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Short Communication

Use of Fruit Waste as Natural Dyes in pH-Sensitive Colorimetric Sensors for Tilapia Fillets Quality Decline

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Abstract

Tilapia fillet aimed to prolonging shelf life, may still experience quality deterioration posing food safety risks. Colorimetric pH indicators offer a simple and affordable solution to the food industry to evaluating fish spoilage. Natural dyes reduce potential health risks associated with synthetic dyes. Anthocyanin sources which haven't been explored like dragon fruit rind, mangosteen rind, and red onion skin serve as real time quality and safety assessment tools for consumers. This study explores the potential of anthocyanin extracted from dragon fruit rind, mangosteen rind, and red onion skin as colorimetric sensors for evaluating the quality and safety of tilapia fillets. The anthocyanin-based sensors developed using kappa carrageenan and corn starch polymers to achieve sustainability fisheries program and were characterized according to the Japanese Industrial Standard for bioplastics. The results show that anthocyanin from dragon fruit skin exhibited the best color change in response to pH changes in the tilapia fillet, indicating its potential as a reliable indicator of spoilage. This research highlights the feasibility of using natural dyes as colorimetric sensors, reducing the risk of health hazards associated with synthetic dyes. This study also shows that different polymers give different characteristic of bioplastic. Carrageenan bioplastic shows best thickness values of 0,118 mm; Carrageenan and corn starch bioplastics show best tensile strength values of 20,62 MPa; Carrageenan bioplastic shows best elongation values of 254%; and all polymers show the same biodegradation rate values of 14,29%. Further studies are needed to explore other natural dyes and optimize the polymers for optimal bioplastic characteristics.

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1. Introduction

Tilapia is an Indonesian aquaculture commodity with large production volumes, second most abundant after seaweed, with a production value of 1,374,230 tons in 2019 (Central Bureau of Statistics, 2022) and a temporary amount of 401,767 tons in 2022 (Ministry of Marine Affairs and Fisheries, 2023), with the highest demand in the market domestically being fresh tilapia and in the United States market being tilapia in fillet form (Hadiroseyani *et al*., 2023), as supported by data from National Oceanic and Atmospheric Administration (NOAA) showing 89,104 tons of tilapia imports in the first half of 2022. Tilapia fillets, as a processed food product designed to extend shelf life, may still experience a decline in quality due to microbial growth and biochemical reactions. These reactions can reduce shelf life and increase food safety risks, prompting the food industry to seek simple, inexpensive, and non-destructive methods to evaluate freshness (Aghaei *et al*., 2020). Colorimetric pH indicators have been used in smart packaging due to their practical, simple, and inexpensive nature. These indicators are integrated into food packaging to evaluate fish spoilage and typically employ synthetic dyes (Amorim *et al*., 2022; Liu *et al*., 2022). Using natural dyes in food products reduces the risk of potential hazards associated with synthetic dyes, such as genotoxicity, carcinogenicity, allergenicity, and mutagenicity (Berlina *et al*., 2019). Natural dyes like anthocyanin, which can be found in various plants with a range of colors from red to purple, have been identified as a potential alternative (Sankaralingam *et al*., 2023) Anthocyanin is a flavonoid compound with antioxidant properties dissolves in polar solvents. It has maximum absorption UV-Visible wavelength of 490-550 nm and can undergo significant color changes influenced by pH, temperature, and light (Aprillia *et al*., 2020).

The issue of waste disposal is a common problem faced by both developed and developing countries, including Indonesia. Organic waste, such as fruit peels, is often left unmanaged and can lead to unpleasant odors and health hazards. Therefore, the use of natural dyes like anthocyanin from fruit rinds and peels not only increases its value but also utilizes it for something more useful. Smart packaging films containing natural colorants are proposed as a suitable alternative for consumers to assess food quality and safety in real-time (Alizadeh *et al*., 2020). Anthocyanin-based pH-sensitive smart packaging films are made using anthocyanin-rich extracts from natural sources such as dragon fruit rind, mangosteen rind, and red onion skin, which act as pH indicators. These natural sources change color in response to pH changes, making it suitable for colorimetric pH sensors. The films are environmentally friendly to achieve sustainability fisheries program. In addition, the use of natural sources can improve the physical and mechanical properties of composite films (Zeng *et al*., 2023). Previous research has been done by Guo *et al*. (2024) and Wu *et al*. (2024) using the same natural dyes but different polymers. The purpose of this article is to identify the best natural dyes with the widest color range to be used in colorimetric sensors with kappa carrageenan and cornstarch-based polymers.

