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Modification of Cassava Peel Starch, Substituting Chitosan and Seaweed: Production of High Quality Edible Film

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Abstract: Biodegradable packaging materials from biomass have been proven to be effective and efficient in improving the quality of materials food because it has the advantages of long shelf life, biodegradable, safe for health and cheap easy to use. The aim of the research is to determine the best formulation and water vapor rate properties as well as the mechanical properties of edible film based on cassava peel starch with the addition of palm oil and chitosan. The method used to determine the best formulation of edible film by making cassava peel starch, mixing raw materials and printing edibles film. Next, the water vapor rate and mechanical properties tests were carried out which included tensile strength tests, elongation tests and biodegradation tests. Results What was obtained from this research was the optimum concentration, namely 5 gr of cassava starch, 2 ml glycerol, 2 grams chitosan and 1 ml Palm oil with edible film characteristics was obtained respectively for the water vapor transmission rate test of 15.37 gr/hour.m2, tensile strength test of 0.66 MPa, elongation test of 4.05%, thickness test of 0.17 mm, and biodegrability test for 2 Sunday.

Keywords: Cassava peel starch; Chitosan; Edible film; Palm oil

Introduction

The quality of food ingredients is determined by the packaging materials used which affect their shelf life (Perera et al., 2023). The packaging material commonly used is plastic packaging material because it has the advantages of being transparent, flexible, light, not easily broken, waterproof and non-corrosive (Channa et al., 2022). The increasing awarenesses of environmental problems caused by plastic packaging has led to the used of ecofriendly and biodegradable products, including edible film. This film can be consumed and incorporated into food products as a cover or packaging. The benefit of edible film is protected food quality by maintaining moisture, avoiding spoilage, and preventing contamination. In addition, edible film or coating supply as a barrier to environmental disturbances that can potentially reduce the quality and appearance of the product.

The material used to make plastic is a material with a very complex molecular structure, making it difficult for plastic to be degraded by microorganisms and causing pollution and damage to the environment. It takes hundreds of years for microbes to recycle plastic waste, causing pollution (Tang et al., 2023). Overcoming this problem, we can develop more environmentally friendly packaging, namely edible film.

Edible film is a thin layer that functions as packaging for food products that are biodegradable, namely environmentally friendly, easily decomposed naturally and do not have a negative impact on health (Szabo et al., 2022). Edible film functions as a barrier to protect food products from light, humidity of a product, transmission of water vapor, preventing significant reduction in quality. Edible film is composed of three main components, namely hydrocolloids, lipids and composites. Hydrocolloid edible films can be formed using polysaccharides

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(starch, alginate, pectin, gum arabic) and proteins (gliten, gelatin, casein, soy protein). Edible films with the main component of lipids can be formed from fat-based ingredients such as glycerol, beeswax, essential oils. Meanwhile, the main component of the composite is a combination of hydrocolloid and lipis to form an edible film that has better physical characteristics (Kulkarni et al., 2021).

One of the best breakthroughs is count material that has bioactive compounds that contain antioxidant project. In packaging technology, usied antioxidant compounds can reduce the oxidation process of fats and oils and food spoilage. In addition, it can give food's shelf life and fat stability, and prevent nutritional qualities reduction. Since starch- based film lacks antioxidant compounds, incorporating antioxidant compounds will create the ability of the film to scavenge free radicals, and converting purple DPPH radicals into yellow (Colussi et al., 2021).

Antimicrobial packaging films play a crucial role in saving food from microbial contamination and grow their shelf life .. The most important quality of packaging film is that it should have good mechanical properties and enough permeability to water vapour and oxygen. Conventional petroleum based polymeric materials, such as, polyethylene, polycarbonate, polyethylene eterephthalate, polyvinylchloride, polypropylene, polystyrene and polyamides have been showing to be most suitable packaging material. However, environmental worries with approval to their production and disposal have necessitated the search for another, environmentally benign counterparts with comparable thermal, mechanical and barrier properties. In this regard, bio-based polymer films can be termed as successful alternatives on account of their easy availability, low cost and biodegradability. However, these polymers still suffer from demerits like low tensile strength and high degree of water absorption. These demerits of biopolymers can be overcome by combining them with other polymeric materials that escalate the desired properties of the film.

