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INTELLIGENT PACKAGING AS A pH-INDICATOR BASED ON CASSAVA STARCH WITH ADDITION OF PURPLE SWEET POTATO EXTRACT *(IPOMOEA BATATAS L.)*

Safinta Nurindra Rahmadhia*
Yanas Anggana Saputra
Titisari Juwitaningtyas
Wahidah Mahanani Rahayu

Department of Food Technology, Faculty of Industrial Technology, Universitas Ahmad Dahlan, Yogyakarta, Indonesia.

ABSTRACT

Intelligent packaging is an indicator that has the capability to the condition of packaged foods and their environment through an indicator. This study aims to determine anthocyanin levels, pH sensitivity, solubility, swelling, and water content in intelligent packaging based on cassava starch with the addition of purple sweet potato extract. The method of this research is separated into two stages, which are the extraction of purple sweet potato and the development of indicator film. Furthermore, this research is conducted to analyze the physico-chemical properties of the film. This research is used Completely Randomized Design (CRD) with various extracts on indicator films that are 0%; 5%; 10%; 15% (v/v). The results showed that the indicator film with 15% extract has the best pH sensitivity with the color result 76.07 (L), 23.56 (a), 7.88 (b), solubility 52.15%, swelling power 133.90%, and it has 15.46% water content. The addition of purple sweet potato extracts significantly affected the total anthocyanin of the film indicator and pH sensitivity. The more extracts added to the film, the more obvious the color changes, the higher the water content and solubility but the lower the swelling properties.

Keywords: anthocyanin; cassava starch; intelligent packaging; pH indicator; purple sweet potato

ABSTRAK

Kemasan cerdas merupakan indikator yang memiliki kemampuan terhadap kondisi pangan yang dikemas dan lingkungannya melalui suatu indikator tersebut. Penelitian ini bertujuan untuk mengetahui kadar antosianin, sensitivitas pH, kelarutan, daya pengembangan, dan kadar air dalam kemasan cerdas berbasis pati singkong dengan penambahan ekstrak ubi jalar ungu. Metode penelitian ini dibagi menjadi dua tahap, yaitu ekstraksi ubi jalar ungu dan pembuatan indicator film. Selanjutnya, penelitian ini dilakukan untuk menganalisis sifat fisiko-kimia film. Penelitian ini menggunakan Rancangan Acak Lengkap (RAL) dengan variasi ekstrak pada indikator film yaitu 0%; 5%; 10%; 15% (v/v). Hasil penelitian menunjukkan bahwa indikator film dengan ekstrak 15% memiliki sensitivitas pH terbaik dengan hasil nilai warna 76,07 (L), 23,56 (a), 7,88 (b), kelarutan 52,15%, daya pengembangan 133,90%, dan memiliki kandungan air 15,46%. Penambahan ekstrak ubi jalar ungu berpengaruh nyata terhadap total antosianin dan sensitivitas pH pada indikator film. Semakin banyak ekstrak yang ditambahkan ke dalam film, semakin jelas perubahan warnanya, semakin tinggi kadar air dan kelarutannya, tetapi semakin rendah sifat pengembangannya.

Kata kunci: antosianin; indikator pH; kemasan cerdas; tepung singkong; ubi ungu

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Corresponding author:
Safinta Nurindra Rahmadhia
Yogyakarta, Indonesia, 55166
Email:
safinta.rahmadhia@tp.uad.ac.id

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INTRODUCTION

Food safety and quality are two factors affecting consumers in food selection. A factor impacting food safety and quality is food packaging. Food packaging aims to wrap and afford protection to food materials from external environmental conditions, e.g., heat, light, water, air, and microorganisms (Marsh & Bugusu, 2007). Changes in consumer preference for food safety breed innovations in food packaging technology, one of which is intelligent packaging (Biji et al., 2015).

Intelligent packaging is one of the developments of packaging concepts. It can monitor the condition and environment of the food using an indicator (Sitanggang et al., 2020). Intelligent packaging is an interactive indicator, the component of which is natural dye-based which can assess product quality from its chemical components. Freshness indicator was used to monitor fishes based on the number of total volatile basic nitrogen (TVB-N). Moreover, the detection of cod flesh was also observed using food freshness indicators made from natural sources such as curcumin, grape peel, and beetroot extract. The result shown that the applications of intelligent packaging which was incorporated with natural bio-based extract can effectively show freshness level based on color difference (Tichoniuk et al., 2017).

