IMPROVING EDIBLE FILM QUALITY USING MODIFIED WATER YAM (*DIOSCOREA ALATA L*) STARCH

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Submitted : 8 July 2021 Revised : 11 February 2022 Accepted : 20 July 2022

ABSTRACT

The existed edible films made of water yam starch tend to have a low quality which is indicated by high WVTR value and low mechanical strength. Addition of modified starch can decrease the WVTR value and improve the mechanical properties. The purpose of this study is to obtain the ratio of starch paste volume to ethanol volume which produces modified water yam starch and to improve the quality of the edible film by adding modified water yam starch. This research was conducted in 2 stages, which are the modification of water yam starch using the precipitation method and the making of edible films using several levels of modified water yam starch concentration. The first stage of the study was designed to produce modified starch using the treatment of the ratio of starch paste volume to ethanol volume. There are 5 treatment ratios used, which were 1:5, 1:7.5, 1:10, 1:12.5, and 1:15. The results showed that the different ratios of starch paste volume to ethanol volume resulted in different sizes of modified starches. The ethanol volume ratio of 1:5 resulted in a granular starch with the most damage in its morphology, and the smallest particle size detected was 1.135x1.767 μ, with the yield of modified starch was 80.5%. The addition of modified water yam starch of as much as 20%, which was modified using a ratio of paste volume to ethanol volume of 1:5, succeeded in improving the quality of the edible film of water yam starch by reducing the value of the water vapor transmission rate and increasing the compressive strength.

Keywords: Ethanol; Precipitation; Volume Ratio

INTRODUCTION

Water yam (*Dioscorea spp.)* is one of the most grown tubers in Indonesia. This plant is a type of shrub that vines. Water yam has brown to blackish skin and a white, creamy, or purplish flesh color (Hapsari, 2014). Water yam has not been widely cultivated in Indonesia and it is mostly only processed in the traditional ways, such as by steaming, frying, baking, or making it into chips.

Water yam contains carbohydrates (81.6-87.6%), water (75%), protein (6.7%), fat (0.2%) and crude fiber (9.37%) (Sakthidevi and Mohan, 2013). Generally, tubers of water yam have a high starch content with high amylose levels ranging from 24-26% (Nadia *et al.,* 2014). Based on these characteristics, water yam starch is suitable to be used as a raw material in making edible films.

Water yam starch – based edible film tends to have low quality such as fragile and tearable, with compressive strength values and water vapor transmission rates that do not meet JIS (Japanese Industrial

Standard) (Rugchati and Thanacharoenchanapas, 2015; Ulyarti *et al.,* 2019). To improve the physical, mechanical, and edible film barrier characteristics of water yam starch, this can be done by using native starches formulated with modified starches. Edible film of corn starch with the addition of modified corn starch in the form of donuts as much as 15% caused a decrease in the rate of water vapor transmission from 217.81 to 196.38 g/m^2.hour (Farrag *et al.,* 2018). Decreased water vapor transmission rate also occurred in edible films derived from pea starch with the addition of modified pea starch in the form of donuts (Farrag *et al*., 2018). Kaewpool (2010) reported that the addition of as much as 15% of nano-sized modified starch in film making using rice starch resulted in an increase in water vapor transmission and the mechanical strength of the resulting film.

Several modification methods produce modified starches with different morphologies, namely acidic, enzymatic hydrolysis, and mechanical treatment (Le corre *et al*., 2010). According to Ma *et al.* (2008), changes in starch morphology can be produced through mechanical treatment, one of which is through the precipitation method using organic solvents such as ethanol, butanol, and acetone. This precipitation method is carried out by the principle of heating when glatinizing starch and precipitating starch by slowly adding a solvent such as ethanol and stirring it rapidly using a magnetic stirrer. This process will result in the starch being rapidly retrograded and the formation of water-insoluble starch particles. In this study, the organic solvent used is ethanol beause it is polar, harmless, affordable, easy to obtain and can bind water well. Starch modifications using the precipitation method showed better results than modifications using other acids or other methods. The reason is that it does not use materials that are not safe to consume such as strong acids, also since this method is not complicated, even though the processing time takes slightly longer, it does not require sophisticated tools (Winarti *et al.,* 2011).

