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EFFECTS OF CHEMICAL MODIFICATION AND RATIO OF DRAGON FRUIT (*Hylocereus polyhizus*) PEEL EXTRACT ON THE DYED-ARROWROOT (*Maranta arundinacea*) STARCH

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Abstract*. Color is the main attraction in choosing food; therefore, manufacturers do various ways to produce good colors without considering the consumer's health. Natural colors are a safe choice for health; besides being safe, natural dyes can also provide functional properties to those who consume them. The study aimed to determine the activity of natural dyes and their functional properties when mixed with chemically modified arrowroot starch. This study used a factorial design with chemical modification factor A and factor B, a ratio of dragon fruit extract to the amount of starch, with three repetitions. The best treatment was A2B3 (cross-link, starch with dragon fruit 1:2) because it had the treatment with the most visible color. This treatment has an antioxidant content of 38.95%, a polyphenol content of 106.19 mg GAE/g, a color of 76.88, and an anthocyanin content of 0.71 mg/100g.*
Keywords: *anthocyanins; antioxidants; arrowroot starch; dragon fruit peel; modified starch; polyphenols*

1. Introduction

The color of food is an attraction for consumers to consume it because the color is closely related to freshness, taste, and perceived quality (Galaffu *et al*., 2015). Every effort is made to improve the color quality of food. One way that can be done is to use synthetic dyes. Synthetic dyes have advantages, including lower costs, better appearance, high color intensity, color stability, and better and more uniform pH (Dey & Nagababu, 2022). However, its use in the community is beyond the threshold, causing health risks to those who consume it. Dey and Nagababu (2022) say this can cause severe health problems such as mutations and cancer, reduced hemoglobin concentration, and allergic reactions.

Natural dyes are the right solution to overcome this problem. Besides being safe in use, natural dyes provide added value to food through functional properties. The addition of polyphenols can also reduce the digestibility of starch. Polyphenols reduce digestibility by inhibiting the action of the amylase enzyme so that it is not recognized (Thompson & Yoon, 1984). Meanwhile, natural dyes have weaknesses, including experiencing a reduction in color due to exposure to light, temperature, or air in storage (Amchova *et al*., 2015). Natural dyes are used in society by using fresh ingredients directly. This makes people lazy to use this natural dye because

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preparing the material is challenging. Direct coloring on starch will make it easier to use so that it is more practical, and its use is also wider, safer, and healthier. Staining of this starch is still rarely done, and what has been done includes coloring rice flour (Ridawati & Alsuhendra, 2019), rice (Sari *et al*., 2020), fiber (Peng *et al*., 2020), and paper (Mandegeni *et al*., 2016).

Dyes can come from fruit types rich in shades and have no economic value, namely dragon fruit skin. Red dragon fruit skin contains betacyanin and anthocyanin pigments. This red dragon fruit contains the pigment betacyanin (150.46 ± 2.19 mg/100 g), pectin (10.8%), and dietary fiber (Jamilah *et al*., 2011). Compounds are contained in dragon fruit peels, such as dyes (anthocyanins and betacyanins, phenolics), and dietary fiber in the form of pectins and oligosaccharides (Le, 2022). The dragon fruit peel extract phenolic content is 28.16 mg GAE/100g (Nurliyana *et al*., 2010).

The starch must first be modified to absorb the dye more completely. According to (Cao *et al*., 2023), native starch has low water solubility at room temperature and poor stability, so a modification was made to overcome this. In this study, the change chosen modifications produced resistant starch, namely chemical modifications, especially esterification, and cross-linking. Type IV resistant starch (RS4) is formed when starch is modified using ester modifications or cross-linked starch (Fuentes-Zaragoza *et al*., 2010; Sajilata *et al*., 2006).

The modifications to the starch require further study so that the color mixed with the starch does not change or get damaged. In addition, the modification is also expected not to reduce the amount of polyphenolic compounds contained in the dragon fruit peel extract. The research aimed to examine the effect of modification on the color quality of arrowroot starch and to find a comparison of starch with dragon fruit peel extract, which gave the best color to arrowroot starch.

2. Methods

2.1. Material

The materials used include arrowroot starch of the creole type, which is extracted with microwave-assisted extract (MAE), and has been modified by esterification and cross-linking. The dye comes from the MAE method's fresh red dragon fruit peel extract. The ratio of aquadest to red dragon fruit peel is 1: 30, and the extraction time is 80 seconds (Ying, 2019). The equipment used for extracting was a microwave (AQUA AEM-S2612S, 400 W), oven (DHG9070A), blender, 80 mesh sieve, and rotary vacuum evaporator.

