

## Physical Properties of Tapioca Starch-Based Film Indicators with Anthocyanin Extract from Purple Sweet Potato (*Ipomoea batatas* L.) and Response to pH Changes

(Sifat Fizikal Penunjuk Filem Berasaskan Kanji Ubi Kayu dengan Ekstrak Antosianin daripada Ubi Keledek Ungu (*Ipomoea batatas* L.) dan Tindak Balasnya terhadap Perubahan pH)

SAFINTA NURINDRA RAHMADHIA\*, AFIST AZKIYA SIDQI & YANAS ANGGANA SAPUTRA

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

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### ABSTRACT

Intelligent packaging comes in the form of interactive film indicator using a natural pigment compound sensitive to pH changes. The development of intelligent packaging as an indicator film by utilizing natural pigment compounds that are related to pH change and food safety is motivated by increased consumer awareness of food safety. Purple sweet potato (*Ipomoea batatas* L.) is the source of anthocyanin flavonoid compounds sensitive to pH changes, demonstrated by color changes in film indicators. This research aims to determine physical properties and the pH response of tapioca starch-based film indicators with anthocyanin extract variation from purple sweet potato. Purple sweet potato anthocyanin (PSPA) indicator film was made using tapioca starch as biopolymer by casting method with the addition of anthocyanin extract at concentrations of 0, 5, 10, and 15 g. Furthermore, this research is conducted to analyze the physical properties of the film, and response to pH changes of fresh cow milk, Gindara fish fillet and chicken sausage stored at 7 °C and 25 °C under 48-hour observation. The results showed that the film indicator thickness was 0.72-0.74 mm, tensile strength was 1.23-9.86 MPa, elongation was 14.83-55.74%, and water vapor permeability (WVP) was  $1.32-1.78 \times 10^{-14}$  kg.m/m<sup>2</sup>.s.Pa. The results of this study indicated that the PSPA indicator films have the potential to be used as smart packaging to monitor food freshness and quality for safe consumption. That was supported by the good physical properties of PSPA indicator films.

Keywords: Anthocyanin; food applications; intelligent packaging; pH indicator; purple sweet potato

### ABSTRAK

Pembungkusan pintar datang dalam bentuk filem penunjuk interaktif menggunakan pigmen semula jadi yang sensitif kepada perubahan pH. Membuat pembungkusan pintar sebagai filem penunjuk dengan menggunakan komponen pigmen semula jadi yang berkaitan dengan perubahan pH dan keselamatan makanan didorong oleh peningkatan kesedaran pengguna terhadap keselamatan makanan. Ubi keledek ungu (*Ipomoea batatas* L.) ialah sumber flavonoid antosianin yang sensitif kepada perubahan pH, ditunjukkan oleh perubahan warna dalam filem penunjuk. Penyelidikan ini bertujuan untuk menentukan sifat fizikal dan tindak balas pH filem penunjuk berasaskan kanji ubi kayu dengan variasi ekstrak antosianin daripada keledek ungu. Filem penunjuk antosianin ubi keledek ungu (PSPA) dibuat menggunakan kanji ubi kayu sebagai biopolimer dengan kaedah percetakan dengan penambahan ekstrak antosianin pada kepekatan 0, 5, 10 dan 15 g. Seterusnya, kajian ini dijalankan untuk menganalisis sifat fizikal filem dan perubahan tindak balas terhadap pH susu lembu segar, ikan Gindara dan sosej ayam yang disimpan pada suhu 7 °C dan 25 °C di bawah pemerhatian selama 48 jam. Keputusan menunjukkan bahawa ketebalan filem penunjuk ialah 0.72-0.74 mm, kekuatan tegangan ialah 1.23-9.86 MPa, pemanjangan ialah 14.83-55.74% dan Kebolehtelapan Wap Air (WVP) ialah  $1.32-1.78 \times 10^{-14}$  kg.m/m<sup>2</sup>.s.Pa. Hasil kajian ini menunjukkan bahawa filem penunjuk PSPA sebagai penunjuk pH yang dihasilkan berpotensi untuk digunakan sebagai pembungkusan pintar untuk memantau kesegaran dan kualiti makanan untuk penggunaan yang selamat. Itu disokong oleh sifat fizikal yang baik bagi filem penunjuk PSPA.

Kata kunci: Antosianin; aplikasi makanan; pembungkusan pintar; penunjuk pH; ubi keledek ungu

## INTRODUCTION

Packaging plays a crucial role in food industries in maintaining food safety, quality, and shelf-life (Balbinot-Alfaro et al. 2019). It functions to protect and nourish products packaged, as well as to nurture convenience and communication with consumers (Choi et al. 2017). Food product quality can be impacted by a variety of circumstances, including incorrect handling and temperature which cannot be determined only by the expiry date printed on the packaging. As a result, intelligent packaging has received a lot of attention in response to customers' growing worries about accurate food quality information (Choi et al. 2017; Zhang et al. 2020).

