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Physical and Mechanical Properties of Biodegradable Film from Taro Starch (Xanthosoma Sagittifolium) and Duck Bone Gelatin

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KEYWORDS

Biodegradable film;
Taro starch;
Duck bone gelatin;
Physical properties;
Mechanical properties.

ABSTRACT

This study describes the use of duck bone gelatin in a taro starch mixture to produce biodegradable films. Films were produced using duck bone gelatin in various percentages (0%, 5%, 15%, 25%, and 35% of the total solid weight) and added with glycerol as a plasticizer and the solution casting method. Parameters observed were tensile strength, percent elongation, thickness, moisture content, and surface morphology of the product was observed by scanning electron microscopy (SEM) analysis. Adding duck bone gelatin to biodegradable films based on taro starch had a significant effect ($p<0.05$) on tensile strength, percent elongation, and moisture content, but it did not affect ($p>0.05$) on thickness. The biodegradable film with a 5% duck bone gelatin concentration produced the highest tensile strength of 11.333 MPa and the highest percent elongation of 17.100%. Thickness values for all additions of duck bone gelatin concentration ranged from 0.191 to 0.194 mm, and the highest moisture content produced at 35% duck bone gelatin concentration was 4.079%. The surface morphology of the biodegradable film with a 5% duck bone gelatin concentration with the highest tensile strength value shows a flat, solid but slightly rough cross-section which may be caused by the flexibility of the gelatin film.

1. Introduction

Gelatin is an animal protein derivative product obtained by collagen hydrolysis [1]. It is mostly produced from pork skins (46%), cow skins (29.4%), beef, and pork bones (23.1%) [2]. This study used gelatin from duck bone waste, due to the increasing number of duck bone waste produced. Statistical data shows an increase in the duck population from 2019 to 2020. The duck population increased from 47,783 to 48,588 [3]. The increasing duck population is due to the increasing consumption of duck meat. Consumption of duck meat produces waste in the form of duck bones. The utilization of duck bone waste is still not optimal and most of it is thrown away in vain. Duck bone waste can be used as a gelatin product.

Duck bone gelatin is a product of the collagen hydrolysis from duck bones. Nowadays, gelatin is widely used in the food and non-food industries. The food industry uses gelatin as a foaming agent, stabilizer, gelling agent, binder, thickener, and viscosity enhancer [4]. The non-food industry uses gelatin as a mixture for making biodegradable films.

Biodegradable film is a material used to make environmentally friendly packaging because it can be decomposed naturally by microorganisms without any toxic substances that harm the environment [5]. Biodegradable films are made to reduce abundant non-biodegradable plastic waste that is difficult to decompose, and their raw materials are running low. Therefore, innovation is needed in making biodegradable films. The basic ingredients of biodegradable films are cellulose, chitosan, starch, gelatin, or a combination of components [6]. Among these natural polymers, starch is an ideal polysaccharide material that is widely used to make biodegradable films. It is a type of carbohydrate found in nature and can be obtained from plant body parts such as seeds, stems, and tubers [7]. Starch can form a

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colorless and transparent polymer matrix, so it is widely used in food packaging. It is a heterogeneous material containing amylose and amylopectin [8]. Amylose is a starch component with straight chains, is soluble in water, and is hard, while amylopectin is a starch component with branched chains, is sticky, and does not dissolve in cold water [9]. In this study, taro starch with around 80% starch content was used. It is used as a material for making biodegradable films because the taro plant is abundantly available. This natural material is inexpensive and renewable. The productivity of taro tubers reaches 30 tons/hectare [10]. The starch content in talas is greater than garut starch and cassava starch [11]. Similarly, the amylopectin content of 74.45% and amylose of 25.55% so it can affect the scientific and mechanical properties of the film [12]. The starch content in taro tubers can affect the strength of biodegradable films to become stiffer, however brittle [13]. Therefore, another additive, i.e., duck bone gelatin with different concentrations of total solids, is needed to improve the mechanical properties and quality of biodegradable films. Adding gelatin can make biodegradable films more flexible or easier to shape, bend [14], and decompose. This is because protein has a unique structure, namely, there are 20 kinds of amino acids that can provide various types of intermolecular bonds so that it expands the functional properties of the plastic formed [15]. In this study, duck bone gelatin was added to the biodegradable taro starch film to increase its tensile strength and flexibility. Several studies generally use commercial gelatin which has a disadvantage, namely it is expensive, so further research needs to be done to find alternative sources of other gelatin. One of them is by utilizing gelatin from duck bone waste in a biodegradable film based on taro starch.