The characteristics of edible film are determined by the concentration of hydrophilic and hydrophobic molecules. Several parameters are usually used to determine the characteristics of edible film, namely film thickness, water content, tensile strength and elongation, biodegradation and water vapor transmission rate. Film thickness is influenced by the concentration and size of the printer. The higher the concentration of dissolved solids, the thicker the film produced, resulting in the longer the ability to protect the product and increase the shelf life of the product (Widodo et al., 2019). Thickness determines the film's resistance to the rate of transfer of water vapor, gas and other volatile compounds. The thickness of the film is influenced by the total amount of solids in the solution and the thickness of the mold. With the same mold, the film formed will be thicker if a larger volume of solution is poured into the mold. The total solids will form a thicker film with greater amounts.

The advantages of an edible film include inhibiting the diffusion of oxygen and water vapor into the coated material, inhibiting spoilage by microbes and being safe for consumption. The raw materials for making edible film are protein (polypeptides), carbohydrates (polysaccharides) and fats (lipids). These three materials are thermoplastic, so they are easy to print as edible films. This polymer has the advantage of being renewable and easily biodegradable. Starch is a type of polysaccharide that is abundantly available in nature, is biodegradable, easy to obtain, and more economical (Díaz-Montes, 2022). The characteristics of cassava peel starch are water content 10.14%, gelatinization temperature 63^0C, starch content 93.46%, and amylose content 9.69% (Mudaffar, 2021). So this makes it possible to use cassava peel starch as a basic ingredient for making edible films. Starch can be found in various types of root crops or grains. The main components of starch are amylose and amylopectin. Amylopectin and amylose levels affect the properties of the starch. The higher the amount of amylopectin, the starch will absorb very little water, be wetter and stickier. In making edible film, cassava skin is used as a raw material from which its starch content will be utilized. The starch content in cassava skin ranges from 44%-59% (Kong et al., 2022). Chitosan is a natural polysaccharide that has a positive charge with a pKa value of around 6.3 – 7.3 and has been used for various purposes such as in the field of health care (Murphy et al., 2023) food and beverage products (Pangestika et al., 2021), waste water treatment (Mustafiah et al., 2018), separation membrane (Kulkarni et al., 2021), anti-oxidant (Lee et al., 2019), dissolved CO_2 gas sensor (Usman et al., 2020), and inhibitor of bacterial growth because it can suppress the metabolic processes of bacteria (Murphy et al., 2023). Based on the things described, the aim of the research is to determine the optimization of the composition of edible film made from cassava peel starch substituted with chitosan and seaweed to produce high quality packaging materials and determine the characteristics of edible film including water vapor transmission rate, tensile strength and biodegradable properties.

The functional properties and characteristic of starch will depend on type, environmental factors, botanical origin and growing conditions. The main chemical component of starch is cereal grains, comprising about 90% of the dry weight of rice grains. Mush starch has weaknesses in terms of mechanical deficiencies and liquid holding ability. However, mush starch has much lower hygroscopic properties than cassava starch, rice starch, or even potato starch. Mush starch is a biopolymer that is widely used because of its high biodegradability so that it is able to form a film matrix. The more starch used, the denser the film matrix formed. This has an impact on increasing the tensile strength of the film. Generally, corn starch has about 75% branched amylopectin polymer and 25% linear amylose. The chemical properties of corn starch is: carbohydrate (84.07%), protein (2.76%), fiber (0.85%), ash (1.97%) and fat (3.02%). Corn starch films have proven to be excellent at form an oxygen-resistant films used to coat fish steaks.

The thickness of the film is one of the parameters that affect the biological, physical properties and shelf life of the coated material or product. Film thickness is often used to calculate the permeability, mechanical and optical properties of films. The thickness of the starch-based film increased with increasing amylose matter in starch. Thickness will affect the mechanical properties of the film included tensile power, elongation, water vapor transmission rate, flavor and texture of the product. The size and shape of the granules also significantly affect the thickness of the starch-based films. In research by colussi et al., film forming solutions for several types of starch with the same concentration obtained variations in film thickness values. The thickness of the potato starch-based film was the thickest (0.227 mm), then mush starch (0.196 mm) and the thinnest was wheat starch film (0.189 mm). The difference in layer thickness was caused by the amylose content in the type of starch, which was 20.90%, 25%, 26.90% for wheat, corn and potato starch, respectively. This is because the high amylose content will increased the interaction between amylose molecules to form stronger hydrogen bonds. This increasing the thickness of the film matrix and the heterogeneity of the film.