2. Materials and Methods

2.1 Materials

2.1.1 The equipments

The equipments used in this research are a blender (Y45, Moulinex, France); sieve; plastic funnel; electronic scale (Sf-400, China), analytical scale (PA313, Ohaus Pioneer, US); centrifuge (Rotanta 460R, Hettich, Germany); UV-Visible spectrophotometer (N4S, Nanbei, China); micrometer (device); pH meter (WD3541903, Oakton, Singapore); Universal Testing Machine; beaker glass; measuring cup; magnetic stirrer; hot plate; stative clamp; thermometer; oven; silica; dark glass bottle; plastic clip; and square glass mold.

2.1.2 The materials

Materials used in this research are dragon fruit and mangosteen, later, only the rind was taken for experimental needs, red onion skin; double distilled water; tap water; aquadest; ‘Dolphin’ salt; fresh fillet tilapia; Kappa carrageenan from Kappa Carrageenan Nusantara; glycerol; and ‘Maizenaku’ corn starch.

2.1.3 Ethical approval

This study does not require ethical approval because it does not use experimental animals.

2.2 Methods

This research was structured objectively based on literatures with the research topic of colorimetric sensor packaging. The method used in this research is an experimental method with a completely randomized factorial design using two factors, namely the composition of polysaccharide content and the natural dyes. The research design, particularly regarding the content of complex carbohydrates, is based on Mangala’s (2024) study on creating colorimetric sensor packaging using bioplastics with a plasticizer made from used cooking oil. This bioplastic was made by combining 37% kappa carrageenan and 25% corn starch through an extrusion process. This formulation did not provide maximum results with the casting method, so the formulation in Table 1 was used.

Table 1. Research Design

Factor 1. Polysaccharides Composition		Factor 2. Natural Dyes	
A1	Carrageenan 100%	B1	Dragonfruit rind
A2	Carrageenan 50% Corn Starch 50%	B2	Mangosteen Rind
A3	Corn Starch 100%	B3	Red Onion Skin

2.2.1 Extraction method

The extraction of anthocyanins was conducted using a method modified where the process involved blending the source materials (dragon fruit rind, mangosteen rind, and onion skin) with double distilled water (dd water) in a 1:3 ratio. The resulting dye was filtered using a sieve and then centrifuged at 4600 rpm for 30 minutes using a Rotanta centrifuge. The supernatant was collected and analyzed using UV-Vis spectrophotometry to confirm the presence of anthocyanins. Once the anthocyanin content was confirmed, the extract was used to produce pH-sensitive packaging films (da Silva Filipini *et al*., 2020).

Table 2. Composition of Materials for Making Colorimetry Sensors

Treatment	Carrageenan (g)	Corn Starch (g)	Dragonfruit rind dye (ml)	Mangosteen Rind Dye (ml)	Red Onion Skin Dye (ml)	Glycerol (g)	Aquadest (ml)
A1B1	2.5	-	70	-	-	1.8	30
A1B2	2.5	-	-	70	-	1.8	30
A1B3	2.5	-	-	-	70	1.8	30
A2B1	1.25	3.5	70	-	-	1.8	30
A2B2	1.25	3.5	-	70	-	1.8	30
A2B3	1.25	3.5	-	-	70	1.8	30
A3B1	-	7	70	-	-	1.8	30
A3B2	-	7	-	70	-	1.8	30
A3B3	-	7	-	-	70	1.8	30

2.2.2 pH sensitive colorimetric sensors method

Making colorimetric sensor packaging in the form of bioplastic follows the modified theory of making colorimetric sensors by Ismed *et al.* (2018). First, all the ingredients needed are weighed according to the composition of each treatment and can be seen in Table 2. In all treatments, distilled water is initially heated to 40°C on a hotplate. Subsequently, carrageenan, corn starch, glycerol, and dye are added to the water in a beaker, and the mixture heated to 60°C while stirring using a magnetic stirrer. The resulting colorimetry sensor solution is then poured into square glass molds and dried in an oven at 50°C for 24 hours.