One of the packaging indicators is acid-based. This indicator is called a pH indicator. The color of the indicator, which is pH-based, is influenced by the environment where it is applied (Moradi et al., 2019). Plant-based dyes contain natural compounds at large, e.g., anthocyanin, β-carotene, curcumin, and chlorophyll. Some of these compounds are sensitive to pH and will exhibit a clear color spectrum when applied in two different environments, i.e., acid and base (Alizadeh-Sani et al., 2020). One of the natural dyes in a packaging indicator is anthocyanin.

As having a dominant hydroxy cluster, anthocyanin is blue in color and relatively unstable (a high pH), whereas if having a dominant methoxy cluster, it will be red in color and relatively stable (a low pH). Therefore, anthocyanin is suitable for being a bio-indicator and give a color change effect. Anthocyanin stability in water or polar solvent, which is neutral or basic, can be augmented with acid addition (Sipahli et al., 2017). A proper combination of polar solvent and an organic acid which breeds a very acidic pH condition (a pH of 1-2) can elevate anthocyanin stability in the form of red flavylium cation (Pedro et al., 2016). Accordingly, the process of anthocyanin extraction deploys an ethanol solvent and citric acid.

One of the raw materials containing anthocyanin is purple sweet potato. Anthocyanin contained in a purple sweet potato is an acyl chain in form. Acylation in anthocyanin of a purple sweet potato can indicate unique properties, e.g., pH stability, heat resistance, antioxidant capability, and anti-inflammation (Yaningsih et al., 2016). A purple sweet potato contains 19.61% of carbohydrates, 1.04% of protein, and 0.33% of fat. Additionally, it also contains 93.64 mg/100 g of anthocyanin (Chen et al., 2019).

Tapioca starch is extracted from cassava. Among polysaccharide products, tapioca starch affords many benefits, such as being affordable, reachable, colorless, odorless, non-toxic, and eco-friendly. Tapioca contained 96.74% starch and 37.43% amylose which is higher than corn starch and purple sweet potato starch (Lopattananon et al., 2012; Palacios-Fonseca et al., 2013; H. Wang et al., 2020). Amylose plays an important role in the crystallization of starch to form a film in the starch gelatinization process (Wang et al., 2022).

However, a starch-based film has a poor and inflexible mechanic property (Silva et al., 2019). So, the addition of plasticizer is an interesting formulation to improve film mechanical properties. The mixture of plasticizer and starch are tends to decrease the interaction between starch chains and increase their mobility (Yang et al., 2017).

The focal points of this research are total anthocyanin contents, pH sensitivity, solubility, swelling capacity, and water content in intelligent packaging in the form of a pH indicator. The indicator is made from a tapioca starch-based film layer added with the extract of purple sweet potato.

MATERIALS AND METHOD

Materials

Tools used were a hot plate stirrer (Thermo Scientific Cimarec, USA), magnetic stirrer, water bath shaker (B-ONE SWB 30), blender (Philips HR2115, Indonesia), oven (Memmert, Germany), glass mold, analytical balance (Ohaus, Indonesia), desiccator, UV-vis spectrophotometer (Thermo Scientific Genesys 15), digital pH meter (Nutron Tech), and chromameter (Konika Minolta CR-400).

The raw materials used were a Gunung Kawi purple sweet potato (CV Sarana Meraih Berkah, Indonesia), tapioca starch (PT Budi Starch, Indonesia), aquadest, glycerol, 96% of ethanol, HCl-KCl buffer (pH of 1), acetate buffer (pH of 4.5), 60% of citric acid, and whatman paper no. 41.

Methods

Extraction of anthocyanin from a sweet purple potato

The extraction method referred to (Chen et al., 2019) with a modification. The purple sweet potato was washed and cut into small cubes. The cubes were smashed using a blender until reaching a porridge-like texture and weighed as much as 20 g. Purple sweet potato was added with a mixture of ethanol 96% and citric acid 60% with a ratio of purple sweet potato and solvent 1:5. Then the mixture was extracted using a water bath shaker at 60℃ for 60 minutes. The extract was filtered using a piece of Whatman paper no. 41. Supernatants yielded were stored in a dark bottle at 4℃.

