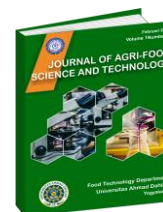


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Edible Film Based on Arrowroot Starch and Glycerol

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ABSTRACT

The edible film aims to improve the shelf life and safety of food by providing a physical barrier to external influences to prevent food deterioration. Arrowroot starch has high amylose content and excellent gelling ability, and it is massively produced in Indonesia. At the same time, glycerol has water-soluble and polar properties. So, studies were carried out on edible film based on arrowroot starch as the polysaccharide and glycerol as the plasticizer. This study was contributed to investigate the effect of the concentration of arrowroot starch (3, 4% w/v) and glycerol (1.25, 1.5, and 1.75% w/v) on physical properties (thickness by micrometer, functional group by FTIR, molecular structure surface by SEM) of edible film. The results showed that increasing the concentration of arrowroot starch and glycerol could increase tensile strength. Besides that, the formulations of 30% arrowroot starch with 12.5% glycerol and 40% arrowroot starch with 17.5% glycerol had the best tensile strength of 27.24 and 22.88 MPa, respectively. However, the results of the morphological analysis of the edible film on the arrowroot starch-glycerol formulation still contained pores and cracks.

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1. INTRODUCTION

Edible film is a thin layer made from edible materials that can be applied as a protective coating for food or placed on top of or between food components (Nogueira et al., 2021). Edible film can be produced from hydrocolloids, fats, and composites (Nogueira et al., 2018a). Hydrocolloid films exhibit superior mechanical properties, making them ideal for strengthening the structure of films and preventing their easy breakage. They are also highly effective as barriers to transferring oxygen, carbon dioxide, and fat (Nasution et al., 2019). Edible films can be made from polysaccharides like starch as raw materials. Due to its affordability, renewable nature, and favorable physical properties, starch is frequently utilized in the food industry as a biodegradable film substitute for plastic polymers. Several previous studies have examined edible film, namely edible film made from sorghum starch (Nogueira

et al., 2021) and edible film made from starch from cassava peel waste and using glycerol as a plasticizer (Cengristitama & Ramlan, 2022). Therefore, research on starch-based edible films with good mechanical properties and high water resistance is good (Tafa et al., 2023).

This study examines an edible film produced from starch derived from arrowroot starch and glycerol plasticizer (Nogueira et al., 2018b). Arrowroot starch is used as a source of starch because its utilization is not optimal, and it is abundant in almost every region (Nogueira et al., 2018b). Arrowroot starch is generally only used as a substitute for wheat in food processing, such as cakes, cakes, and bread (Fidianingsih et al., 2022).

Gelatinization is essentially used to create edible films made of starch (Maharijaya et al., 2020). Gelatinization happens if a specific amount of water is added and heated to a high temperature (Utama et al., 2018). The presence of hydrogen bonds during gelatinization causes the amylose bonds to tend to be close to one another. The drying process will result in shrinkage (Mahiuddin et al., 2018) as a result of the release of water so that the gel will form a stable film using glycerol as a plasticizer (content between 20-70%) for edible films based on a mixture of starch, gelatin, and sodium alginate. Plasticizers are organic materials with low molecular weight that are added to weaken the stiffness of the polymer while increasing flexibility and extensibility (Eslami et al., 2023).

Glycerol is one type of plasticizer that can be used to create edible films, and glycerol provides higher solubility compared to other plasticizers, such as sorbitol in starch-based edible films (Żolek-Tryznowska & Cichy, 2018). The purpose of using glycerol is to break down internal hydrogen bonds in intermolecular bonds, which helps to prevent water from evaporating from the product (Hamzah et al., 2021). It also dissolves in each polymer chain to help the molecules move around more easily, has a lower O₂ permeability, and is abundant, inexpensive, and non-toxic (Nasution et al., 2019). This study contributes to determining the effect of adding glycerol on the physical and mechanical characteristics of arrowroot flour edible film, as well as determining the concentration of added glycerol in arrowroot flour edible film, which shows the best physical and mechanical characteristics of edible film.