2.2.3 Colorimteric sensor characterization

Characterization of colorimetric sensor packaging includes color change test, thickness test, tensile strength test, elongation test, and biodegradation rate test. The color change test was carried out on the colorimetric sensor packaging which had been applied to tilapia fillets. Next, the colorimeter sensor was tested for color changes using digital camera. The thickness test was carried out using a micrometer device with a precision of 0,01 mm where the colorimetric sensor is inserted into the tool and the scale is rotated until the tool clamps it. The thickness value of the resulting colorimetry sensor will then be compared with the thickness of the plastic based on the Japanese Industrial Standard and determine its suitability as packaging.

Tensile strength test and elongation test were carried out using a Universal Testing Machine. The tensile strength test in this study was carried out by preparing the colorimetric sensor first. The colorimetric sensor packaging was cut to 6 cm long and 1.5 cm wide in a dogbone shape. The sample was then clamped on the testing machine before being pulled until it broke. The biodegradation rate test was carried out by cutting the sample into a square shape measuring 2x2 cm and weighing the initial weight (W0). After recording the initial weight, the samples were buried in the ground. Stockpiling was carried out for seven days and samples were checked again every 24 hours. After seven days, weight was weighed after stockpiling (W). Weight loss was then calculated using the weight loss percentage formula.

2.3 Analysis data

The data obtained were first tested for normality and homogeneity. All data were then analyzed using Analysis of Variance (ANOVA) and continued with Duncan Test with a level of 0.05. If the calculated F value is greater than the table F value with a confidence level of 95%, then the hypothesis is rejected.

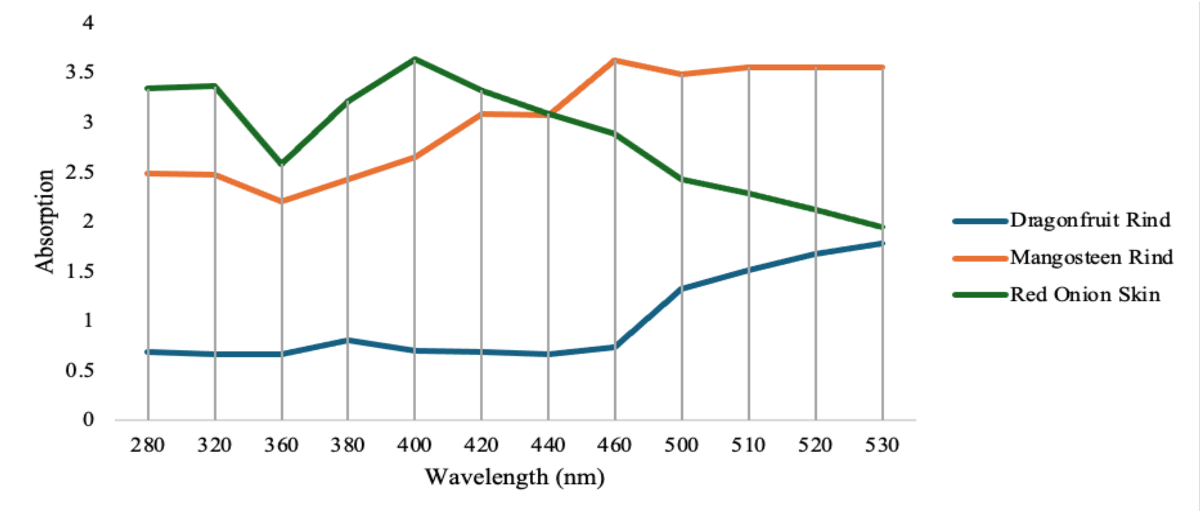


Figure 1. Ultra violet - visible absorption wavelength of extracted dragonfruit rind, mangosteen rind, and red onion skin.