In the precipitation method, one of the factors that influence the starch morphology is the solvent ratio. Farraq *et al.* (2018) used the ratio of paste volume to ethanol volume of 1:2 to produce a donut-shaped modified starch that has the ability to improve the barrier and mechanical properties of edible films made of corn starch and pea starch. Qin *et al*. (2016) reported the modification of cassava starch by comparison of solvent ratio (1:10) at 100 °C for 30 minutes producing starch particles with a size of 30-110 nm, while Chin *et al.* (2011) used the comparison of starch paste and solvent (1:20) to produce modified sago starch with a starch size of 300-400 nm.

The purpose of this study is to obtain the ratio of starch paste volume to ethanol volume in the production of modified water yam starch which can be used to improve the quality of the edible film.

METHOD

The ingredients used in this study are water yam starch obtained from the extraction of freshwater yam tubers, glycerol (*Merck, Germany*), aquadest, absolute ethanol (*Merck, Germany*) and 96% technical ethanol (Sandi Mitra Kimia, Indonesia). Chemicals used for analysis included $Mg(NO3)_2$ (*Merck, Germany*), NaCl, and $CaCl_2$. The equipments used in this study were a scanning electron microscope (JEOL JSM 6510 LA), Fourier-Transformed Infrared Spectroscopy (*Shimadzu* prestige 21), and LFRA Texture Analyzer (Brookfield).

Methods

The research was conducted in 2 stages which are the starch modification stage and the edible film making stage. In the first step, a starch modification experiment was carried out using a Completely Randomized Design (CRD) which was repeated 4 times with variations of the volume ratio of starch paste to ethanol volume consisting of 5 levels of ratio, in which paste volume:ethanol volume = 1: 5; 1:7.5; 1:10; 1:12.5 and 1:15.

In the second stage, the application experiments of modified water yam starch for the manufacture of edible films were carried out. At this stage, a Completely Randomized Design (CRD) was used and repeated 4 times. The modified starch concentration treatment consisted of 5 treatment levels which were 0%, 5%, 10%, 15%, and 20%.

Starch Modification by Precipitation Method (Ulyarti *et al*., 2022)

Water yam starch was weighed at 1 g and then mixed with 100 ml of aquadest. The mixture was then heated on a hot plate at 90 °C for 30 minutes while being stirred continuously using a stir bar. Subsequently, the solution was immediately cooled and added ethanol gradually at a ratio of 1:5, 1:7.5, 1:10, 1:12.5, and 1:15 ml while continued to be stirred. Then, the solution was left at room temperature for 8 hours while still being stirred constantly. After that, the solution was centrifuged for 15 minutes at 2500 g, and the precipitate obtained was washed using absolute ethanol 3 times. The precipitate obtained was then dried using a heatless drying process (cold air dry), and accordingly modified starch was obtained.

Edible Film Preparation (Panjaitan *et al*., 2019)

A 4 g of water yam starch was dissolved in 143.55 g aquadest, stirred for 10 minutes, and heated on a hot plate for 30 minutes at gelatinization temperature (80 °C) with constant stirring using a magnetic stirrer. The solution was then added with 2% glycerol (3 g) at 10 min to the reactor. Subsequently, the solution was supplemented with modified starch according to the treatment and homogenized. A film solution of 25 g was then printed using petri dish with a diameter of 9.2 cm and a height of 1.7 cm and then dried using an oven at 50 °C for 24 hours. The dried edible film was then stored in a desiccator at 52% RH made using saturated $Mg(NO_3)_2$ before being analyzed.

