Biodegradable film based on taro starch with variations of duck bone gelatin concentration: physical and barrier properties

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Abstract

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Introduction. This study aims to improve the physical properties of biodegradable films based on taro starch added with duck bone gelatin and glycerol as a plasticizer by using the solution casting method.

Materials and methods. A non-factorial completely randomized design (CRD) with ANOVA was used, followed by the DUNCAN test to determine the effect of different treatments. Various concentrations of duck bone gelatin were used, including 0, 5, 15, 25 and 35% of the total weight of solids (taro starch and duck bone gelatin). The functional groups of biodegradable films were analyzed using Fourier Transport Infrared (FTIR). The morphological structure of the product was studied using Scanning Electron Microscope (SEM).

Results and discussion. The parameters observed included the water vapor transmission rate test, which resulted in a value of 0.465 – 0.529 g/m²·hour. The highest water vapor transmission rate was at a concentration of 35% duck bone gelatin of 0.529 g/m²·hour. The factor that affects the value of the water vapor transmission rate is the chemical nature of the material polymer. Water absorption was 85.17–40.47%, and the highest water absorption was at a concentration of 0% duck bone gelatin. The percentage of water absorption is used to assess the value of the water resistance of the resulting film. Water solubility was 31.08–64.60%. The highest water solubility value was at a concentration of 35% duck bone gelatin of 64.60% because the addition of duck bone gelatin can reduce the intermolecular forces of the film and increase water activity so that free water molecules can occupy the matrix in the film.

The FTIR spectrum for biodegradable film products showed a peak wavenumber at 3600–3000 cm⁻¹, indicating the presence of N-H and O-H groups. The presence of N-H and O-H groups in biodegradable films can help the degradation process because they have hydrophilic properties that enable water molecules to access and enter the film matrix.

The results of SEM analysis showed that the taro starch film with the addition of duck bone gelatin produced a homogeneous and flexible biodegradable film with compact structure.

Conclusions. The addition of duck bone gelatin had an insignificant effect on the water vapor transmission rate but a very significant effect on water solubility and a significant effect on water absorption.

Introduction

Plastic is a common packaging material but is not decomposable (Muin et al., 2017). It has advantages, including practical, transparent, flexible, economical, strong, and multifunctional. This is what causes plastic packaging to be widely used by the community, resulting in increasing waste. This can cause environmental damage. Therefore, an alternative for making biodegradable plastics, called biodegradable films, is needed. Biodegradable film is a biodegradable and environmentally friendly plastic (Aripin et al., 2017). The main ingredients for making biodegradable films come from cellulose, chitosan, starch, protein, or a combination of components (Asngad et al., 2018). Among these natural polymers, starch is a polysaccharide that is widely used as a material for making biodegradable films. It is one type of carbohydrate that is found in nature, one of which is from plant body parts such as seeds, roots, stems, and cereals (Aryanti et al., 2017). Starch contains compact-structure amylose and amylopectin (Udjiana et al., 2019). Different levels of amylose and amylopectin can affect film formation (Rosseto et al., 2019). Starch from taro tubers was used because it has a starch content of 80%, which is greater than corn starch content of 71.3% and cassava starch content of 72.17% (Rahmawati et al., 2012); besides, taro starch is a natural renewable material. The use of starch in biodegradable films has the advantages of being easy to decompose (biodegradation), easy to form films (plastic), easy to obtain (Winarti et al., 2012), and not easily penetrated by water vapor (Aripin et al., 2017). However, starch also has drawbacks, including low mechanical properties and being easy to dissolve in water (Bani, 2019). Other additives are needed to improve these, one of which is duck bone gelatin to reduce the stiffness of the polymer and its hydrophilic properties to water (Resalina et al., 2013). Taro starch and duck bone gelatin is renewable in making biodegradable films. Gelatin added to the biodegradable film will provide flexural and elastic properties (Illing et al., 2017). Gelatin can also increase its solubility in water to facilitate the degradation process (Kumar et al., 2019).