2. MATERIALS AND METHODS

2.1. Materials

The materials used: Arrowroot starch, glycerol, and aquadest. Equipment used such as spatula, beaker glass, thermometer, cabinet dryer, magnetic stirrer (Tensolab 5000, MesdanLab Italy), FTIR (Thermo Scientific Nicolet iS10, ThermoFisher Scientific America), and Scanning Electron Microscopy (JEOL JSM-6360LA, JEOL Japan).

2.2. Research Methods

The first stage is preparation, in which the arrowroot starch is weighed according to the treatment at 3% and 4% w/v. The second stage is preparing and characterizing edible films (Abdullahi et al., 2014). In this research, some variables are varied, and variables are determined. The varied variables are arrowroot starch concentration (3% and 4% w/v) and glycerol concentration (1.25, 1.5, and 1.75% w/v) based on the total mixture of edible film formulation (Warkoyo, 2019).

Arrowroot starch is dissolved in a certain amount of distilled water with a total volume of 50 ml, stirring for \pm 20 minutes using a stirrer. The mixture was then heated to 70°C, the gelatinization temperature of arrowroot starch, using a magnetic stirrer. A certain amount of 3% glycerol concentration solution was added to the mixture and stirred for five minutes after the mixture had been heated for twenty-five minutes. Air bubbles or other mixed-in impurities are eliminated when the solution is cooled. Next, a glass mold measuring 14 x 2.5 cm was filled with \pm 150 grams of solution and dried for approximately 4 hours at 70°C. After that, the mold

is removed and allowed to cool for about 20 minutes. The Edible Film is next taken out of the mold and prepared for different types of characterization analysis (Subando et al., 2023).

2.3. Thickness Analysis of Edible Film

The thickness of the resulting film was measured using a micrometer with an accuracy of 0.0001 mm. Measurements were conducted at five different places to obtain an average thickness representing the sample (Zamani, 2015).

The tensile strength testing process used a MesdanLab strength tool type, the Tensolab 5000, manufactured from MesdanLab Italy. The test was carried out by clamping the end of the sample with a tensile testing machine (Karlán & Rahmadhia, 2022; Shamsuri & Darus, 2020). Next, the initial thickness and length of the sample are recorded. When the start button on the computer is pressed, the tool will pull the sample at a speed of 100 mm/minute until the sample breaks (Dewi & Husni, 2020). The tensile strength value is obtained by dividing the maximum stress by the cross-sectional area. The cross-sectional area is obtained by multiplying the initial length of the sample by the initial thickness of the sample. The tensile strength test was carried out on three edible film samples, and the average was calculated. The tensile strength of bioplastics is calculated by the equation (1).

$$\tau = \frac{F_{max}}{A} \quad (1)$$

Where τ is Tensile Strength (MPa), F_{max} is Maximum Voltage (N), and A is cross-sectional area (mm²). Elongation at break measurements was carried out in the same way as tensile strength testing. Elongation is expressed as a percentage and calculated by the equation (2). Meanwhile, elasticity (Young's modulus) is obtained by comparing tensile strength with elongation (Ndukwe et al., 2021).

$$Elongation (\%) = \frac{\text{Strain at break (mm)}}{\text{Initial length (mm)}} \times 100\% \quad (2)$$

2.4. Functional Group Analysis with FTIR (Fourier Transform Infrared Spectroscopy)

Samples are examined using FTIR. The relevant spectrum is found after the sample is inserted into the set holder. The end product will be a diffractogram showing the relationship between wave number and intensity. A spectrophotometer operating at room temperature was used to record the FTIR spectrum (Montoya-Escobar et al., 2022).

2.5. Morphological Analysis with SEM (Scanning Electron Microscope)

Scanning electron microscopy (SEM) JEOL JSM-6360LA manufactured by JEOL Japan. The edible film samples were fastened to the set holder following a double adhesive application and vacuum-coated gold metal coating. The topographic picture was then examined and magnified 500 times after the sample had been placed into the SEM (Rianto, 2022).

