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Physic-mechanical properties of edible film based on taro starch (*Colocasia esculenta* L. Schoott) with glycerol addition

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**Abstract.** Food packaging has substantial roles, such as product protection from microbial contamination, chemical pollution and physical harm. While environmental pollution caused by plastic packaging become concerning, hence there are innovation to produce films that are convenient to decompose. Edible film is a thin layer that serves as food protector, and barriers to the transfer of mass, such as water vapor, and oxygen. Taro tuber is one of the potential raw materials, which has high starch content. While the most edible films that made from starch have deficiencies alike high fragility, therefore plasticizers need to be added. Glycerol addition is more efficient in improving physical and mechanical properties such as tensile strength and elongation, because it improve flexibility, strength to against physical damage, and better transparency. This study aimed to determine the concentrations of taro starch and glycerol effect on edible film physics-mechanical properties. Randomized complete block design factorial (RCBD) with two factors and 3 replications were applied to analyze the data. The first factor was taro starch concentration (2, 2.5 and 3%) and the second factor was glycerol concentration (25, 30, and 35%). The results indicated that there were interaction between taro starch and glycerol concentration to the parameters of tensile strength, elasticity, solubility, WVTR and transparency. Moreover, the best combination treatment was 3% taro starch and 25% glycerol, with characteristics of thickness (0.15 mm), tensile strength (10.63 MPa), elasticity (60.21%), solubility (23.48%), WVTR (25.37 g/m²/24 hours) and transparency (2.23 mm⁻¹).

**1. Introduction**

Plastics food packaging causes’ environmental restlessness, it has several properties as difficult to be degraded naturally (non-biodegradable), so that it can produce midden, and environment ravage [1–4]. Edible films can be solution of this matter, it made from hydrocolloids such as polysaccharides (gum, starch, and pectin), fat and protein. The protein based edible film has higher barrier properties than lipids and polysaccharides based on Bourtoom [5]. But it has poor stability to water vapor and low mechanical strength for food packaging.

Starch is one of hydrocolloids from polysaccharides whose physical characteristics resemble to plastic. Starch considered as one of the most promising natural renewable resources due to relatively low cost, its large availability, and biodegradability. Starch consisted to amylopectin and amylose. Most starches contain between 15% and 35% amylose [6]. Starch characteristics are insoluble in water, and in the form of white, fresh and odorless powder. Starch is the main ingredient produced by plants to save the excess glucose (as a photosynthetic product) in the long term. Starch is the main energy source for adults in the entire world population, especially in developing countries because it is consumed as a staple food [6]. Amylose has the ability to form crystals because of its simple polymer
chain structure. This simple structure can form strong molecular interactions. This interaction occurs in the hydroxyl group of amylose molecules. The formation of hydrogen bonds is easier to occur in amylose than amylopectin. Basically, the structure of amylopectin is same as amylose, which consists of a short chain of α- (1.4) -D-glucose in large quantities [7, 8].

Starch has a fairly large hydroxyl group content and it is hydrophilic. Normally, starch granules do not dissolve in cold water due to the fact that hydrogen bonds maintain starch chains with each other but when starch is heated in water, the crystalline structure will be damaged and water molecules will interact with hydrogen bonds. This hydrophilic characteristic causes starch to have low stability in water, thus limiting the starch-based films development. Secondly because the amorphous region of structure formed by amylose and amylopectin branching, starch has low mechanical properties, such as low tensile strength, low elongation. Several approaches have been made to improve starch-based products for industrial use.

Improving the granular structure of starch can used physical modification, so it will enhance its solubility in cold water. This modification does not involve the chemical, and the process is easier. There are several processes of physical modifications: annealing, heat moisture treatment, freezing, retrodegradation, high pressure treatment and gelatinization. However, this modification type is not able to provide chemical changes of starch, so it can not be applied to film production. In addition, chemical modification involving the etherification, esterification, oxidation, hydrolysis, and crosslinking processes are methods commonly used to increase film-forming capacity by starch. Chemical modification facilitates intra-molecular and intermolecular bonds in a random position in the structure.

