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The Impact of Incorporating Varying Amounts of Anthocyanin from Purple Sweet Potato Peel and PVA on the Physical and Mechanical Characteristics of Smart Packaging Film

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Abstract
Smart Packaging Film (SPF) from biopolymer with the addition of anthocyanin extract from purple sweet potato peel (*Ipomoea batatas L. Poir*) as a freshness quality indicator is the main focus of this research. Anthocyanin extract was chosen due to its sensitivity to pH changes, functioning as an indicator of the fish's freshness quality. This research aims to assess the effect on the characteristics of SPF, including the testing of physical properties (thickness, moisture content, and water solubility), and mechanical properties (tensile strength and elongation). The anthocyanin indicator film from purple sweet potato peel was made using corn starch as the biopolymer with the addition of anthocyanin extract at concentrations of 0.5, 1, 1.5, and 2 g and variations of PVA at 1% and 2% (w/v). The results showed a thickness of 0.12 – 0.19 mm, moisture content of 14.58 - 18.93%, water solubility of 46.76 – 55.15%, tensile strength of 1.193 – 2.926 MPa, and elongation of 89.423 – 96.822%. The results of this research are expected to contribute to the development of innovative smart packaging technology and provide solutions to food safety issues with an environmentally friendly approach.

Keywords: Anthocyanin, pH Indicator, *Smart Packaging Film*

Introduction

Smart packaging film is a packaging technology equipped with natural or synthetic dyes incorporated into a polymer-based film. Synthetic dyes are sensitive to pH changes, but their use has some disadvantages, such as easy degradation, being inedible, and potentially toxic [1]. The search for alternative materials that can be used as indicators has led to the use of natural dyes. Some examples of natural dyes include anthocyanins [2], curcumin [3], betalains [4], chlorophyll [5], and carotenoids [6]. Anthocyanins are one of the most widely used natural dyes in smart packaging films [7]. Anthocyanins are a group of phenolic compounds belonging to the flavonoid family, and they provide red, orange, blue, and purple colors in flowers, fruits, vegetables, and leaves [8]. Anthocyanins are highly sensitive to pH changes, as indicated by different colors in acidic, neutral, and alkaline conditions [9].

The potential source of anthocyanin as a natural dye is purple sweet potato. This vegetable belongs to the *Convolvulaceae* family and is widely cultivated in tropical and subtropical countries, including Indonesia. In 2020 approximately 89.5 million tons of sweet potatoes were cultivated across approximately 7.4 million hectares worldwide [10]. In Indonesia, about 89% of sweet potato production is used as food, with a consumption rate of around 7.9 kg/capita/year. At the same time, the remaining portion is used as raw materials for industries, especially for sauces and animal feed production. However, this usage primarily focuses on the flesh of the tuber, while the peels are rarely utilized, becoming waste that is discarded without further processing. The anthocyanin content in sweet potatoes depends on the intensity of the tuber's color [11]. The anthocyanin content in sweet potatoes counts on the intensity of the tuber's color. The more purple the color, the higher the anthocyanin content. Based on research by [12], boiled purple sweet potatoes contain 696.1 mg of anthocyanin per 100 g, with about 77% of the anthocyanin compounds being peonidin derivatives. Research by [8] also reported that the anthocyanin content in purple sweet potato extract is 311 mg per 100 g.

Some of the biopolymers used in the creation of smart packaging films include starch [13], gelatin [14], chitosan [15], and cellulose [16]. Among these materials, starch is the most promising for use as a film matrix due to its high stability to heat, acidic and alkaline conditions, and ease of solubility in water. This study uses corn starch as the film matrix because corn starch has lower hygroscopicity, around 11%, compared to cassava starch at 13%, rice starch at 14%, and potato starch at 18%. Furthermore, corn starch has an amylose content of 27% compared to 22% in potato starch and 17% in cassava starch. Amylose contributes to the flexibility and strength of film in SPF formulation [17]. However, starch-based smart packaging films have some drawbacks, such as low mechanical strength [18]. Thus, adding ther biodegradable materials is necessary to address these physical and functional properties. One alternative material that can be used is polyvinyl alcohol (PVA), a hydrophilic, water-soluble, non-toxic, and biocompatible biodegradable polymer. The combination of starch/polyvinyl alcohol (SPVA) in smart packaging films (SPF) can produce films with good transparency [19], and they are non-toxic, renewable, and biodegradable [20].