Modified Starch Yield (Panjaitan *et al*., 2019)

Measurement of starch yield was calculated based on the comparison of the final weight obtained after modification to the weight of starch used before modification expressed in percent (%), using Equation 1.

$$\text{Yield (\%)} = \frac{Final\ weight}{Initial\ weight} \times 100\% \quad \text{...(1)}$$

Morphology and Size of Starch Granules (Panjaitan *et al*., 2019)

Image of native and modified water yam starch was taken using Scanning Electron Microscope (SEM). Previously, the starch was dispersed using alcohol. The sample was placed on an aluminum stab using double-sided tape and coated with gold powder to avoid charging under an electron beam. After the alcohol evaporated, the starch granules were observed at 250× and 1000× magnification. The size of the starch is determined by measuring the size of the SEM image in the photoshop.

Film Thickness (Ulyarti *et al*., 2020)

The film thickness was measured using a micrometer screw gauge at 5 different randomly selected places. The average of the five values obtained was determined as the film thickness.

Solubility (Ulyarti *et al*., 2020)

The filter paper that has been dried was weighed. The film sample was then cut to a size of 2×2 cm, put into 50 ml of water containing aquadest and immersed for 24 hours while being stirred periodically. The solution was then filtered, and the filter paper is dried for 24 hours at 105 °C. The amount of insoluble film was then weighed. Solubility percentage can be calculated using Equation 2.

$$\text{\% Solubility} = (\frac{(w1-w2)}{w1}\ x\ 100\%) \text{..................(2)}$$

Notes:
w1= Initial weight of edible film (g)
w2= Weight of insoluble edible film (g)

Transparency (Pineroz-Hernandeze *et al*., 2017)

The film was cut into squares (50×10 mm), placed in a spectrophotometer cell. Transmittance percentage was measured with a UV-Vis spectrophotometer at a wavelength of 600 nm. The transparency of the edible film was calculated using Equation 3.

$$\text{Transparency} = \log \%T/\text{Film} \text{.........(3)}$$

***Water Vapour Transmission Rate*/WVTR (Pineroz-Hernandez *et al.*, 2017)**

A reaction tube containing $CaCl_2$ was sealed using a film. The weight of the tube was then weighed. The tube was placed in a saturated desiccator using saturated NaCl (75% RH). The tube weight changes were then recorded and plotted as a function of time. The WVTR calculation following the Equation 4.

$$WVTR=\frac{Slope}{A} \quad (4)$$

Notes:
WVTR = Water Vapour Transmission Rate (g/m^2.hour)
Slope = Weight gain linear function/unit of time (g/hour)
A = Area of film area (m^2)

Pressure Strength

The compressive strength was measured with a Brookfield brand LFRA Texture Analyzer. The step to measure the edible film compressive strength test works is (ASTM, 1997) :

1. Determine the type of probe to be used for the edible film, which is TA 7 60 mm type, and the blade to be used in the compressive strength test of the edible film.
2. The LFRA Texture Analyzer tool is set to:
   Trigger : 2 g
   Distance : 2 mm
   Speed : 2 mm/s
3. The probe is installed in place and the "start" button is pressed to start pressing the edible film.
4. A sample of the edible film that has been cut with a size of 5×2 cm is placed under the probe and the probe will press the film until the amount of probe force used appears on the screen.

RESULTS AND DISCUSSION

Modified Starch Yield

The modified starch yield obtained in this study was quite large ranging from 76−80.5% (Table 1). The values obtained showed lower results than the modification of sago starch and tapioca starch using the same method as the yields, which ranged from 92.84-94.54% for sago starch and 85.38-87.17% for tapioca (Wulandari, 2013). The low yield produced can be caused by the differences in the temperature and the length of the gelatinization process. During gelatinization of starch, water enters the amorphous region of the starch granule and binds to amylose and amylopectin, but the bond with amylose is much greater than with amylopectin. This is because amylose is more hydrophilic. When the gelatinization temperature is reached, the starch granules will continue to absorb water and swell. Starch granules cannot withstand the exceeded amount of water; therefore, they will eventually break. When the granular starch breaks down, granular molecules in the form of amylose and amylopectin dissolve in water and possibly undergo further destruction from heating so that they cannot be deposited during the precipitation process.

