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The Effect of Starch Concentration on the Characteristics of Modified Purple Yam Starch Using the Precipitation Method

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Abstract :
Purple yam starch-based edible film experienced high water vapor transmission rate. The use of composite starch using addition of modified starch of smaller size particles may decrease the rate. This study aimed to obtain starch concentrations that produced smallest size of modified starch, to produce composite starch for edible films and to determine the characteristics of edible films made out of native starch and edible films from composite starch (mixture of native starch and modified starch with the smallest size). This research was conducted in 2 stages: modification of purple yam starch by precipitation method and production of edible films from native starch and composite starch. This study was designed to produce modified starch using 5 levels of starch concentration (0,2%, 0,4%, 0,6%, 0,8%, and 1%) and 2 repetitions. The results showed that the treatment of 0,8% starch concentration produced the smallest particle size of starch (3,211 x 10,340 µm) up to (20,876 x 25,437 µm) with starch yield of 76,25%. Edible films made out composite starch have different characteristics than native starch alone. Edible films from composite starch produced higher thickness values of 0.161 ± 0.007 mm, lower solubility 32,080 ± 4,671%, lower transparency of 10.644 ± 0.357% / mm and lower water vapor transmission rate (WVTR) of 24,701 ± 7,05 g / m^2. hour and higher compressive strength 356,87 ± 7,38 gF than native starch edible films.

Keywords: Edible film, precipitation, composite starch, yam starch

1. Introduction

The purple yam plant (Dioscorea alata) is a type of tuber that is widely found in Indonesia, one of which is in the province of Jambi. Purple yam is rich in carbohydrates so it can be developed as an alternative food, because it contains 75-84% starch (dry weight), carbohydrates (76.57%) and energy (357.65 kcal/100g) (Ezeocha & Ojimelukwe, 2012). Most of the carbohydrates in the form of starch in cassava tubers consist of amylose and amylopectin. Yam starch contains around 19-20% amylose. High starch production and high amylose content make yam starch very potential as a base material for edible films (Rubatzky and Yamaguchi, 1998). According to (Garcia et al, 2011) materials that have high amylose can be made into edible films. A high amylose content will make the film more compact because amylose is responsible for the formation of the film matrix.

Edible film is a thin layer that can be used as food packaging or coating which can also be eaten together with the packaged product (Guilbert and Biquet, 1990) in (Yulianti and Ginting, 2012). Maryana (2018) conducted research on making edible film from two different types of cassava starch. The best research results, the use of 2.67% yam starch produced an edible film thickness of 0.12 mm, transparency of 13.245%, solubility of 16.7%, water vapor transmission rate (WVTR) of 41.9 g/m2. O'clock. From the results of this research, it is known that the water vapor transmission rate of edible film from yam starch is still high. The quality of edible film is still low because the edible film is made from natural cassava starch only. One way that can be done to improve the characteristics of edible film from starch is to use a composite starch mixture of native starch and modified starch.

Small sized starch can be applied as a matrix or filler which functions as reinforcement in the formation of nanocomposites either with natural rubber or synthetic plastic. Farrag et al. (2018) succeeded in improving the water vapor transmission rate of edible film from corn starch by adding 15% corn starch which had been modified using the precipitation method thereby reducing the water vapor transmission rate of the film from 9.07 g/m2.hour to 8.18 g/m¬ 2 hours. The same results also occurred in edible film from pea starch where there was a decrease in the water vapor transmission rate from 8.33 g/m2.hour to 6.68 g/m2.hour (Farrag et al., 2018). Research by Gonzàlez et al., (2015) which made edible film from a mixture of corn starch, modified corn starch and also microcrystalline cellulose with acid hydrolysis modification also succeeded in improving the mechanical properties and reducing the water vapor transmission rate by 54.2 g/m2.Hour.