In previous research [21], developed SPF from chitosan-anthocyanin derived from purple sweet potato peel with varying mass. Based on that study, the physical properties of the film, such as color, thickness, moisture content, and solubility, were influenced by the concentration of anthocyanin added. As the anthocyanin concentration increased, the transparency and thermal stability improved, while the water vapor barrier and tensile strength decreased [22]. developed SPF from nanofiber-anthocyanin-essential oil. The incorporation of this film composite enhanced pH sensitivity and transparency but reduced its tensile strength and elasticity. Research by [23] developed SPF based on potato starch/ PVA /anthocyanin from purple sweet potato/limonene. The results showed that the addition of anthocyanin and limonene concentrations improved the mechanical properties of the film. In that study, the potato starch/PVA/anthocyanin film from purple sweet potato/limonene could prevent milk spoilage. Furthermore, research by [24] developed SPF from carboxymethyl cellulose/starch/anthocyanin. In that study, the addition of anthocyanin concentrations increased tensile strength and thickness, while decreasing elasticity, moisture content, and light transmission. The application of this research demonstrated that the film could monitor fish freshness in real-time.

Based on the previous studies mentioned above, there is potential for developing anthocyanin as an interactive indicator for SPF with varying anthocyanin and PVA mass added to corn starch biopolymer, as well as the characterization of the physical and mechanical properties of the film.

Experimental

Materials and chemical reagents

Purple sweet potato peels were sourced from Trawas, Mojokerto. Corn starch was sourced from the Mergan market in Malang, East Java, Indonesia. Ethanol (96%) was supplied by Smart-Lab, Indonesia. Glycerol (99%), polyvinyl alcohol, HCl, were acquired from Merck, Germany. All other chemicals used were of analytical grade.

Instrumentals

The experiment instruments used in this research are an instance scale, thermometer, porcelain dishes, glassware, pH indicators, electric heater, rotary evaporator, volume pipette, magnetic stirrer, desiccator, and oven. The tools for analysis and testing are a tensile strength and elongation test, micrometer, spectrometer FTIR, and UV-Vis spectrophotometer.

Anthocyanin Extraction from Purple Sweet Potato Peel (*Ipomoea batatas L. Poir*)

Anthocyanin extraction from purple sweet potato peel was carried out using the method by [22] with some modifications. The purple sweet potato peel was washed under running water. A 160 g of purple sweet potato peel was cut into small pieces and blended. The blended peel was diluted with 360 mL of 96% ethanol and 40 mL of 1% acetic acid. The mixture was then macerated for 2 x 24 hours at room temperature. The liquid extract was then evaporated using a rotary evaporator to remove ethanol from the filtrate at 50°C. The result from the rotary evaporator was a concentrated extract.

Preparation of Corn Starch and PVA Film as Control

Corn starch (2% w/v) and varying PVA concentrations (1% w/v, 2% w/v) were added to 100 mL of distilled water. The mixture was heated on a hotplate with a magnetic stirrer at 80°C and 500 rpm for 1 hour until a gel formed. Glycerol (1% w/v) was then added to the film mixture, and homogenized for 1 hour. The resulting gel was cast onto a glass plate and dried for 24 hours at 40°C using an oven.

Preparation of Corn Starch and PVA Film with Added Purple Sweet Potato Peel Extract (PSPPE)

Concentrated purple sweet potato peel extract was mixed with 100 mL of distilled water. This mixture was stirred at 50°C for 30 minutes in a 250 mL Pyrex glass beaker on a magnetic stirrer at 500 rpm. Starch (2% w/v), varying PVA concentrations (1% w/v, 2% w/v), and glycerol (1% w/v) were added to the pure liquid extract of purple sweet potato peel, then dissolved at 80°C using a magnetic stirrer at 500 rpm for 2 hours until gelatinization occurred. The resulting gel was then dried for 24 hours at 40°C using vacuum oven drying. Before characterizing the samples, all dried films were stored in a desiccator with 50% relative humidity. Samples were prepared with varying masses of PSPPE extract: 0.5, 1, 1.5, and 2 grams, each in pure starch and PVA.