Preparation of indicator films from a sweet purple potato

In making the film, a casting method by (Piñeros-Hernandez et al., 2017) was applied with a modification. A 7.5g of tapioca starch was mixed with 1.5 g of glycerol and 91 ml aquadest. The mixture was stirred on a hot plate stirrer for 60 minutes at 90℃. The extract of purple potato (5, 10, 15% w/v) was then added and stirred for ten minutes at 50℃. The mixture was cast using a glass mold and left to dry at 50℃ for 15 minutes using an oven.

Total anthocyanin analysis

A pH differential method was employed to calculate the total anthocyanins content in indicator films containing the extract of purple sweet potato (Lee et al., 2005). The absorbance of the sample and total content of anthocyanins in the sample were determined using the following equation.

$A = (A_{max} - A_{700})\ pH = 1 - (A_{max} - A_{700})\ pH = 4.5$ (1)

$$\text{Total anthocyanin (mg/L)} = \frac{A \times BM \times FP \times 1000}{\varepsilon \times b} \quad (2)$$

Descriptions:

A: Absorbance

A_{max}: Maximum absorbance

A_{700}: Absorbance at 700 wave length

BM: Molecular weight (449,2 g/mol)

FP: Dilution factor

ε : Molar absorptivity (26900L/mol.cm)

b: Thick of cuvette

Color measurement

The color test on the indicator film contained the extract of sweet purple potato (5 x 5 cm^2) was carried out using a chromameter (Konika Minolta CR-400) at a CIE color system (Sitanggang et al., 2020). The film was put on a plate reader which was on a white table. Then the plate reader was shot using a chromameter. The measured value on the screen as L^* (lightness), a^* (appearance), b^* (blueness).

pH sensitivity

The test for the extract's pH sensitivity was conducted by preparing 18 ml of buffer solvent with a pH of 2-11. A 2 mL of anthocyanin extract was added and stirred using a magnetic stirrer for 30 minutes. Conditioning the pH value from acid to basic was critical to examine levels of color changes of anthocyanin extracts at each pH value.

The film was cut into a size of 2 x 2 cm and soaked in a mold containing a buffered solvent with a pH of 2-11 for five minutes. The acid-basic indicator film was measured for its pH using a pH meter (Moradi et al., 2019).

Solubility

The film was cut into a size of 1x 2 cm and put on a cup prior to put into an oven at 100℃ for an hour. The film was weighed for its initial dry weight (w_0) and soaked for 24 hours. An insoluble film was put into an oven for an hour at 100℃, leaving it to dry. It was then stored in a desiccator for 10 minutes and weighed for its post-soaking dry weight (w_1) (Unsa & Paramastri, 2018).

Swelling properties

The indicator film was cut by 1 x 2 cm and aerated for 30 minutes and weighed (w_1). It was then soaked in 30 ml of aquadest (25℃) for 60 minutes. The wet sample was filtered to remove its solvent and weighed (w2) (Popović et al., 2011).

Water content analysis

Water content was measured using a percentage of weight loss of the sample of anthocyanin indicator films after the sample was dried using an oven at 105℃ for 24 hours until a constant weight of the sample was reached (Sitanggang et al., 2020).

RESULTS AND DISCUSSION

Total anthocyanin content

Extracting a purple sweet potato using a water bath shaker for 60 minutes at 60℃ with a mixture of ethanol and citric acid as solvent resulted in an extract of purple sweet potato which was red in color and 42.74% of yields. After extraction, the total anthocyanin was measured, yielding a mass of 118.17 mg/100 g. Table 1 points out the total anthocyanins of the indicator film of purple sweet potato.

The highest total anthocyanin was showcased by the indicator film F3 as a result of a 15% of extract addition. Meanwhile, the lowest one was presented by the indicator film F1 because of the least extract addition of 5%. The indicator film F0 did not contain anthocyanins. The absence was marked by the measurement result of < 1 by virtue of no anthocyanins contained in the materials added to the film. Anthocyanin content of the extract of sweet purple potato, which was initially 118.17 mg/100 g, declined after being added to indicator films. The decline came about due to anthocyanin degradation after the heating process since one of the anthocyanin natures was heat-unresistant (Husna et al., 2013). In other research, anthocyanin from Hibiscus sabdariffa was extracted from four different solutions then treated to 80 and 50ºC for 6 hours. That extract declined generally in the pigmen retention. The number of anthocyanin degradation rose following the increase of heat temperature (Sipahli et al., 2017). In addition, the hydrogen peroxide which formed through oxidation of ascorbic acid oxidized anthocyanin pigments (Mercali et al., 2013).