According to Le Corre et al. (2010) there are three ways to make smaller starch, namely acid or enzymatic hydrolysis, regeneration (precipitation) and mechanical treatment. Modification of starch by precipitation is a modification of starch which is classified as physical and mechanical modification, where the principle of the precipitation method is to heat above the gelatinization temperature and precipitate the starch using a solvent (Winarti, 2011). According to Sauyana (2014), the acid hydrolysis process in starch modification only changes the chemical structure of the starch, but slightly changes the physical structure of the starch granules so that further modification is required, namely precipitation. In the precipitation process, heating above the gelatinization temperature and rapid stirring causes the starch amylose and amylopectin chains to break so that smaller particle sizes are formed.

In the precipitation method, the starch concentration greatly influences the size of the modified granules. (Saari et al. 2016) stated that increasing the starch concentration from 1 mg/mL to 2 mg/mL increased the particle size of modified starch, but if the concentration continued to be increased from 2 to 4, 8 and 10 mg/mL, the size of the starch particles decreased. up to 0.3 µm. If the starch concentration continues to be increased to 20 mg/mL, the size of the starch particles produced increases again. Research by Ma et al. (2008) who used 0.05% corn starch produced starch granule sizes of 50-300 nm, while Kim et al. (2009) used high amylose corn starch with a starch concentration of 0.5% producing granule particle sizes <100 nm. This research aims to obtain starch concentrations that can produce modified starch with the smallest size and determine the characteristics of edible films made from starch composites plus the best modified starch as a result of research.

2. Research Methods

Materials

The materials used in this research were yam starch obtained from previous research, glycerol, distilled water, 96% technical ethanol and absolute ethanol, while the materials used for analysis included $Mg(NO_3)_2$, NaCl and CaCl.

The tools used in this research are analytical scales, sieves, 200 mesh sieves, basins, baking sheets, electric ovens, measuring cups, beakers, stirrers, magnetic stirrers, centrifuges, test tubes, petri dishes, plastic clips and plastic wrap as well as analytical tools in the form of a scanning electron microscope, screw micrometer, thermometer, spectrophotometer, screw tube, desiccator, cup and vortex.

Research Design

This research was designed to produce modified starch using 5 levels of starch concentration and carried out in 2 repetitions. The levels of treatment in this study are as follows:
K0 = Control (native starch)
K1 = Starch concentration 0.2%
K2 = Starch concentration 0.4%
K3 = Starch concentration 0.6%
K4 = Starch concentration 0.8%
K5 = Starch concentration 1%

Modification of Starch using the Precipitation Method (Qin et al., 2016 with modification)

Starch was weighed according to the treatment concentration (0.2%, 0.4%, 0.6%, 0.8% and 1%) and added to 100 mL of distilled water. The mixture was then heated at 90°C for 30 minutes using a stirring rod. The solution was cooled immediately and 1000 mL of ethanol was added little by little while continuing to stir. The solution was then left for 8 hours at room temperature while continuing to be stirred constantly using a stirrer. After the stirrer process, the solution was then centrifuged at a speed of 2500 rpm for 15 minutes. The precipitate was washed with absolute ethanol 3 times. The precipitate was dried using a drying process without heat (cold air drying). Modified starch was stored in polyethylene plastic at room temperature.

Preparation of Edible films (Gonzalez et al., 2015)

There are two types of edible films made in the research, namely edible films from natural purple cassava starch and edible films from composite starch. The composite starch in question is a mixture of natural purple yam starch and modified purple yam starch with the smallest particle size. Both edible films were made using similar procedures with the difference that there was the addition of modified cassava starch at the end of the process of making edible films from composite starch.

Purple yam starch with a concentration of 2% (3 g) was dissolved in distilled water in a beaker and stirred without heating for 10 minutes. After that, the solution was heated on a hot plate using a magnetic stirrer until gelatinization occurred (temperature 80ºC) for 10 minutes. The heated solution was added with 2% glycerol (3 g) and heated again for 10 minutes. The dissolved solution is added with modified starch as much as 15% (0.45 g) of the amount of native starch used and homogenized. This step is omitted for making edible film from native starch of purple yam. The film solution was then removed and printed using a petridish with a diameter of 9.2 cm as much as 25 g. Next, the petridish containing the edible film solution is dried using an oven at a temperature of 50ºC for 24 hours. The dried edible film was then stored in a desiccator with RH 52% at room temperature before analysis.

