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Impact of glycerol concentration on the physicochemical properties of phosphorylated Banggai yam starch edible films

[1,*]Chaniago, R., [2]Hasanuddin, A., [2]Rahim, A. and [1]Lamusu, D.

[1]*Faculty of Agriculture and Fisheries, Muhammadiyah University Luwuk 94711, Indonesia*
[2]*Faculty of Agriculture, Tadulako University Palu 94226, Indonesia*

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Abstract

Edible films have gained significant attention as an environmentally friendly alternative to traditional plastic packaging. Their applications cover many areas, especially food packaging, which offers advantages such as biodegradability and incorporation of active ingredients. The physicochemical characteristics of edible films are essential because they directly affect the function of the material and consumer appeal. This study evaluated the physicochemical attributes of edible phosphorylated Banggai yam films with different glycerol concentrations. Edible films were made with glycerol concentrations (10, 30, and 50% (w/v)) in phosphorylated Banggai yam starch (PBYS) edible films. The observation variables in this study were colour, viscosity, water solubility, and pH. Data obtained from various measurements were analyzed to determine the relationship between glycerol concentration and the physicochemical properties of edible films. The results showed that the resulting edible film has a relatively low brightness with a tendency towards red and blue. Varying thickness allows adjustment according to needs; there is an increase in solubility, and a pH that tends to be neutral or slightly acidic. The results of the study showed that the edible film produced had a relatively low brightness level with a tendency towards red and blue colours, varying thickness (0.10-0.65 mm), increased solubility (10.39-39.26%), and a pH that tended to be neutral or slightly acidic (5.5-7.9). Overall, this edible film exhibits properties that can be utilized in certain applications, although it is worth noting the variation in thickness and low brightness levels.

1. Introduction

In the food business, edible films have drawn much interest as an eco-friendly packaging option (Chawla *et al*., 2021; Petkoska *et al*., 2021). Using natural materials as the foundation for edible films is driven by the growing demand for sustainable products worldwide (Ali and Ahmed, 2018). Phosphorylated Banggai Yam Starch is one of the possible ingredients; it has unique physicochemical properties and can raise the calibre of edible films.

Research is now concentrating on the physicochemical properties of edible films made from phosphorylated Banggai yam starch, specifically about colour, thickness, water solubility, and pH (Nadia *et al*., 2014; Rahim *et al*., 2023). The physicochemical properties of the films, such as their viscosity and colour stability, can be enhanced by adding plasticizers such as sorbitol and glycerol (Sofia *et al*., 2017; Afriyanti *et al*., 2021). Furthermore, the film's mechanical and functional qualities may benefit from adding additives such as chitosan and essential oils (Putri *et al*., 2022; Wijaya, 2022).

The efficacy of edible films in various food applications is significantly influenced by colour, thickness, water solubility, and pH variables (Ferdiansyah *et al*., 2021; Lestari, 2022). While thickness influences permeability and mechanical qualities, colour stability influences aesthetic appeal (Siregar and Irma, 2012). The bioavailability of active chemicals is determined by their water solubility, while stability and resistance to microbes are influenced by pH (Wati *et al.,* 2023). To provide the best possible formulation for use in the food business, this study intends to investigate how glycerol concentration affects the physicochemical properties of edible films made from phosphorylated Banggai yam starch (PBYS).

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*Corresponding author.
Email: *idhon86chaniago@gmail.com*

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2. Materials and methods

2.1 Materials

The materials used in making PBYS include Banggai yam starch, distilled water, 3% NaOH, Sodium Trimetaphosphate (STMP), Sodium Tripolyphosphate (STPP), HCl, and 95% ethanol. Meanwhile, the materials used in making PBYS edible films include PBYS and glycerol. Edible film was made using the method described by Rahim *et al*. (2011) with slight modifications. The PBYS edible film was made in 2 stages. In the first stage, starch was modified using STMP, STPP and STMP: STPP 99:1 reagent with three concentration levels, namely 2, 6, and 10% (w/v) for each phosphorylation reagent. In the second stage, the edible film was made with the highest degree of substitution, namely 10% STMP, 2% STPP, and a 99:1 ratio of 10% STMP: STPP. Edible films were made in 2 stages. In the first stage of making PBYS, starch was modified using STMP, STPP and STMP: STPP 99:1 reagent with three concentration levels of 2, 6, and 10% (w/v) for each phosphorylation reagent. In the second stage, edible films were made with the highest degree of substitution: 10% STMP, 2% STPP, and a 99:1 ratio of 10% STMP: STPP.