Intelligent packaging is a packaging system by which we can monitor and afford real-time information about the packaged food product condition to consumers (Choi et al. 2017). It can be developed into a film indicator that is sensitive to pH fluctuations, earning the term pH indicator. One of the elemental factors which identify spoiled food products is pH changes (Silva-Pereira et al. 2015). The pH changes of products packaged are caused by microorganism activity, enzymatic autolysis, protein hydrolysis and fatty acid oxidation. The volatile compounds in packaging as a result of those reactions will be recorded by anthocyanin compounds on film indicator (Šojić et al. 2014; Tavares et al. 2021). Film indicators sensitive to pH changes, by principle, monitor the quality of packaged products by reading or recording pH changes in packaging. The changes are demonstrated by color changes in film indicators (Balbinot-Alfaro et al. 2019).

Film indicators sensitive to pH changes are commonly composed of two materials, i.e., polymers or indicator solids and pigment extracts sensitive to pH changes (Park et al. 2015). Polymers assembling film indicators as intelligent packaging are polysaccharides, namely tapioca starch. Tapioca starch, one of the film indicator materials, comes with several features: easily findable, affordable, abundant, consumable, and decomposable (Qin et al. 2019). The other film indicator materials are pigment extracts sensitive to pH changes. Purple sweet potato (*Ipomea batatas* L.) is the source of anthocyanin flavonoid compounds sensitive to pH changes exhibited by color changes in film indicators (Singh, Gaikwad & Lee 2018). The pigment extracts' color stability representing anthocyanin presence in a commodity and affected by pH changes make the extracts employed as an indicators to monitor food quality in a

packaging application or system (Singh, Gaikwad & Lee 2018). Jiang et al. (2020) found that pH indicators with anthocyanin extracts from purple sweet potato are used to check the freshness of fish fillets held at 25 °C for 48 h, causing the pH indicators to change from pink to purplish-blue. The changes happened caused by anthocyanins' protonation and deprotonation reaction (Barnes et al. 2009). At conditions more than pH 3 or in line with the increase in pH to pH 7, the red flavylium will change into blue quinoneoidal bases or to colorless carbinol pseudo bases (Yong & Liu 2020).

Film indicators from purple sweet potato were specific or special indicators in the form of labels or films which could be applied or administered into a food packaging system. The indicators, which were chemical sensors or indicators, could detect physical and chemical changes, e.g., aroma and flavor changes of food products as a result of decaying processes during storage at room temperature, which created pH changes (Kuswandi & Jumina 2020). pH change indicators could be applied to food products, especially meat, fish, and milk and its derivative products (Dodero et al. 2021).

Chemical sensors can act as receptors, detecting certain gases or chemical substances using adsorption sensors that induce changes to the sensor's surface (Vanderroost et al. 2014). Food with a high protein and moisture content easy to promote microbial growth, incurring organic acid, which could decrease pH of food product. In addition, carbon dioxide (CO<sub>2</sub>) is one of the microorganism metabolism products which could be dissolved in food products and form carbonate acid subsequently diminishing pH (Saliu & Della Pergola 2018). Chemical sensors detected volatile organic compounds, too, such as trimethylamine, dimethylamine, and ammonia which could augment pH because of their alkali properties (Kuswandi et al. 2020).

This research aims to develop tapioca starch-based film indicators sensitive to pH changes. The film indicators are made using 0 g (F1/control), 5 g (F2), 10 g (F3), and 15 g (F4) anthocyanin extract variation from purple sweet potato to investigate their physical properties, i.e., thickness, tensile strength, elongation, water vapor permeability, biodegradability, and changes to the pH of fresh cow milk, Gindara fish fillet, and chicken sausage stored at 7 °C and 25 °C over the course of 48 h. The products chosen because they are perishable and, this research was conducted to determine the differences color changes in film indicators caused by the pH changes of spoiled product.

## MATERIALS AND METHODS

The materials used were Gunung Kawi purple sweet potato (CV Sarana Meraih Berkah) purchased from Superindo Parangtritis Yogyakarta, tapioca starch (Pak Tani, PT Budi Starch) acquired from Superindo Parangtritis Yogyakarta, fresh cow milk supplied by Toko Susu Sapi Segar Pak Tri Kotagede Yogyakarta, Gindara fish fillet (*Lepidocybium flavobrunneum*) acquired from Superindo Parangtritis Yogyakarta, chicken sausage homemade at Laboratorium Terpadu Universitas Ahmad Dahlan Yogyakarta, ethanol 96%, citric acid 60%, filter paper (Whatman no. 41), aquadest, glycerol, and technical NaCl.

### EXTRACTION OF ANTHOCYANIN

Anthocyanin extraction from purple sweet potato was carried out using method (Chen et al. 2019) with some modifications. To commence the extraction, the purple sweet potato was washed under running water. The 20 g-weighed purple sweet potato was cut into a small size and mashed into a slurry form using a blender. The purple sweet potato slurry was diluted in 85mL of ethanol 96% and 15 mL of citric acid 60%. The mixture was subsequently extracted using a water bath shaker at 60 °C and a speed of 150 rpm 60 minute-long. The extraction result was filtered using the Whatman filter paper no. 41. The supernatant derived was stored in a dark bottle at 4 °C.