2. Materials and Methods

Taro tubers (Rogojampi traditional market, Banyuwangi, Indonesia), duck bones (duck bone waste obtained from food scraps), sodium hydroxide (NaOH, analytical grade, Merck KGaA, Darmstadt, Germany), Hydrochloric Acid (HCl, analytical grade, PT. Smart Lab Indonesia), aquadest (CV. Makmur Sejati, Malang, Indonesia), glycerol (CV. Sahabat Lab, Indonesia), petri dish, oven, Universal Testing Machine (UTM) Tensilon RTF-1310, micrometer screw refers to the standard JIS B7502, Scanning Electron Microscope (SEM) are the materials and apparatus used in this study. The method used in making biodegradable films was solution casting. Five different loading of duck bone gelatin of 0%, 5%, 15%, 25%, and 35% to the total weight of solids (taro starch and duck bone gelatin). Taro starch and duck bone gelatin were weighed according to the treatment. The taro starch was baked in an oven before making the biodegradable film solution. The 5 grams of starch was then dissolved in 10 ml of aquadest using a hot plate and a magnetic stirrer at 70°C at a speed of 375 rpm, and the gelatin was also dissolved in 10 ml of aquadest. After that, a solution of taro starch and duck bone gelatin was mixed while heated and stirred; then, 0.5 ml of glycerol and 5 ml of aquadest were added. The solution was heated, stirred, and kept at 70°C. Then, it was left to gelatinize for 40 minutes. The biodegradable film solution was left for 3 minutes. After that, it was poured into a glass petri dish mold with a diameter of 9.8 cm and baked in an oven for 24 hours at 60°C. The sample was left for some time at room temperature before it was peeled off. The finished biodegradable films were ready for tensile strength, percent elongation (elongation), thickness, moisture content, and structure morphology (SEM).

Tensile strength was measured using a Universal Testing Machine (UTM) at a crosshead speed of 50 mm/min maintaining a gauge length of 50 mm. In each case, three specimens were tested and the average values are reported. Tensile tests were performed as per the ASTM D882 standard. The thickness of the 2.5 x 5 cm biodegradable film was measured using a micrometer; then, both ends of the biodegradable film were clamped between the grips. The initial length before tension or force was recorded, and the recording ink tip was placed at a position of 0 of the graphs. The start button on the application was pressed and the tool pulled the biodegradable film until it broke. The force that could be received by the biodegradable film was then recorded. The tensile strength was calculated by the maximum force applied to the film until it tore (N) divided by the unit area of the film (mm2) [12].

$$\sigma = F/N \quad (1)$$

where σ = tensile strength (MPa), F = maximum stress (N), and N = cross-sectional area (mm^2).

2.1. Elongation

The percent elongation test results were measured using a Universal Testing Machine (UTM). The working principle of the percent elongation test on biodegradable films is almost the same as the tensile strength test. The biodegradable film was prepared with a size of 2.5 x 5 cm. The percent elongation was calculated when the film broke or tore [16].

Before the film was pulled, its length was measured up to the grip limit (1_0) and tested.

The length after being pulled or broke (1_1) was calculated with the formula:

$$\varepsilon = (l_1 - l_0)/l_0 \times 100\% \quad (2)$$

where ε = percent elongation or elongation (%), l_1 = length after breaking (mm), l_0 = initial length (mm).

2.2. Thickness

The thickness of the biodegradable film product was measured using a digital micrometer with an accuracy of ± 0.001 mm (Mitutoyo, Japan). The thickness of each film was measured at five different positions, and then the average thickness was calculated to obtain the final thickness data [17].

$$Thickness = (point1+point2+point3+point4+point5)/5 \quad (3)$$

Each point was taken randomly, namely on each side of the biodegradable film.