2.2 Extraction process of arrowroot tubers

The extraction process begins with cleaning the damaged parts of the tubers and continues with washing. Arrowroot tubers are washed until clean and then grated with a grater. The results of grated arrowroot tubers are then extracted with a ratio of water to ingredients 1: 33.81 g/ml

(Putri *et al*., 2021). The extraction was performed in a microwave (AQUA AEM-S2612S, 400 W) for 4 minutes at ±49.2ºC. The extraction process is then followed by filtering using a filter cloth. The filter results were left for 24 hours at room temperature with three water changes. The residue is further modified by esterification and cross-linking.

2.3. Methods

Starch chemically modified through esterification (Amalia & Kumoro, 2015) and cross-linking (Retnaningtyas, 2014) in a wet state is mixed with dragon fruit extract in the ratio according to the treatment. This mixture is then stirred using a spoon until the color is even. This wet starch is then dried using an oven DHG9070A at 50ºC. During drying, it is reversed for even heat distribution so that it dries faster. The dried starch was then pounded using a mortar and sieved using an 80 mesh sieve. Observations on colored starch were antioxidant by spectrophotometry method, polyphenol content by spectrophotometric form, color by Hunter-Lab method, and anthocyanin content by the spectrophotometric process.

2.4. Data Analysis

This study used a 2X3 factorial design, repeated three times with factor A, a type of chemical modification where A1 was esterified, and A2 was cross-linked. Factor B was the ratio of starch to dragon fruit extract, namely B1 1: 1, B2 1: 1.5, and B3 1: 2. The interaction between the two factors resulted in 6 treatments are A1B1, A1B2, A1B3, A2B1, A2B2, and, A2B3. Follow-up test using 5% DNMRT.

3. Results and Discussion

3.1. Antioxidant levels

Antioxidant levels in arrowroot starch with dragon fruit peel extract coloring. Antioxidants are expected in the food consumed because they can prevent free radicals in the body. The interaction between treatments significantly affected the antioxidant levels in dyed starch. Antioxidant levels in the interaction treatment had a significant effect on all treatments. Antioxidant levels of each treatment are shown in Table 1.

Table 1. Antioxidant levels from the interaction of chemical modification treatment (Factor A) and comparison of dragon fruit peel extract with arrowroot starch (B)

Types of chemical modification	Comparison of dragon fruit peel extract with arrowroot starch		
	1:1 (B1)	1.5:1 (B2)	2:1 (B3)
Esterification (A1)	34.30±0.86[a]	45.96±0.78[b]	54.51±1.12[c]
Cross-link (A2)	30.16±1.19[a]	34.26±1.16[b]	38.95±1.64[c]
KK=2.9%			

Numbers in the same column following the same lowercase letters are not significantly different according to DMNRT 5%

In Table 1, it can be seen that the highest antioxidant levels were found in treatment A1B3 (esterification, peel extract 1:2) and the lowest in treatment A2B1 (cross-link, skin extract 1:1). Antioxidant levels in colored starch products come from the raw materials used. Antioxidants in arrowroot starch come from phenol compounds and the colors found in arrowroot tubers and dragon fruit. Polyphenols can act as antioxidants, reducing the risk of atherosclerosis and coronary heart disease. Intake of polyphenols in food has been associated with a reduced risk of chronic diseases, such as heart disease and cancer (Savikin *et al*., 2009). Anthocyanins and dyes are also known as antioxidants and anti-diabetics (Jiao *et al*., 2012; Takahata *et al*., 2011).

There was color damage in the treatment of factor A1 (esterification modification), but with reduced color, actually higher antioxidants. Antioxidants in this treatment also come from a modification process carried out on A1 (esterification). According to (Li *et al*., 2023), in vitro antioxidant experiments showed that esterification modification (acetylation) can effectively increase hydroxyl radicals, DPPH, and the ability to scavenge radicals and lipid peroxidation of polysaccharides.

In addition, the antioxidant content is also influenced by the amount of polyphenols present in materials other than dyes because polyphenols are also a source of antioxidants. According to Wu *et al.* (2006), dragon fruit peel extract is a better antioxidant than the fruit flesh because the peel polyphenols are slightly higher than the flesh. The increase in phenolic content is directly proportional to the amount of antioxidants.