### PREPARATION OF INDICATOR FILMS

The preparation of PSPA film indicators were made using method by Rahmadhia et al. (2022). A 7.5 g of tapioca starch was mixed with aquadest and 1.5 g of glycerol. The mixture was stirred using a hot plate stirrer over 60 min at 90 °C. After the first stir, 0 g (F1/control), 5 g (F2), 10 g (F3) dan 15 g (F4) of anthocyanin extracts from purple sweet potato were added to the mixture. The mixture was stirred on a hot plate stirrer at 50 °C for ten min. A 100 mL solution was moulded in a glass tray and dried in an oven at 50 °C for 15 h.

### FILM THICKNESS

Each PSPA film indicator was cut into a 5 × 5 cm size and measured for its thickness using a manual screw micrometer (Mitutoyo, Japan) in five film sides, namely top right, top left, middle, bottom right, and bottom left, and the mean sample thickness (mm) was defined.

## MECHANICAL PROPERTIES

The tensile strength and elongation of samples were observed using a universal testing machine with the ASTM D 882 standard method (ASTM D 882-02, 2002). Samples were cut to a length of 50 mm and a width of 5 mm. The sample was placed on the top and bottom holding grips. Then the sample was locked by turning the handwheel until the sample cannot be released. The test speed on the universal testing machine was set to 10 mm/min and then the machine was started. The universal testing machine will pull the tested sample until it breaks and the value was displayed on the screen in the form of the tensile strength (MPa) value and the percent elongation.

### WATER VAPOR PERMEABILITY

The water vapor permeability (WVP) of PSPA film indicators was researched using the ASTM E 96 gravimetric method (ASTM E 96, 1995) with some modifications. The samples were cut into 3 × 3 and adhered to a ceramic dish containing 3 g of silica gel. The silica gel had been left to dry using a microwave at 105 °C over the course of 2 h. The dish was placed into a desiccator at 25 °C and RH of 75% with a saturated solution of NaCl. Observation of sample weight changes was performed every 12 hours five-day-long. The WVP in the sample were determined using the following equation.

$$WVP = \frac{WVTR \times thickness}{\Delta P}$$

where WVTR is the water vapor transmission rate (kg/m<sup>2</sup>); and ΔP is the pressure difference (Pa).

### THE FILM INDICATOR RESPONSE TO THE PH OF FOOD SAMPLES

The PSPA film indicator response to the food sample's pH were analyzed using method of Jiang et al. (2020) with some modifications. The food products, i.e., fresh cow milk, Gindara fish fillet, and chicken sausage, were weighed at 20 g each. The milk was stored in a capped plastic container, whereas the Gindara fish fillet and chicken sausage were stored in petri dishes. PSPA film indicators were cut in 2 × 2 cm and placed on the lid or the top (headspace) of the plastic container and petri dishes. The food products were stored at 7 °C and 25 °C

henceforth. pH changes were measured three times, and color changes of PSPA film indicators were examined every 24 hours for two days and taken for pictures using the iPhone 6 camera. The image of the application of the PSPA film indicator on food samples can be seen in Figure 1. The distance of the PSPA indicator film with the surface of the milk is about 3 cm, while the distance between the PSPA indicator film with Gindara fillet and sausage was 1 cm.

#### STATISTIC ANALYSIS

In this study, the data from the analysis has been written as the mean  $\pm$  standard deviation. The research data has been processed using Microsoft Excel 2021 and SPSS 21. One-way analysis of variance (ANOVA) was used to determine the significance of variations in anthocyanin extract on film indicators on thickness, tensile strength, elongation, and WVP. Two-way ANOVA to determine the significance of storage time and film indicators on changes in pH in food products on the response of film indicators to pH. The ANOVA used a 95% significance level ( $p = 0.05$ ) and Duncan's test was used to determine the significant difference between each treatment.

## RESULTS AND DISCUSSION

#### FILM THICKNESS

Measuring film indicator thickness was to study the effect of anthocyanin extracts from purple sweet potato on the thickness of the tapioca starch-based film indicators. Thickness was one of the elemental characteristics because affecting WVP and the mechanical properties of film indicators (Qin et al. 2019). The result of the thickness, tensile strength, elongation and WVP of the PSPA film indicators are mentioned in Table 1.

The thickness of all the PSPA film indicator formulations did not differ significantly. The values obtained ranged from 0.72 to 0.74 mm. The addition of anthocyanins to the PSPA film indicator had no significant effect on its thickness because the total volume of film solution had the same amount of 100 mL. In several studies, it was also stated that the film with the addition of anthocyanins did not have a significantly different thickness. This is because the amount of anthocyanin added is small and the anthocyanins are evenly distributed on the entire surface of the film (Abedi-Firoozjah et al. 2022; Chen et al. 2021; Musso, Salgado & Mauri 2016). According to Chen et al. (2021), adding different amounts of red cabbage anthocyanins (RCAs) had little to no effect on the film's thickness.