2.6. Data Analysis

The data obtained were analyzed using descriptive analysis methods using IBM SPSS 22 methods. Then, data processing in this research used ANOVA analysis of variance at = 5% to ascertain whether the therapy had a notable impact. A differentiating test using the DMRT was performed on the data to determine if there was a significant effect.

3. RESULT AND DISCUSSION

3.1. Color of Arrowroot Starch and Glycerol Edible Film

Starch gelatinization can be accelerated by high levels of amylopectin and low levels of amylose by making starch less soluble in water, which is necessary for the starch to swell only in hot water (Jiang et al., 2022). There is a lot of space when amylopectin levels are high, and the mixing biopolymer will fill this space. A comparison of amylose and amylopectin levels in arrowroot starch in this study shows that arrowroot starch can be utilized as a potential ingredient to produce certain types of edible film (Alcázar-Alay & Meireles, 2015).

Water content analysis is conducted to determine the amount of water contained in food ingredients, in this case, the water content of arrowroot starch, and to simplify the subsequent process. The water content of the edible arrowroot starch produced in this study was 11.16% (w/w). Based on the result, the water content of arrowroot starch is very close to the SII standard water content of arrowroot starch, which is 14%. The water content of starch as an essential ingredient in edible film affects its shelf life. The higher the water content of starch, the shorter its shelf life because it becomes contaminated with microbes faster. In making this edible film with a relatively high-water content of arrowroot starch, this is not a big problem because, in making an edible film, arrowroot starch is dissolved in water, and then to prevent microbial growth, it is recommended that the basic ingredient of arrowroot starch not be stored for too long so that the starch from arrowroot remains ideal for use as a base material for edible films (Fakhouri et al., 2019).

The humidity of the surrounding air is another determining factor, and it is connected to the type and nature of the material, its storage location, and any treatments it has received. Knowing the starch brightness value, which will influence the edible product or preparation results, the whiter the starch used, the more transparent the edible film produced will be. This degree of brightness was measured using a chromameter on several samples based on the mesh sieve results (Marta et al., 2023).

Table 1. Color of arrowroot starch and glycerol edible film.

Sample	L	a*	b*
A1B1	88.81 ± 0.37 ^{ab}	-0.82 ± 0.108 ^a	-1.77 ± 0.48 ^a
A1B2	89.18 ± 0.24 ^{ab}	-0.76 ± 0.021 ^a	-1.48 ± 0.11 ^a
A1B3	89.36 ± 0.21 ^b	-0.79 ± 0.071 ^a	-1.72 ± 0.24 ^a
A2B1	88.60 ± 0.099 ^{ab}	-0.72 ± 0.028 ^a	-1.5 ± 0.23 ^a
A2B2	89.05 ± 0.081 ^{ab}	-0.73 ± 0.035 ^a	-1.64 ± 0.17 ^a
A2B3	88.14 ± 0.099 ^a	-0.83 ± 0.049 ^a	-1.53 ± 0.16 ^a

Different letter notations indicate significant differences ($p < 0.05$) in response values due to treatment.

The test results show that the degree of brightness for the edible film produced from arrowroot starch measuring > 200 mesh has a brightness value of L*89.36, a* -0.79, and b* -1.71 with a pale gray starch color, which, when viewed from the a and b values, displays the distinctive bright color and bluish-red color. The L value indicates the brightness level of the edible film with arrowroot starch. The higher the measured L value, the brighter the actual visible color. With an L value of arrowroot starch of 89.36, arrowroot starch is suitable for making edible films because it will produce transparent edible films that are supportive of aesthetics and marketing (Nogueira et al., 2019).

3.2. FTIR of Arrowroot Starch and Glycerol Edible Film

The characteristics of edible films produced from glycerol and arrowroot starch are presented, and each mixing step is completed using the FTIR spectroscopy technique.