Edible PBYS films were prepared in two stages. In the first stage, starch was modified using STMP, STPP, and a 99:1 ratio of STMP:STPP with three concentration levels, namely 2, 6, and 10% (w/v) for each phosphorylation reagent. In the second stage, edible films were prepared with the highest substitution levels, namely 10% STMP, 2% STPP, and a 99:1 ratio of 10% STMP: STPP.

2.2 Synthesis of edible film from phosphorized Banggai yam starch

Glycerol was added in different concentrations (10, 30, and 50%) (w/v) of the weight of PBYS (Table 1), then heated on a hot plate stirrer until it reached a temperature of 85°C and maintained for 11 min while continuing to stir. Glycerol was added in different concentrations (10, 30, and 50%) (w/v) of the weight of PBSS, then heated on a hot plate stirrer while continuing to stir until it reached a temperature of 85°C for 11 min. The solution was then poured into a plastic plate and dried in an oven at 50°C for 18–24 h. The drying process was stopped after the edible film could be easily removed from the plate. After drying, the film was cooled at room temperature for 15 min before being stored in a desiccator.

Table 1. Preparation stages and treatment codes of phosphorylated Banggai yam starch (PBYS) and its edible films

PBYS (g)	Concentration Level Reagent (% w/v)	Glycerol (%)
2 (B1)	STMP 10% (R1)	10 (P1)
	STPP 2% (R2)	30 (P2)
	STMP: STPP (99: 1) 10% (R3)	50 (P3)
3 (B2)	STMP 10% (R1)	10 (P1)
	STPP 2% (R2)	30 (P2)
	STMP: STPP (99: 1) 10% (R3)	50 (P3)
4 (B3)	STMP 10% (R1)	10 (P1)
	STPP 2% (R2)	30 (P2)
	STMP: STPP (99: 1) 10% (R3)	50 (P3)
5 (B4)	STMP 10% (R1)	10 (P1)
	STPP 2% (R2)	30 (P2)
	STMP: STPP (99: 1) 10% (R3)	50 (P3)

2.3 Colour

Colour analysis is a standard method for determining the visual characteristics of phosphate Banggai yam starch (PBYS). Film clarity was measured using the method developed by (Kerr and Cleveland, 1953), as described in Wattanachant *et al*. (2002). The 1% starch solution was heated in a water bath at 95°C for 30 min while stirring, then cooled to 25°C for one hour. Film clarity was measured using a spectrophotometer with a per cent transmittance (%T) at a wavelength of 650 nm, with distilled water as a blank.

2.4 Thickness

Thickness measurement is an important parameter to evaluate the physical properties of films produced from PBYS. The edible thickness of the film was measured using callipers according to the method described by Turhan and Sahbaz (2004). The sample was placed between the calliper's jaws, and the thickness was measured at five places. The average was calculated.

2.5 Solubility in water

Measuring the solubility of PBYS in water provides information about its potential application in water-based food systems. The solubility of the film in water is measured as the percentage of dry weight of the film that has been dissolved in water for 24 h (Bertuzzi *et al.*, 2007). This solubility was determined using a method developed by Colla *et al.* (2006) and modified by Chiumarelli and Hubinger (2012). The three discs, each 2 cm in diameter, were cut, weighed, and immersed in 50 ml of distilled water, stirred gently for 24 h at 25°C. Next, the samples were dried at 105°C for 24 h to determine the dry mass content of the initial and final samples.

2.6 Degree of acidity

Degree of acidity (pH) analysis can provide information about the chemical characteristics of PBYS that influence its properties. Stability to acids was measured using a method described by Wattanachant *et* *al.* (2002) by comparing the gelatinization pattern of the modified starch solution at pH 3.5. According to standard procedures, the starch solution was tested using a Rapid Viscosity Analyzer (RVA). pH adjustment to reach 3.5 was carried out by adding 0.003% acetic acid solution, using a pH meter to ensure accuracy.

3. Results and discussion

3.1 Colour

When producing the edible film, one of the parameters checked was colour using a Konica Minolta CR-400 chromameter with a measurement method referring to the CIE system (Precision Colour Communication Book: Konica Minolta). The L* value indicates the colour brightness level; the higher the L* value, the brighter. Values represent red and green; a positive value means the colour becomes redder, and a negative value means the colour becomes greener. Meanwhile, blue or yellow is described with a b value; a positive b value means the colour is getting yellower, and a negative b value means the colour is getting bluer (Coultate, 2002). The L* value, i.e. whiteness, is the Lightness between 0 and 100. The corresponding a* and b* values are red, green, and blue, respectively, between 0 and -60, and yellow, blue, and between 0 and -60.