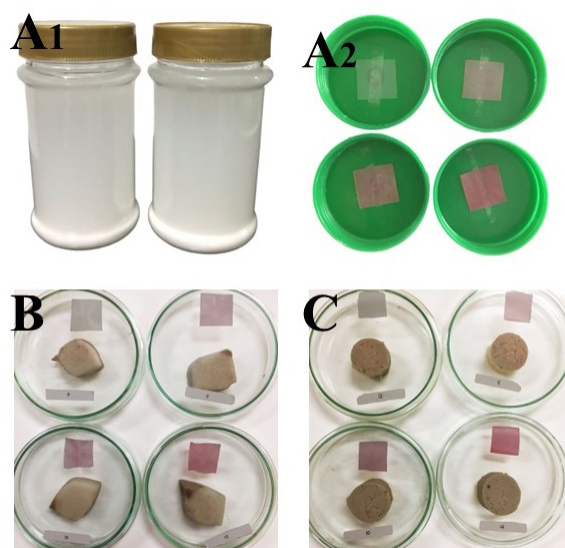


FIGURE 1. Application of PSPA indicator film in (A1) fresh milk, (B) Gindara fish fillet, (C) chicken sausage. (A2) is the appearance of the film indicator on the milk bottle cap

TABLE 1. Physical properties of film indicators

Sample	Thickness (mm)	Tensile strength (MPa)	Elongation (%)	WVP ( $\times 10^{-14}$ kg.m/ $m^2.s.Pa$ )	Water content (%)*
F1	$0.72 \pm 0.002^a$	$9.86 \pm 0.38^d$	$14.83 \pm 0.78^a$	$1.32 \pm 0.07^a$	$14.77 \pm 0.27^b$
F2	$0.74 \pm 0.001^a$	$4.44 \pm 0.02^c$	$32.95 \pm 3.35^b$	$1.78 \pm 0.11^b$	$14.13 \pm 0.13^a$
F3	$0.74 \pm 0.002^a$	$1.80 \pm 0.04^b$	$55.47 \pm 1.81^c$	$1.72 \pm 0.08^b$	$15.11 \pm 0.25^b$
F4	$0.73 \pm 0.002^a$	$1.23 \pm 0.38^a$	$54.81 \pm 6.14^c$	$1.68 \pm 0.04^b$	$15.46 \pm 0.26^c$

The different superscripted letters show a significant difference ( $p < 0.05$ ) in the same column. F1 = control, F2 = film with 5 g extract, F3 = film with 10 g extract, and F4 = film with 15 g extract. \*water content data refer to Rahmadhia et al. (2022)'s research

This is presumably because the RCAs were properly compatible with the chitosan/oxidized chitin nanocrystals composite matrix and were present in very small amounts. Similar to Park et al. (2022)'s findings, there was no discernible variation in the thickness of edible chitosan-based films, which may have been caused by the extract's modest volume and low dry matter content. That is the same as Novita and Rahmadhia (2021)'s study using films made of gelatine and infused with *Muntingia calabura* leaf extract.

#### MECHANICAL PROPERTIES

Tensile strength values of F1 to F4 showed a significant decrease, with each value  $9.86 \pm 0.38^d > 4.44 \pm 0.02^c > 1.80 \pm 0.04^b > 1.23 \pm 0.38^a$  MPa. The highest value was F1 and the lowest was F4. The addition of anthocyanin extracts from purple sweet potato had a significant influence on the tensile strength of film indicators. The decreased tensile strength of film indicators was because of increased volumes of active materials, which in this research were anthocyanin extracts administered to the film indicator matrix, bringing on a weakened interaction between tapioca starch molecules and lessening the strength and stiffness of the films made (Netramai et al. 2020). The impact of intramolecular interaction between anthocyanins and the biopolymer has been observed to cause the tensile strength to decrease with anthocyanin inclusion. The anthocyanins' addition decreased the intramolecular interactions between the biopolymer molecules and increased the structure's disorder, which led to a decrease in the tensile strength (Chi et al. 2020; Ezati et al. 2019; Ge et al. 2020; Lee,

Soloi & How 2021). Furthermore, the lessening could break out by virtue of high amylopectin levels in tapioca starch, disrupting the stability of film indicators made (Nisah 2017).

In the making of tapioca starch-based film indicators, a structure with strong intermolecular forces was formed after gelatinization. And yet, anthocyanins administered to the film matrix could attenuate the forces, impacting the mechanical properties of the films made (Prietto et al. 2017). The same phenomenon was argued in Zhai et al. (2017) as well, that administering anthocyanin extracts (30, 60, 120 mg/100 g starch) from rosella flowers to potato starch-based films caused the tensile strength of the control group to reduce from 48.97MPa to 41.85MPa, considering that the administration could interfere with molecular interactions in polymers. In research Prietto et al. (2017), the tensile strength of corn starch-based pH sensors decreased from 1.2MPa to 0.8MPa due to the administration of anthocyanin extracts from black beans. The research pointed out that increases in percent elongation or extension of PSPA film indicators were proportional to increased anthocyanin extract levels administered. When the film is generated exclusively with starch, after the gelatinization, the molecules form a three-dimensional network ordered with intermolecular connections. However, the presence of anthocyanins might impair the intermolecular connections and so influence the mechanical characteristics of the films (Buchweitz, Carle & Kammerer 2013; He et al. 2016).