Physical Properties of Corn Starch/PVA-Purple Sweet Potato Peel Films

Thickness and Moisture Content

The thickness of the film was measured using a digital micrometer, randomly selecting five points on each film with two replications. Each purple sweet potato peel film indicator was cut into a 5 × 5 cm size, and the thickness was measured using a digital micrometer (Mitutoyo, Japan) at five random points: Top right, top left, center, bottom right, and bottom left, and the average sample thickness (mm) was determined. The moisture content of the film was determined by drying the film at 105°C until a constant weight was achieved.

$$\text{Moisture content (\%)} = \frac{(M_i - M_t)}{M_i} \times 100 \qquad (3.1)$$

Where Mi and Mt are the initial and final weights of the film sample respectively.

Mechanical Properties of Corn Starch/PVA-purple Sweet Potato Peel Films

Tensile strength and Elongation

For the measurement of mechanical properties, film strips (6 cm × 1 cm) are analyzed using TMS-Pro with an initial gauge length of 4 cm and a test speed of 1 mm/s. The tensile strength (TS) and elongation at break (EAB) of the film samples are calculated as follows:

$$\text{Tensile strength} = \frac{F}{(X \times W)} \times 100 \quad (3.3)$$

$$\text{Elongation (\%)} = \frac{\Delta L}{L_0} \times 100 \quad (3.4)$$

Where F is the maximum tensile force (N), X is the thickness of the film (mm), and W is the width of the film (mm); ΔL and L_0 are the extended and original lengths (mm) of the film sample, respectively.

Results and Discussion

SPF (Smart Packaging Film) Preparation

The preparation of the smart packaging film begins by weighing 2 gr of corn starch (C), PVA with variations of 1 gr (P1) and 2 gr (P2), 1 gr of glycerol, and anthocyanin dye with variations of 0.5 gr (A1), 1 gr (A2), 1.5 gr (A3), and 2 gr (A4). The weighed materials are then dissolved in 100 mL of distilled water at a temperature of 80°C. This temperature is chosen because at 80°C, polymers such as starch and PVA (Polyvinyl Alcohol) can dissolve and mix better, ensuring an even distribution of each film component [25]. The dissolution process uses a magnetic stirrer at a speed of 500 rpm. The speed of 500 rpm is used as it sufficiently ensures that the mixture of materials such as starch, PVA, and bioactive components like anthocyanins is homogenized evenly. Good homogenization is crucial for enhancing the mechanical properties and functionality of the film [26]. The film preparation process is carried out for 2 hours until gelatinization occurs. The resulting gel is then cast onto a glass plate and dried for 24 hours at 40°C in an oven.

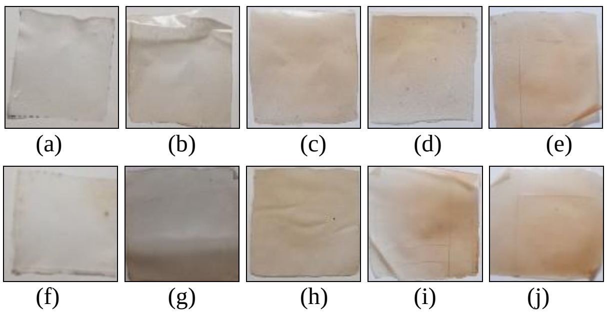

Fig 1: CP2 (a), CP2A1 (b), CP2A2 (c), CP2A3 (d), CP2A4(e), CP1(f), CP1A1 (g), CP1A2 (h), CP1A3 (i), CP1A4 (j) film, which C (corn starch), P (PVA), A (anthocyanin)

Physical Characteristics of Smart Packaging Film (Thickness, Water Content)

Thickness is one of the fundamental characteristics of a film that can influence the physical properties of an indicator film (Qin *et al*., 2019). The results of thickness measurements are shown in Fig 1, where the thickness of CP films ranges between 0.10-0.11 mm, and CPA films measure 0.12-0.19 mm. Based on these measurements, CPA films have a greater thickness compared to CP films. This is because the addition of anthocyanin mass affects the film's thickness; the higher the concentration of purple sweet potato peel extract, the thicker the resulting film. This finding aligns with the research by [27], which also reported an increase in thickness due to the addition of anthocyanin extract.