Based on the research results obtained (Figure 1), the B2R2P2 treatment (3 g PBYS, 10% STMP, 30% glycerol) gave the highest value for the L* value, namely 36.12±0.63, B3R1P3 treatment (4 g PBYS, STPP 2%, Glycerol 50%) gave the highest value in the a+ value, namely 0.84±0.11, and treatment B2R2P3 (3 g PBYS, STMP 10%, Glycerol 30%) gave the highest value for the b- value, namely -3.19±0.01. Whereas B1R2P3 treatment (2 g PBYS, 10% STMP, and 50% glycerol) gave the lowest L* value, namely 30.70±0.48, treatment B2R2P2 (4 g PBYS, STMP 10% STMP, and 30% glycerol) provide the lowest value the a+ value of 0.09±0.06, B4R1P2 treatment (5 g PBYS, STPP 2%, Glycerol 30%) gives the lowest value b- value is -1.31±0.18.

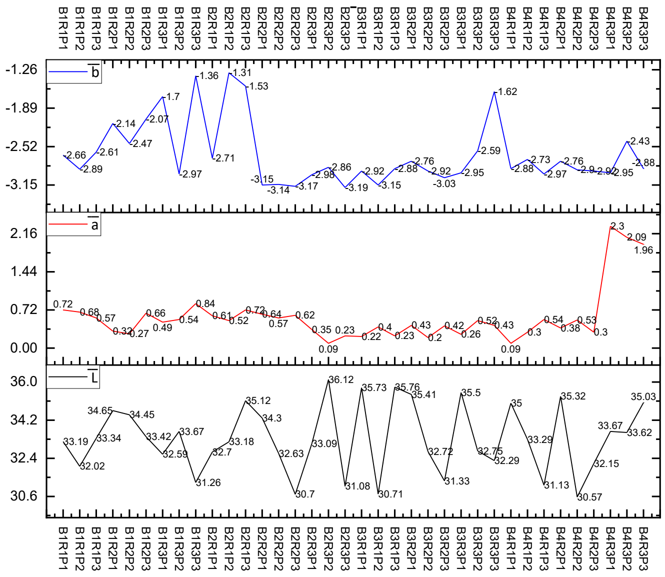

Figure 1. Colour of PBYS edible film.

Figure 1 shows the colour value of PBYS edible film with an L* value between 30.70 and 36.12. This value is still blurry because the more significant the L* value, the more transparent the colour level is; conversely, if the L* value is lower, it shows the colour level, which is blurry. The average colour value of a+ (positive) edible film is between 2.30-0.09, showing the colour is getting redder. Adding glycerol decreases the greenish and yellowish colours, whereas increasing the concentration of glycerol affects the a and b colour values but not the L colour values (Fatma *et al*., 2015). When glycerol is added, the film's colour becomes more vibrant. Consequently, the optical qualities of the film can be improved by adding a plasticizer (Jouki *et al.*, 2013).

Meanwhile, the average (negative) colour value of the edible film produced was between -1.31 and -3.19, which means the colour is getting bluer. According to Nikmah *et al*. (2020), the colour decreases as the edible film becomes thicker because it can increase the amount of film-forming polymer that can filter light, so it looks dull and blurry. This is thought to occur because there is a relationship between the brightness level and the thickness of the edible film, where the thicker it is, the lower the brightness level (Sitompul and Zubaidah, 2017). In addition, several conditions, such as heating food, changes in pH, and oxidation during storage, can cause degradation or even loss of colour pigments in food (Dewi *et al.,* 2022). Besides that, the colour or brightness of edible films can be reduced by increasing the starch concentration because higher starch concentrations cause the suspension to become more viscous, which increases the amount of dissolved solids in the film and the polymers that make it up. This increases the thickness of the film, which scatters light and makes the edible film appear opaque and dull (Nikmah *et al*., 2020).

3.2 Thickness

In making edible film and its intended use for coating or packaging food, thickness is a significant factor. The size of the film mould and the concentration of dissolved particles in the film solution affect the thickness. According to Rusli *et al.* (2017), thickness impacts edible film's mechanical and physical characteristics, including elongation, solubility, tensile strength, and water vapour permeability. According to Putri *et al.* (2022), thickness is a crucial factor that influences the use of film on products, including packaging and coatings (Safitri *et al*., 2020). The higher

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the plasticizer concentration in the mould and edible film solution, the higher the thickness value (Alfatahillah *et al*., 2021). Figure 2 shows the average combination value of PBYS weight treatment, phosphate reagent, and glycerol concentration on the thickness value of edible film.

