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Characterization of Bioplastic Packaging from Tapioca Flour Modified with the Addition of Chitosan and Fish Bone Gelatin

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ABSTRACT

This research aimed to characterize bioplastic packaging made from modified tapioca flour so that it has the best thickness, transparency, tensile strength, elasticity and biodegradability values. This research was conducted at the Laboratory of Fisheries Products Processing, Faculty of Fisheries and Marine Sciences, Padjadjaran University, Jatinangor, starting from February-April 2019. Bioplastic were tested namely bioplastic in four treatments and three repeat with the addition of chitosan (K) and gelatin (G) concentrations, namely treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (4% K, 20% G). The data obtained were analyzed using a comparative descriptive method. The parameters observed included thickness, transparency, tensile strength, percent elongation, and biodegradability. Thickness testing with measurement methods at 5 different spots using a digital screw micrometer. Transparency testing was done by the method of inserting a sample in the spectrophotometer and the absorbance value was determined. Tensile strength and elongation tests were carried out by the withdrawal method at both ends of the sample using the Instron tool. Testing the value of biodegradability was carried out using the soil burial test method. The best bioplastic packaging produced is using the addition of 2% chitosan and 5% gelatin concentration which is seen from the thickness parameter values of 0,072 mm, transparency value of bioplastic 2,09, the tensile strength of 19,05 MPa, percent elongation 28,333% and can be degraded by soil after 14 days about 99,84% based on Japanese Industrial Standard (JIS).

Keywords: bioplastics, modified tapioca, chitosan, gelatin, biodegradability

1. INTRODUCTION

The packing is one of the important things that must be considered in the process of packaging a product, especially in food. The function of packaging is to be a physical protector between the outside environment and food so that it could avoid dirt and reduce oxidation [7]. Generally, the packaging used commonly is from synthetic plastic because it is lightweight, flexible, strong, and not easily broken [19]. But unfortunately the use of such packaging can cause several problems, such as soil pollution and also if the plastic packaging was heated up, it could produce harmful residues on food and can interfere with health [18].

The development of degradable packaging (bioplastics) is now being carried out. Bioplastics are perishable packaging materials by the activity of microorganisms when disposed into the environment, thereby reducing pollution. The benefit of using the bioplastics packaging is that it can provide protection against product quality and can extend the shelf life [5]. Bioplastic packaging can be made from hydrocolloids (proteins, polysaccharides), lipids (fatty acids, acyl glycerol, wax or wax), and composites (hydrocolloid and lipid mixtures). Polysaccharides that can be used for this package are modified starch, chitosan, carrageenan, cellulose, etc. [22].

Starch is a type of polysaccharide obtained from cassava extract [29]. Starch as a bioplastic forming material is due to having amylopectin levels which can affect the stability of bioplastics and amylose which influence the tensile strength and flexibility. Tapioca starch is easily degraded by microorganisms such as decomposing bacteria by breaking the polymer chain into its monomers [6].

Bioplastics made from tapioca flour have finer structures and high tensile strength values [14]. An edible film made from tapioca flour can produce transparent clear films and a more chewy texture [12]. Starch-based edible films have low water resistance, so they can affect the physical or mechanical properties of the edible film [33]. Starch needs to be added to other biopolymers such as materials that are hydrophobic and antimicrobial.

The making of bioplastic packaging can be added with fishery waste such as shrimp shells which are processed into chitosan and fish skin/bone made into gelatin. The addition of chitosan to the film can maintain the integrity of the film when applied to food products. The addition of gelatin can form a transparent film [10].

Chitosan has hydrophobic properties (waterproof material) and has antimicrobial effect [21]. The characteristic of chitosan can complement the shortcomings of the characteristics of starch-based bioplastic packaging [17]. Chitosan has amin functional groups, primary and secondary hydroxyl groups, so chitosan has high chemical reactivity because it can form hydrogen bonds and become ideal mixers [25].

Gelatin is a result of hydrolysis of collagen in animals, which comes from the skin/bone part which is used as thickener / condensing a liquid [32]. Gelatin has hydrocolloid properties which can form a thin and elastic layer. Gelatin is hydrophilic, which is poor retaining water [10]. Other than that, the addition of gelatin to bioplastics will work effectively if chitosan was added.