Modified Starch Rendement (Juliana, 2007)

Measurement of the yield of modified yam starch is calculated based on the comparison of the final weight obtained after modification to the weight of the sample used before modification, expressed in percent (%), namely:

$$Yield(\%) = \frac{Final\ weight}{Sample} x100\%$$

Morphology of Starch Granules (Juhana, 2018)

Modified yam starch was tested using a scanning electron microscope (model JEOL JSM 6510 LA) which had previously been dispersed using alcohol. The samples were placed on aluminum stabs using double-sided adhesive tape and coated with gold powder to avoid charging under the electron beam after the alcohol evaporated. Starch granules were observed at 250x and 1000x magnification.

Film Thickness (Mendes et al., 2016)

The film thickness was measured using a scrub micrometer at 5 different places and chosen randomly. The average of these five values is then reported as the film thickness.

Solubility (Gontard et al., 1993)

Filter paper was dried and weighed. The film samples were cut into 2x2 cm pieces, put into 50 mL of distilled water and soaked for 24 hours while stirring periodically. The solution was then filtered and the filter paper dried at 1500C for 24 hours. The number of films that did not dissolve was then weighed. % Solubility can be calculated using the formula:

$$\%\ \text{Solubility} = \frac{w2 - (w3 - w1)}{w2} \text{x } 100\%$$

Note:
w1 = Initial filter paper (g)
w2 = Initial edible film (g)
w3 = filter paper and insoluble edible film

Transparency (Pineros-Hernandez, 2017)

The film was cut square (50x10mm), placed in the spectrophotometer cell. Percent transmittance was measured with a UV-Vis spectrophotometer at a wavelength of 600 nm. Edible film transparency is calculated using the formula:

$$\text{Transparency} = \log T / \text{Thickness}$$

Water Vapor Transmission Rate/WVTR Analysis (Pineros-Hernandez, 2017)

A test tube containing calcium chloride is covered using film. The weight of the tube is then weighed. The tube was placed in a desiccator that was saturated using saturated sodium chloride (RH 75%). The change in tube weight is then recorded and plotted as a function of time. WVTR calculations can use the formula:

$$\text{WVTR} = \frac{\text{Slope}}{A}$$

Note:
WVTR = Water vapor transmition (g/m2. hour)
Slope = Linear function of weight gain and time (g/hour)
A = Film area (m2)

Compressive Strength

Compressive strength was measured with a Brookfield brand LFRA Texture Analyzer using the TA 7 60 mm type probe. The Texture Analyzer was set to cycle count with trigger 2 g, distance 2 mm, and speed 2mm/s. The probe was installed in place and the “start” button is pressed to start pressing the edible film. A sample of edible film that has been cut to a size of 5 x 2 cm is placed under the probe and the probe will press the film until the magnitude of the probe force used appears on the screen.

3. Results and Discussion

Modified Starch Yield

The yield of modified starch is calculated as a percentage of the weight ratio of the starch after modification to the weight of starch before modification. The yield of modified starch can be seen in Table 1.

In Table 1 it can be seen that the higher the concentration, the higher the resulting yield. The precipitation method for modifying cassava starch to obtain a smaller starch size begins with the gelatinization process. During starch gelatinization, the starch granules slowly swell as the heating temperature increases. Swelling occurs because water molecules enter and become trapped between the amylose and amylopectin molecules. Swelling of starch granules also causes the hydrogen bonds between amylose and amylopectin to weaken, so that the compactness of the starch granules is also weakened. After the swelling of the starch granules reaches a maximum at a certain heating temperature, the starch granules can no longer hold the amount of water, and finally burst. When starch granules break down, granule molecules in the form of amylose are released from the starch granules. The release of amylose causes damage to the structure of the amorphous area. The damaged amorphous area will change the structure and shape of the starch granules (Faridah, 2011).