Table 1. Modified water yam starch yield

Volume ratio	Yield (%)
1:5	80.5
1:7.5	78.0
1:10	79.0
1:12.5	76.0
1:15	77.0

Starch Granules Morphology

The morphology of the starch granules was obtained through the image obtained from analysis using SEM. The size of the starch granules produced in this starch modification was obtained by measuring the dimensions of the starch granules displayed in the SEM image using the application imageJ version 1.5.2. Figure 1 shows the difference in the granular form of native water yam starch and modified water yam starch using the precipitation method. The image of native starch shows that the starch granules are intact with an oval shape, this shows that the granules in native water yam starch have not suffered damage in the granular structure. In addition, native starch granules still have a smooth and intact surface, like the previously reported by Faridah (2011). At 1000× magnification, the water yam starch granules had a size of 17−33μm, similar to the previous research reported by Mali *et al* (2004), in which the starch granules had a size of 12−37μm.

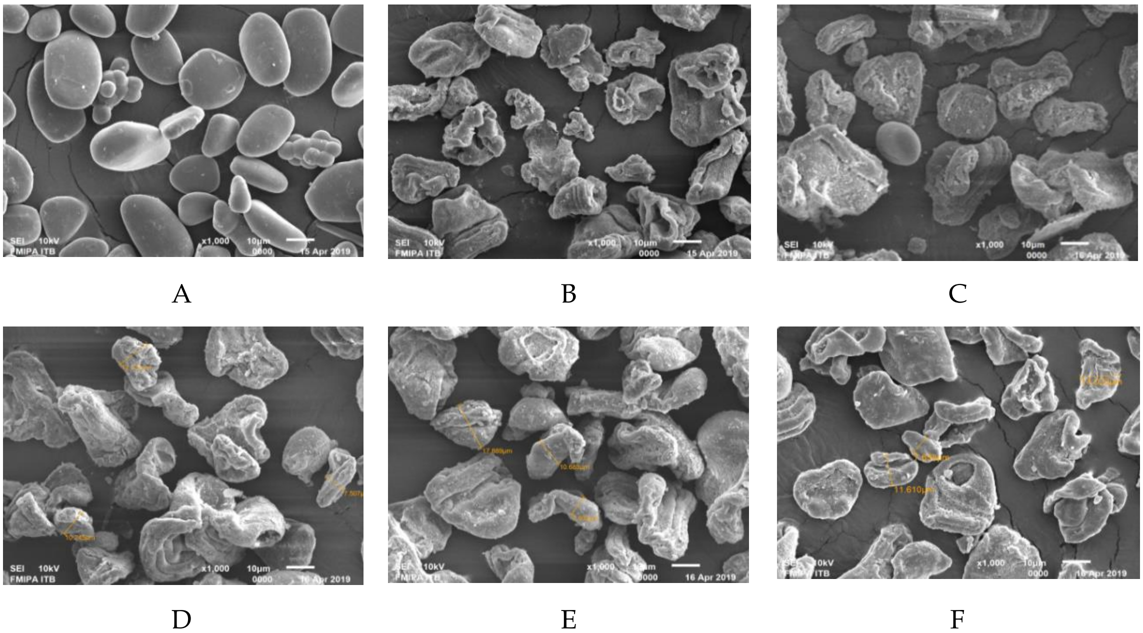

Figure 1. Image Scanning Electron Microscope. (A) Native water yam starch; (B) Modified starch with a ratio of 1:5; (C) Modified starch with a ratio of 1:7.5; (D) Modified starch with a ratio of 1:10; (E) Modified starch with a ratio of 1:12.5; (F) Modified starch with a ratio of 1:15.