3. Results and Discussion

3.1 Results

3.1.1 Absorption

The absorption of the dragon fruit rind extract was measured at length waves from 280 to 530 nm. As seen in Figure 1, the absorption spectrum shows that the extract has highest absorption at 530 nm. The result of this absorption shows similarities with recent studies where the UV-Vis spectrum shows peak in the wavelength range of 520-570 nm (Cyio and Darwis, 2021). The absorption of mangosteen rind extract also measured at length waves from 280 to 530 nm. As seen in Figure 1 the absorption spectrum shows that the extract has highest absorption at 460 nm. Other research shows similarities where the highest absorption is at a wavelength of 520 nm (Munawaroh *et al*., 2016). The absorption of red onion skin extract also measured at length waves from 280 to 530 nm. As seen in Figure 1, the absorption spectrum shows that the extract has highest absorption at 400 nm. This result shows that this anthocyanin is a non-acylated anthocyanin (Saha *et al*., 2019).

3.1.2 Color change

The color change that occurs in the dragon fruit skin dye makes the pink color become yellowish and fades after storing the tilapia fillets for 24 hours. The skin color of the mangosteen rind changed from brownish red to blackish brown after storing the tilapia fillets for 24 hours. Onion skin dye changes its orange-brown color to brown after 24 hours of storage. The color change on the colorimetric sensor packaging occurs in line with the pH change in the tilapia fillet, as shown in Figure 2.

3.1.3 Thickness

The physical property of thickness that can determine the quality characteristics of bioplastics in this research is colorimetric sensor. The thickness of the colorimetric sensor can be seen in Figure 3. The thickness of the colorimetric sensor is not influenced by the addition of natural dyes but influenced by the different polymers used. The highest thickness was obtained in the mixed polymer treatment of corn starch and carrageenan at a value of 0,210 mm. This value meets JIS standards where the maximum thickness is 0,25 mm (Budiman and Tarman, 2022).

3.1.4 Tensile strength

Tensile strength is tested to find out how much force should be used to achieve maximum tensile strength in colorimetric sensor packaging. The lowest tensile strength of the colorimetric sensor is at a value of 7,45. It meets JIS standards where the minimum tensile strength is 3.92 mPa (Budiman and Tarman, 2022). The highest tensile strength of the colorimetric sensor at a value of 20.62 MPa was found in the colorimetric sensor with a mixture of carrageenan and corn starch as polymers. The tensile strength of the colorimetric sensor can be seen in Figure 4. The tensile strength of the colorimetric sensor packaging is greatly influenced by the polysaccharide composition; however, the addition of different dyes does not influence the tensile strength.

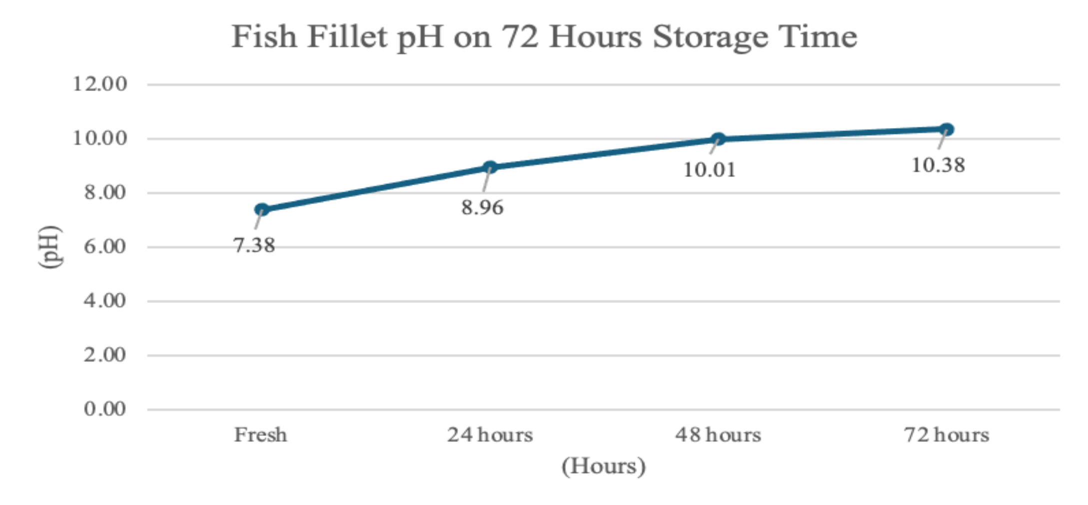


Figure 2. Tilapia fillet pH changes for 72 hours storage time at room temperature