When using concentrations of 0.2, 0.4, and 0.6%, the resulting starch yield increases as the starch concentration increases, but the yield decreases at a concentration of 0.8% to 76.25% and increases again to 80% when the starch concentration increased to 1%. At a starch concentration of 0.8%, the yield of modified starch produced decreased. The decrease in yield was caused by the fact that at a starch concentration of 0.8%, the water was sufficient to make the starch completely gelatinized. Starch that is completely gelatinized will cause many amylose molecules to come out of the granules, because the outermost (amorphous) area in the granules has been damaged. The amylose that comes out can be further degraded into simple compounds such as sugar. Degradation is caused by the starch concentration being too low so that the hot temperature during the gelatinization process can hydrolyze more starch. Simple sugars that are soluble in water cannot be withdrawn and precipitated, so the resulting yield will be low. The higher the concentration used in modifying starch, the more difficult it is to hydrolyze starch into simpler compounds so that the resulting yield will be high.

This was also proven by Wulandari (2013) who modified sago starch and tapioca starch using the precipitation method. The resulting yield ranged between 92.84-94.54% for sago starch and 85.38-87.17% for tapioca starch. The high yield is because during the precipitation process, no further destruction of the amylose and amylopectin chains occurs so that the resulting yield is high.

Morphology of Starch Particles

The shape and size of the starch granules of natural purple yam and modified purple yam appeared to be different. The shape of natural purple cassava starch granules looks very diverse, ranging from round discs, elliptical and also oval. Starch granules also have a smooth surface and are not the same size. The size and shape of starch granules can be seen in Table 1.

The use of concentration in starch modification affects changes in starch particle size. The ratio of water and starch concentration can affect the size of the starch particles. Zhang et al., (2012) also stated that a water ratio that is greater than the starch concentration in the gelatinization process causes uncontrolled swelling of the granules and ultimately causes the structure of the starch granules to be damaged. Sufficient water conditions and the heating temperature that has been reached in the gelatinization process will damage the starch granules and ultimately small sized starch particles will form (Maaruf et al., 2001).

Table 1. Average yield and particle size of modified starch at various concentrations

Starch Concentration (%)	Modified Starch Yield (%)	Modified Starch Particle Size (µm)
0.2	65 ± 0.07	7.767 x 24.486 to 15.200 x 30.348
0.4	75 ± 0.14	5.161 x 6.878 to 14.519 x 25.247
0.6	79 ± 0.07	8.621 x 14.919 to 20.147 x 23.523
0.8	76.25 ± 0.07	3.211 x 10.340 to 20.876 x 25.523
1	80 ± 0.07	8.497 x 17.119 to 21.115 x 21.621