The principle of the precipitation method is to provide a heating treatment to the starch gelatinization process and the gradual addition of ethanol while stirring rapidly with a magnetic stirrer. This process results in starch being rapidly retrograded, also the forming of starch particles that have water-insoluble properties (Qin *et al.,* 2016). The gelatinization process will cause the starch granules to swell, therefore the longer heating process is carried out, the more starch granules will break and dissolve the starch molecules. The addition of ethanol and mechanical treatment in form of stirring can reduce the particle size of the deposited starch.

In Figure 1, it can be seen that for a 1:5 to 1:15 solvent ratio treatment, the gelatinization process has damaged the granular structure resulting in a morphology that is different from its native starch. Minimization of granular size can be observed only on the polygonal side, and it results in granules with eroded and folded surfaces. Although generally the size of the modified starch has no difference, in the volume ratio treatment of 1:5 there was found particles with the smallest size of 1.135×1.767 µm. As the paste volume to ethanol volume ratio gradually increases, the process of rapid stirring also lasts longer, therefore it can damage the small particles. It is suspected that these small particles degrade further and dissolve in water so that they cannot be deposited. Consequently, the more ethanol used, the smaller the starch particles that is produced.

Modified Starch Characteristics for Edible Film

Modified water yam starches that was treated using a 1:5 ratio of paste volume to ethanol volume were selected for the manufacture of edible films because they have the most different morphology from their native starches. As seen in SEM image, most of the starch has large sizes with different morphology, which is a rough surface with some portions on the granules eroded (Figure 2). In contrast to starch modification using the precipitation method which generally has an effect on granular size shrinkage (Qin *et al.,* 2016; Saari *et al.,* 2016; Chang *et al.,* 2018; Fan and Picchioni, 2020; Luo *et al.,* 2022; Shokri *et al.*, 2022), in this study, the modification technique using the precipitation method displayed a granular change effect that is similar as reported by Farrag *et al.* (2018), which is the main change in granular morphology.

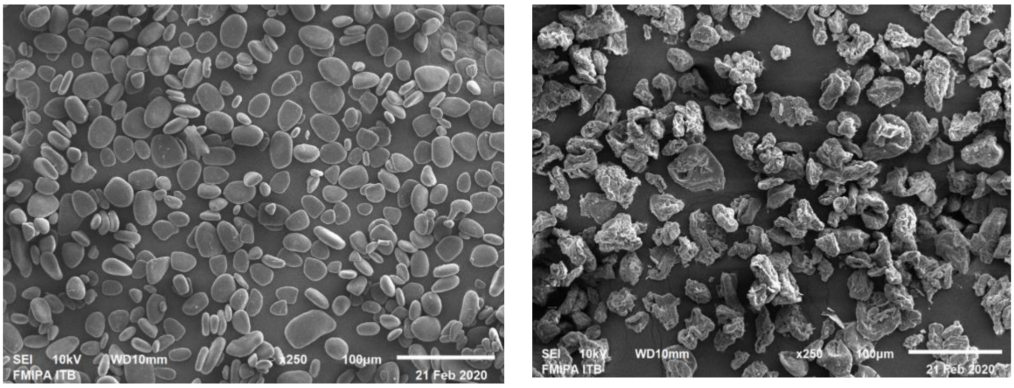

Figure 2. SEM Image Native Water Yam Starch (left) and Modified Water Yam Starch (right)