3.2. Polyphenol content

The value of polyphenol levels in arrowroot starch with the addition of dragon fruit peel extract can be seen in Table 2. All treatment interactions on polyphenol levels were not significantly different from each other. The levels of polyphenols in arrowroot starch come from dyes of dragon fruit and bioactive compounds containing polyphenols found in the tubers.

Table 2. Polyphenol levels from the interaction of chemical modification treatment (Factor A) and the comparison of dragon fruit peel extract with arrowroot starch (Factor B)

Types of chemical modification	Comparison of dragon fruit peel extract with arrowroot starch		
	1:1 (B1)	1.5:1 (B2)	2:1 (B3)
Esterification (A1)	89.43±2.40^{a}	110,07±1.57^{b}	147.39±3.26^{c}
Cross-link (A2)	74.41±0.40^{a}	93.79±0.26^{b}	106.19±0.65^{c}
KK=1.74%			

The same lowercase letters in the same column will not be significantly different according to the DNMRT test at the 5% level.

Table 2 shows that the levels of antioxidants are comparable to those of polyphenolic compounds because antioxidants come from phenolic compounds in the ingredients. Therefore, the highest polyphenolic compound was the A1B3 treatment (esterification, dragon fruit peel extract 1:2). A1B3 is the treatment that has the highest antioxidant. The source of phenol also

comes from dragon fruit which is rich in color and is a source of antioxidants. Dragon fruit is a tropical fruit with a high content of phenolic compounds, a source of antioxidants, and beneficial for health. According to (Chen *et al*., 2021), dragon fruit skin has a higher content of flavonoids and tannins than the fruit flesh.

Phenol compounds also come from arrowroot tubers which are used as a source of starch in the form of bioactive compounds. Bioactive compounds found in local tubers such as gembili, gadung, cassava, arrowroot, and kimpul, namely dioscorin, diosgenin, and phenol, have functions as antioxidants. The antioxidant function of several bioactive compounds present in local inferior tubers is expected to counteract free radicals in the body (Mar'atirrosyidah & Estiasih, 2015).

3.3. Colors

The color of the starch comes from the addition of dragon fruit peel extract. The highest ratio was starch containing the highest dragon fruit peel extract. The color of modified arrowroot starch was significantly different between the interaction treatments. The color value of modified arrowroot starch with dragon fruit peel extract coloring can be seen in the following Table 3

Table 3. Color of modified arrowroot starch with the addition of dragon fruit peel extract

Treatments	Color average±sd		
	*L	*a	*b
A2B3 (cross-link, dragon fruit peel extract 1:2)	76.88±0.07[a]	8.08±0.025	6.36±0.015
A1B3 (esterifikasi, dragon fruit peel extract 1:2)	83.03±0.04[b]	1.61±0	11.56±0.005
A2B2 (cross-link, dragon fruit peel extract 1:1.5)	83.89±0.02[c]	3.20±0.005	6.85±0.015
A1B2 (esterifikasi, dragon fruit peel extract 1:1.5)	85.87±0.08[d]	0.78±0	10.84±0.040
A2B1 (cross-link, dragon fruit peel extract 1:1)	87.04±0.05[e]	2.65±0.015	5.88±0.010
A1B1 (esterifikasi, dragon fruit peel extract 1:1)	89.83±0.08[f]	-0.56±0.005	9.26±0.005

Numbers in the same column with the same lowercase letters are not significantly different according to the DNMRT level of 5%

The L* value represents brightness with a value of 0-100. The L* value of red dragon fruit peel dye extract ranged from 76.88-89.83. The modified treatment and the addition of dragon fruit peel extract were significantly different. The highest L* value was 89.83 with the cross-link modification treatment and the addition of dragon fruit peel extract twice the amount of starch, and the lowest L* value was 76.88.

The a* value indicates a red-green color, with a positive value (+) meaning red and a negative value (-) meaning green. The a* value is positive; the highest a* value is in the cross-link modification treatment and the addition of 2:1 dragon fruit peel extract from the amount of starch, namely 8.08. The lowest is in the esterification modification and the addition of 1:1 dragon fruit peel to the amount of starch. According to Hermawati (2015), the red intensity value (a+) is the reddest, averaging 52.84. This value was lower in the study because it was mixed with starch from arrowroot tubers.