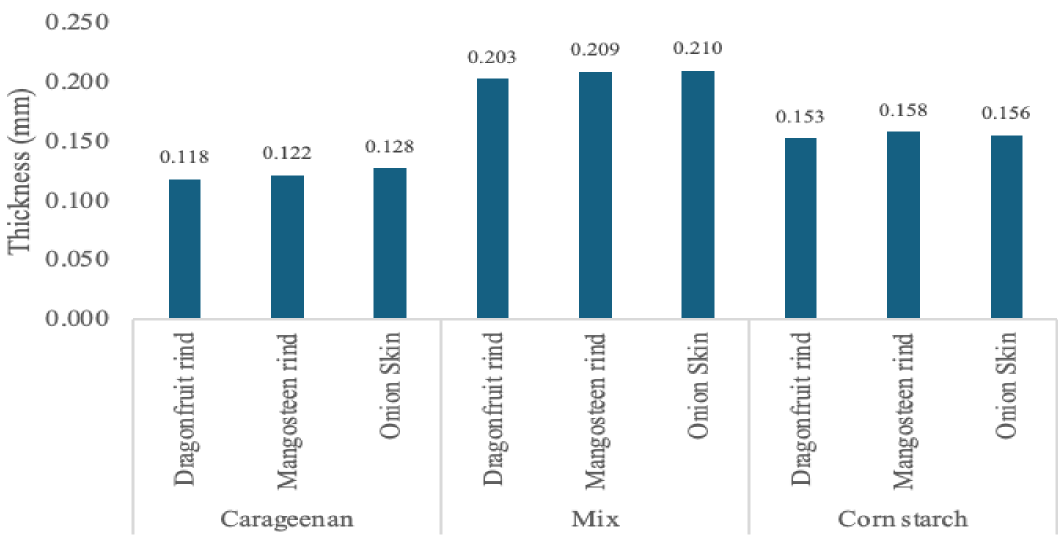


Figure 3. Graphic of colorimetric sensor's thickness

3.1.5 Elongation break

The elongation break of the colorimetric sensor can be seen in Figure 5. The lowest elongation break of the colorimetric sensors is at a value of 118%. The Japanese Industrial Standard (JIS) for bioplastics sets a minimum standard for elongation at break, which is the ability of a natural plastic to resist deformation without cracking or breaking. The standard specifies that the minimum elongation at break for bioplastics should be between 10% and 50% (Budiman and Tarman, 2022). This means that bioplastics should be able to stretch at least 10% of their original length before breaking, indicating their ability to resist deformation without cracking or breaking. The highest elongation break of colorimetric sensor at a value of 254% was found in the colorimetric sensor with carrageenan as a polymer.

The biodegradation rate of the colorimetric sensor did not differ between treatments, either with differences in polymer treatment or with differences in dye treatment. The biodegradation rate of the colorimetric sensors can be seen in Figure 6.

with fresh tilapia fillet with 72 hours storage time on room temperature. The colorimetric sensor undergoes a color change. The color changes that occur in the colorimetric sensor for each treatment can be seen in Table 3.