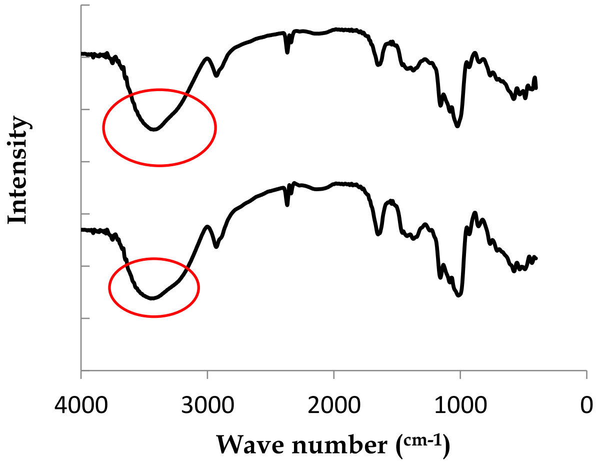

Figure 3. FTIR Spectra of Native Water Yam Starch (lower) and Modified Water Yam Starch (upper)

The starch modification process produced an absorption intensity at a wavelength of 3600–3400 cm^{-1} (OH-stretching) higher than that of native starch as seen in the FTIR spectra (Figure 3). This means that there were more OH groups in the modified starch. In modified starch, the highest absorption intensity of OH stretching occurred at lower wavelengths (3425 cm^{-1}) than native starch (3448 cm^{-1}). This shift indicates a stronger interaction occurred in the OH group of modified starch or in other words a reorganization of starch in which the amorphous region is reduced so that the starch becomes more crystalline (Qin *et al.*, 2016).

Edible Film Characteristics

Thickness is one of the important parameters in edible film (Totosaus *et al.*, 2020; Das *et al.*, 2022). Aside of affecting its application on the product, it can also affect other parameters such as transparency, Water Vapor Transmission Rate (WVTR), solubility and compressive strength (Ulyarti *et al.*, 2019). The edible thickness test is obtained from the average measurement of five different test points, including the corner and the middle of the edible film. This film thickness was obtained from measurements using a micrometer screw.

Table 2. Characteristics of water yam starch based edible film with the addition of several levels of modified water yam starch concentration

Characteristics Edible film	**Modified Water Yam Starch Concentration 0%**	**5%**	**10%**	**15%**	**20%**
Thickness	0.11±0.00	0.11±0.00	0.12±0.00	0.12±0.00	0.12±0.01
Solubility	54.29±2.87	47.50±6.69	42.85±7.38	38.19±4.61	29.86±16.26
Transparency	21.58±0.41	19.63±1.38	18.88±0.49	18.44±0.46	18.03±1.47
WVTR	36.32±1.36	15.79±3.10	15.26±0.61	15.00±1.01	10.79±0.53
Pressure Strength	631.63±3.66	693.83±1.21	743.60±0.78	802.08±3.45	884.20±2.77

The ANOVA results showed that in the interval of modified water yam starch concentrations used, the concentration had no effect on film thickness (Table 2). However, from the linear regression it is indicated that there is a linear correlation between the concentration of modified water yam starch and the thickness of edible film (Figure 4). Increased concentrations of modified water yam starch tended to increase the thickness of the edible film (Figure 4). Ulyarti *et al.* (2019) reported the thickness of edible film using water yam starch at a concentration of 2% at 0.117 mm, similar to the thickness of edible film from this current study with the addition of modified water yam starch up to 20%. This value is in accordance with established standards from the Japanese Industrial Standard (JIS) that edible films can be categorized as packaging materials if they have a maximum thickness of 0.25 mm.

Solubility in water is a physical property of edible film which shows the percentage weight of edible film dissolved after being immersed in water for 24 hours. Edible films that have high solubility are suitable for use in ready-to-eat food products, because of their ease to dissolve when consumed. Although the ANOVA results showed that the solubility of the edible film was not affected by the concentration of modified water yam starch (Table 2), there was a linear correlation between the concentration of modified water yam starch and the solubility of the edible film (Figure 4). The higher the concentration of modified water yam starch tends to decrease the solubility of the edible film (Figure 4). The hydrophobic nature of modified starch results in a decrease in the solubility of the edible film (Murdianto *et al.,* 2005). Modifying the precipitation method of starch damages, the amorphous areas contained in the starch granules and leaves the granules with a crystalline structure. The amorphous region of the modified starch is an area that has more hydrophilic properties than the crystalline region so that the reduced amorphous region of the modified starch decreases the ability of the modified starch to bind water. Edible film which has a low solubility value is one of the important requirements of edible film as food packaging that comes into contact with water and acts as a food product protector.