Table 1. Total Anthocyanins of Film Indicators

Sample	Total Anthocyanin (mg/100 g)
F0	0.78 ± 0.09^{a}
F1	21.82 ± 0.10^{b}
F2	43.42 ± 0.17^{c}
F3	69.97 ± 0.17^{d}

The different superscripted letters show a significant difference ($p < 0.05$). F0 = without-extract film, F1 = film with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.

The extract of sweet purple potato with a combination of ethanol solvent and citric acid yielded better than that with a water solvent and a total anthocyanin of 17.06 mg/100 g (Chen et al., 2019). A low anthocyanin level might be brought about by the sweet purple potato's plant tissue which could not be destroyed and penetrated by water. This brought on poor extraction of chemical compounds. Alcohol and citric acid as solvents could enhance the lysis ability of vacuole membranes, escalate osmosis abilities, and produce anthocyanins in a stable condition in the form of flavylium ions (pH of 1-2) (Li et al., 2013). In the research of (Chen et al., 2019), the difference in total anthocyanins extracted using different solvents was identified using water and ethanol, water and citric acid, and ethanol and citric acid as

the solvents. The highest anthocyanin was acquired with a combination of ethanol and citric acid solvents of 93.64 mg/100 g at an extraction temperature of 80℃ for 40 minutes. Previous Chen's research results were comparably different from ours owing to different variants of the sweet purple potato used and different extraction temperatures. Chen et al., (2019) used a TN73 sweet purple potato from Taiwan, while we used a Gunung Kawi one. In addition, Chen exerted an extraction temperature of 78.9℃ which exceeded the boiling point of ethanol. This caused several chemical compounds to evaporate during the extraction process.

Previous research demonstrated as the best result for sweet purple potato using a concentrated solvent extraction method (Cai et al., 2016). Anthocyanin content derived from the extraction was 215.29 mg/100 g at 70℃ for 90 minutes using ethanol and HCl as the solvents. Chai's research results were different from the current ones considering different variants of the sweet purple potato used. Besides, Chai centrifuged the extract acquired and evaporated the solvents using a rotary evaporator. This method yielded a more concentrated extract, which affected anthocyanin resulted, the amount of which increased. Nevertheless, the anthocyanin was heat labile because the extraction process was conducted in both high temperature and short time (accelerated-solvent extraction method), while the anthocyanin that was extracted using conventional extraction and ultrasound-assisted extraction generate high impurities extract.

Color characteristics of indicator films

Figure 1 presents indicator films of sweet purple potato in all formulations. The indicator film F0 was white in color and slightly transparent. Moreover, indicator films with an addition of sweet purple potato extracts were pink in color. The more the extract of sweet purple potato added, the brighter the red color. The pink color of an indicator film was engendered by the addition of sweet purple potato extract, which contained anthocyanins in the form of flavylium cation from the extraction process using acid (Khoo et al., 2017).

Table 2 exhibits the results of color analysis on indicator films made. The indicator film without the extract of sweet purple potato indicated the brightest color. Meanwhile, the addition of sweet purple potato extract might decrease film brightness but promote the red color as well as add a slightly-yellowish color. The yellowish color was on account of an addition of glycerol, which was then oxidated during the process of film molding. The pinker the indicator film, the more apparent the color change.

Table 2. Color Characteristics of Indicator Films

Sample	L^*	a^*	b^*
F0	85.27 ± 0.03^a	0.50 ± 0.03^a	3.47 ± 0.03^a
F1	82.94 ± 0.01^b	9.64 ± 0.05^b	5.82 ± 0.00^b
F2	76.69 ± 0.01^c	21.31 ± 0.03^c	8.12 ± 0.06^d
F3	76.07 ± 0.15^d	23.56 ± 0.44^d	7.88 ± 0.08^c

The different superscripted letters show a significant difference ($p < 0.05$). F0 = without-extract film, F1 = film with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.

Previous research figured out that a chitosan-based indicator film incorporated with alizarin extracts declined in terms of brightness level after extract addition (Ezati & Rhim, 2020). The film, which was initially colorless and transparent, turned its color after the alizarin addition. Alizarin addition to a chitosan-based film could decrease its brightness but heighten its red and yellowish colors.