The edible film based on modified starch and carrageenan that added chitosan has a thickness, elongation value, and high water vapor value [28]. Starch-based biodegradable packaging with chitosan has good water resistance and can be degraded perfectly in shorter periode [17]. The edible film with mixture of chitosan and gelatin can extend the shelf life of the food and has good gas, oxygen, and aroma barrier properties [4].

The unfit addition of chitosan and gelatin can affect the quality or characteristics of the bioplastic produced. This study aimed to determine the best concentration of chitosan and gelatin addition to the manufacture of bioplastic packaging to produce the best characteristics.

2. MATERIALS AND METHODS

2. 1. Tools and Materials

The tools used include analytic balance, stove, pan, knife, jar, beaker glass, measuring cup, water bath, filter paper, measuring pipette, plastic tray, pH meter, hot plate, magnetic stirrer, oven blower, spatula, thermometer, plastic mold size 23 cm × 18 cm. The materials used include fresh tilapia bones, modified tapioca flour, chitosan, gelatin, 6% hydrochloric acid (HCl), 1% acetic acid (CH₃COOH), glycerol, aquades.

2. 2. Research Method

The research method used was experimental which consisted of four treatments and three repeat.

- A = the concentration of 2% chitosan and 5% gelatin (2% K, 5% G)
- B = the concentration of 3% chitosan and 10% gelatin (3% K, 10% G)
- C = the concentration of 4% chitosan and 15% gelatin (4% K, 15% G)
- D = the concentration of 5% chitosan and 20% gelatin (5% K, 20% G)

The formulation of bioplastics used in this research is presented in Table 1 below.

Table 1. Bioplastic Formulations

Materials		Treatment			
		A (2% K, 5% G)	B (3% K, 10% G)	C (4% K, 15% G)	D (5% K, 20% G)
Making Chitosan Solution	Chitosan (gram)	2	3	4	5
	Acetic acid 1% (ml)	100	100	100	100
	Glyserol (ml)	1,2	1,2	1,2	1,2
Making Starch Solution	Tapioca fluor (gram)	1	1	1	1
	Distilled water (ml)	100	100	100	100
	Glyserol (ml)	1,2	1,2	1,2	1,2
Making Gelatin Solution	Gelatin (gram)	5	10	15	20
	Distilled water (ml)	100	100	100	100
	Acetic acid 1% (ml)	2	2	2	2

The process of manufacturing bioplastic packaging refers to the research method used [28] with modifications. The process of making bioplastic packaging includes mixing 1% starch solution with chitosan solution with different concentrations (2%, 3%, 4%, 5%) then adding gelatin solution with different concentrations (5%, 10%, 15%, 20%). Starch-chitosan-gelatin solution (bioplastic solution) then stirred and heated using a magnetic stirrer on the hot plate with a speed of 4 rpm and a temperature of 65 °C for 20 minutes. The solution was then poured into a plastic mold size 23 cm × 18 cm as much as 90 ml then dried using an oven blower (T = 55 °C) for ± 24 hours. Bioplastics that had been formed were released from the mold to be tested for their characteristics.

2. 3. Parameters Observed

2. 3. 1. Thickness

Bioplastics thickness is measured using a digital screw micrometer Insize a brand with an accuracy of 0.001 mm. Thickness was measured at 5 different spots, namely one in the middle and four in the four each corners of the bioplastic packaging [23].

2. 3. 2. Transparency

Transparency is the ability of a material that can indicate the level of clarity of a material marked by the ability of the material to transmit light [31]. The transparency of bioplastics was measured using a spectrophotometer ($\lambda = 550$ nm). Transparency is measured using the Al-Hassan and Norziah method (2012) [3] and calculated using the formula:

$$T = A_{550} / x$$

Information :

$$\begin{aligned} A_{550} &= \text{Absorbance } (\lambda) 550 \text{ nm} \\ x &= \text{Bioplastic thickness (mm)} \end{aligned}$$