Identifying the functional groups present in each stage of the edible film mixing process attempts to figure out the mixing mechanism and determine whether or not the edible film contains new functional groups. The FTIR spectrum displayed in Figure 1 indicates that no new functional groups were formed during the edible film formation process (Choque-Quispe et al., 2021).

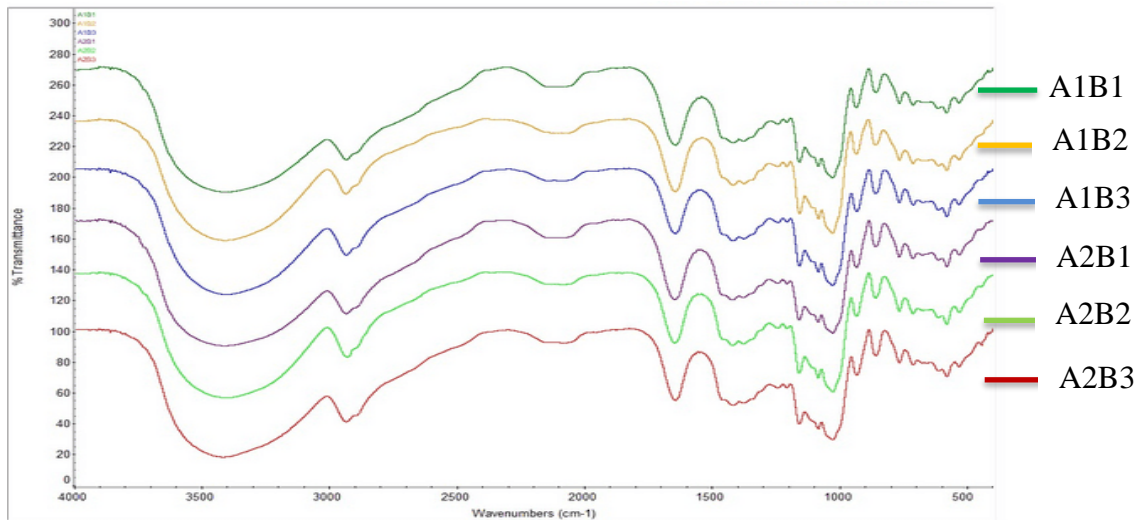


Figure 1. FTIR spectrum of arrowroot starch and glycerol edible film. A1B1 (arrowroot 3% + Glycerol 1.25%); A1B2 (arrowroot 3% + Glycerol 1.5%); A1B3 (arrowroot 3% + Glycerol 1.75%); A2B1 (arrowroot 4% + Glycerol 1.25%); A2B2 (arrowroot 4% + Glycerol 1.5%); A2B3 (arrowroot 4% + Glycerol 1.75%).

Figure 1 (A) shows that the FTIR results of starch show the absorption of C-O after the addition of arrowroot 3% + glycerol process in Figure 1 (B and C). After the edible film in Figure 1 (D) was formed, After the addition of arrowroot 4% + glycerol process in Figure 1 (B and C), the absorption of the OH group experienced a widening due to interactions and the possibility of water absorption in the edible film (Basiak et al., 2018). Figure 1 (A) shows that the FTIR results of starch show the absorption of C-O groups at a wave number of 858.66 cm^{-1} ; OH group at absorption $3398.11 - 3414.11\text{ cm}^{-1}$; NH at absorption 1638.68 cm^{-1} ; and C-O 2930.22 cm^{-1} , but with a minimal absorption intensity. After the addition of arrowroot 3% and glycerol too much (Figure 1 (B)), the NH absorption intensity widened, increased, and was quite sharp at the wave number 1638.68 cm^{-1} and the wave number 1548.22 cm^{-1} . The intensity of OH absorption is also wider and stronger, which shows that NH and OH groups are influenced by glycerol.