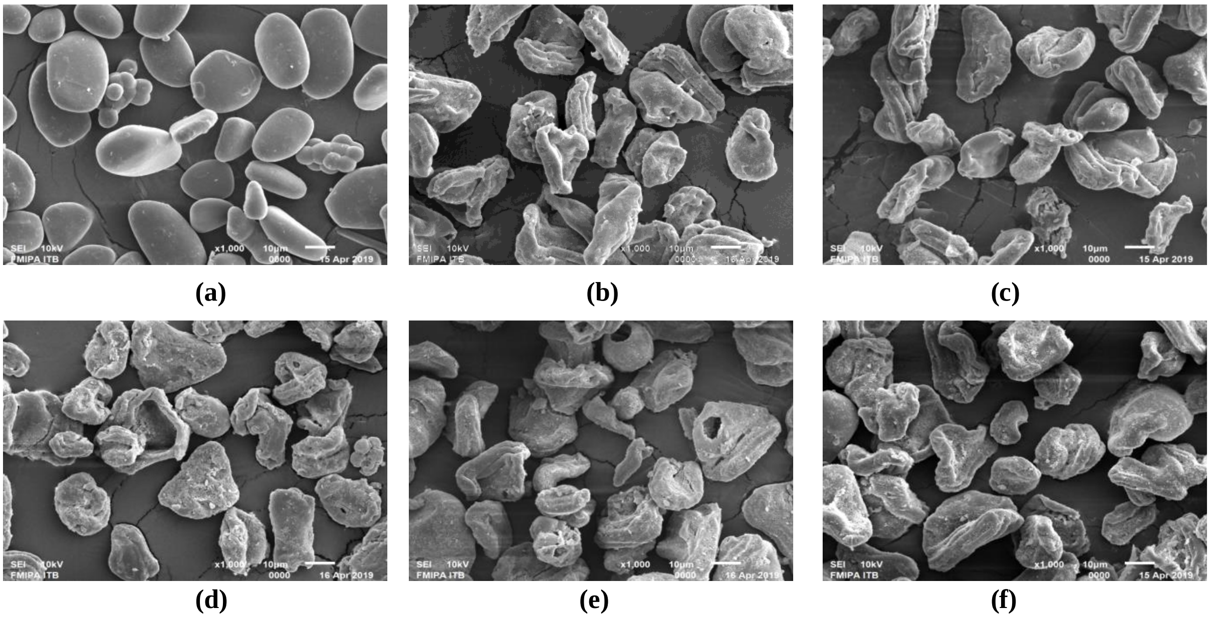

Figure 1. Scanning Electron Microscope Results (a). Native starch of purple yam; (b). Modified starch concentration 0.2%; (c) Modified starch concentration 0.4%; (d). Modified starch concentration 0.6%; (e). Modified starch concentration 0.8%; (f). Modified starch concentration 1%.

In Figure 1 it can be seen that all starch concentration treatments produce granules that have the same shape, namely in the form of polygonal structures that are hollow, dredged and folded. The size of the starch granules after precipitation appears to decrease. The treatment that produced the smallest size was at a concentration of 0.8%, producing size of 3.211 x 10.340µm to 20.876 x 25.437µm. However, the increase in the concentration to 1%, will increase the size of the starch particles. This is in line with research conducted by Saari et al., (2016) which states that increasing the starch concentration from 1 mg/mL to 2 mg/mL increases the size of the starch particles. If the starch concentration is increased to 4 to 10 mg/mL, size of the starch granules will decrease. Further increase in the concentration to 20 mg/mL, will increase the size of the starch granules.

Table 2. Average values for thickness, solubility, transparency and WVTR of edible films

Parameter	Native starch	Composite starch
Thickness (mm)	0.123 ± 0.013	0.161 ± 0.007
Solubility (%)	36.051 ± 4.946	32.080 ± 4.671
Transparency (%/mm)	15.480 ± 0.132	10.644 ± 0.357
WVTR (g/m^2. jam)	49.402 ± 9.98	24.701 ± 7.05
Compressive Strength (gF)	219.85 ± 3.46	356.87 ± 7.38

Thickness of Edible Film

Thickness is an important parameter in edible film, because it can affect its application to a product. Thickness can also affect other parameters such as transparency, solubility, water vapor transmission rate (WVTR) and compressive strength. The edible film thickness value was taken from the average at 5 different film points using a screw micrometer. The average value of edible film thickness can be seen in Table 2.

The thickness values obtained in Table 2 show that the addition of modified starch concentration to composite edible films affects the film thickness values. The edible film thickness value increased from 0.123 ± 0.013 mm to 0.161 ± 0.007 mm with the addition of smaller sized starch resulting from precipitation modification. This is in accordance with Santoso (2018), Mudaffar (2018), and Supeni & Irawan (2012) who state that the higher the total solids, the greater the thickness of the composite edible film produced.