Biodegradable film based on gelatin is usually very good as a barrier to oxygen, carbon dioxide, and some aromatic compounds, and has excellent mechanical properties (mechanical strength of the film) and barrier properties (the ability to inhibit the entry and exit of certain substances through the film) (Jagadeesh et al., 2016). In addition, gelatin also basically has good barrier properties against UV rays due to the presence of aromatic amino acids (the structure resembles a ring) which is able to absorb UV light, so that it can prevent oxidation in food products wrapped in plastic from the protein. Mixing two different polymers, such as gelatin and polysaccharides, proteins and lipids, polysaccharides and lipids or synthetic polymers and natural polymers aims to improve the biodegradable film properties.

Gelatin is an animal protein product from the hydrolysis of collagen (Chaves da Silva et al., 2018). It is widely used by the food and non-food industries. The food industry uses gelatin as an emulsifier, binder, stabilizer, and thickener (Miskah et al., 2010). The non-food industry uses gelatin as an additive for making films or plastics (Puspawati et al., 2017). The raw materials of gelatin come from cow bones, cow skins, pig skins (Santosa et al., 2018), and poultry including ducks, chickens, and birds (Puspawati et al., 2017). Duck bone waste was used as a source for making gelatin. Generally, duck bone waste has not been utilized optimally. Therefore, it is necessary to conduct research on making biodegradable films based on taro starch and duck bone gelatin to produce better physical properties, namely water vapor transmission rate, water solubility, and water absorption.

The aim of research is to improve the physical and barrier properties of biodegradable films based on taro starch added with duck bone gelatin.

Materials and methods

Materials

The main ingredients used were taro tubers and duck bone gelatin obtained from markets and restaurants in Banyuwangi Regency, Indonesia. 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). Thermometer, hot plate, magnetic stirrer, food dehydrator, dry box, beaker glass, spatula, desiccator, petri dish, measuring cup, dropper, pot, stove, knife, oven, scale analytics, Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscope (SEM) were used in research.

Synthesis of Biodegradable Film

In this study, the biodegradable films of taro starch and duck bone gelatin were made using the solution casting method. Five treatments were used with different concentrations of duck bone gelatin, namely 0, 5, 15, 25 and 35% of the total solids weight (taro starch and duck bone gelatin). Taro starch and duck bone gelatin were weighed according to the treatment. Starch was dissolved in 10 ml of distilled water using a hot plate and stirred with a magnetic stirrer at 70 °C with a speed of 375 rpm. Gelatin was also dissolved in the same way as taro starch. The solutions of taro starch and duck bone gelatin were then mixed, heated, stirred, and added with 0.5 ml of glycerol and 5 ml of distilled water. The solution was heated, stirred, and kept a constant temperature of 70 °C. Then, the gelatinization occurred for 40 minutes. The biodegradable film solution was left for 3 minutes. It was poured into a petri dish mold and put in the oven for 24 hours at 60 °C. The sample was left at room temperature after being removed from the mold. The biodegradable film was stored in a zip bag log and given silica gel so that it was not easily damaged and was ready for Fourier Transform Infrared (FTIR), SEM, water vapor transmission rate, water solubility, and water absorption tests.

Fourier Transform Infrared (FTIR)

Infrared spectra of biodegradable film samples were measured using KBr pellets with the FTIR Prestige-21 Shimadzu spectrophotometer at wavenumbers between 4000 cm^{-1} to 400 cm^{-1} . The sample was placed into the set holder and searched with the appropriate spectrum. The result was obtained in the form of spectra of the relationship between wavenumber and intensity. The FTIR spectrum was recorded using a spectrometer at room temperature (Darni et al., 2010).

Water Vapor Transmission Rate Test

Measurement of the water vapor transmission rate was carried out using a porcelain cup and a glass jar. The porcelain cup was filled with 15 ml of water and covered with a biodegradable film. Then, the porcelain cup was placed in a glass jar containing 30 g of silica gel with known initial weight and left in a closed glass jar for hours (Sunardi et al., 2019). The water vapor transmission rate test was calculated using the following formula:

$$WVTR = \frac{\Delta W}{A \times T}$$

Remark:

WVTR – water vapor transmission rate; ΔW – change in weight of silica gel (gram);
A – film area (m²); T – storage time interval (hours).