2. 3. 3. Tensile Strength

Tensile strength is the maximum pull that can be achieved until the film can remain before breaking [23]. Tensile strength was measured using Instron tools. The calculation of tensile strength values was calculated using the following formula [34].

$$\tau = \frac{F_{maks}}{A}$$

Information :

$$\begin{aligned} \tau &= \text{Tensile strength (MPa)} \\ F_{maks} &= \text{Maximum voltage (N)} \\ A &= \text{Cross-sectional area (mm}^2\text{)} \end{aligned}$$

2. 3. 4. Percent Elongation

Percent elongation is the percentage of length increment of a bioplastic/film which is measured from the initial length when withdrawal until the film breaks [11]. Elongation is measured using the same tool to measure tensile strength. The amount of elongation value was calculated using the following formula [34].

$$E = \frac{\Delta L}{L_0} \times 100\%$$

Information :

- %E = Extension break (%)
- ΔL = Length of specimen (mm)
- L_0 = Initial specimen length (mm)

2. 3. 5. Biodegradability

Biodegradability testing aims to determine the resistance level of bioplastics to the influence of decomposing microorganisms, soil moisture, temperature and Physico-chemical factors found on the soil. Biodegradability tests were measured using the Zulferiyenni et. al. method (2014) [34], namely bioplastics cut to size 10 cm x 10 cm and buried in the soil (12 cm depth) contained in plastic jars for 14 days, then visually carried out. The biodegradation value of bioplastics can be calculated using the formula:

$$\text{Losing Weight} = \frac{W_1 - W_2}{W_1} \times 100\%$$

Information :

- W_1 = Weight of plastic before biodegradation test
- W_2 = Weight of plastic after biodegradation test

2. 4. Data Analysis

The values of thickness, transparency, tensile strength, percent elongation, and biodegradability of bioplastics that have been observed are then analyzed. Data were analyzed using descriptive comparative analysis.

3. RESULT AND DISCUSSION

3. 1. Thickness

Thickness is included in physical properties that can determine the quality of the characteristics of bioplastics. Thickness must be adjusted to the product to be packaged. Thick films will increase the value of tensile strength but will reduce the elongation value [22]. The results of testing the thickness of bioplastics with the addition of chitosan and gelatin are presented in Table 2.

Table 2. Bioplastic Packaging Thickness Test Results Based on Chitosan and Gelatin Concentrations

Treatment	Average (mm)
A (2% K, 5% G)	0,072
B (3% K, 10% G)	0,148
C (4% K, 15% G)	0,227
D (5% K, 20% G)	0,332

Information: K = Chitosan, G = Gelatin.

Based on the thickness test results of bioplastics in each treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (5% K, 20% G) has values that they are equal to 0.072 mm, 0.148 mm, 0.227 mm and 0.332 mm. The lowest bioplastic thickness was found in treatment A (2% K, 5% G) and the highest was found in treatment D (5% K, 20% G). The thickness value of bioplastics increases with increasing concentration of the material used (chitosan and gelatin). Bioplastic packaging in treatment A (2% K, 5% G) has a lower thickness value than other treatments. Increasing the concentration of the material used will increase the total solids contained in bioplastics after drying so that the bioplastic produced will be thicker. This is in accordance with [8] that the higher the concentration of the solution used, the higher the bioplastic formed. Thickness can be influenced by the mold area, the volume of the bioplastic solution printed and the addition of glycerol [13].

The thickness of bioplastics in this research has values ranging from 0,072 mm – 0,332 mm. The thickness of the bioplastic packaging that met the JIS (Japanese Industrial Standard) standard is the thickness value in treatment A (2% K, 5% G), B (3% K, 10% G), and C (4% K, 15% G). Maximum thickness value based on the JIS of 0.25 mm. The thickness of bioplastics in this research has a value that is not much different from research recent [20] which has a thickness value in his research regarding the addition of gelatin to biodegradable which is in the range of 0,089-0,535 mm.