Method

Edible film production using cassava peel as raw material begins with the preparation stage, the stage of making edible film from cassava peel starch using methods, and the analysis stage of water vapor rate and mechanical properties; Making Starch from Cassava Skin; Wash 100 grams of cassava skin using running water and soak for 24 hours and change every 8 hours until the color of the soaking water is slightly clear; Then the crushing process is carried out using a blender and 100 ml of water is added to make the crushing easier; The cassava pulp is filtered and left for 30 minutes to get a precipitate of cassava skin pulp; The precipitate is then dried in the sun for about 1-2 days or in the oven at 70^{o}C for 30 minutes; Making edible film. Put 50 grams of cassava peel starch into a glass beaker. Add distilled water to the limit of 300 ml. Heated using a hot plate at 7^{0}C until gelatinization occurs. Stirred using a magnetic stirrer at a speed of 300 rpm. Add 90 ml of glycerol. Add chitosan according to the treatment (4 gr and 8 gr) little by little until homogeneous. Added palm oil according to treatment 15 (4.5 mL and 9 mL). Cast in glass casting with a diameter of 20 x 20 cm and flattened.

Dried in a drying oven at 70^{0}C for 24 hours. Remove the Edible Film and place it in a desiccator for 24 hours and it is ready for analysis. 3.5 Chemical Testing a. Water Vapor Transmission Rate Test The water vapor transmission rate was measured using the gravimetrically determined cup method by cutting the edible film into a circle with a diameter corresponding to the surface of the cup. Weigh the initial weight of the cup and add 3 grams of silica gel. Coated with liquid wax on the surface of the edible film. Conditioned at room temperature for 24 hours. The final weight of the sample is weighed and calculated using the formula: WVRT = (W-W0)/(t x A) Information: W0 = Initial weight W = Final weight after 24 hours t=time (24 hours) A = film area (m2); Tensile Strength Test Cut the sample in the shape of the letter "I" with dimensions of 7x3 cm. placed on the analyzer vertically upwards with the surface area observed. Run the tool and note the amount of compressive force on the edible film. Calculated using the formula: Tensile strength = Information: F= Compressive Force (N) A= Surface Area (cm^2); Elogancy Test Cut the sample into the shape of the letter "I" with a size of 7x3 cm. placed on the analyzer with the initial length observed.

Run the tool until the edible film. Calculated using the formula: % Elongation = P1-P1/P2 x 100 % 16 Note: P1 = Initial Length P2 = Final Length d. Film Thickness Test A thickness test is carried out to determine the thickness of the edible film sample being made. In this test the edible film was measured at three different points using a screw micrometer. Then the measurement results are averaged as a result of the thickness of the edible film samples made. In this research, a biodegradation test was carried out to find out how long it took for samples to degrade using the method of planting samples in soil. Previously, the sample was prepared first with a size of 4 x 1 cm2 which was then placed in a pot containing soil, the sample was left exposed to the open air without being covered with glass. The samples were then observed for physical changes that occurred qualitatively every two days for 15 days.

Cells were sonicated prior to the extraction to kind the process more effective and efficient. The extraction process was repeated until the color of the rest became pale. Furthermore, the ethanol extract was concentrated using a rotary evaporator, while the water extract was dried using a freeze-dryer.

Packaging into active or intelligent packaging, and being some of them edible films/coatings. A film is usually considered as a thin standalone solid sheet manufacturing using at least one processing system, and it is then applied and used to wrap or contain food products. On the other side, a coating is applied directly to the food product surface in liquid form, and using methods such as dipping, spraying, or brushing. The authors would like to clarify that sometimes the term ‘film’ is also used for coatings, since the film formation is made in situ, coating the food product surface.

Result and Discussion

The result of this research is an edible film from cassava peel starch using the heating method. Making edible film in this research uses cassava peel starch as the main ingredient and other supporting ingredients, namely glycerol, palm oil and chitosan. These ingredients give character to edible film. To make cassava husk starch as the main ingredient, clean cassava skin is soaked in water for 24 hours, the soaking water is changed every 8 hours. Then, the cassava skin is blended until smooth and strained. The results of the filter are precipitated, then the precipitate is dried for 24 hours under sunlight.

The starch produced was 18.37% in 300 grams of dry cassava skin. There are several factors that influence the amount of starch content in cassava skin, such as the type of soil used for planting, the nutrients contained in the soil, and the temperature and humidity (Ariani, 2017).