2.3. Moisture Content

The cup is cleaned to be sterile by the way it was washed and dried in an oven for 30 minutes from 105°C to 110°C. It was cooled in a desiccator for 30 minutes and weighed to determine its initial weight (A). A sample of 2 grams was then put in a cup and weighed (B). Then, it was dried in an oven from 105°C to 110°C for 3 to 4 hours until a constant weight was reached. The cup was then cooled in a desiccator for about 30 minutes and weighed (C) [18]. Moisture content was calculated using the formula:

$$Moisture\ Content = (B-C)/(B-A) \times 100\% \quad (4)$$

where A = Weight of the empty cup (grams), B = Weight of the cup + sample before drying (grams), C = Weight of the cup + sample after drying (grams).

2.4. Morphological Structure Analysis (SEM)

Scanning Electron Microscope (SEM) brand FEI INSPECT S50 with 20 kV voltage and magnification of 2,000 times. SEM was used to observe the surface structure of biodegradable films, taro starch, and duck bone gelatin. All samples were prepared by placing them in a desiccator with 54% humidity at room temperature. Observations were made according to standard procedures.

2.5. Statistical Analysis

Sample analysis was carried out 3 times test. Furthermore, the data obtained is analyzed statistically by ANOVA using the IBM SPSS Statistics 24 program.

3. Results and discussion

3.1. Tensile Strength

Figure 1 shows that adding duck bone gelatin with different concentrations to the biodegradable film based on taro starch had a significant effect ($p<0.05$) on the tensile strength of the biodegradable film. Different notations on the graph showed a significant effect ($p<0.05$), while the same notation on the graph showed no significantly different effects ($p>0.05$). The tensile strength increased at a duck bone gelatin concentration of 5%. The highest tensile strength value at 5% duck bone gelatin concentration was 11.333 MPa due to the hydroxyl group (OH) of starch with the amine group (NH) of gelatin that can bind well so that the film structure became compact and mechanical resistance increased [19].

The tensile strength value in this study was included in the moderate properties group. Reference [20] stated that the biodegradable film with a tensile strength of 10-100 MPa is included in the moderate properties group. Moderate properties are a standard used for biodegradable plastics [21].

The more addition of duck bone gelatin resulted in a decrease in tensile strength from concentrations of 15%, 25%, and 35%. The highest tensile strength value was produced by addition of 15% gelatin is 11,333 MPa, 25% gelatin is 8.133 MPa, and 35% gelatin is 3.767 MPa. The lowest tensile strength value was produced by P4 of 3.267 MPa. The decrease in tensile strength is due to the formation of intramolecular hydrogen bonds rather than intermolecular hydrogen bonds [22].

Intramolecular hydrogen bonds are hydrogen bonds between molecules of duck bone gelatin. Intermolecular hydrogen bonds are hydrogen bonds between taro starch molecules and duck bone gelatin molecules. In addition, it is due to that the number of hydrogen bonds between duck bone gelatin is more than the hydrogen bonds of other molecules.