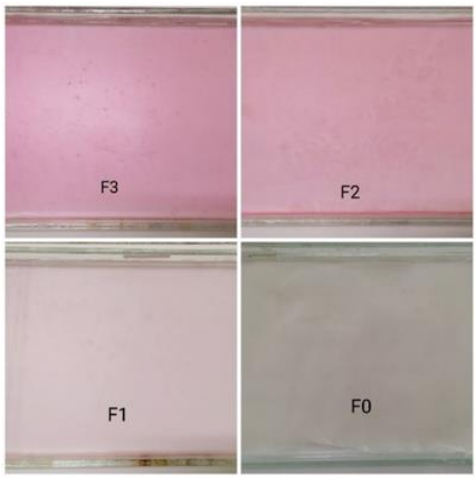

Figure 1. Colors of Indicator Films. *F0 = without-extract film, F1 = film with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.*

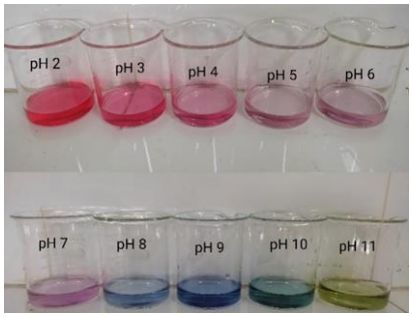

Figure 2. pH Sensitivity of Sweet Purple Potato extracts. *F0 = without-extract film, F1 = fifilm with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.*

Figure 3. pH Sensitivity of Indicator Films. *F0 = without-extract film, F1 = film with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.*

pH sensitivity of the extract and indicator films

Figure 2 shows color changes in the extract of sweet purple potato at a pH of 2-12. The extract color at an acid pH of < 5 was reddish, whereas that at a pH of 5-7 was purplish pink. Furthermore, the extract colors at basic pH of 8-9 and 10-11 were blue and yellowish-green, respectively.

Anthocyanin solution with the extract of sweet purple potato at different pH conditions (2-12) showcased a decreased value from +23.09 to -1.10, and the color parameter *a* showed off a color change from red to green (Chen et al., 2020). Moreover, with an increased value from +4.24 to +24.95, the color parameter *b* demonstrated a change in its yellow color on account of an increased pH. Hence, the extract of sweet purple potato was sensitive to pH changes and thereby effective for being used as a natural pH indicator.

Figure 3 exhibits color changes in indicator films. The test of pH sensitivity on the indicator film F0 did not indicate any color changes in different pH conditions. Meanwhile, indicator films with an extract addition changed in color in different pH conditions. The more significant the color change, the more the extract concentration added. The indicator film F3 pointed out the most significant color change. It was red in color at a pH of 2 but turned to pink on grounds of gradual pH increases to 5. At a pH of 6-9, its color was purple to blue, and at a pH of 10-11, the color was green. Similarly, Sitanggang et al., (2020) presented research results which stated that films made from gelatin with the extract of sweet purple potato were red in color at a pH of 2 and gradually turned their color when the pH was gradually increased to 5. At pH of 6-9 and 10, they turned to blue and green, respectively. Film that was incorporated with oxalic acid and anthocyanin extract was chosen because it can show a clear color change in pH 2-11.

Solubility of indicator films

The result of the test for the solubility of indicator films is shown in Table 3. The lowest and highest solubility was reached by indicator films F0 and F3, respectively. It attested that the more the amount of extract added, the higher the indicator film's solubility. An indicator film's solubility showcased that each formulation resulted in significantly different solubility in water. A test for solubility was crucial for film-based food packaging to identify film resistance against water (Mustafa et al., 2020).

The high solubility of an indicator film might be owing to the addition of sweet purple potato, which

contained anthocyanins. Anthocyanin increased the number of hydroxyl clusters which could promote affinity for water. High solubility was a result of the weak bond of starch molecules and an increased number of hydrophilic clusters which could absorb water after a starch-anthocyanin interaction. In a sweet purple potato starch-based film with an addition of sweet purple potato extract, the more the extract added, the higher the solubility (Sohany et al., 2021; Zhang et al., 2020).