3. 2. Transparency

Transparency is the ability of a material that can indicate the level of clarity of a material marked by the ability of the material to transmit light [31]. The results of the transparency testing of bioplastic packaging with the addition of chitosan and gelatin are presented in Table 3. Based on the results of bioplastics transparency tests on each treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (5% K, 20% G) has a value consecutively by 2,09, 2,864, 3,05, 3,07 and the clarity degree values are 97,91%, 97,14%, 96,95%, and 96,93%. The lowest transparency value of bioplastics was found in treatment A (2% K, 5% G), but the value of the degree of clarity was highest. The highest transparency value of bioplastics was found in treatment D (5% K, 20% G), but the value of the degree of clarity was the lowest. The transparency value of the film is inversely proportional to the degree of clarity [3]. The appearance of the bioplastic packaging was shown in Figure 1.

Table 3. Bioplastic Packaging Transparency Test Results Based on Chitosan and Gelatin Concentrations

Treatment	Average Transparency	Degree of Clarity (%)
A (2% K, 5% G)	2,09	97,91
B (3% K, 10% G)	2,864	97,14
C (4% K, 15% G)	3,05	96,95
D (5% K, 20% G)	3,07	96,93

Information: K = Chitosan, G = Gelatin.

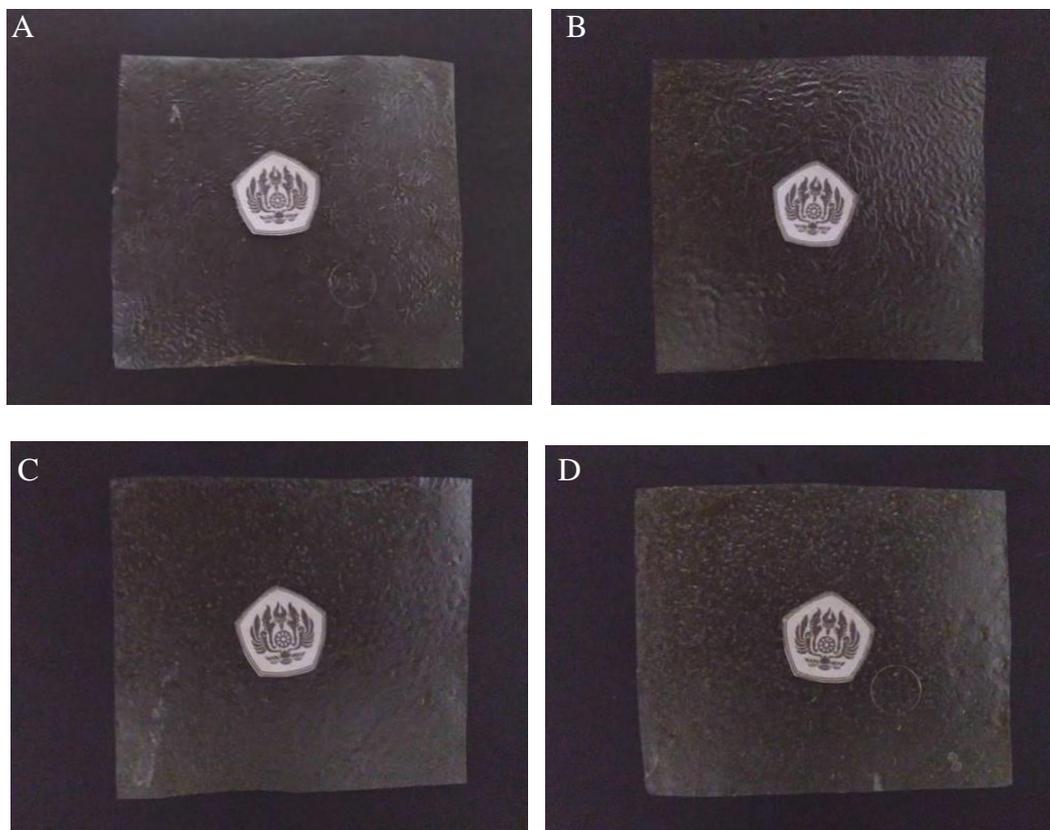


Figure 1. Bioplastics of Tapioca Modified with Addition of Chitosan and Gelatin to Each Treatment