Transparency describes the amount of light that can pass through the edible film. The transparency test of the edible film is determined from the amount of light passed on by the edible film (transmittance). In Table 2, it can be seen that the percentage of transparency value decreases along with the increasing concentration of modified starch. Although this decrease was not statistically significant, there was a negative correlation between the concentration of modified water yam starch and the transparency of the edible film (Figure 4). Light passing through the film cannot penetrate gaps or voids that were previously contained in the edible film, because the gaps have been filled by modified starch to reduce the transparency value in the edible film (Shi *et al.,* 2013; Dai *et al*., 2019; Choi *et al*., 2022).

Water Vapor Transmission Rate (WVTR) is a very important parameter in assessing the quality of edible film. A good edible film is a film with a low water vapor transmission value (Amaliya and Putri, 2014).

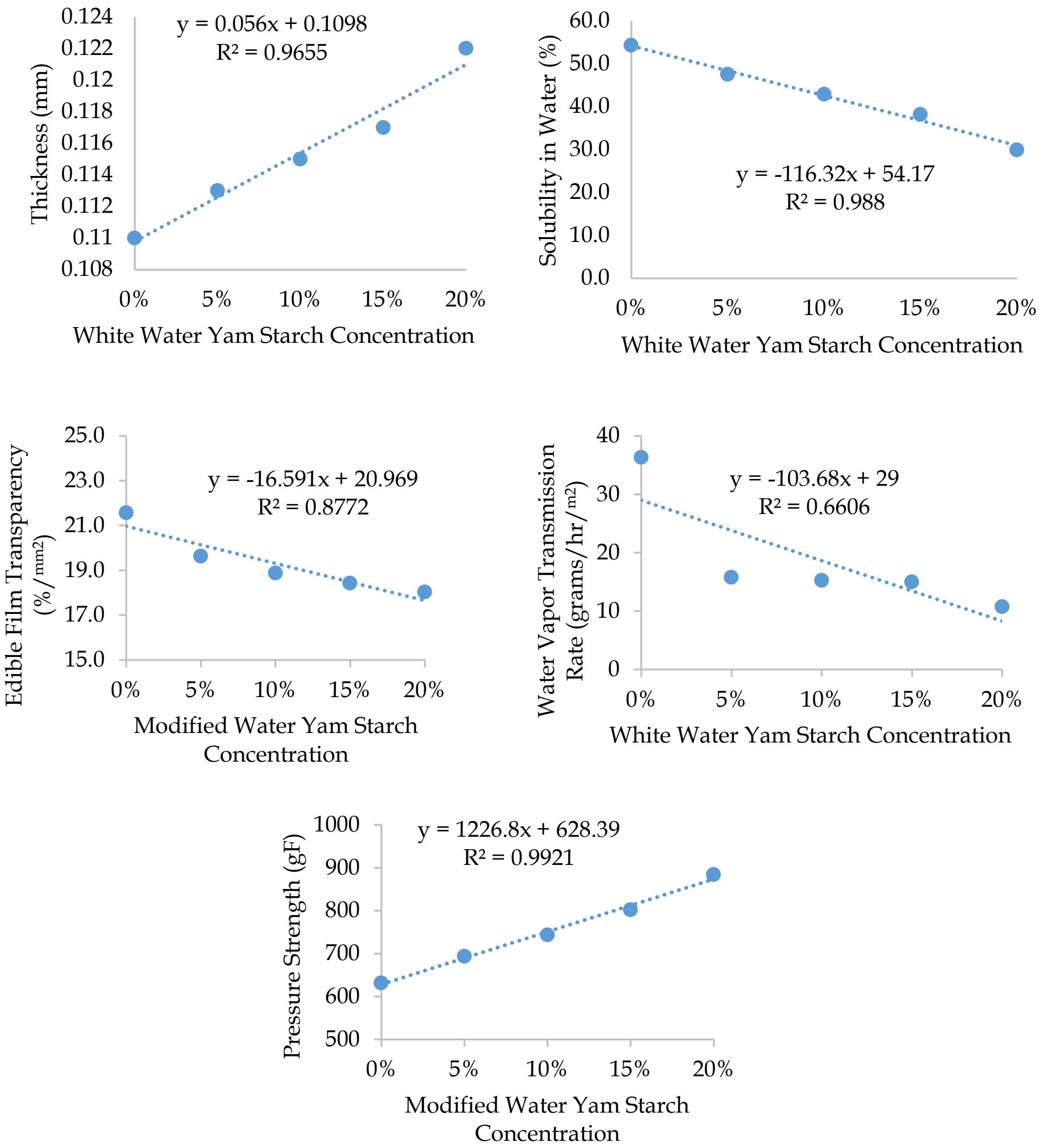

Figure 4. Correlation Between Modified Water Yam Starch Concentration and Edible Film Characteristics

WVTR shows the velocity at which water vapor passes through the film (gram per hour per area of the edible film). The WVTR value will indicate the ability of the film to inhibit water vapor. In Figure 4, it can be seen that the higher the concentration of modified starch in the edible film, the lower the WVTR value, although statistically through the ANOVA results the concentration of modified water yam starch does not affect the edible film WVTR. The use of this modified starch results in an increasingly tight film structure which results in the edible surface of the film being difficult to penetrate by water vapor. The results of this study are in line with Farrag *et al*. (2018) who made edible films with the addition of micro-particle corn starch as much as 15% producing edible films with WVTR values decreased from 217.81 g/m^2.hour to 196.38 g/m^2.hour. WVTR is also influenced by the thickness of the film tested where the thicker the film, the lower the WVTR value (Moulia, 2018). As seen in Figure 4, an increase in the amount of modified starch increases the film thickness and simultaneously decreases the WVTR.