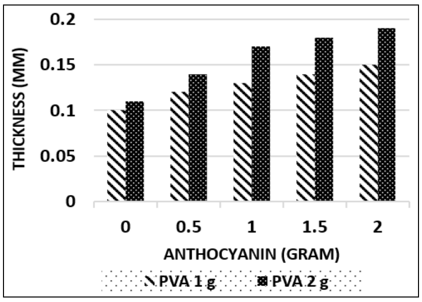

Fig 2: Film thickness of Corn Starch/PVA-Purple Sweet Potato Peel Films

In this study, the increased film thickness is attributed to molecular interactions between anthocyanin and polymers like starch and PVA. These interactions form hydrogen bonds between the hydroxyl groups of anthocyanin and the polar groups of the polymers (such as -OH in starch and PVA) [28]. Additionally, adding a certain amount of anthocyanin extract increases the total solids in the solution, thus increasing the film's thickness [29]. The addition of PVA to the SPF film also influences its thickness. As shown in Fig 1, the greater the amount of PVA added, the thicker the film becomes. This is because more hydrogen bonds are evenly distributed throughout the film, increasing its thickness. In this study, the addition of PVA enhances the physical properties of the film.

Moisture content refers to the percentage of water present in packaging materials, which can affect their functional properties. Moisture content is usually measured to ensure that the packaging remains effective in preserving product quality, preventing damage caused by microbial growth, and extending the product's shelf life. An ideal moisture content can help optimize the interaction between the packaging material and active components, such as anthocyanin extract, which may play a role in detecting changes in the quality of the packaged product [21]. The ideal moisture content for smart packaging ranges from 10% to 20% [30].

The moisture content test results for CP films, as shown in Fig 2, are in the range of 14-15%, while the results for CPA films range from 15-18%. The figure indicates that anthocyanin extract affects the moisture content of the films. The more anthocyanin extract added to the film, the higher the moisture content. This increase in moisture content is due to the hydrophilic nature of anthocyanin. Anthocyanin has a molecular structure rich in hydroxyl groups (-OH), which are polar and can form hydrogen bonds with water molecules. As the concentration of anthocyanin in the film increases, the number of hydroxyl groups available to bind water also increases, causing the film to bind and retain more water molecules from the environment, thereby increasing the moisture content [31].

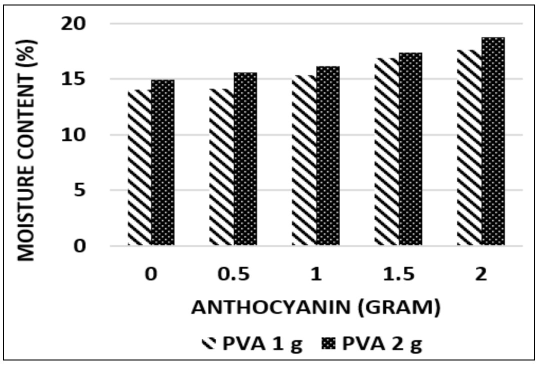

Fig 3: The moisture content of Corn Starch/PVA-Purple Sweet Potato Peel Films

The hydrogen bonds in anthocyanin can also bond with hydroxyl groups in polymers like starch and PVA. This bonding results in a more complex matrix structure within the film, creating micro-spaces that enhance the film's capacity to absorb and retain water, thus increasing its moisture content (Liu *et al.*, 2019). In this study, the addition of PVA mass to the SPF film influenced the moisture content of the film. The moisture content of the film was found to increase in proportion to the amount of PVA used. Theoretically, PVA is a polymer with a simple chemical structure containing irregular hydroxyl groups, which form hydrogen bonds with water molecules. Thus, the more PVA added to the film, the greater the film's ability to absorb and retain water [32].