Water Solubility Test

The cup was cleaned and dried at 100 °C for 30 minutes. A sample of 1 gram was poured in a cup and placed in an oven at 100 °C for 30 minutes. The dried sample was weighed as the initial dry weight (W_0) before being immersed for 24 hours. The sample that was not dissolved in water was removed and dried in an oven for 2 hours at 100 °C; then, it was stored in a desiccator for 20 minutes. The dried sample was weighed as the sample weight after immersion (W_1) (Gontard et al., 1993). The percentage of water solubility of the sample was calculated by the following formula:

$$S = W_0/W_1 \times 100\%$$

Remark:

S – Water solubility (%); W_0 – Initial dry weight (gram); W_1 – Weight after immersion (gram).

Water Absorbition Test (Swelling)

The biodegradable film was cut and weighed the initial weight (W_0), then put into a glass beaker containing 15 ml of distilled water for 2 minutes. The sample was removed and there was water on the surface of the plastic then drained using a tissue for 5 minutes, after which it was weighed. Weighing and immersion of the biodegradable film was repeated until the sample had a constant final weight. The water absorption test (swelling) can be calculated using the following formula:

$$Swelling = \Delta W/W_0 \times 100\%$$

Remark:

ΔW – Weight change of biodegradable film (grams);
 W_0 – Initial weight of biodegradable film (grams).

Morphological Structure Analysis (SEM)

Observation of the cross sectional morphological structure of biodegradable film products was carried out using Scanning Electron Microscope (SEM) analysis (ZEOL, Germany). Before testing, the samples were placed in a desiccator with 54% humidity at room temperature. The biodegradable film was prepared by breaking it down in liquid nitrogen and then observing it according to standard procedures.

Results and discussion

Fourier Transform Infrared (FTIR) Analysis

Figure 1 shows the FTIR spectrum of taro starch, duck bone gelatin, glycerol, and biodegradable film.

Figure 1. Spectrum FTIR of taro starch, glycerol, duck bone gelatin, and biodegradable film

The biodegradable film was made of three ingredients, namely taro starch, duck bone gelatin, and glycerol; therefore, the functional groups contained in these three ingredients must be present in the biodegradable film. The FTIR spectrum for biodegradable film products showed the peak wavenumber at 3600-3000 cm^{-1} , indicating the presence of N-H and O-H groups. This causes water to be easily absorbed due to the presence of inter- and intra-molecular hydrogen bonds. Therefore, there is an interaction and molecular solubility between taro starch, duck bone gelatin, and glycerol in biodegradable films (Tongdeesoontorn et al., 2012). The peak wavenumber at 1,589 cm^{-1} indicated the C=C group contained in gelatin (Radiyahatullah et al., 2015). The peak wavenumber at 1,468 cm^{-1} represented the C-H group found in starch and glycerol (Iling et al., 2017). The peak wavenumber at 1,350 cm^{-1} indicated the C-N group in gelatin (Kong et al., 2007). The mixture of materials in the biodegradable film showed the functional groups contained in the three constituent materials and can be found in the biodegradable film product. This shows the presence of hydrogen bonds between polymers so that the biodegradable film is still hydrophilic (Aritonang et al., 2020). This FTIR analysis is in line with research (Iling et al., 2017) using sago waste and gelatin materials, showing the presence of functional groups in the form of C-H, O-H, C-O, C=O, and N-H. Tongdeesoontorn et al. (2012) stated that the functional groups contained in cassava starch and gelatin in the film had such functional groups as O-H, C-H, C=O, C-N, and N-H. The presence of N-H and O-H groups in biodegradable films can help the degradation process because they have hydrophilic

properties that enable water molecules to access and enter the film matrix (Aritonang et al., 2020).

Biodegradable Film Water Vapor Transmission Rate Test

Figure 2 shows that the different concentration of duck bone gelatin has no significant effect ($P > 0.05$) on the water transmission rate of the biodegradable film.