Compressive strength is one of the important mechanical properties of edible film, because it is related to the ability of edible film to protect the product it is coating. Edible film with high compressive strength value is suitable for use in food packaging because it is more able to protect food ingredients during handling, food product transportation to marketing. In Table 2 it can be seen that the increase in the concentration of modified water yam starch increases the compressive strength of the edible film even though this influence is not significant according to ANOVA. Based on the value of the regression coefficient, there is a positive correlation between the concentration of modified water yam starch and the compressive strength of the edible film (Figure 4). This result is in line with Gonzalez *et al*., (2014) where the addition of 10% microcrystalline cellulose produced an edible film with an increase in compressive strength from 3.23 MPa to 4.41 MPa. The increase in compressive strength of the edible film is due to the interaction between the modified starch and the native starch matrix.

CONCLUSIONS

The morphology and granular size of starch can be modified using the precipitation method where the lower the starch volume to ethanol volume ratio during the modification process resulted in increasingly damaged granules and smaller starch particle size. In this study, the process conditions that resulted in the smallest size of modified starch were the ratio of starch volume to. non-reagent solvent volume of 1:5. Although statistically insignificant, the concentration of modified water yam starch tended to improve the characteristics of the edible film in form of a decrease in WVTR and an increase in the compressive strength of the edible film. This improvement is thought to be significant if the concentration of modified starch were increased.

ACKNOWLEDGMENT

We would like to express our gratitude to the Dean of the Faculty of Agricultural Technology of Jambi University who has provided research funding support through the Non-Tax Revenue scheme of the Faculty of Agricultural Technology in 2020.

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