After the addition of 4% arrowroot in Figure D, the absorption intensity of the NH group is not too wide, nor does the intensity of the OH group decrease due to the loss of water molecules. After adding too much glycerol in Figure 1 (D), the FTIR spectrum is almost the same and does not show a significant difference but experiences a slight shift, while the OH becomes sharper and stronger due to glycerol, which has many OH groups. After the edible film in Figure 1 (E and F) was formed, the absorption of the OH group experienced a widening due to interactions and the possibility of water absorption in the edible film. The NH absorption intensity and peak sharpness increase significantly, indicating that a membrane has formed.

The findings of the FTIR analysis show that physical mixing and hydrogen interactions within chains are necessary for the production of edible film. The proposed hydrogen interactions between amylose, amylopectin, and glycerol chains in edible film are shown in Figure 2. The illustration of the presence of hydrogen bonds in the form of edible film is shown

in Figure 2. This hydrogen bond occurs when an O or N atom molecule in glycerol interacts with an H atom from amylose, amylopectin, or glycerol. This hydrogen interaction can also happen between amylose and amylose with amylopectin. Based on the interactions, it can be inferred that arrowroot starch and glycerol can enhance the mechanical properties of the edible film by forming hydrogen bonds between chains, causing the edible film to become tighter and stiffer. Glycerol is added to the edible film to decrease hydrogen interactions, make it less stiff, and become elastic (Choque-Quispe et al., 2021).

Table 2. Mechanical properties of arrowroot starch and glycerol edible film.

Sample	Fmax (N)	Tensile Strength (MPa)	Strain at Tmax (%)
A1B1	21.48 ± 0.14 ^c	26.54 ± 0.98 ^c	1.71 ± 0.03 ^a
A1B2	16.33 ± 0.66 ^b	25.27 ± 1.03 ^c	2.02 ± 0.38 ^a
A1B3	7.28 ± 3.12 ^a	15.58 ± 6.24 ^a	2.38 ± 0.56 ^a
A2B1	8.73 ± 0.35 ^a	14.15 ± 0.82 ^a	2.36 ± 0.09 ^a
A2B2	16.73 ± 0.21 ^b	17.84 ± 0.03 ^{ab}	2.10 ± 0.69 ^a
A2B3	18.91 ± 2.37 ^{bc}	22.42 ± 0.65 ^{bc}	2.21 ± 0.51 ^a

a, b, c Different letter notations indicate significant differences ($p < 0.05$) in response values due to treatment.

Different starch concentrations produce significantly different Fmax responses, namely 30% starch concentration produces Fmax 21.48 ± 0.14 N, tensile strength 26.54 ± 0.98 MPa, while 40% produces Fmax 8.73 ± 0.35 N, tensile strength 14.15 ± 0.82 MPa. The mechanical characteristics of the final edible film are significantly influenced by the components, which include glycerol as a plasticizer and arrowroot starch as a mixing biopolymer. The three variables that influence the mechanical properties of edible film are elongation, tensile strength, and Young's modulus. Table 1 is a table of the mechanical properties of the edible film, while the arrowroot starch and glycerol formulations can all be tested for their mechanical properties. This shows that edible film made from starch alone produces low mechanical properties of around 8 MPa. The following is a table of test results analyzing the mechanical properties of arrowroot starch edible film. Table 1 shows that the samples A1B1 (30% of arrowroot starch and 12.5% of glycerol) and A2B3 (40% of arrowroot starch and 17.5% of glycerol) have the best tensile strength values of 26.54 MPa and 22.42 MPa, respectively. Glycerol addition quantity and tensile strength are directly correlated.

Characteristics of arrowroot starch are the result of extracting arrowroot tubers from the arrowroot plant (*Maranta arundinaceae* L.), which is a type of tuber that has a starch content of around 80 - 85%, so arrowroot tubers are not inferior to other tubers which are considered a source of starch, such as cassava starch tree (85%), cassava starch (63%), and potato starch (8%) (Nogueira et al., 2021). Starch is composed of two important fractions: amylose, a linear fraction, and amylopectin, a branch fraction. Planting is still widespread in rural areas, but its sustainability is increasingly threatened. In arrowroot tubers, the amylose and amylopectin content is 15.21% and 84.79%, respectively (Malki et al., 2023; Rahmadhia et al., 2023).