Figure 2. Water vapor transmission rate of biodegradable film

Higher concentration of duck bone gelatin increases the value of the water vapor transmission rate. The highest water vapor transmission rate was 0.529 g/m²hour at a concentration of 35% duck bone gelatin. The factor that affects the value of the water vapor transmission rate is the chemical nature of the material polymer (Budianto et al., 2019). The main ingredients for making biodegradable films in the form of polysaccharides (taro starch) and protein (duck bone gelatin) are polar polymer components that are hydrophilic because they have long hydrogen bonds and can increase the matrix or empty space, thereby accelerating the water vapor transmission rate (Deden et al., 2020). Thicker films will result in less water evaporation (Wijayani et al., 2021). The film structure with many air bubbles can cause water molecules to easily diffuse through the film surface (Widyaningsih et al., 2012). The value of the water vapor transmission rate was low from 0.465 g/m²hour to 0.5296 g/m²hour and did not exceed the steam transmission rate standard set by the Japanese Industrial Standard (JIS) (1975) for biodegradable film of 7 g/m²hour. The result of this study is lower than that of the previous research (Santoso et al., 2020) with low water vapor transmission values ranging from 1.07 g/m²hour to 1.60 g/m²hour with catfish bone gelatin and breadfruit starch as the main ingredients. The low value of water evaporation makes it difficult for water to penetrate the film so that the product is safer and not easy to damage (Salimah et al., 2016). The higher value of the water vapor transmission rate influenced by duck bone gelatin will result the faster film decomposition because gelatin can be hydrolyzed by adsorption of water in the soil and the polymer chains of the film will be broken and easily decomposed (Silviana et al., 2018).

Biodegradable Film Solubility Test

Figure 3 shows that the different concentration of duck bone gelatin has a very significant effect ($P > 0.01$) on the water solubility of the biodegradable film.

Figure 3. Solubility of biodegradable film

The more concentration of duck bone gelatin increases the value of water solubility. The highest water solubility value was at a concentration of 35% duck bone gelatin of 64.60% because the addition of duck bone gelatin can reduce the intermolecular forces of the film and increase water activity so that free water molecules can occupy the matrix in the film (Sutra et al., 2020). Protein-based films (gelatin) have hydrophilic properties due to the presence of polar groups such as hydroxyl, carbonyl, and amino acids that can form hydrogen bonds that can bind to water molecules (Putri, 2018). In addition, gelatin has almost all amino acids except tryptophan (Otoni et al., 2017). The large number of hydrophilic amino acids can increase the affinity of gelatin to water, causing the film to easily bind water molecules (Dhirisma et al., 2014). Therefore, the amount of water that diffuses into the film can cause the breakdown of the film cavity, increasing its water solubility (Wang et al., 2017). This is following Darni et al. (2008) stating that the biodegradable film structure using gelatin has many cavities so that the ability to absorb water is higher.

The solubility value in this study is quite high, from 31.08% to 64.60%. The solubility value is in line with the finding of Kumar et al. (2020) using corn starch and gelatin, resulting in a solubility value ranging from 20% to 60% with increasing concentration of added gelatin. Zulferiyani (2014) reported that the high solubility value could facilitate the biodegradation of the film due to the hydrophilic component because it can absorb water in the soil so that the film is easily broken into polymer units.

Biodegradable Film Water Absorption Test (Swelling)

Figure 4 shows that the different concentration of duck bone gelatin has a very significant effect ($P > 0.05$) on the water absorption of the biodegradable film.