The b* value is blue-yellow with values (-) to (+). The b* (+) value means the dye extract produces a yellow chromatic value. The b* value of red dragon fruit skin dyes ranged from 5.88 to 11.56. Modifying esterification and adding dragon fruit peel 2:1 with starch resulted in the highest b* value of 11.56, and cross-link modification and addition of dragon fruit peel 1:1 with starch produced the lowest b* value of 5.88.

The color of modified arrowroot starch comes from dragon fruit peel extract. However, when mixing with starch, some colors are lost, and some have changed. Table 3 shows that the positive a* value, red, is found in the A2B3 treatment (cross-link, dragon fruit peel extract 1:2). The highest a* color is the most colorful starch. The color of arrowroot starch comes from the ingredients found in the dragon fruit peel, namely anthocyanin and betacyanin. Dragon fruit color is stable in acidic conditions, namely pH 2 (Babaloo & Jamei, 2017).

Red dragon fruit peel contains the pigment betacyanin (150.46 ± 2.19 mg/100 g) and pectin (10.8%) and has very high dietary fiber (Jamilah *et al*., 2011). Betacyanin in dragon fruit peel gives a red-purple color (Strack *et al*., 2003). According to (Sambasevam *et al*., 2020), betacyanin pigment is more stable at low temperatures and in acidic conditions. Degradation is associated with discoloration or browning. The stability of pigment extracts to light shows a gradual deterioration over time.

Dragon fruit peel also has anthocyanin pigment, which has the potential as a natural dye. Anthocyanins are natural dyes widely used in food but sensitive to heat. The anthocyanin in dragon fruit peel is very soluble in polar solvents, so extraction is done using distilled water. Still, distilled water cannot stabilize it because anthocyanins are stable at acidic pH (Nizori *et al*., 2020).

3.4. Anthocyanin levels

Anthocyanin is an observation of the dyes in the arrowroot starch produced. The anthocyanins in starch decreased when mixed with arrowroot starch, and the interactions between treatments differed significantly. Anthocyanin observation data can be seen in Table 4.

Anthocyanin is a dye present in dragon fruit skin apart from betacyanin. Anthocyanins are bioactive components of the flavonoid group, which can give red, purple, and blue colors to flowers, leaves, tubers, fruits, and vegetables (Jensen *et al*., 2011; Torskangerpoll & Andersen, 2005). The amount of anthocyanin was different in each treatment because the amount of dragon fruit extract in the starch was also different. The modification process also causes another thing to be carried out on this arrowroot starch beforehand. In all A1 treatments (esterification), there was a loss of color in the arrowroot starch produced. It was possible because residual NaOH is still used in the modification, damaging the color of the dragon fruit peel extract. The color is getting lost the more the color enters mixed starch. According to (Marco *et al*., 2011), the properties of

anthocyanins, including color changes and antioxidant activity, are affected by the pH and structure of the anthocyanins. Anthocyanin structure changed at pH 1, 4,5, and 7 (Lee *et al*., 2005).

Table 4. The amount of anthocyanins in modified arrowroot starch with the addition of dragon fruit peel extract

Treatments	Average anthocyanin (mg/100g)±sd
A1B1(esterifikasi, dragon fruit peel extract 1:1)	0.00±0.00[a]
A1B2 (esterifikasi, dragon fruit peel extract 1:1.5)	0.17±0.001[b]
A2B1 (cross-link, dragon fruit peel extract 1:1)	0.22±0.018[c]
A2B2 (cross-link, dragon fruit peel extract 1:1.5)	0.24±0.007[d]
A1B3 (esterifikasi, dragon fruit peel extract 1:2)	0.44±0.089[e]
A2B3 (cross-link, dragon fruit peel extract 1:2)	0.71±0.036[f]

Numbers in the same column with the same lowercase letters are not significantly different according to the DNMRT level of 5%

Anthocyanins break down more quickly at high pH, and high temperature accelerates this reaction. During storage, there was a decrease in quality which resulted in damage to anthocyanins (Sari *et al*., 2020). This condition will cause anthocyanins to be damaged. The drying process also causes the process of reducing anthocyanins in arrowroot starch after mixing with starch. Drying takes a long time, and contact with light causes the color to fade. Anthocyanins are stable at 50˚C without light and pH 4 (Vargas *et al*., 2013).

4. Conclusions

The chemical modification affects the color of the starch produced, and this occurs because the reagent used causes the color to become unstable. The best treatment in this study was A2B3 because it had the best color. This cross-link modification treatment adds dragon fruit peel extract 2:1 with the amount of starch.

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