The addition of smaller sized starches to edible film produces good edible film thickness because it complies with the Japanese Industrial Standard (JIS), where the maximum thickness for films categorized as food packaging is 0.25 mm (Saputra, 2015). The thickness of the edible film obtained in this study was 0.12 mm for natural edible film and 0.16 mm for composite starch edible film which had added modified starch. The results of this research are in line with Cerqueira's (2017) research on edible film from yam starch composite with the addition of coconut fiber extract cellulose nanocrystals. The film thickness increases with increasing concentration of cellulose nanocrystals from 0.8 to 0.9 mm.

According to Yulianti and Ginting (2012), the thicker the edible film, the higher the ability of the edible film to inhibit the flow of water, so the product's shelf life will be longer. However, Jatmiko (2019) stated that if it is too thick it will affect the taste of the product when eaten, so the thickness of the edible film must be adjusted to the product being packaged.

Solubility of Edible Film

Solubility is the percentage of edible film that dissolves in water during 24 hour immersion. Film solubility is an important factor in determining the biodegradability of edible film when used as packaging material. According to Jatmiko (2019), edible film with high solubility is very good for use in ready-to-eat food products because it dissolves easily when consumed. On the other hand, low solubility is one of the important requirements for edible film as food packaging that comes into contact with water and acts as a protector for food products.

As can be seen in Table 2, the addition of smaller starch into the ingredients that make up the edible film reduces the solubility value from 36.05% in native starch edible film to 32.08% in composite edible film. This research produced a higher value compared to research by Liu et al., (2015) who made a composite edible film with the addition of 25% corn starch which was smaller in size, namely 22.33%. Jania et al., (2012) stated that the addition of smaller starch to edible film can increase the stability of the film in water, causing the film to not dissolve easily in water. Composite edible film has higher hydrophobicity properties than edible film made from native starch because smaller starch is added to its manufacture.

Smaller sized starches are more hydrophobic because during the starch modification process, the amorphous areas in the starch granules have been damaged.

The amorphous area in starch is an area that is more hydrophilic than the crystalline area, so starch's ability to bind water becomes more difficult. The smaller crystalline areas remaining in starch when mixed with native starch when making edible film will make the crystalline structure stronger, so that the solubility of the edible film decreases. This is in line with research by Warkoyo (2014) which states that the lower the hydrophilic groups in the ingredients that make up edible film can cause a decrease in the solubility of edible film.

Transparency of Edible Film

Transparency describes the amount of light that can pass through edible film (Maryana, 2018). Light from the UV-Vis Spectrophotometer passes through the edible film so that the transmittance value can be obtained. Based on the research results, the percentage of transparency value decreased with the addition of modified starch resulting from precipitation.

Based on Table 2, it can be seen that the addition of 0.5 grams of modified starch from the previous stage of research from 150 grams of material in making composite edible films, reduces the transparency value from 15.48%/mm in native starch edible films to 10.64%/mm in edible film from composite starch. This result is in line with research by Liu et al., (2015) who made edible film with the addition of smaller sized corn starch resulting in a transparency value that decreased from 80%/mm for natural corn starch to 66%/mm for corn starch with the addition of starch. smaller sized corn.

The addition of smaller sized starch as a result of modification by precipitation causes the level of transparency of the edible film to decrease. This is in accordance with the opinion of Shi et al., (2013) that when light passes through the film, the light cannot penetrate the gaps (interspaces) of the edible film because the gaps in the edible film have been filled with small starch particles. This will produce an edible film with a low level of transparency.

WVTR (Water Vapor Transmission Rate) Edible Film

Water vapor transmission rate (WVTR) or water vapor transmission rate is the movement of water vapor entering the edible film at a certain time, temperature and humidity. The WVTR value will indicate the film's ability to inhibit water vapor. The greater the WVTR value, the greater the rate of water vapor in the film which inhibits water vapor less well.