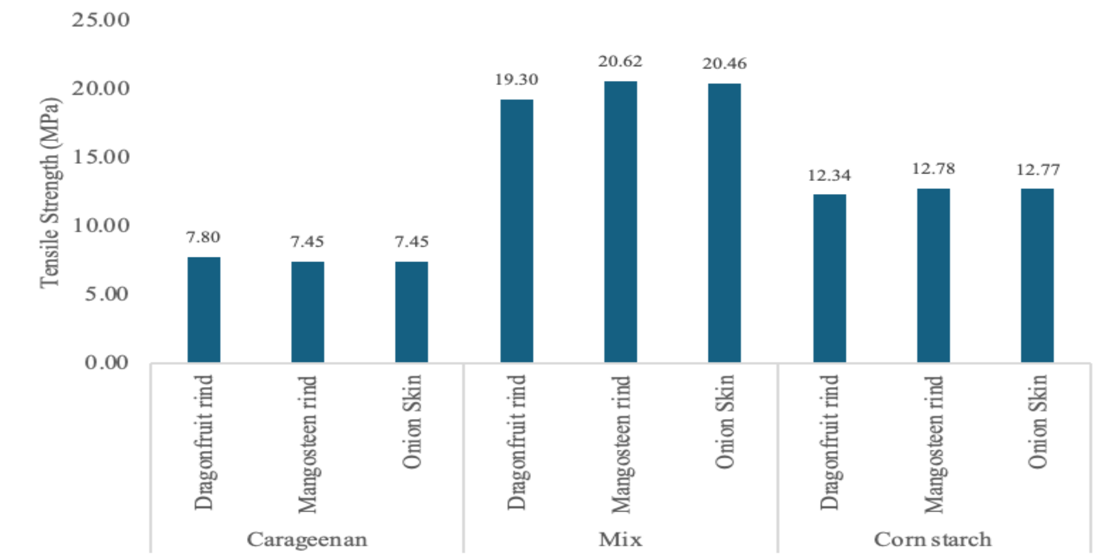


Figure 4. Graphic of colorimetric sensor's tensile strength

3.2 Discussion

3.2.1 UV-Vis absorption

Dragon fruit rind extract, mangosteen rind extract, and red onion skin extract show the UV-Vis peak of anthocyanin. Anthocyanin generally shows UV-Vis spectrum absorption maxima at around 400-600 nm. The UV Vis absorption spectrum of anthocyanin is divided into two, where at a wavelength of 260-280 nm there is absorption in the UV visible region and at a wavelength of 490-550 nm there is absorption in the visible region. There's an additional barely detectable peak (may appear as a small bump) in the wavelength range of 310-340 nm when the sugar moiety is acylated. Additionally, there's a hump at 400-450 nm. The size of the hump depends on the number of sugar moieties attached to the anthocyanidin moiety (Saha *et al.*, 2019). Aquabidest was used in the extraction following previous research on optimum extraction conditions showing highest anthocyanin content with the use of aquadest as solvent. Aquadest has polar properties which polarity is close to dragon fruit rind anthocyanin and anthocyanin has a polar aromatic ring so it will dissolve more easily in polar solvents (Kwartiningsih *et al.*, 2016). Uv-Vis result shows that anthocyanin in the extracts can be used as natural dyes of pH-sensitive smart packaging films.

Anthocyanin used in this research then used in colorimetric sensor packaging placed in a lunchbox

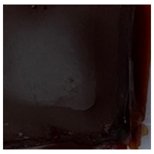
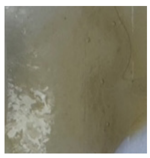
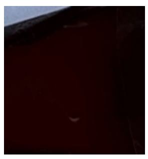
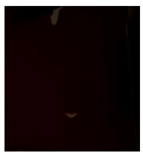
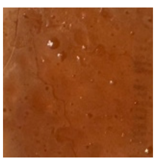
3.2.2 Changes of the color and pH

The pH of fish fillets changes during storage, gradually increasing over time. This increase in pH is attributed to the accumulation of alkaline compounds such as ammonia and amines derived from microbial action during fish muscle spoilage (An *et al.*, 2023). The pH of fillets in the 0-hour holding group was lower than the other after storage time. This increase in pH is associated with the spoilage of the fish, and the rate of pH increase can be used as an indicator of the rate of spoilage. The pH of fish fillets can also be influenced by the storage temperature, with higher temperatures leading to a faster increase in pH (Kim *et al.*, 2023). The anthocyanin's stability is significantly influenced by pH, with the molecules being more stable at lower pH levels. This is because anthocyanins exist in an equilibrium between their colored cationic form and the colorless pseudo base, and this equilibrium is directly influenced by pH. At low pH, the cyanidin molecule is protonated and forms a positive ion or cation, which is more stable and soluble in water. As the pH increases, the molecules become deprotonated, and, at high pH, the molecule forms a negative ion or anion, which is less stable and more susceptible to degradation (Saha *et al.*, 2019).