Based on Figure 1, the transparency of bioplastics can be seen that treatment A (2% K, 5% G) had the clearest transparency appearance compared to other treatments. The relationship between the value of transparency and the degree of clarity showed that the high value of transparency will cause a low degree of clarity of bioplastics so that the bioplastic was more

turbid. Chitosan used in the coarse flaky form and white, which when dissolved will form a clear suspension. The gelatin used was yellowish, so that if the higher concentration of gelatin is added it will produce bioplastics that have a yellowish appearance (Figure 1). The higher concentration of chitosan and gelatin were added, the higher the value of transparency. The more gelatin added will produce a film with a higher yellow level and produce a less clear film. The results of the transparency test in this research are in accordance with [24], namely the increasing concentration of bioplastics (chitosan and gelatin) which was added, the transparency value tends to increase as well.

Bioplastics thickness also affects the value of transparency. The higher the thickness value, the higher transparency value. The use of glycerol can also affect transparency. The more glycerol used, the value of transparency will tend to decrease [31]. The amount of glycerol added to this research was equal for all treatments. However, even though the amount of glycerol added was equal, the concentration of the solution of bioplastics were increasing so that its use did not work optimally [27].

The transparency value based on the degree of clarity of the bioplastic packaging contained in this research has a value that is not much different from the results of the research [2], which ranges from 95,74 - 97,92%. The value of this research transparency is greater than the results [3], which has transparency values ranging from 0,86 - 2,12.

3. 3. Tensile Strength

The measurement of tensile strength is used to determine the magnitude of the force achieved to achieve the maximum pull in each unit area of the film to extend [1]. The results of testing the tensile strength of bioplastic packaging are presented in Table 4.

Table 4. Bioplastic Packaging Tensile Strength Test Results Based on Chitosan and Gelatin Concentrations

Treatment	Average (MPa)
A (2% K, 5% G)	19,05
B (3% K, 10% G)	16,26
C (4% K, 15% G)	11,91
D (5% K, 20% G)	7,625

Information: K = Chitosan, G = Gelatin.

Based on the results of the bioplastic tensile strength test on each treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (5% K 20% G has an average value of 19.05 MPa, 16.26 MPa, 11.91 MPa, and 7.625 MPa, respectively. The lowest value of tensile strength of bioplastics was found in treatment D (5% K, 20% G) and the highest tensile strength of bioplastic was found in treatment A (2% K, 5% G).

The more chitosan and gelatin added to bioplastics, the greater the value of tensile strength of bioplastics. This is different from Nurhayati et. Al. statement (2013) [15] namely,

the more chitosan added, the higher tensile strength produced. The decrease in tensile strength can be caused by too much chitosan being used so that the chitosan formed a very thick film and the added gelatin is also increasing. The increasing concentration of chitosan and gelatin causes the bioplastic formed to be more rigid and reduce the value of tensile strength. Another factor that decreases the value of tensile strength is the equality of glycerol for all treatments so that the use of glycerol did not work optimally [27]. Glycerol functions to reduce intermolecular forces in bioplastic polymer chains so as to produce flexibility [22]. A small amount of glycerol produces less elastic/cracking bioplastics [31].

The decrease in tensile strength can also be due to stirring and heating a less homogeneous bioplastic solution because the chitosan formed is very thick. The less homogeneous solution can cause a lot of space in bioplastic so that the bonds between bioplastic constituent molecules were not able to bind well [16]. The value of the attractiveness of bioplastics in this research ranges from 7,625-19.05 MPa. These results can be categorized as bioplastics because they met the JIS (Japanese Industrial Standard), which is a minimum of 3.92 MPa. The value of tensile strength in this research was not much different from Ratri research (2018) [20] which has tensile strength values ranging from 8,550 MPa-14,601 MPa, but lower with tensile strength results [27] which have values ranging from 0,99-38,79 MPa.

3. 4. Percent Lengthening

The measurement of tensile strength values is usually followed by a measurement of the percent elongation value. Percentage elongation illustrates the value of the plasticity of the film produced. The results of testing the elongation of bioplastics are presented in Table 5.