Elongation values of F1 to F4 showed a significant increase, with each percentage  $14.83 \pm 0.78^a$  (F1) <

$32.95 \pm 3.35^b$  (F2) <  $54.81 \pm 6.14^c$  (F4) <  $55.47 \pm 1.81^c$  (F3) %. The highest value was F3 and the lowest was F1. The addition of active ingredients can increase the homogeneity of tapioca flour with plasticizers so as to produce films with good extensibility (Zhai et al. 2017). It was because such administration of active compound could promote water molecule reactions during film making and generate intermolecular stretching in the film matrix tissue, decreasing film fragility, which increased percent elongation (Li et al. 2021; Warkoyo et al. 2014).

Additionally, glycerol administration as a plasticizer to a film matrix could produce a more extended hydrogen bond and diminish the attractive forces of molecules on the polymer chain, resulting in a film with high flexibility (Maran et al. 2013). Augmented percent elongation in a film indicator was conveyed in research Zhai et al. (2017), too. In the research, the percent elongation of the control group films, which was 44.15%, was elevated to 88.28% and proportional to anthocyanin extract volumes from rosella added to the film indicator formulation. The same phenomenon was also shown off in research by Prietto et al. (2017), that elongation was enhanced after the administration of anthocyanin extracts from black beans (the initial elongation of the control group films was 78.3%, and it escalated to 88.8%).

Elongation would increase since it was inversely proportional to film tensile strength. It was commensurate with this research's analysis of tensile strength and elongation. In addition, administering anthocyanin extracts could debilitate hydrogen molecule structures in films, and the effects of anthocyanin plasticity with water could induce less tensile strength and more elongation (Prietto et al. 2017).

#### WATER VAPOR PERMEABILITY

Water Vapor Permeability (WVP) of PSPA film indicators significantly ( $p < 0.05$ ) increased after the administration of anthocyanin extracts from purple sweet potato. The least WVP was  $(1.32 \times 10^{-14})^a$  kg.m/m<sup>2</sup>.s.Pa (F1) while the highest was  $(1.78 \times 10^{-14})^b$  kg.m/m<sup>2</sup>.s.Pa (F2). The advanced statistical analysis using Duncan's multiple range test signified that F1 significantly differed from F2, F3, and F4, whereas F2, F3, and F4 did not.

Increases in WVP of F2, F3, and F4 from F1 (control) came about as administering anthocyanin

extracts from purple sweet potato could damage the structure of molecules making up films between starch and glycerol and homogeneity of the tapioca starch-based films and constituted pores on the film surface, allowing films to absorb water molecules in the packaging environment efficiently (Luchese et al. 2018; Zhang et al. 2020). A greater WVP should be investigated in order to develop the film as an indication. For monitoring the degradation of food goods, the films should have a high volatile permeation ability. The addition of the hydrophilic anthocyanin extract may have contributed to the rise in WVP for the pH indicator films. Polyphenolic compounds in purple sweet potato extract may have resulted in a decrease in intermolecular connections in the film network. Thus, the hydroxyl groups of anthocyanin extract molecules may form hydrogen bonds with water and intervene in the network, increasing water vapor permeability (Acevedo-Fani et al. 2015; Sai-Ut et al. 2021).

The phenomenon was congruent with the research (Zhang et al. 2020), that corn starch-based pH indicators demonstrated an increased WVP from  $20.8 \times 10^{-11}$  g.m/m<sup>2</sup>.s.Pa (control) to  $23.2 \times 10^{-11}$  g.m/m<sup>2</sup>.s.Pa after anthocyanin extracts 0,5 and 1% (w/v) of purple sweet potato were administered. Furthermore, the tapioca starch-based pH indicators increased WVP from  $23 \times 10^{-11}$  g.m/m<sup>2</sup>.s.Pa (control) to  $23.2 \times 10^{-11}$  g.m/m<sup>2</sup>.s.Pa after the addition of blueberry residue powder (Luchese et al. 2018).

In our previous published research, the PSPA film has a water content value that increases with the addition of anthocyanin extract. The water content values from F1 to F3, respectively, were  $14.13 \pm 0.13^a$ %;  $15.11 \pm 0.25^b$ %; and  $15.46 \pm 0.26^c$ % (Rahmadhia et al. 2022). Water levels could deflect water vapor absorption in films. The lower the water levels, the lower the floating capability of film indicators on grounds of lower water vapor diffusion (Benbettaïeb et al. 2014). The results were same with argument Benbettaïeb et al. (2014), that the higher the water levels, the higher the WVP. In this research, F1, with a thickness of 0.72mm and a water level of 14.77%, had WVP of  $1.32 \times 10^{-14}$ kg.m/m<sup>2</sup>.s.Pa, while F4, with thickness of 0.73 mm and a water level of 15.46% had WVP of  $1.68 \times 10^{-14}$ kg.m/m<sup>2</sup>.s.Pa (Table 1). Furthermore, WFP was also affected by a range of factors, such as hydrophilic and hydrophobic compounds, pores on the surface, damaged or debilitated structures, and crystallized starch owing to amylose levels (Andretta et al. 2019; Luchese et al. 2018; Versino & García 2014).