3.2.3 Thickness of the intelligent packaging

The thickness of the colorimetric sensor is susceptible to variations in the type of hydrocolloid,

Table 3. Color Changes in Colorimetric Sensor Packaging

Treatment	Storage Time			
	0 hour	24 hours	48 hours	72 hours
A1B1				
A1B2				
A1B3				
A2B1				
A2B2				
A2B3				
A3B1				
A3B2				
A3B3				

plasticizer, and concentration of the material employed, which collectively contribute to its dimensional properties. Furthermore, the size of the bioplastic mould utilized also exerts an influence on the thickness of the bioplastic, underscoring the multifaceted nature of this complex system (Budiman and Tarman, 2022). that repels water. When combined in bioplastics, the opposing properties of corn starch and carrageenan interact, resulting in a balance between the two that is crucial for creating bioplastics with improved water resistance and mechanical properties (Wang *et al*., 2022).

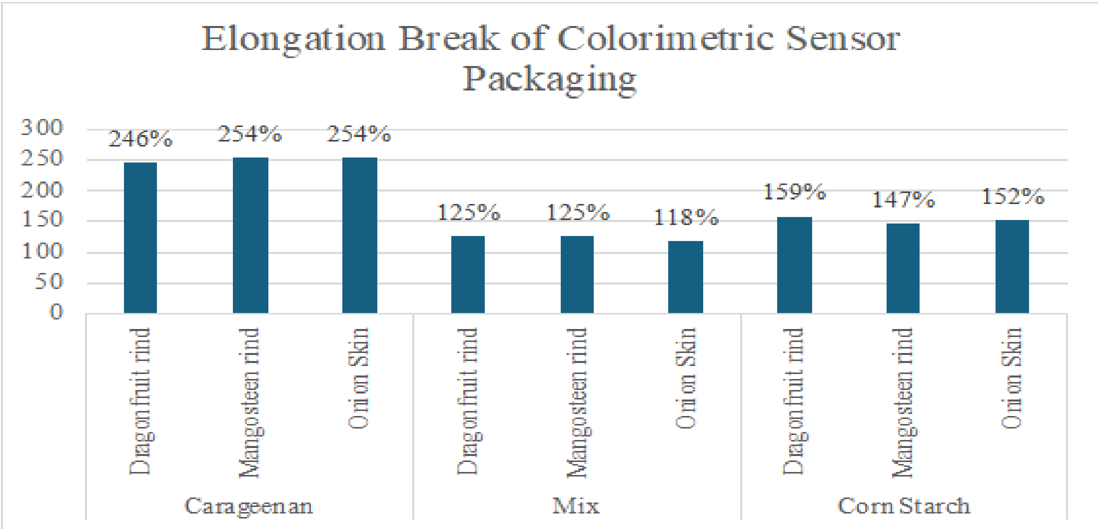


Figure 5. Graphic of colorimetric sensor's elongation break

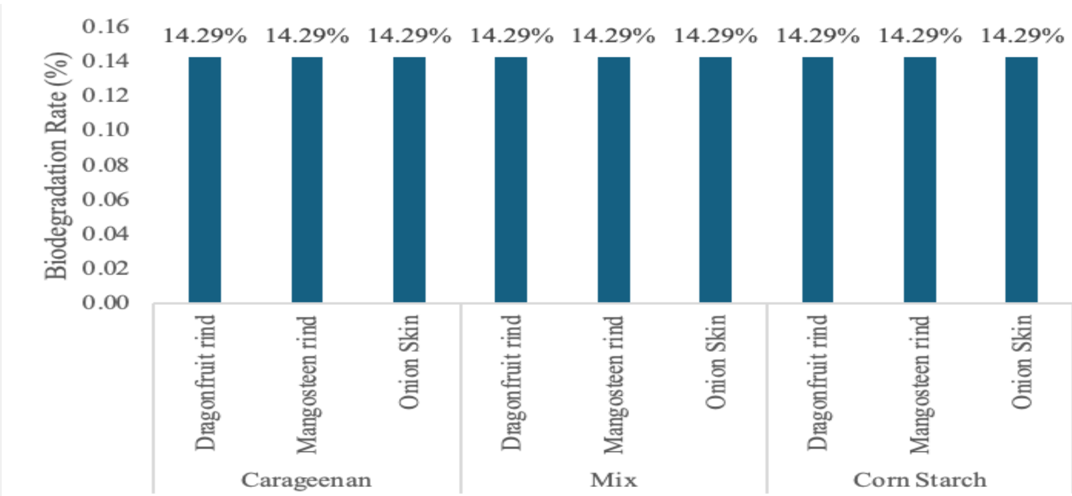


Figure 6. Graphic of colorimetric sensor's biodegradation rate

3.2.4 Tensile strength of the intelligent packaging

The tensile strength of bioplastics is affected by several factors, including the type of bioplastic, how it is made, and the presence of additives. The bond between bioplastics made from corn starch and carrageenan is primarily formed through chemical bonds called hydrogen bonds and non-polar interactions. Carrageenan, a carbohydrate derived from red seaweed, is naturally attracted to water due to the presence of free hydroxyl groups that can form bonds with water, allowing it to create a gel-like structure that is beneficial for bioplastic applications (Genecya *et al*., 2023). In contrast, corn starch is a material

3.2.5 Elongation Break of the Intelligent Packaging

An elongation break is defined as the extendibility of film length from the initial length to the point of the break. The effect of different polymers exhibits an opposite action as compared with their correspondent Tensile strength. The elongation at break of carrageenan-based bioplastics can vary significantly depending on the concentration of carrageenan used, the type of carrageenan, and the presence of additives or other biodegradable polymers.

3.2.6 Decomposition of the intelligent packaging

The decomposition of organic matter, a