Taro becomes potential raw material, because it has high starch content. The previous finding showed that starch content of taro was 70- 80% [9, 10]. It has been found that the carbohydrate content of taro cultivated in different locations varied [11]. In general, starch is not able dissolved in cold water due to crystal structure of starch. Through the temperature enhancement, starch molecule started to disintegrate in water. The starch solubility was increased, in case amylose and amylopectin dissociated in suspension. The hydration contributed to the insoluble starch swelling [12]. In spite, taro starch forms rigid coating layer, but its solution has high clarity at high solid concentration. It has high gel strength, peak viscosity, and swelling power [13]. It also has small granule size (0.5 - 5 μm), smooth textural gel, and easily digestible [14]. Taro starch also was analyzed as fat substitute and the biodegradable polyethylene film-filling agent [11, 15]. According to Htwe [12], taro starch characteristics included: pH of taro starch about 7.81 (slightly alkaline), moisture content (13.78%), protein content (5.61%), and hydration capacity (1.70) which means, it was capable to absorb 1.7 times of its own weight of water. While true density (1.81), and moisture sorption capacity (15%) showed material moisture sensitivity. Taro starch was less soluble under the gelatinization temperature. The taro starch solubility was less than 10% at 50ºC to 70ºC, but it increased up to 29.2% at 80ºC. The loss of granular structure, release of amylose fraction, and it preferentially solubilized also leached from swollen.

Fragility is the weakness of edible film made from starch, so plasticizers addition become crucial and able to enhance the elasticity. Starch based films were found have good carbon dioxide and oxygen barrier properties which relevant for effective and biodegradable packaging. On the other hand, it had drawbacks in mechanical and water vapor permeability properties. In addition, native starches are brittle and not processable. Therefore, plasticizers are generally added to native starch to overcome brittleness as well as improve flexibility, workability [16, 17], and it able to reduce inter-molecular forces along polymer chain [5]. Glycerol is one of plasticizers, which commonly act as fat compiler, and has great hydrophilic, and hygroscopic properties. Edible film produced from starch a glycerol addition provide higher solubility [18-22], glycerol is appropriately added because it is hydrophilic like starch, and water-soluble plasticizer [23-26]. Glycerol addition is more efficient in improving edible film physic-chemical properties especially in tensile strength, elongation, and transparency compared to glucose syrup. Therefore, this study aimed to determine the concentrations of taro starch and glycerol effect on edible film physics-mechanical properties.
2. Materials and Methods

The main material, taro (*Colocasia esculenta* L. Schoot.) were obtained from Kendalpayak Village, Pakisaji District, Malang Regency. It was harvested in terms of 9 months when the tubbers already brownish, and diameter ± 20 cm. Glycerol, STPP (Sodium Tri Poly Phosphate), technical NaCl, and silica gel were also prepared. The main equipment used 80 mesh sieves, cabinet dryers, micropipettes, magnetic stirrers, hotplate stirrers, micrometers, texture analyzer, texture gauges, and spectrophotometer UV-Vis.

Randomized Complete Block Design factorial (RCBD) with 2 factors (starch and glycerol concentration) were applied. The first factor was starch concentration *P* (2%, 2.5%, 3% b/v), and the second factors included glycerol concentration *G* (25%, 30%, 35% v/b starch). Therefore, 9 combination treatments were obtained which repeated 3 times. The samples (edible films) were determined for thickness, tensile strength, elasticity, solubility, transparency, and water vapor transmission rate (WVTR).

2.1 Analysis of Starch Content

71.9 g of anhydrous *Na₂CO₃* dissolved in 300 mL heated distilled water. After dissolving, 25 g citric acid was added to the 25 mL distilled water gradually. Then 8 g *CuSO₄*·5*H₂O* mixed in 100 mL of distilled water gradually, followed with decreasing the temperature and left for 30 minutes, and treated to 500 mL and left for one night in a dark place.