Table 5. Bioplastic Packaging Elongation Test Results Based on Chitosan and Gelatin Concentrations

Treatment	Average (%)
A (2% K, 5% G)	28,33
B (3% K, 10% G)	16,77
C (4% K, 15% G)	8,33
D (5% K, 20% G)	5

Information: K = Chitosan, G = Gelatin.

Based on the results of the percent elongation test for each treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (5% K, 20% G) has a consecutive value of 28.33%, 16.77%, 8.33% and 5%. The lowest percent value of elongation of bioplastic was found in treatment D (5% K, 20% G) and the highest percent elongation value was found in treatment A (2% K, 5% G). If it is associated with the tensile strength value in Table 4, treatment A (2% K, 5% G) also has the highest tensile strength and treatment D (5% K, 20% G) was the lowest tensile strength value. The percent elongation results in this research showed that the percent elongation value is directly proportional to the tensile strength value. This is different from

Rusli statement (2017) [22] that the percentage of elongation was inversely proportional to the value of tensile strength.

The addition of gelatin can provide a flexible nature of the film produced, but if the gelatin added is high, the resulting film will become stiff and the percent extension value is low [20]. This is in accordance with the bioplastics produced in this research, which is in line with the increasing concentration of gelatin added to bioplastics, the thicker the film and stiffness than the lower concentration gelatin. Bioplastics with the addition of low concentration gelatin are more elastic.

The percent value of elongation that meets the standards in JIS (*Japanesse Industrial Standard*) is in treatment A (2% K, 5% G) and B (3% K, 10% G), which is 28,33% and 16,77%. The standard percent value for elongation specified by JIS is 10-50%. With this standard, the percent length value for treatment C (4% K, 15% G) and D (5% K, 20% G) did not met the standard. The results of the elongation of this research are not very different from the results of [2], which range from 6,21% -22,02%.

3. 5. Biodegradability

Biodegradation is the process of organic matter broken down by microorganisms such as bacteria and fungi that are live in the soil [20]. Tests for degradation of bioplastic packaging were carried out using a soil burial test method. The higher concentration of chitosan, the longer bioplastic will be degraded bioplastics. The results of biodegradability testing observations are presented in Table 6.

Table 6. Observation Results of Bioplastic Biodegradation Tests Based on the Concentrations of Chitosan and Gelatin.

Treatment	Day Biodegradability Percentage	
	1	14
A (2% K, 5% G)	11,15%	99,84%
B (3% K, 10% G)	9,77%	99,21%
C (4% K, 15% G)	9,27%	98,87%
D (5% K, 20% G)	8,48%	98,61%

Information: K = Chitosan, G = Gelatin.

Based on the biodegradability test results after 1 day of bioplastic burial in each treatment A (2% K, 5% G), B (3% K, 10% G), C (4% K, 15% G), and D (5% K, 20% G) has a percentage value of 11,15%, 9,77%, 9,27%, and 8,48% and after 14 days of bioplastics burial which has a percentage value of 99,84%, 99,21%, 98,87%, and 98,61%. Based on visual observations, bioplastics are damaged in texture so that bioplastics became softer and then ripped.

The results of the biodegradability percentage of this research after 1 day burial ranged from 8.48-11.15%. These results are greater than the results of Ratri (2018) [20] which have a

percentage of biodegradability on the first day ranging from 3-4%. The results of the biodegradability percentage of this research after 14 days burial ranged from 98.61%-99.84%. Gelatin is hydrophilic, although chitosan is hydrophobic, it turns out that the greater concentration of gelatin causes bioplastics to degrade faster.

Hydrophilic properties make it easier for microorganisms to absorb nutrients from bioplastics so that bioplastics can be degraded. Overall, bioplastics in this research tend to be rapidly degraded by soil. The results of observing bioplastic packaging through visual physical observation are presented in Figure 2.