THE RESPONSE OF THE FILM INDICATOR TO THE FOOD  
SAMPLE'S pH

























The analysis of response changes of PSPA film indicator was carried out as a simulation and aimed to research color change responses of purple sweet potato-based film indicators to pH changes of food products in containers. Table 2 points out response changes of film indicators from purple sweet potato and pH measurement of fresh cow milk during storage.

Based on observations using the sense of sight in fresh cow's milk stored at 7 °C for 48 h, the PSPA indicator film did not experience a significant color change in each formulation because low temperatures can prevent spoilage due to microorganism metabolism and enzymatic activity. However, PSPA as an indicator of the F4 sample film stored at 25 °C at 48 h of observation gave a color change response from pink to a more concentrated red. Milk that was stored at room temperature will more quickly degrade its nutritional content which causes color changes in smart packaging (Kuswandi et al. 2020; Zhao et al. 2022). The color change of PSPA indicator film in this study was in line with research by Ramadhan and Rusdianto, (2021) which showed that purple sweet potato smart packaging changed from dark purple to pink

due to changes in pH to acid caused by spoilage of milk stored at 27 °C.

The pH of milk gradually fell as storage time increased, owing to the formation of lactic acid from microbial activity. In general, milk was deemed fresh when the pH obtained from the experiment at various storage intervals was 0 h (pH 6.62) or 24 h (pH 6.53) (Gao et al. 2022; Ma et al. 2017). This statement was in accordance with this study that at 0, the pH of milk ranged from  $6.63 \pm 0.02$  to  $6.94 \pm 0.02$  at 7 °C and 25 °C storage temperatures. Meanwhile, at 24 h, the pH of milk at 7 °C storage ranged from  $6.54 \pm 0.02$  to  $6.88 \pm 0.01$ . However, at 25 °C, the pH of cow's milk was detected to be lower than 6.53, namely  $5.23 \pm 0.02$  to  $5.98 \pm 0.02$ . Based on this theory, in this study, cow's milk stored at 7 °C at 0 and 24 h could still be categorized as 'fresh', while at 25 °C the freshness of cow's milk decreased at 24 h storage. These beneficial phenomena for milk freshness monitoring were consistent with Buchweitz, Carle and Kammerer (2013) claim that proteins were better suited for coloring with anthocyanin chelates. Furthermore, milk contains protein, which might interact with anthocyanins. It was also observed that some compounds, such as proteins, had an effect on the excited states of

TABLE 2. Response changes of film indicators from purple sweet potato and pH of fresh cow milk

Sample Time	F1		F2		F3		F4	
	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C
0 h								
	$6.94 \pm 0.02^{3d}$	$6.66 \pm 0.01^{3d}$	$6.72 \pm 0.01^{3c}$	$6.64 \pm 0.02^{3a}$	$6.66 \pm 0.02^{3c}$	$6.67 \pm 0.02^{3c}$	$6.63 \pm 0.02^{3b}$	$6.70 \pm 0.01^{3d}$
24 h								
	$6.75 \pm 0.01^{2d}$	$5.84 \pm 0.01^{2d}$	$6.88 \pm 0.01^{2c}$	$5.23 \pm 0.02^{1a}$	$6.54 \pm 0.02^{2a}$	$5.91 \pm 0.01^{2c}$	$6.60 \pm 0.01^{2b}$	$5.98 \pm 0.01^{2b}$
48 h								
	$5.78 \pm 0.02^{1d}$	$5.29 \pm 0.02^{1d}$	$5.80 \pm 0.01^{1c}$	$5.27 \pm 0.02^{1a}$	$5.74 \pm 0.01^{1a}$	$5.12 \pm 0.01^{1c}$	$5.80 \pm 0.01^{1b}$	$4.84 \pm 0.01^{1b}$

The green color in the sample is the effect of packaging lid, and the F1 sample color is white. The different superscripted number shows a significant difference ( $p < 0.05$ ) in pH between time in the same column. The different superscripted letter shows a significant difference ( $p < 0.05$ ) in pH between sample in the same column. F1 = control, F2 = film with 5 g extract, F3 = film with 10 g extract, and F4 = film with 15 g extract

























chromophores, which might modify the features of UV-Vis absorption spectra to higher or lower wavelengths, resulting in the color of indicator films changing (Buchweitz, Carle & Kammerer 2013; He et al. 2016; Madeira et al. 2019). Table 3 shows response changes of film indicators from purple sweet potato to pH changes of Gindara fish fillet, and pH changes of Gindara fish fillet during storage.