One gram flour sample dissolved in 40 mL 3% HCl, and refluxed for 3 hours in 200 - 250 °C. Then sample was cooled and neutralized by adding a few drops of 3% NaOH through assisted by PP indicator until it was pink. 3% HCl was used for acidified process, which was about 6.5 mL filtrate was piped into a sharpened erlenmeyer, then it was added 25 mL Luff Schrool solution, 20 mL distilled water and re-refluxed for 10 minutes (calculated when boiling started), then chilled in the ice box for a few minutes. The 25 mL of 25% H₂SO₄ and 15 mL of 20% KI solution were added on cold sample, then immediately titrated with standardized Na₂S₂O₃ 0.1 N. The addition of 0.5% starch indicator was carried out during titration and stopped when the solution changed from purple to cloudy white. Blank determination was made by mixing 25 mL of Luff Schrool solution and 25 mL of distilled water (without samples).

\[
\text{Starch} = \frac{G \times Fp \times 0.9}{W} \times 100\%
\]

Remark: *G* = glucose (mg) (Vol Na₂S₂O₃ Blanko - Vol Na₂S₂O₃ sample); *Fp* = dilution factor; *W* = sample weight (mg)

2.2 Thickness of Edible film

The film thickness was measured by a micrometer instrument with a precision screw 0.001 mm. The micrometer was placed on a compact and clean table, set to zero then the pressing feet was lowered on the sample. Pressing feet was lifted slightly then removed from the first measurement location. Measurements were repeated in five different places, and edible film thickness value average of the five measurement sites.

2.3 Solubility in Water

The samples were cut (size 1 cm x 1 cm), weighed as the initial weight (W0), and put into petri dish containing 15 ml of distilled water for 10 minutes. The samples which had been soaked, removed and the water on the plastic surface was cleaned with paper tissue. Then sample weighed as final sample weight (W).
2.4 Transparency
Edible films were cut (1 cm x 4 cm), measured the thickness and recorded. The samples were put in glass cuvettes, measured using a spectrophotometer at wavelength (λ) 546 nm. Then the data of transparency (T) edible film was calculated.

\[ T = \frac{\text{Absorbance at } 546 \text{ nm}}{X} \]

2.5 Edible Film Mechanical Test
Edible films were cut (20 mm x 50 mm) and clamped 30 mm on both sides. Tensile strength and elongation of edible films were tested by Universal Testing Machine. Tensile strength was the maximum pull that could be achieved until edible films remain before breaking or tearing. Tensile strength values were recorded and calculated following equation:

\[ \text{Tensile Strength} (\tau) = \frac{\text{Maximum Stress} (F_{\text{max}})}{\text{Area} (A)} \]

Elongation is the ability of the edible range of film produced. Elongation was calculated by the formula:

\[ \text{Elongation} (\%) = \frac{d_{\text{after}} - d_{\text{before}} (\text{mm})}{d_{\text{before}} (\text{mm})} \times 100 \]

2.6 Water Vapor Transmission Rate (WVTR)
The film tested was mounted on a cup containing 2 g of silica gel. The edge of the cup and the film were covered with wax or insulation. The cup and film were weighed, put in a plastic jar containing 100 mL of 40% NaCl solution, then tightly closed. Each hour the cup was weighed and observed for 24 hours. The data obtained were made linear regression equations, so that slope enhancement in the cup weight (g day\(^{-1}\)) was divided by the film area (m\(^2\)).

3. Results and Discussion
3.1. Basic Properties of Raw Material (Taro)
Taro were comprised starch (67.2%) and contained 32.15% amylose and 35.05% amyllopectin. Amylose structure will establish strong hydrogen bonds among the constituents of glucose molecules, and in heating process will form three dimensional tissues which produce strong gel, because it can trap water. One of starch constituent molecules is amylose, it is responsible in matrixes formation of edible films, as the result it can improve film texture become more tightly and compact. Amylose content of taro starch was greater than cassava which was 8.92%.