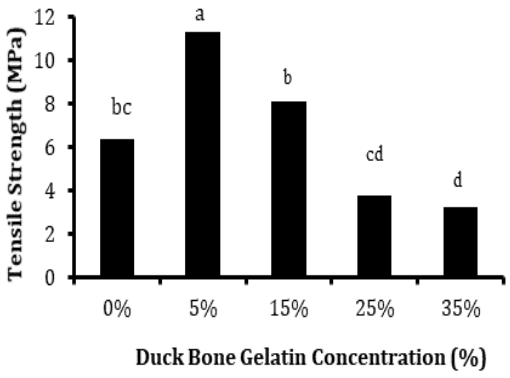


Fig. 1. Tensile strength of the biodegradable film

Reference [23] showed that gelatin can weaken the forces between adjacent molecules along the polymer chain, resulting in decreased tensile strength. The increase in gelatin will reduce the cohesive bonds between the polymer that forms the film. This is because the OH group in gelatin will form intermolecular bonds with reduced polymer chains. Reduced hydrogen bonds internal will weaken the intermolecular attractions of adjacent polymer chains thereby reducing the tensile strength of the film. In addition, it was also caused by the number of hydrogen bonds in gelatin more than the hydrogen bonds of other molecules. Gelatin is a polypeptide consisting of covalent bonds and bonds peptides between the amino acids that make them up. This polypeptide has two terminal atoms, the left end contains an amino group and the right end contains a carboxyl group. The two ends allow the gelatin to form a bond hydrogen with other gelatin molecules, or with water molecules. Amino acid 4-hydroxyproline has two functional groups that allow it to form hydrogen bonds, namely the H atom of the OH functional group, the H atom, and the O atom of the carboxy group. So it is possible that there are more hydrogen bonds in gelatin than in other molecules. Another factor was that the chemical quality of the duck bone gelatin used in this study was thought to be poor. Each gelatin had a different chemical quality depending on the type of material and the extraction process. The quality of gelatin was not good because it had not been purified. The gelatin extraction was not precise so the moisture content was more than the protein. In this study, duck bone gelatin was not purified, so it was suspected that the quality of the gelatin protein was low because there were still many impurities. In duck bone gelatin, the fat content is still above 5% and the ash content is above 3%. It is an impurity in the manufacture of gelatin. Gelatin which is high in ash content will affect its gelation properties.

Gelatin protein is a protein that forms a triple helix woven to increase tensile strength [24]. Reference [25] showed that excessive protein can reduce the tensile strength due to interactions between the hydroxyl groups of starch and gelatin that reduce interactions between starch molecules.

This study is different from the study of reference [26] stating that more gelatin increases tensile strength. The increase occurred because the two polymers could form a structural network through the interaction between the anionic groups of polysaccharides and cationic groups of gelatins that strengthen the film structure. Tensile strength and percent elongation are related to each other because higher tensile strength and elongation will result in a better quality of the biodegradable film.

3.2. Elongation

Figure 2 shows that adding duck bone gelatin with different concentrations to biodegradable films based on taro starch had a significant effect ($p<0.05$) on the percent elongation. The percent elongation value was produced by addition of 0% gelatin is 12.18%, 5% gelatin is 17.10%, 15% gelatin is 14.39%, 25% gelatin is 11.38%, and 35% gelatin is 8.77%. The highest percent elongation was produced when 17.100% of 5% gelatin concentration was added. The percent elongation from the duck bone gelatin concentration of 0% to 5% increased because the long-chain polysaccharide macromolecules could cross-link with gelatin, allowing macromolecular relaxation to occur [27].

The increase in the gelatin concentration means a decreased percent elongation. This occurred in the addition of 5% to 35% gelatin concentration which had decreased. The decrease in the percent elongation depends on the intermolecular structure of the matrix. When there is an excess of gelatin concentration, the polymerization between collagen and other components is not optimal [28]. There is an optimum level for interaction between polysaccharides and gelatin, where gelatin is the main and dominant phase in the film system used [27]. The percent elongation value in this study was in the moderate properties group. Reference [20] showed that the mechanical properties of biodegradable films with a percent elongation value of 10-20% are classified as moderate properties. The higher the percentage of elongation, the better the quality of the biodegradable film because it has more elastic and flexible properties [29].

The study of reference [30] reported that the more gelatin is added, the more the tensile strength and percent elongation are obtained. This shows that previous research is not in line with this study. The increase or decrease in mechanical properties is influenced by the amount and type of material used in making biodegradable films and their thickness

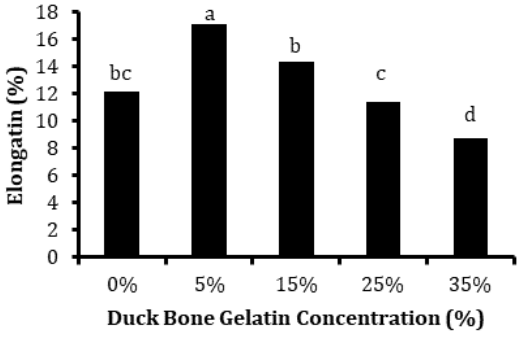


Fig. 2. Elongation of biodegradable film

From these results, it is assumed that the duck bone gelatin used is not pure yet and there are still impurities so that by adding more duck bone gelatin, its physical properties will decrease.