Figure 1. Stages of starch making

Then the stage of making edible film uses cassava peel starch as the main ingredient with the addition of palm oil and chitosan, as well as glycerol as a plasticizer. At this stage the materials are heated until they gelatinize, the film is printed and then dried to form a film. The results of the formulation can be seen in the following image:

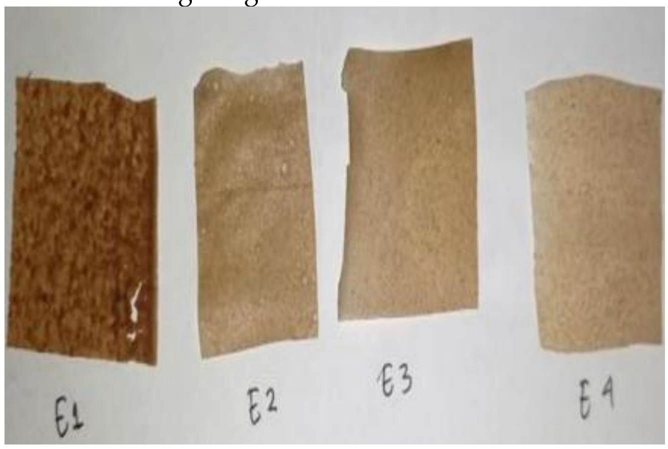

Figure 2. Edible film formulation results

Based on Figure 2, an analysis of the texture of edible film was carried out using the five senses, namely by looking, touching and looking at the edible film. Edible sample 1 has a texture that is too elastic so the film becomes brittle, has a rough surface, has a dark brown color and is quite oily due to the amount of oil used being too much. Then in edible 2, it has a stiff texture, there are lumps of chitosan that do not dissolve due to the low heating temperature and stirring speed, the film is perfectly formed, not oily and produces a light brown color.

In edible sample 3, a fairly elastic film was formed, there were no chitosan lumps, the edible film was perfectly formed, not oily and had a light brown color. This is because starch successfully binds chitosan and glycerol. Then, edible sample 4 has an elastic texture, there are no lumps, it is not oily and the film is perfectly formed.

A study showed higher antioxidant activity in microalgae that extracted with ethanol than that with water. High antioxidant activity may originate from high polyphenols compound.

Water Vapor Transmission Rate Test

Testing the vapor transmission rate related to the water vapor permeability of the film where the edible film is able to hold water vapor and gas particles in a unit of material under certain conditions. However, in reality, edible film is not absolutely capable of holding gas and water vapor, so in this research it is hoped that the addition of palm oil to cassava peel starch-based edible film can reduce the rate of water vapor transmission.

The results of the water vapor transmission rate test above show the influence of the addition of glycerol and palm oil. Edible-1 sample with the addition of 3 ml of glycerol and 5 ml of palm oil, showed the lowest water vapor transmission rate value of 2.9865 g/hour.m². Meanwhile, the edible-3 sample with the addition of 1 ml of glycerol and 2 ml of palm oil showed the highest water vapor transmission rate value, namely 17.6148 g/hour.m². Based on the Japan Industrial Standard (JIS), the standard water vapor transmission rate on film is a maximum of 7 g/hour.m2, so edible 1 and edible 2 samples meet the standard with a water vapor transmission rate value below 7 g/hour.m2, namely 2.9865 g /hour.m² and 6.7665 g/hour.m².

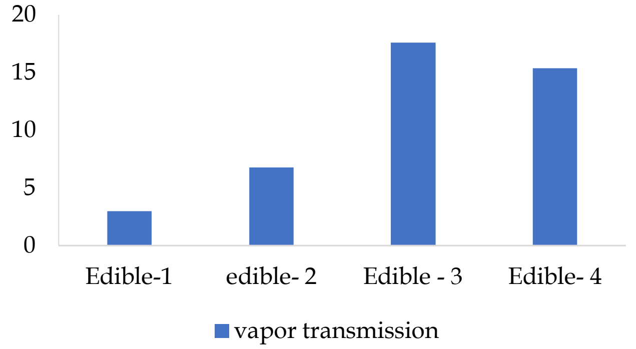

Figure 3. Test results for edible film water vapor transmission rate

Water vapor permeability of a packaging film is the rate of speed or transmission of water vapor through a unit area of material with a flat surface of a certain thickness. This occurs because of the difference in vapor pressure units between two surfaces at certain temperature and humidity conditions (Creswell, 2017).