3.2. Thickness
Analysis of Variance indicated that there was not interaction, but taro starch and glycerol concentration separately gave significant effect to edible film thickness. The result defined that edible film thickness proportionately increased with taro starch and glycerol formulation (Table 1). The high viscosity of film compiler, making total solids (density) increase, which influences edible film thickness enhancement and become thicker [27]. Dissolved solids concentrations in film-forming solutions also able to influence edible film thickness. Based on JIS [28], edible film requirements become good packaging whenever, it has thickness value <0.25 mm.

3.3. Tensile Strength, Elasticity and Solubility
The interaction between taro starch and glycerol inflicted significant effect on edible film tensile strength. There was positive correlation between tensile strength and taro starch concentration (Table
2). Tensile strength values were ranging from 3.80-10.63 Mpa, which was higher than standard of JIS [28] stating that minimum tensile strength should be 3.92 MPa. More concentration of starch given can make stronger matrix, and as the results, edible film tensile strength become greater. This result was in line with Rodriguez et al. [23] reporting that higher glycerol addition would increase elongation and decrease tensile strength value. Increasing amylose content would enhance the suspension retrogradation properties of edible film after heated, then tensile strength value become greater. Edible film stability and compactness were due to the large number of polysaccharides. Amylose and amylpectin levels in starch would affect to polymeric bonds formation, and it could increase edible film produced and decrease glycerol requirements.

Table 1. Thickness of edible film based on taro starch and glycerol concentration

| Treatment | Thickness (mm) |
|-----------|----------------|
| **Taro starch concentration (%)** | |
| 2         | 0.10 a         |
| 2.5       | 0.13 b         |
| 3         | 0.17 c         |
| **Glycerol concentration (%)** | |
| 25        | 0.12 a         |
| 30        | 0.13 b         |
| 35        | 0.15 c         |

Numbers followed by the same letter in each column shows insignificant differences according to Duncan's test α 5%.

The data identified that increasing elasticity would make edible film become more easily elongated due to great flexibility value (Table 2). There was interaction between taro starch and glycerol concentration significantly on edible film elasticity. Increasing concentration of taro starch and glycerol causes matrix formed and edible film shape will be stronger. Hydroxyl group along glycerol chain is the main factor of hydrogen bonds formation between starch polymers and plasticizers, which replace hydrogen bonds among starch polymers during film biopolymers formation [18]. Glycerol is an effective plasticizer because it has ability to reduce intermolecular bonds and increase flexibility [29]. Elasticity value (53.57-64.56%) categorized very well based on JIS [28]. The standard states that elasticity categorized bad (<10%), good (10-50%), and it is very well whenever the percentage >50%.

Based on Analysis of Variance (ANOVA), there were interaction of taro starch and glycerol significantly on edible film solubility (Table 2). Biodegradability of film is an important property, which determined by solubility. According to Mehyar and Han [30] hydrophilic components has positive correlation with solubility percentage, in other word increasing starch formulations (ratio of glycerol: starch) in edible films will reduce solubility percentage. Starcch formulation declining will increase hydrocolloid and the number of hydroxyl groups which enhancing in water level become easier and faster to improve solubility.

3.4. Water Vapor Transmission Rate (WVTR) and Transparency of Edible Film

Water vapor transmission rate value exceed packaging films standard (21.73 - 31.54 g m⁻² 24 hours⁻¹). Taro starch and glycerol formulation had significant effect to edible film WVTR (water vapor transmission rate) (Table 3). Enhancement of WVTR value along to glycerol concentration indicated that moisture component that could pass through edible film was greater, but the ability to resist water evaporation become lower. This was presumably caused by glycerol properties, which is hydrophilic, able to reduce film matrix voltage, matrix molecule widened, and penetrate to edible film cavity by moisture. Hydrophilic characteristics defines that glycerol is easily soluble in water, which improve
polarity. Thus, glycerol formulation enhancement would increase water vapor permeability. While hydrophilic/hydrophobic properties and plasticizer (type, degree and suitability) influenced inhibitory ability [23, 31, 32].