Where A1B1 is 30% of arrowroot starch and 12.5% of glycerol; A1B2 is 30% of arrowroot starch and 15% of glycerol; A1B3 is 30% of arrowroot starch and 17.5% of glycerol; A2B1 is 40% of arrowroot starch and 12.5% of glycerol; A2B2 is 40% of arrowroot starch and 15% of glycerol; A2B3 is 40% of arrowroot starch and 17.5% of glycerol; and average aO is 0,146 mm; bO is 5 mm; Lc is 50 mm; Fmax is 15,5563 N.

Adding glycerol can raise the tensile strength, but the tensile strength value tends to decrease with increasing starch. Because glycerol can create hydrogen bonds between chains to make the edible film tighter, glycerol, as a plasticizer, is inclined to increase the tensile

strength value in specific formulations. According to the research findings, the function of glycerol can be seen as indicated by the greater elongation value in small arrowroot starch and glycerol formulations. In contrast, in small arrowroot starch formulations, the role of glycerol is less visible due to the lack of starch, which tends to be less active in interacting with hydrogen and other monomers in edible film (Rahmadhia et al., 2022; Wulandari et al., 2017).

This tensile strength analysis is used to determine the strength and deformation of the film at the breaking point. A higher percentage of arrowroot starch (40%) results in a lower tensile strength value. However, glycerol addition can raise tensile strength. The tensile strength value is inversely proportional to the amount of arrowroot starch added. This is because more hydrogen interactions will be contained in the edible film, so the bonds between chains will be stronger and more difficult to break because a large amount of energy is required to break the bond.

The tensile strength values of A1B1 (30% of arrowroot starch and 12.5% of glycerol) and A2B3 (40% of arrowroot starch and 17.5% of glycerol) samples, namely 26.54 MPa and 22.42 MPa, respectively. This result meets the standard tensile strength values, with a biodegradable plastic tensile strength value of 10-100 MPa and seen from the tensile strength value of polypropylene of 10-100 MPa, namely 24.7, which is quite close to (Nogueira et al., 2017). When glycerol is present, the plasticizer molecules cause the starch to become less compact, decrease intermolecular interactions, and increase polymer mobility (Nogueira et al., 2021). Tensile strength decreases, and elongation rises with an increase in glycerol concentration.

3.3. Edible Film Surface Morphology of Arrowroot Starch and Glycerol Edible Film

The findings of the surface morphology analysis of the arrowroot starch-glycerol edible film are shown in Figure 2. Based on Figure 2(b), it can be seen that the molecular structure surface of the arrowroot starch edible film does not appear to be dense. Starch fibers with a relatively large particle size (20–30 mesh) that do not dissolve completely are thought to cause cracks in the edible film. More water will be absorbed if the structure is less dense or the fibers are cracked. The image also shows a less smooth and porous surface. A surface that is not smooth indicates that the film is less homogeneous.

However, the tensile strength value tends to increase with increasing glycerol concentration. Because glycerol contains several active compounds that can influence the tensile strength value of the edible film produced, it also has to be adjusted by adding the right amount of starch. This follows (Warkoyo, 2019) statement that the tensile strength value of edible film will weaken as the concentration of active ingredients increases because there are interactions between molecules, which can weaken as the amount of active ingredients added increases. The tensile strength values of arrowroot starch concentration and glycerol concentration meet JIS (Japanese Industrial Standard) standards. Whereas, according to the JIS 1975 Standard (Japanese Industrial Standard), the tensile strength value of the edible film is at least 3.92 Mpa (Rahmiatiningrum et al., 2019).