Figure 2 illustrates how the addition of PBYS weight, phosphate reagent, and glycerol concentration as a plasticizer produces varying thickness values for the average thickness of the edible film. The highest PBYS edible film thickness value was found in the B3R2P1 treatment (4 g PBYS, 10% STMP, and 10% glycerol), namely, 0.65 mm, and the lowest PBYS edible film thickness value was found in the B2R2P1 B2R2P2 B2R2P3 treatments (3 g PBYS, 10% STMP, 10% glycerol, 30%, 50%) B1R1P1 B1R1P2 B1R1P3 (2 g PBYS, 2% STPP, 10% Glycerol, 30%, 50%), each 0.10 mm. Specific film thicknesses are suitable because they are within the maximum thickness standard of 0.25 mm set by Japanese Industrial Standards. In contrast, other thicknesses are categorized as exceeding this maximum requirement. Adding a modified starch content can increase the edible film's thickness (Nikmah *et al*., 2020). This happens because adding more starch will increase the amount of polymer that makes the film matrix, increasing the edible film's total solids and making the final product thicker (Ulfayanti *et al*., 2018).

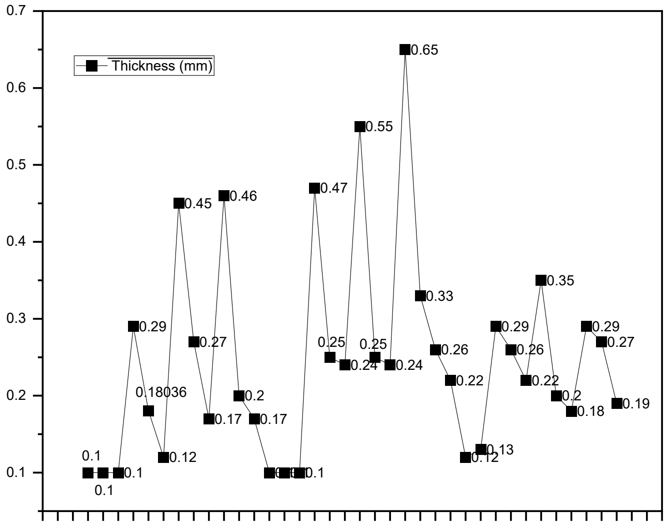

Figure 2. The thickness of PBYS edible film.

This research observed that the thickness of edible film tends to decrease as the glycerol content increases. According to Faizin *et al.* (2023), glycerol is easily soluble in water and can reduce the solution's viscosity; the higher the glycerol concentration applied, the thinner the edible film. Because glycerol is hydrophilic, it binds more water, evaporating during the drying process, thus affecting the viscosity value. With increasing glycerol concentrations, the thickness value also increases. The resulting edible film will be thicker if more edible film solution is added to the small mould (Lestari *et al*., 2022).

3.3 Solubility in water

The ability of a food ingredient to dissolve after consumption is called solubility, which also determines the properties of biodegradable films that can be used to package food. Because it dissolves easily when swallowed, an edible film with high solubility is ideal for food products (Febianti *et al*., 2020). Water solubility is essential in packaging materials, and water insolubility, water resistance, and product integrity are critical. The average value of the combination of heavy PBYS treatment, phosphate reagent, and glycerol concentration on the water solubility value of edible film can be seen in Figure 3.

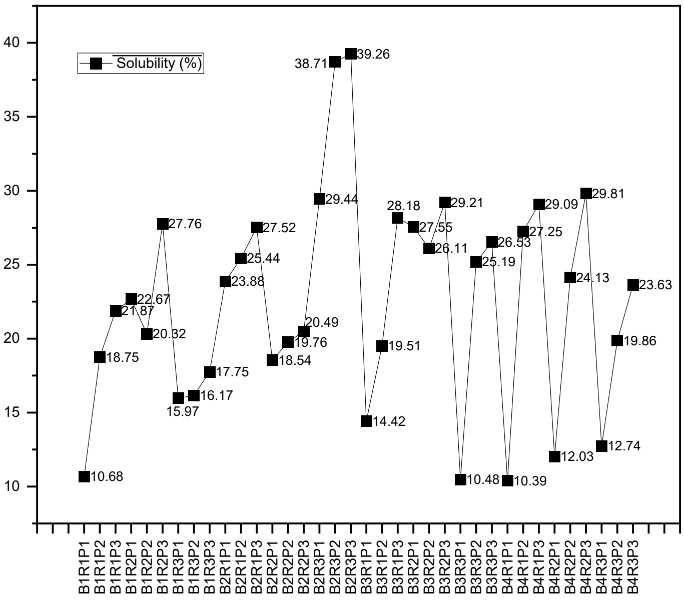

Figure 3. Solubility in water of PBYS edible film.