Edible films clarity or brightness defined by transparency, it become cloudy whenever the value was high. Taro starch and glycerol concentration interaction significantly contributed on edible film transparency (Table 3). Transparency influenced by thickness and constituent components of edible film, and it had positive correlation. It was related to component quantity, which absorbed by waves fired of spectrophotometer to film sheets in cuvette. Transparency value tend to increase through starch and glycerol concentration enhancement, because the absorbance reader absorbed more colors. Despite edible films based on starch and glycerol commonly would be clear in color, but the result showed pink color, and it probably influenced by raw material concentration.

### Table 2. Tensile strength, elasticity and solubility of edible film.

| Treatments                  | Tensile Strength (MPa) | Elasticity (%) | Solubility (%) |
|-----------------------------|------------------------|----------------|----------------|
| P1G1 (Starch 2% + Glycerol 25%) | 5.58 a                  | 53.57 a        | 60.40 c        |
| P1G2 (Starch 2% + Glycerol 30%) | 4.06 a                  | 54.44 b        | 66.23 c        |
| P1G3 (Starch 3% + Glycerol 35%) | 3.80 a                  | 56.40 c        | 67.06 c        |
| P2G1 (Starch 2.5% + Glycerol 25%) | 6.52 a                  | 57.27 d        | 31.36 ab       |
| P2G2 (Starch 2.5% + Glycerol 30%) | 6.20 a                  | 58.59 e        | 36.34 b        |
| P2G3 (Starch 2.5% + Glycerol 35%) | 5.65 a                  | 59.64 f        | 41.69 b        |
| P3G1 (Starch 3% + Glycerol 25%) | 10.63 b                 | 60.21 f        | 23.48 b        |
| P3G2 (Starch 3% + Glycerol 30%) | 9.43 b                  | 61.52 g        | 26.25 a        |
| P3G3 (Starch 3% + Glycerol 35%) | 6.76 a                  | 64.56 h        | 30.94 ab       |

Numbers followed by the same letter in each column shows insignificant differences according to Duncan's test α 5%.

### Table 3. WVTR and transparency of edible film

| Treatments                  | WVTR (g m⁻² 24 h⁻¹) | Transparency (mm⁻¹) |
|-----------------------------|----------------------|---------------------|
| P1G1 (Starch 2% + Glycerol 25%) | 21.73 a              | 1.61 a              |
| P1G2 (Starch 2% + Glycerol 30%) | 23.46 b              | 1.74 b              |
| P1G3 (Starch 3% + Glycerol 35%) | 26.19 e              | 1.94 c              |
| P2G1 (Starch 2.5% + Glycerol 25%) | 24.35 c              | 2.07 d              |
| P2G2 (Starch 2.5% + Glycerol 30%) | 27.32 e              | 2.10 e              |
| P2G3 (Starch 2.5% + Glycerol 35%) | 30.40 g              | 2.13 f              |
| P3G1 (Starch 3% + Glycerol 25%) | 25.37 d              | 2.23 f              |
| P3G2 (Starch 3% + Glycerol 30%) | 28.57 f              | 2.34 g              |
| P3G3 (Starch 3% + Glycerol 35%) | 31.54 h              | 2.45 h              |

Numbers followed by the same letter in each column shows insignificant differences according to Duncan's test α 5%.

### 4. Conclusion

Taro starch and glycerol concentration had significantly interaction effect to the water vapor transmission rate (WVTR), tensile strength, elasticity, solubility, and transparency of edible film. The increasing taro starch concentration was able to enhance thickness, tensile strength, elasticity, WVTR, and transparency. Otherwise the best treatment was 2.5 % taro starch and 25 % glycerol with edible film properties, thickness (0.17 mm), tensile strength (5.65 MPa), elasticity (57.27 %), solubility (31.36 %), WVTR (24.35 g m⁻² 24 hours⁻¹), transparency (2.07 mm⁻¹).
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