process facilitated by microorganisms such as Aerobacter and Clostridium, is a natural phenomenon that occurs in soil environments. In the context of bioplastic, which is composed of natural ingredients susceptible to decomposition, a significant weight loss was observed after a 7-day burial period, suggesting that biodegradation had taken place. The rate of biodegradation is influenced by various factors, including the presence and activity of decomposing microorganisms and the water content in the soil.

4. Conclusion

Anthocyanins extracted from dragon fruit peel, mangosteen peel, and onion coolies can be used as colorimetric sensors with the ability to change color. This color change occurs in line with changes in the pH of the tilapia fillet which increases. The best color changes are obtained from anthocyanins from dragon fruit skin. The characterization of bioplastics as colorimetric sensors meets the Japanese Industrial Standard for bioplastics. The best value for thickness was obtained in the carrageenan polymer treatment, the best value for tensile strength was obtained in the mixed polymer treatment of corn starch and carrageenan, the best value for elongation was obtained in the carrageenan treatment, and the biodegradation rate value had no difference between polymer treatments. More exploration needs to be done regarding natural dyes that can be used as colorimetry sensors as well as the best treatment of polymers to get the best bioplastic characteristics.

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Authors' Contributions

All authors have contributed to the final manuscript. The contribution of each author is as follows: Brigitta; collected the data, drafted the manuscript, and designed the figures. Prof. Amin and Dr. Kismiyati; revised the main conceptual ideas and critical revision of the article. All authors discussed the results and contributed to the final manuscript.

Conflict of Interest

The authors declare that they have no competing interests.

Declaration of Artificial Intelligence (AI)

The author(s) acknowledge the use of Perplexity for language refinement in preparing this manuscript. All AI-generated content was rigorously reviewed, edited, and validated to ensure accuracy and originality. Full responsibility for the manuscript's final content rests with the author(s). To ensure transparency and support the review process, a comprehensive delineation of the tool's application is furnished in the "Results and Discussion" section of this manuscript in compliance with the publisher's ethical guidelines.

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