Based on Table 2, it can be seen that the addition of smaller starch reduces the WVTR value of the composite edible film. The WVTR value of edible film decreased from 49.402 ± 9.98 g/m2. hours on natural edible film to 24.701 ± 7.05 g/m2. hours on composite edible film. The results of this research are in line with research by Suàrez et al., (2013) who made edible film from potato starch with the addition of cellulose nanoparticles up to 1.2% resulting in a WVTR value that decreased from 1.7 x 10-10 to 0.7 x 10- 10 g/m2. O'clock. The WVTR (Water Vapor Transmission Rate) value decreased due to the addition of smaller starch in the manufacture of edible film. The smaller sized starch is more hydrophobic than native starch, so the hydrophobicity of the edible film increases. Garcia et al., (2011) stated that water vapor migration generally occurs in the hydrophilic part of the film.

Garcia et al., (2000), Barus (2002), and De Azeredo et al., (2009) stated that the increase in film water vapor resistance was associated with the addition of smaller starch into the ingredients making up the edible film. During the production process of smaller sized starches, the amorphous part of the starch is lost and the crystalline part remains so that the starch is more hydrophobic. This smaller sized starch, when added to making edible film, will form winding paths on the surface of the edible film. Winding paths are formed in the hydrophilic part of the film, while the more hydrophobic part will resist the diffusion of water vapor in the film. The part of the film that is more hydrophobic is due to the addition of smaller starch which has filled the empty spaces in the edible film, so that water will find it difficult to penetrate that part of the film.

Muller et al., (2011) also stated that the decrease in the water vapor transmission rate (WVTR) of starch-based films was caused by the incorporation of small starch into the edible film, due to the winding paths that were also visible in the resulting films for the diffusion of water vapor.

The WVTR (Water Vapor Transmission Rate) value is also influenced by several factors such as the chemical properties of the material, polymer structure, and test conditions (McHugh and Krochta, 1994). Polnaya et al., (2006) stated that the decrease in the WVTR value in edible film was due to the increase in total solids in the film solution and the increase in the concentration of amylose molecules which form stronger hydrogen bonds, resulting in a compact structure. The compact structure can inhibit the diffusion of water vapor through the edible film (Breemer, et al., 2012).

Compressive Strength of Edible Film

Compressive strength is the ability of edible film to hold or protect the product it coats. Compressive strength is a mechanical property of edible film. The greater the compressive strength, the better the edible film is at resisting mechanical damage.

Based on the compressive strength results in Table 2, the addition of smaller starch to the composite edible film produces higher compressive strength values. The compressive strength value of edible film increased from 219.85 gF on native starch edible film to 356.87 gF on composite edible film which used 15% purple cassava starch which was smaller in size. This research is in line with Gonzalez et al., (2015) who made composite edible film with the addition of microcrystalline cellulose resulting in a compressive strength of 3.23 MPa in native starch edible film to 4.41 MPa in edible film with the addition of 10% coconut cellulose nanocrystals.

According to Hulleman et al., (1998) the strength of starch films is influenced by their botanical origin, especially the content of amylose and amylopectin. The compressive strength of a film is influenced by the interactions between amylose and amylopectin molecules, plasticizers, and the distribution of small starch in the film. Apart from that, a strong film is also influenced by the stronger intermolecular attractive forces on the surface of the film components, which include starch, glycerol and smaller starches. (Cerqueira et al., 2017).

Conclusion

Based on the results of the research that has been carried out, it can be concluded that the best treatment was obtained at a concentration of 0.8% cassava starch. The 0.8% starch concentration treatment produced the smallest starch particle size, namely 3.211 x 10.340 µm to 20.876 x 25.437 µm and produced a yield of 76.25%. Composite starch is better at improving the characteristics of edible films than native starch. Edible film from composite starch has different characteristics than edible film from native starch alone. Edible film from composite starch produces a higher thickness value of 0.161 ± 0.007, a lower solubility value of 32.080 ± 4.671%, a lower transparency value of 10.644% ± 0.357%/mm, a lower WVTR (Water Vapor Transmission Rate) value of 24 , 701 ± 7.05 g/m2. Hours and compressive strength values were higher 356.87 ± 7.38 gF than edible films from native starch alone.

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