Figure 3 shows that the average value of edible film solubility in water varies. The highest value was found in the B2R3P3 treatment (3 g PBYS, 10% STMP/STPP, 50% glycerol), which was 39.26%, and the lowest value was found in the B4R1P1 treatment (5 g PBYS, 2% STPP, 10% glycerol), which was 10.39%. As the concentration of starch increases, the edible film's solubility in water decreases. This is because the hydrophilic groups in the edible film components will diminish as the starch content increases, resulting in a decrease in the glycerol: starch ratio (Ulfayanti *et al*., 2018). This research shows that the water solubility of edible film increases with increasing glycerol concentration. This is caused by the hydrophilic nature of glycerol, which means that adding more to the edible film will weaken the bonds between starch molecules, causing the molecular density to decrease and the formation of free spaces in the film matrix, increasing solubility (Widodo *et al.,* 2019). In general, the higher water solubility of glycerol-based films is due to their heterogeneous structure, thus potentially retaining more water in the film structure (Jaderi *et al.*, 2023).

3.4 Acidity degree

The degree of acidity of a material or solution can be expressed using the pH scale. This pH test is essential because it is a standard to limit the growth of biological pollutants, such as fungi, bacteria, and other microbes, that can damage the product's texture, taste, and nutritional value (Dewi *et al*., 2022). The acidity indicator of a solution is the standard acidity level. Based on the measurement results, the pH of the edible film was in the physiological pH range of the mouth, which was 5.5 to 7.9 (Wati *et al*., 2023). A pH close to neutral (7) is ideal for making edible films because a pH that is too low can change the taste of food. A pH meter was used to measure pH in this investigation. Research test findings for edible film acidity ranged from 5.25 to 6.21.

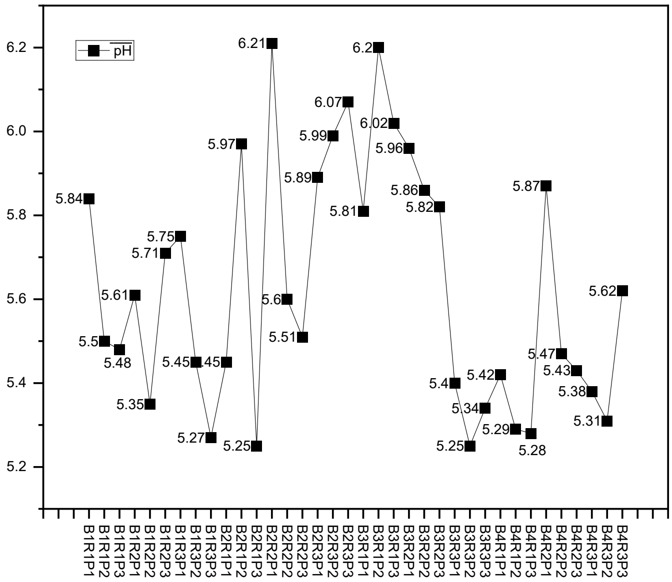

Figure 4. pH of PBYS edible film.

Figure 4 shows a graph representing the average acidity of edible film.

Based on Figure 4, the treatment of starch, different phosphate reagents, and glycerol concentration influences the degree of acidity of the edible film. With each increase in glycerol concentration, acidity decreases as the added glycerol concentration increases. The highest degree of acidity was found in the B2R2P1 treatment (2 g PBYS, 2% STTP, 30% glycerol), 6.21. Meanwhile, the lowest degree of acidity was found in the B2R1P3 treatment (2 g PBYS, 2% STTP, 50% glycerol), at 5.25. The acidity test results show that the average edible film produced is quite good because the pH is close to neutral (pH = 7). If the acidity value decreases, they may become more acidic. The glycerolutral acidity value is around 6-7. This chemical has the release of H+ ions as a byproduct. Esterification is one process that can occur with glycerol. Compounds containing carboxylic acid are produced as the esterification reaction progresses.

4. Conclusion

The results show that the resulting Edible Film has a relatively low brightness with a tendency to red and blue colours. Variations in thickness allow for adjustment as needed, while increased solubility and pH that tend to be neutral or slightly acidic support its application in various food products.

Conflict of interest

The authors declare no conflict of interest.

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