Mechanical Properties of Smart Packaging Film (Tensile Strength and Elongation)

Tensile strength or tensile rupture strength is the maximum force that can be applied to a film before it breaks or tears. The results of this study show that the more anthocyanin extract added, the higher the tensile strength value. This occurs due to the even distribution of anthocyanin within the film matrix. Uniform distribution during the mixing process can eliminate weak points that could serve as the initial cause of cracking or breaking when the material is subjected to tension. With the anthocyanin particles evenly dispersed, the material has a more homogeneous structure, which increases the film's ability to withstand mechanical stress (tensile strength). The uniform distribution from the mixing process can also enhance the hydrogen bonding (-OH) between anthocyanin and the hydroxyl groups in the starch/PVA biopolymer. These bonds strengthen the film matrix's structural network, making the film's polymer network denser, resulting in a mechanically stronger material [33].

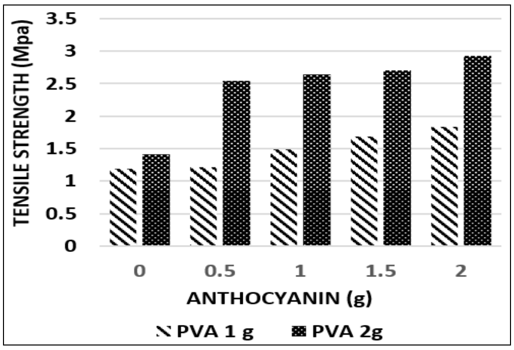

Fig 4: Tensile strenght of Corn Starch/PVA-Purple Sweet Potato Peel Films

The tensile strength test results for the 1-g PVA variation (CP1) showed an increase from 1.212 MPa to 1.835 MPa, while for the 2 g PVA variation (CP2), it increased from 2.541 MPa to 2.926 MPa as more anthocyanin extract was added. The 2-gram PVA variation (CP2) showed higher tensile strength compared to the 1-gram PVA variation (CP1). This is because the higher the PVA content, the longer the polymer chains in the film matrix. Longer polymer chains increase the number of hydroxyl groups in the film, resulting in more hydrogen bonds (-OH) forming within the film, which consequently increases its tensile strength [34].

Elongation or stretch refers to the material's ability to extend when subjected to tensile force until it breaks. Elongation is defined as the ratio of the length change to the initial length of the material undergoing deformation. Elongation test results in this study show that for the 1-g PVA variation (CP1), elongation increased with the addition of anthocyanin extract. The value increased from 93.9% to 95.82%. This is because anthocyanin can form hydrogen bonds with the matrix components. These hydrogen bonds can disrupt the cohesive molecular network in the film, reducing the tensile strength of the film due to weaker intermolecular bonds. However, on the other hand, the film becomes more flexible, as these bonds increase the elasticity of the film, reflected in the increased elongation value [35]. For the 2-g PVA variation (CP2), the elongation results decreased as more anthocyanin extract was added. The elongation value dropped from 91.99% to 89.42%. The reduction in elongation in this variable is due to the increased use of PVA, which results in more hydrogen bonds (-OH) forming with the hydroxyl groups in the starch/PVA molecules, strengthening the film structure. These bonds make the film stiffer, which increases its tensile strength but reduces its elasticity, resulting in a lower elongation value [36].

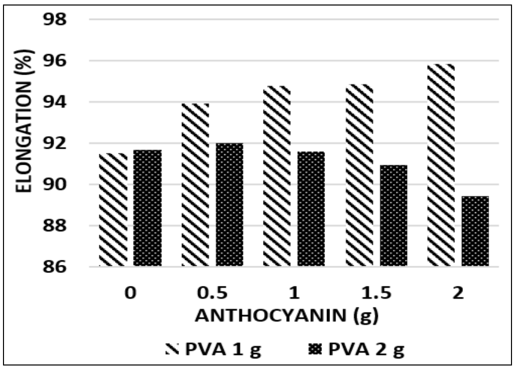

Fig 5: Elongation of Corn Starch/PVA-Purple Sweet Potato Peel Films

Conclusion

Natural dye indicators were extracted from purple sweet potato peel and used to fabricate smart packaging films. The addition of anthocyanin mass from purple sweet potato extract affects the physical and mechanical properties of the film. The higher the mass of anthocyanin extract and PVA added to the film, the higher the tensile strength, moisture content, and thickness. However, the elongation value decreases when larger amounts of PVA are added. The abundance of hydroxyl groups in *Ipomoea batatas* extracts forms hydrogen bonds with the hydroxyl groups in starch, PVA, and anthocyanin from purple sweet potato peel.

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