Samples of PSPA indicator films F1, F2, F3, and F4, did not respond to a significant color change when applied as an indicator to Gindara fish fillet at a temperature of 7 °C. Meanwhile, when Gindara fish fillet was stored at 25 °C, the PSPA indicator film F1 did not respond to color changes. However, samples F2 and F3 at 25 °C experienced a color change from red to light blue, while in sample F4 the color changed to become faded. Similarly, Ezati et al. (2019) found that film indication held at a low temperature (4 °C) showed the best color stability, demonstrating the importance of starch (solid support) in preserving dye for a long period. pH-

sensitive films based on starch were reported to be very stable at refrigerator temperature (Prietto et al. 2017; Sinela et al. 2017). Although some indicator films did not change color, the Gindara fish fillet experienced a significant increase in pH along with the length of storage time at both 7 °C and 25 °C ( $p < 0.05$ ). Then when stored at 7 °C, the pH value did not change significantly to the film formulation, whereas when stored at 25 °C the overall pH value tended to increase significantly ( $p < 0.05$ ).

The research from Alizadeh-Sani et al. (2021) indicates that, after 72 h, the pH of the lamb flesh increased dramatically from 5.8 to 7.5. The rise in pH led the color indicator film based on methylcellulose/chitosan nanofiber and barberry anthocyanins to change from reddish to light peach, indicating that the lamb had degraded during storage. The breakdown of protein by microbes and enzymes was the primary cause of meat and seafood deterioration. Protein breakdown produces volatile nitrogenous chemicals such as ammonia and amines, which raise the pH of the package (Ezati et al. 2019; Huang et al. 2019).

TABLE 3. Response changes of film indicators from purple sweet potato and pH of Gindara fish fillet

Sample Time	F1		F2		F3		F4	
	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C
0 h								
	5.78 ± 0.01 <sup>1a</sup>	5.92 ± 0.01 <sup>1a</sup>	5.80 ± 0.01 <sup>1ab</sup>	5.95 ± 0.01 <sup>1c</sup>	5.77 ± 0.01 <sup>1b</sup>	5.99 ± 0.05 <sup>1b</sup>	5.74 ± 0.01 <sup>1ab</sup>	6.06 ± 0.00 <sup>1d</sup>
24 h								
	5.85 ± 0.03 <sup>2a</sup>	6.14 ± 0.01 <sup>2a</sup>	5.91 ± 0.02 <sup>2ab</sup>	6.12 ± 0.02 <sup>2c</sup>	5.90 ± 0.01 <sup>2b</sup>	6.09 ± 0.01 <sup>2b</sup>	5.92 ± 0.02 <sup>2ab</sup>	6.10 ± 0.02 <sup>2d</sup>
48 h								
	6.05 ± 0.03 <sup>3a</sup>	7.13 ± 0.06 <sup>3a</sup>	5.99 ± 0.02 <sup>3ab</sup>	7.30 ± 0.00 <sup>3c</sup>	6.07 ± 0.01 <sup>3b</sup>	7.20 ± 0.00 <sup>3b</sup>	6.05 ± 0.01 <sup>3ab</sup>	7.40 ± 0.00 <sup>3d</sup>

The different superscripted number shows a significant difference ( $p < 0.05$ ) in pH between time in the same column. The different superscripted letter shows a significant difference ( $p < 0.05$ ) in pH between sample in the same column. F1 = control, F2 = film with 5 g extract, F3 = film with 10 g extract, and F4 = film with 15 g extract



























The main factors of spoilage are microorganisms and enzymes in fish which can even carry out metabolism and work at low temperature storage (Rahman et al. 2016). The high-water content and the high amount of nitrogen compounds will accelerate the growth of spoilage microorganisms such as *Shewanella putrefaciens*, *Pseudomonas* sp., *Photobacterium phosphoreum*, *Vibrionaceae*, *Enterobacteriaceae*, and lactic acid bacteria causing unwanted physical changes that result in the production of amines and acids, organic compounds, alcohols, sulphides, ketones, and aldehydes formed from free amino acid decarboxylation. These conditions cause off-flavors in fish and changes in pH to alkaline or alkaline pH (Devarayan et al. 2020; Silbande et al. 2018; Tavares et al. 2021). Decomposition by microorganisms can increase the pH of fish to 7.0-8.0 even more if there is very severe decay. Based on observations using the sense of sight, Gindara fish filet stored at 7 °C for 48 h, intelligent packaging of purple sweet potato did not experience significant color changes in each formulation. Response changes of film indicators from purple sweet potato and pH measurement in chicken sausage is demonstrated in Table 4.

Samples of PSPA indicator films F1, F3, and F4, did not respond to color changes when applied as an indicator to chicken sausage at a temperature of 7 °C while when stored at 25 °C, F1, F2, F3 also did not respond to color changes that were so striking, but at F4 undergoes a color change to a more intense red. Based on research Rahmadhia et al. (2022), the appearance of a darker red color response to the PSPA indicator film indicates a decrease in pH (to become acidic).

In contrast to Gindara fish fillet, which experienced an increase in pH, the pH of chicken sausage decreased significantly with increasing storage time at 7 °C and 25 °C. The trend of decreasing pH in chicken sausages occurred in all PSPA indicator film applications (F1 - F4). Glycogen which turns into lactic acid causes the pH of the meat to decrease (acidic) around 5-4. The process of decaying organic matter will release the carbon contained in it, causing the pH to become acidic. During sausage preservation, intact proteins are often proteolyzed by endogenous enzymes and microorganisms, resulting in tiny peptides, single amino acids, dipeptides, and tripeptides that have a significant impact on meat product quality (Han et al. 2022).