Figure 4. Water absorption (swelling) of biodegradable film

The higher the concentration of duck bone gelatin is, the lower the value of water absorption (swelling) will be. This is because proteins in the form of gelatin have hydrophilic properties due to the presence of polar groups in the form of hydroxyl, carbonyl, and amino acids that can form hydrogen bonds that can bind to water molecules (Putri, 2018). Hydrophilic starch and gelatin can bind to water molecules more quickly so that water easily diffuses, and the film is easy to swell (Kumar et al., 2020). The percentage of water absorption is used to assess the value of the water resistance of the resulting film. This is stated by Iling et al. (2017) that the lower the water absorption value is, the higher the water resistance of the biodegradable film will be. However, this theory is different from the research results. This study found that lower water absorption did not result in higher but lower water resistance values. This is presumably because the increasing concentration of added gelatin can affect the affinity of gelatin. According to Kumar et al. (2019), gelatin can increase the hydrophilicity of water molecules so that it is easier to absorb into the surface of the film. Water resistance will decrease, and solubility will increase. This is also supported by the results of the water solubility test and water content which increase with the higher concentration of duck bone gelatin. The higher solubility causes the film to hold less water so that the water absorption is low and affects the value of water resistance. Phenomenon found in the present study made this study inverse to other studies such as research of Kumar et al. (2020) and Iling et al. (2017). The lower water absorption is caused by the amount of dissolved film. The easier it is for the film to dissolve into water, the faster the process of decomposition (degradation) of the film will be (Asngad, 2018).

Analysis of Biodegradable Film Cross-Section Morphological Structure (SEM)

The cross-sectional view of the biodegradable film is shown in Figure 5.

Figure 5. Cross section of biodegradable film with different content of duck bone gelatin: (a) 0%, (b) 5%, (c) 35%

To obtain the observed cross-sections, the biodegradable films were cryogenically cut in liquid nitrogen. The micrographs of biodegradable taro starch films with the addition of content of 0%, 5%, and 35% duck bone gelatin are shown in Figures 5a, 5b, and 5c, respectively. SEM of biodegradable taro starch film (Fig. 5a) shows a relatively rough, dense, and continuous cross-section without pores and cracks, which confirm the solid and homogeneous structure. The micrograph of the biodegradable film with 5% duck bone gelatin can be seen in the cross-section showing dense and rough results with multiple folds. However, neither duck bone gelatin nor taro starch was seen, meaning that the addition of 5% duck bone gelatin resulted in a homogeneous and flexible biodegradable film with a compact structure. The micrograph of the biodegradable film with 35% duck bone gelatin had an uneven, rough, and multiple-fold cross-section (Fig. 5c), which may be due to the flexibility of the gelatin film (Wang et al., 2017). Figure 5c shows a cross-section that is very rough and duck bone gelatin, which is not bound to taro starch, meaning that many hydroxyl groups in duck bone gelatin are bound to free water. This is in accordance with the results of

the analysis of solubility, water absorption, and water vapor transmission rate. Mixing taro starch with duck bone gelatin aims to improve the internal structure of the taro starch film, where the biodegradable taro starch film with 0% gelatin shows less roughness and is denser and more compact, which may be caused by its less flexibility (Tongdeesoonporn et al., 2012). Biodegradable films with the addition of duck bone gelatin are a more flexible and will be easily decomposable and environmentally friendly.

Conclusion

1. Based on the findings, in making biodegradable films based on taro (*Xanthosoma saggitifolium*) starch and duck bone gelatin, there are some functional groups including NH at a wavenumber of 3.250 cm^{-1} , OH at 2.892 cm^{-1} , C=C at 1.539 cm^{-1} , CH at 1.438 cm^{-1} , and CN at 1.350 cm^{-1} . The biodegradable film functional groups have similarities in the manufacturing materials, namely starch, glycerol, duck bone gelatin, and hydrophilic hydrogen bonds that can facilitate the film degradation.
2. In addition, at a concentration of 35%, duck bone gelatin affects the physical characteristics of the biodegradable film in the form of a water vapor transmission rate of $0.53\text{ g/m}^2\cdot\text{hour}$ and a water solubility value of 64.60%. It also affects the value of water absorption (swelling) of 40.47%. The value of water absorption is depend on the number of films that dissolved in water and cause the solubility value to increase.
3. The results of the micrograph of the biodegradable film in the cross-section with SEM analysis show that dense and rough results with many folds, which means that the biodegradable film added with duck bone gelatin gives more flexibility and accelerates degradation.

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