Increasing the plastcitizer results in an increase in the concentration of gel formation which can reduce the rate of water vapor transmission due to an increase in the solution structure which causes the film matrix to increase so that the film structure becomes stronger with the network structure of the film becoming more compact and sturdy which can increase the strength of the film in withstanding the rate of water vapor transmission. Thus, the higher the plasticizer concentration, the lower the water vapor transmission value produced. Apart from that, the polymer components that make up this occur because the polymer components that are aligned in a straight line will form a dense network and the space between cells in the edible film becomes narrower, making it difficult for water, enzymes and chemicals to penetrate. The polymer component of this research is composed of cassava peel starch, chitosan, and glycerol (Sitompul et al., 2017).

Tensile Strength Test

Tensile strength is the maximum tensile force that a film can withstand before the film breaks or tears. A tensile strength value that is too small indicates that the film cannot be used as packaging, because its physical characteristics are not strong enough and it breaks easily.

Table 1. Test Results for Edible Film Tensile Strength Values

Sample	Tensile strength value (MPa)		Average
	I	II	
Edible – 1	0.08	0.04	0.06
Edible – 2	0.37	0.30	0.33
Edible – 3	0.46	0.45	0.45
Edible – 4	0.80	0.52	0.67

The best edible film is assessed based on tensile strength, the higher the tensile strength, the better the characteristics of the edible film, because edible film with a high tensile strength value is able to protect the product from various pressures and damage. The highest tensile strength of edible film in this study was 0.45 Mpa-0.66 MPa, which meets the Japanese Industrial Standard (JIS) standard, namely a minimum of 0.39 MPa. The tensile strength analysis results in table 7 show the average test results of edible film where the greater the plasticizer concentration, the lower the tensile strength of edible film. This was proven in edible samples 1 and 2 where 3 ml of glycerol was added. The effect of adding plasticizers can cause the resistance to mechanical treatment of edible films to decrease due to a decrease in the tensile force between polymers when water evaporates.Another factor that influences tensile strength is the type and concentration of edible film constituents. The use of surfactant with the right concentration and type will influence the homogeneity of the film suspension formed (Santoso et al., 2017). A homogeneous film suspension will produce an edible film that is dense, dense and even, thus affecting the mechanical properties of the edible film.

Elongation Test

Softness or elongation is a characterization of edible film based on the increase in length of a material when tensile strength treatment is carried out. The analysis results were obtained by comparing the length of the film before being pulled by the tool and the length of the film when it broke.

Based on, it can be seen that the higher the addition of palm oil and glycerol, the higher the percent elongation produced. The research results showed that edible film with the addition of palm oil and glycerol of 5 ml Mand 3 ml respectively, the elongation value was 15.32%, with the addition of palm oil and glycerol of 2 ml and 1 ml respectively, the elongation value decreased to 4.52 %. According to the Japanese Industrial Standard (JIS), the percent elongation value of edible film is at least 10%, so that edible samples 1 and 2 in this study meet the standard. The higher the oil concentration, the greater the decrease in intermolecular forces between the polymer chains so that the film will be more elastic. Apart from preventing the rate of water vapor, the function of lipids can also increase the elasticity of the film.

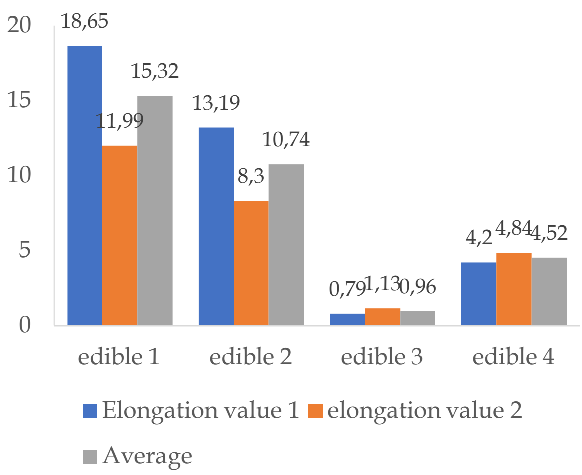

Figure 4. Test results for edible film elongation values

Test Film Thickness

The thickness of an edible film is influenced by several factors such as the area of the mold, the volume of the solution, and the total amount of solids in the solution. In this test the edible film was measured at three different points using a screw micrometer. Then the measurement results are averaged as a result of the thickness of the edible film samples made. The average value of edible film thickness can be seen in the table 5.