Swelling capacity of indicator films

A test for swelling capacity was undertaken to measure the capacity of film swelling when the film was in a water environment. Table 3 demonstrates the result of the test for the swelling capacity of indicator films. The highest and lowest swelling capacities were reached by F0 and F3, respectively. In addition, the more the extract added, the lower the capacity of indicator film swelling. The swelling capacity of an indicator film with an addition of sweet purple potato extract was lower than that of swelling in an indicator film without the addition of the same extract. It was on the grounds that the film decreased in mass because of high solubility. It was proven by the result of the test for solubility, that the more the extract of sweet purple potato added, the higher the solubility.

A starch-based film could absorb more water since starch was a hydrophilic polymer which could strongly bind with water (Qin et al., 2020). A decreased capacity of swelling in a film could break out because of the hydrogen bond interaction between polyphenol compounds in the extract and film matrix. A high extract level might generate a potent interaction between starch molecules. Starch molecules would bind more densely, preventing them from floating when in water.

Nevertheless, (Sohany et al., 2021) demonstrated a higher capacity of swelling in a film based sweet potato starch powder/sweet potato peel with an addition of 2% commercial sweet purple potato anthocyanin, while the sweet potato starch film with 0% commercial sweet purple potato anthocyanin showed lower value. The higher capacity was by virtue of the addition of sweet purple potato skin which contain fiber, to the film. The latter addition would expand the surface, surface porous, and channels which could augment water absorption and capacity of film swelling. The higher the swelling capacity, the higher the water-resistance capability of a film (Cornejo-Ramírez et al., 2018).

Table 3. Water Content, Solubility, and Swelling Capacity of the Film Indicators

Sample	Solubility (%)	Swelling Capacity (%)	Water Content (%)
F0	15.40 ± 0.16^{a}	173.80 ± 1.55^{c}	14.77 ± 0.27^{b}
F1	27.06 ± 0.46^{b}	142.20 ± 1.78^{b}	14.13 ± 0.13^{a}
F2	37.95 ± 0.79^{c}	141.55 ± 1.36^{b}	15.11 ± 0.25^{b}
F3	52.15 ± 0.52^{d}	133.90 ± 1.55^{a}	15.46 ± 0.26^{c}

The different superscripted letters show a significant difference ($p < 0.05$). F0 = without-extract film, F1 = film with 5% of extract, F2 = film with 10% of extract, F3 = film with 15% of extract.

Water content of indicator films

Water content, solubility, and swelling capacity were investigated to observe how extract addition influenced the water resistance capability of indicator films. The results of the investigation are exhibited in Table 3. The lowest and highest water content (14.13% and 15.46%, respectively) was reached by indicator films F1 and F3, respectively.

The results of the test for water content indicated that the addition of the extract of ≥ 10% could elevate water content in indicator films. Water content in the indicator film F1 was lower than that in the indicator film F0 due to an extract addition of 5% and decreased amount of aquadest. Therefore, when indicator films were left to dry, more film solvents evaporated as the extract was in ethanol whose boiling point was lower than that of water. Besides, an extract addition at a certain concentration into indicator films would likely form a hydrogen bond which could decrease hydrophilic compounds for the bond of hydroxyl clusters and amino clusters which could induce a restrained binding capability of starch and water (Nguyen et al., 2020). Previous research pointed out that the addition of oleic acid and anthocyanin extract at a certain concentration could decrease water content. This was a result of interactions between protein and water and between protein and lipid. Additionally, it was also possible because

hydrogen bonds created on account of the interaction between anthocyanins and protein could decrease the water content of a film. Hydrogen bonds between film molecules could decrease free water content in that the extract still contained total solids which could affect the water content of the film (Sitanggang et al., 2020).

This section can either be separated (Results and Discussion in different section) or mixed together as one section (Results/Discussion). The results section provides the scientific findings instead of presenting whole/raw data. The discussion should focus on exploring the significance of the work. In general, those sections should describe the results of the experiments, the interpretation of the scientific results, and the conclusions that can be generated. The author may state the weakness of the study here. All tables and figures must be in separated page after the references page.

CONCLUSION

The conclusion that can be drawn from this study is that the addition of purple sweet potato extract had a significant effect on the total film indicator anthocyanins. The film indicator has a pH sensitivity which is indicated by a change in color at different pH conditions. The best formula is F3 with higher anthocyanin extract. The more extract added to the film, the more obvious the color change will be. The more extracts added, the higher the water content and solubility but the lower the swelling strength.

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