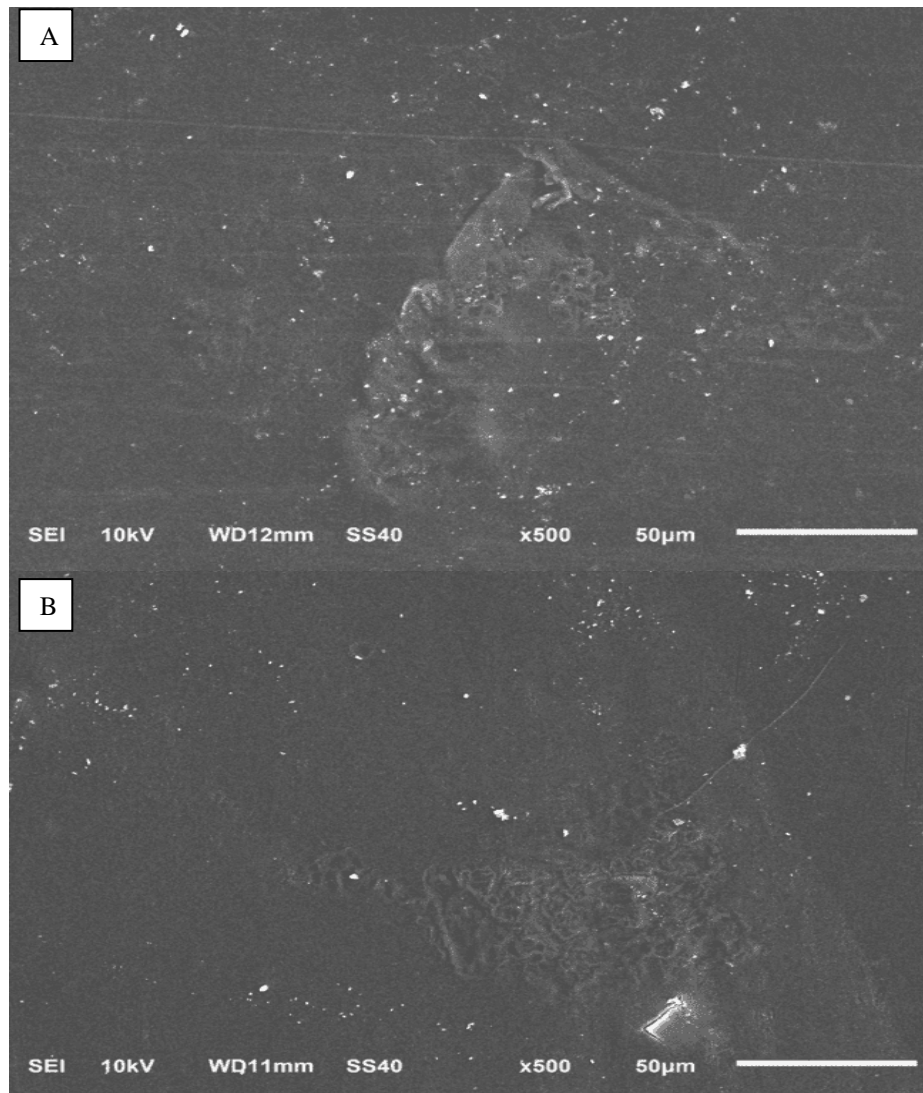


Figure 2. A) Scanning electron microscopy of edible film with arrowroot starch 3% and glycerol 1.5%; B) Scanning electron microscopy of edible film with arrowroot starch 4% and glycerol 1.5%.

4. CONCLUSIONS

In the range of the arrowroot starch (3, 4% w/v) and glycerol concentration (1.25, 1.5, and 1.75% w/v), the increasing concentration of arrowroot starch and glycerol could increase tensile strength. Besides that, the formulation of 30% of arrowroot starch with 12.5% of glycerol and 40% arrowroot starch with 17.5% of glycerol produces high tensile strength of 26.54 MPa and 22.42 MPa, respectively. However, the results of the morphological analysis of the edible film in the arrowroot starch-glycerol formulation still contain pores and cracks.

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