. Edible films should be with good characteristics for the criteria as packaging that meet the standards according to the Japanese Industrial Standard (JIS). Characteristics of a good edible film are as follows: a maximum thickness of 0.25 mm, a minimum tensile strength of 40 kgf/cm^2, elongation of more than 10%, low transparency with high clarity, and high solubility.

Based on Table 1 it is noticed that the thickness of the catfish surimi protein edible film has met the Japanese Industrial Standard (1975) standards. Thickness affects the use of film in the formation of products to be packaged and as a protector in physical damage. The addition of glycerol plasticizer of concentration 1.0% is the best treatment in the thickness parameter with a value of 0.068 mm.

The tensile strength parameters of catfish surimi protein edible films have values ranging from 402.02 - 722.85 kgf / cm^2 (Table 1). The value of tensile strength in this research can be categorized as a food packaging film because it meets the Japanese Industrial Standard (1975) standards. Edible films that have a high tensile strength value can protect packaged products against mechanical interference. Based on the research results, the tensile strength value of edible film of 722.85 kgf/cm^{2}with the best treatment is the addition of a 0.6% glycerol plasticizer concentration.

The elongation value of catfish surimi protein edible films ranged between 15.0 - 33.3% (Table 1); this value indicates that the elongation of edible films in this study met the Japanese Industrial Standard (1975) standards. Based on the results of the edible film, the addition of 1.0% glycerol plasticizer concentration is the best treatment with a value of 33.3%.

Transparency value of catfish surimi protein edible films ranged from 1.94 to 3.20 and clarity values ranged from 96.80 - 98.06% (Table 1). Edible film which is categorized as a food packaging that has a low transparency value and high clarity value looks more attractive [14]. Based on research results, the addition of 1.0% glycerol plasticizer concentration is the best treatment with a transparency value of 1.94 and a clarity value of 98.06%.

The solubility value of edible protein from catfish surimi film ranged from 14.3-17.5% (Table 1). Based on the research results, the solubility value of catfish surimi films cannot be used as a parameter to determine the best edible film characteristics, because the analysis of variance of the 5% test level showed no significant effect. Edible films with a high solubility indicate that the film's resistance to water is lower and indicates the film is easy to consume.

Based on the recapitulation of research results, the best collection of edible film protein gained was obtained from the acquisition of 1.0% glycerol plasticizer. This choice is due to the addition of edible film with the addition of 1.0% glycerol plasticizer concentration which has a high elongation value and transparency value, as well as good thickness and tensile strength value so that the edible film is easily consumed and applied as packaging.

4. CONCLUSIONS

Based on the results of research that has been done it can be concluded that the addition of glycerol plasticizer concentration significantly affects the characteristics of the thickness value, tensile strength, elongation, and transparency. The results which have no significant effect on the solubility of edible film are shown. The effect of adding glycerol plasticizer concentration on the characteristics of catfish surimi protein is presented. It results in increasing the value of thickness, elongation, transparency, solubility, and decreasing the tensile strength value. Edible films produced in this research have met the JIS (Japanese Industrial Standard) standard. The addition of glycerol plasticizer by 1.0% produces an edible film of catfish surimi protein with the best characteristics having a thickness value of 0.068 mm, tensile strength of 402.02 kgf/cm^2, elongation of 33.3%, clarity of 98.06%, and transparency of 1.94.

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