TABLE 4. Response changes of film indicators from purple sweet potato and pH measurement in chicken sausage

Sample Time	F1		F2		F3		F4	
	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C	7 °C	25 °C
0 h								
	6.70 ± 0.00 <sup>1b</sup>	6.57 ± 0.06 <sup>3b</sup>	6.63 ± 0.06 <sup>1c</sup>	6.87 ± 0.06 <sup>3d</sup>	6.67 ± 0.06 <sup>1b</sup>	6.63 ± 0.12 <sup>3b</sup>	6.27 ± 0.06 <sup>1a</sup>	6.40 ± 0.00 <sup>3a</sup>
24 h								
	6.33 ± 0.01 <sup>2b</sup>	6.21 ± 0.01 <sup>2b</sup>	6.36 ± 0.02 <sup>2c</sup>	6.18 ± 0.01 <sup>2d</sup>	6.15 ± 0.01 <sup>2b</sup>	6.16 ± 0.01 <sup>2b</sup>	6.20 ± 0.01 <sup>2a</sup>	6.15 ± 0.01 <sup>2a</sup>
48 h								
	5.63 ± 0.12 <sup>1b</sup>	4.96 ± 0.00 <sup>1b</sup>	5.97 ± 0.06 <sup>1c</sup>	5.01 ± 0.01 <sup>1d</sup>	5.80 ± 0.00 <sup>1b</sup>	5.00 ± 0.01 <sup>1b</sup>	5.70 ± 0.00 <sup>1a</sup>	4.97 ± 0.01 <sup>1a</sup>

The different superscripted numbers show a significant difference ( $p < 0.05$ ) in pH between time in the same column. The different superscripted letters show a significant difference ( $p < 0.05$ ) in pH between sample in the same column. F1 = control, F2 = film with 5 g extract, F3 = film with 10 g extract, and F4 = film with 15 g extract

In this research, film indicators from purple sweet potato acted as pH sensors or indicators which could capture volatile compounds in packaging by virtue of fat oxidation inflicting free radical compounds and amine and ammonia compound formation from protein hydrolysis during storage processes in fresh cow milk, Gindara fish fillet, and chicken sausage (Šojić et al. 2014; Tavares et al. 2021). The color change in the indicator films F2 and F3 to a greenish color when applied to Gindara fillet occurred due to an increase in pH in Gindara fillet. In a study conducted by Rahmadhia et al. (2022) showed that the pH sensitivity of the PSPA indicator film had a pink fading and turned green when the PSP indicator was dipped in a buffer solution. The extract hue was crimson at an acid pH of 5, and purplish pink at a pH of 5-7. Furthermore, the extract hues were blue and yellowish-green at basic pH levels of 8-9 and 10-11, respectively. The quantity of hydroxyl groups connected to anthocyanidin molecules determines its hue. Changes in pH can also produce reversible structural changes in anthocyanin molecules, affecting their color dramatically. The ionic nature of anthocyanins contributes to their pH sensitivity. The structure of anthocyanins changes in acidic solutions to flavylium cation (red) and quinoidal anhydrase (purple). The structure of anthocyanins changes to quinoidal (green) and chalcone in alkaline solutions (yellow) (Sohany et al. 2021; Wahyuningsih et al. 2017).

Anthocyanin compounds as natural color pigments sensitive to pH changes in PSPA film indicators featured significant color response changes due to pH changes of Gindara fish fillet stored at 25 °C 48 hour-long (REF). They indicated color changes from red to blue. Color changes were in light of the structural transformation of anthocyanins as they captured volatile amine compounds formed on account of fish rotting (Lee et al. 2019; Sohany et al. 2021). Besides, color response changes in PSPA film indicators from purple sweet potato also occurred when the film indicators were applied to fresh cow milk and chicken sausage during storage at 25 °C over 48 h. Color changes transpired from red to dark red on grounds of pH lessening. It could take place owing to the formation of volatile compounds from fat oxidation, which posed thiobarbituric acid breeding rancid aroma, causing color changes in samples, from red to dark red.

Response changes to pH, which manifested color changes of film indicators from purple sweet potato, were results of protonation and deprotonation in anthocyanins because of pH changes affected by compounds brought on by food product spoilage (Chen et al. 2019; Choi et al.

2017). During an acid pH condition, anthocyanins would form positive ions, i.e., flavylium cations, which were red. Moreover, during an alkaline or high pH condition, they would experience deprotonation, causing an anhydrous base to form and engendering a blue color (Barnes et al. 2009).

## CONCLUSIONS

Anthocyanin extract from purple sweet potato in film indicators impacted their physical properties. It elevated thickness and elongation, reduced tensile strength, and enhanced WVP. Furthermore, film indicators from purple sweet potato presented response changes by showing color changes because of pH changes of fresh cow milk, Gindara fish fillet, and chicken sausage stored at 25 °C for 48 h.

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\*Corresponding author; email: safinta.rahmadhia@tp.uad.ac.id