3.3. Thickness

Figure 3 shows that adding duck bone gelatin with different concentrations to the biodegradable film based on taro starch had no effect ($p>0.05$) on the thickness. The thickness value was produced by addition of 0% gelatin is 0.191 mm, 5% gelatin is 0.192 mm, 15% gelatin is 0.192 mm, 25% gelatin is 0.194 mm and 35% gelatin is 0.194 mm. The average thickness of the biodegradable film in this study ranged from 0.191 to 0.194 mm. This thickness value is close to the Japanese Industrial Standard (JIS) [31], in which the maximum film thickness is 0.25 mm. The insignificant increase in thickness was due to the total amount of solids in making the same biodegradable film, namely 5 grams, and the same type and size of the mold. The increase in thickness was also due to the increase in the amount of gelatin concentration in each treatment. The thickness of the biodegradable film is influenced by the type of raw material, the concentration of the material in the biodegradable film solution, the volume of the solution poured [24], and the presence of air bubbles in the biodegradable film. The study of reference [32] reveals that making biodegradable films using 5 grams of solids with different raw materials and concentrations, namely cassava starch and gelatin, produces a thickness of about 0.12-0.16 mm. Biodegradable films from cassava starch and gelatin with a total solid of 5 grams produce a thickness of about 0.043–0.075 mm [30]. The study of reference [33] found the thickness of potato starch and gelatin with a total solid of 5 grams was 0.073-0.096 mm. The different thickness value of the biodegradable film in this study was caused by several factors; they are the type of material mixture, the volume of the solution poured, and the different sizes of the molds. Thickness value affects the moisture content of a film. It is thought that the moisture content of the duck bone gelatin used is still high, which causes the film to become heavy and increases its thickness.

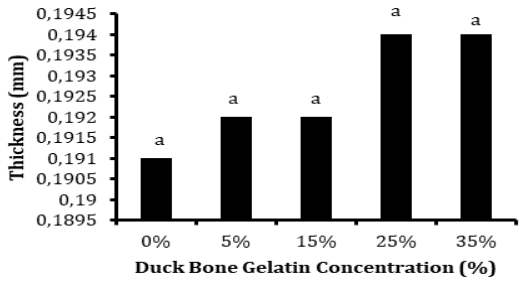


Fig. 3. The thickness of the biodegradable film

3.4. Moisture Content

Figure 4 shows that adding duck bone gelatin with different concentrations to biodegradable films based on taro starch had a significant effect ($p<0.05$) on the moisture content. The moisture content value was produced by addition of 0% gelatin is 9.764%, 5% gelatin is 9.820%, 15% gelatin is 11.143%, 25% gelatin is 12.082% and 35% gelatin is 14.079%. The increase in moisture content occurred from the addition of 0% to 35% duck bone gelatin concentration.

The highest moisture content was produced at a 35% duck bone gelatin concentration with a value of 14.079%. The increase in moisture content was caused by the addition of hydrophilic gelatin concentration [17].

Hydrophilic properties are the properties of a material that can absorb or bind water well so that the moisture content in the biodegradable film increases. This causes the moisture content to increase. The increase in moisture content was also caused by differences in the molecular weight of the materials used, namely starch and gelatin. Reference [34] reported that the molecular weight of gelatin ranged from 15,000 to 400,000 g/mol. The molecular weight of amylose is about 10,000–60,000 g/mol and amylopectin 60,000–100,000 g/mol [9]. Reference [35] showed that the greater the molecular weight in a material is, the greater the gap or space between the molecules will be. Therefore, water can enter between the gaps which increases the amount of moisture content.

The study of reference [19] states that the moisture content decreases with increasing gelatin concentration due to the formation of intermolecular hydrogen bonds, namely hydrogen from the hydroxyl group of starch and hydrogen from the amine group of gelatin, hiding the resulting hydrophilic film. The increased moisture content causes the water content in the biodegradable film to increase, thereby accelerating or facilitating the decomposition process in the soil [6].

3.5. Morphological Structure Analysis (SEM)

The micrograph of taro starch looked like granules resembling spherical shapes with varying granule sizes and smooth surfaces [36].