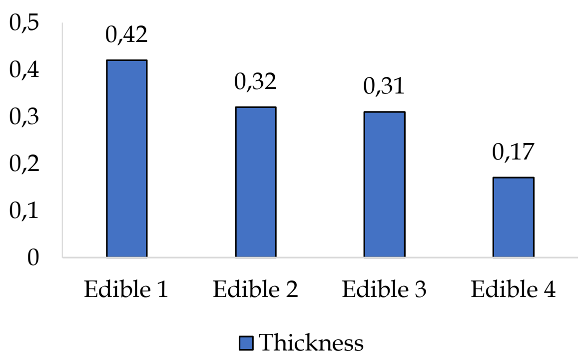

Figure 5. Test results for edible film thickness values

The figure 5 above shows that the thickness values of the edible films produced vary. The thickness of the edible film in this study was influenced by the concentration of glycerol and palm oil used. The greater the concentration of glycerol and palm oil in the edible film, the thicker the resulting edible film.

The thickness of the edible film is also influenced by the drying location of the sample. This is because during drying the sample has uneven areas resulting in different thicknesses. Based on the Japanese Industrial Standard (JIS), the standard thickness of edible film is ≤ 0.25 *mm*. So, it can be seen that edible sample 4 with a thickness of 0.17 mm has met the thickness standards.

Biodegradation Test

Biodegradation is a bioplastic process where the sample being tested is decomposed by microorganisms resulting in physical changes. In this test, the bioplastic is buried for 3 days, after which the sample is weighed. And here are the data results from biodegradation testing

Table 2. Biodegradation Results of Edible Film

Sample	$M_{Begin\ ing}$(gr)	M end	Lost heavy (%)
Edible 1	0.35	0.24	31
Edible 2	0.21	0.16	23
Edible 3	0.16	0.12	25
Edible 4	0.11	0.02	81

Based on the results of the biodegradation test in table 2, it shows that the edible film has lost weight. Physical degradation occurs with a decrease in weight, color changes and brittleness of the edible film. Chitosan in the solid phase experiences degradation because a number of NH2 groups from chitosan experience chain breaks and turn into ammonia gas after reacting with hydrogen radicals (Sarni et al., 2016). Glycerol and starch have OH groups which can initiate hydrolysis reactions after absorbing water from the soil. So the starch polymer will decompose into small pieces until it disappears in the soil. Polymers will be degraded due to the process of damage or reduction in quality due to breaking of chain bonds in the polymer. Soil is a growing medium for most bacteria and microbes, which will cause the edible film to become more easily gradable. The length of time for edible film to be fully graded can be determined by calculating the regression value for each sample. The x value at y=0 indicates the time when bioplastics completely decompose in the soil.

Table 3. Edible Film Destruction Time

Sample	Equality regression	Time is destroyed (Sunday)
Edible 1	y = -0.11x + 0.46	4.10
Edible 2	y = -0.05x + 0.26	5.20
Edible 3	y = -0.04x + 0.20	5.00
Edible 4	y = -0.09x + 0.20	2.20

Table 3 shows that the complete disintegration time for edible film is the fastest in edible 4 and edible 2 samples with the longest disintegration time. According to the European standard EN 13432, biodegradable packaging materials must decompose at least 90%, through biological activity, within a period of 6 months or approximately 24 weeks (Michalska da Rocha et al., 2018). Meanwhile, in this study, the time for complete decomposition was found to be around 2-5 weeks. So the edible film in this research complies with environmentally friendly packaging material standards.

Conclusion

Based on the results of this research, it can be concluded as follows: The optimum concentration in this experiment was the Edible 4 sample with a concentration of 5 grams of cassava peel starch, 2 ml of glycerol, 2 grams of chitosan and 1 ml of palm oil; Based on the results of testing the characteristics of edible film, data on the water vapor transmission rate value was 15.37 gr/hour.m2, tensile strength 0.66 MPa, elongation 4.05%, thickness 0.17 mm, and biodegradation for 2 weeks.

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Author Contributions
Conceptualization, H., F. D., H., I. S., R. H; methodology, H.; validation, F. D.and.; H. formal analysis, I. S.; investigation, R. H., and H.; resources, H. and. F. D; data curation, H.: writing—original draft preparation, I. S and R. H.; writing—review and editing, H.: visualization, F. D and H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest
The authors declare no conflict of interest.

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