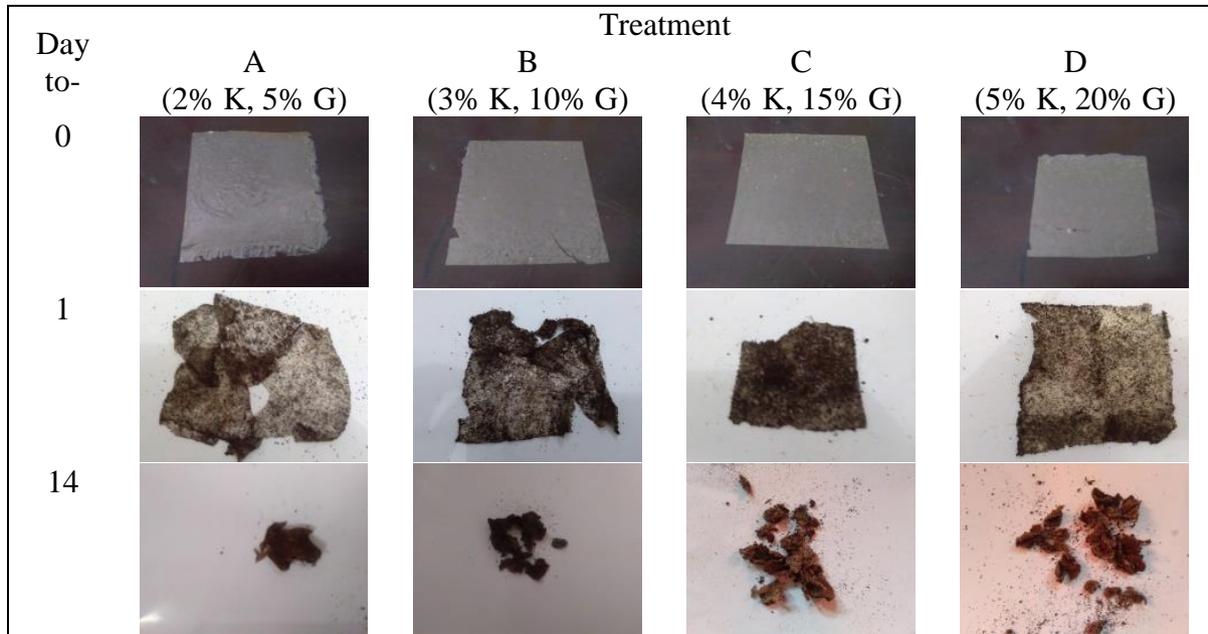


Figure 2. Physical Observation Result of Bioplastic

Based on Figure 2, the addition of chitosan and gelatin with lower concentrations decomposed faster than the addition of chitosan and gelatin with greater concentration. This was indicated by damage to the bioplastic texture in treatment A (2% K, 5% G) and B (3% K, 10% G) on the first-day observation after being buried and supported by the percentage of weight loss (Table 6). The value of biodegradability in this research tends to decrease, which means that the bioplastics are degraded longer. This is due to an increase in the amount of concentration of the film-forming materials (chitosan and gelatin). The more amount of chitosan added to the manufacture of bioplastics, the lower water content in bioplastics so that bioplastics will be degraded longer in the soil [17]. Longer time degradation in bioplastics with greater concentration is also due to microorganisms found in the soil requiring large energy to damage proteins in bioplastics.

After 14 days of burial, observations showed that bioplastics had decomposed in the soil even though there was still a little leftover. This can be due to decomposing microorganisms factors, soil moisture and soil water content [28]. The material for making bioplastics consists of natural ingredients so that it can be destroyed and decomposed naturally as well. The process of bioplastics biodegradation begins with a reduced amount of weight in bioplastics.

Furthermore, microorganisms contained in the soil, such as decomposers and fungi microbes can cause damage to the texture of bioplastics which have biodegradable base material. Examples of microorganisms found in the soil include *Aerobacter* and *Clostridium* microbes [30].

4. CONCLUSION

Based on the research results it can be concluded that the best characteristics of bioplastic packaging was in treatment A, namely by the addition of 2% chitosan and 5% gelatin. The addition of chitosan and gelatin concentrations in bioplastics increased the thickness and transparency value and decreased the tensile strength, elongation, and biodegradation. The results of the best treatment were obtained by referring to JIS. Treatment A has an average thickness value of 0.072 mm, a transparency value of 2.09, the tensile strength of 19.05 MPa, a percent elongation value of 28.333% and can be degraded by soil after 14 days about 99.84%.

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