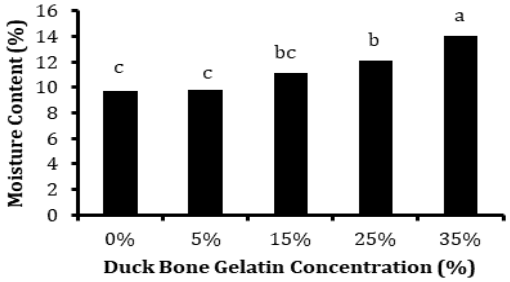
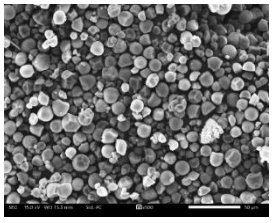
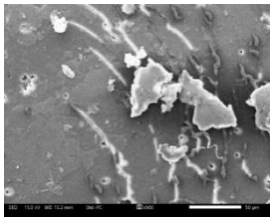


Fig. 4. Moisture Content of biodegradable film

Meanwhile, the appearance of duck bone gelatin looked irregular in shape and the surface was rougher with pores. In the micrograph of the biodegradable film product, granules were no longer found, but the surface was rather rough, tight, and solid because when the duck bone gelatin was mixed, the taro starch was fully gelatinized, causing the granule structure of the taro starch to be damaged and the duck bone gelatin to react on the taro starch backbone and produce a solid biodegradable film product. The SEM image of the taro starch biodegradable film with a 0% duck bone gelatin concentration (Figure 5c) showed a relatively smooth and non-porous film surface with no cracks, which means the surface structure of the biodegradable film is solid and homogeneous. Micrographs of the biodegradable film with a 5% duck bone gelatin concentration showed an even but slightly rough cross-section (Figure 5d), which might be due to the flexibility of the gelatin film. Duck bone gelatin was added to the biodegradable film based on taro starch to improve the internal structure of the biodegradable film. The biodegradable film of a mixture of taro starch and 35% duck bone gelatin showed more roughness and was also uneven compared to the 5% duck bone gelatin biodegradable film (Figure 5e). On the micrograph of the biodegradable film with the addition of 35% duck bone gelatin, there was still gelatin that was physically dispersed on the surface of the biodegradable film. This indicated that the duck bone gelatin had agglomerated so that its distribution in the film layer was not even. This might be due to the presence of residual gelatin that was not bound to taro starch and the less homogeneous mixing process between duck bone gelatin and taro starch, causing uneven distribution of duck bone gelatin. If the stirring technique is long enough and homogeneous during the mixing process at the gelatinization temperature, the taro starch will easily incorporate the duck bone gelatin particles so that the duck bone gelatin can be evenly distributed in the biodegradable film. SEM results showed that the surface of the biodegradable film does not appear to have any phase separation in the film mixture, indicating very good compatibility between taro starch and duck bone gelatin which forms a biodegradable film matrix [17].



(a)



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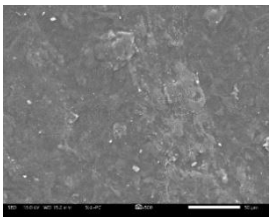

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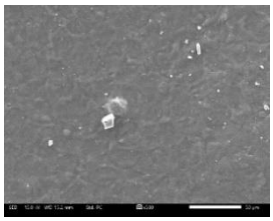

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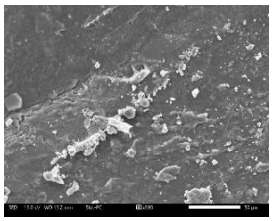

(e)

Fig. 5. Micrograph of (a) Taro starch, (b) Duck bone gelatin, (c) Biodegradable film 0%, (d) 5%, (e) 35% Duck bone gelatin

4. Conclusions

Based on the findings of the study, it can be concluded that making biodegradable films with the addition of duck bone gelatin concentration on taro starch-based biodegradable films (Xanthosoma sagittifolium) had a significant effect ($p<0.05$) on tensile strength, percent elongation, and moisture content, but there was no significant effect ($p>0.05$) on thickness. The highest value of tensile strength and percent elongation was obtained with the addition of a 5% gelatin concentration. The thickness value for all treatments is almost the same, namely 0.19 mm, and still meets JIS (Japanese Industrial Standard). The highest moisture content value was produced by the addition of a 35% gelatin concentration. The higher value of tensile strength and percent elongation will result in a better biodegradable film for packaging food products. The higher value of moisture content will result in the faster decomposition of biodegradable films. The results of SEM micrographs of the biodegradable film with a 5% duck bone gelatin concentration with the highest tensile strength value show a flat, solid but slightly rough cross-section which may be caused by the flexibility of the gelatin film.

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