The effect of encapsulant type on physical and chemical characteristics of anthocyanin extract powder from red dragon fruit *Hylocereus polyrhizus*

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Abstract. Anthocyanin pigment extract from red dragon fruit has the potential to be a natural dye for food and can be used as an alternative to synthetic dyes that are safer for health. Anthocyanins are unstable and easily degraded during processing and storage due to the influence of heat, pH, light, and oxygen. This problem can be overcome by making anthocyanin coloring powders with encapsulation techniques. The objective of this study was to determine the effect of the type of encapsulant on the physical and chemical characteristics of anthocyanin extract powder from red dragon fruit. The encapsulation process of anthocyanin pigment extract was done by using the freeze drying method with the addition of maltodextrin, gum arabic, and a combination of maltodextrin and gum arabic as encapsulant. The physical and chemical characteristics parameters of anthocyanin extract powder analyzed were moisture content, solubility, color intensity, total anthocyanin content, and antioxidant activity. The results showed that the anthocyanin extract powder encapsulated using the freeze drying method obtained the best results using maltodextrin encapsulant with the results of the test parameters: water content of 5.96%, solubility of 94.00%, color intensity of 0.304, total anthocyanin content of 31.17 mgCyE/g, and 84.60% of antioxidant activity.

1. Introduction
Red dragon fruit is a tropical fruit from the *Cactacea* family which belongs to the genus *Hylocereus* which is currently widely cultivated in Asian countries such as Indonesia [1,2]. Red dragon fruits contain high water content. According to Farikha et al., (2013), fresh red dragon fruit cannot be stored for long because it has a high water content of around 90% and a shelf life of 7-10 days at 14 °C, this causes dragon fruit to be easily damaged, so further processing is required such as processing red dragon fruit to be used as a natural dye [3].

The red pigment content found in the flesh and skin of the red dragon fruit is called anthocyanin. Anthocyanins are red, purple, blue pigments found in plants that can be used as natural dyes [4]. Anthocyanin pigments are widely used as food coloring because they can produce colors with a wide pH range [5].
Most of the natural pigments derived from plants, including anthocyanins, have limitations in their use, namely their instability and are easily degraded during processing and storage due to the influence of heat, pH, light, enzymes and oxygen [6–8]. This problem can be overcome by making anthocyanin dye powder using the encapsulation technique.

Encapsulation is a technique of coating active ingredients in solid, liquid, or gas forms into capsules using a carrier matrix which forms a thin wall coating around the encapsulated material [9]. Encapsulation aims to protect the components of sensitive materials so as to reduce the degradation of the active compound in the material [10].

A coating or carrier matrix that can be used as an anthocyanin encapsulation agent is maltodextrin [11,12]. Maltodextrin is used as a coating because it has the ability to form emulsions, is easily soluble in water, has low viscosity, has no color and odor [13,14].

The encapsulation agent that can be used besides maltodextrin is gum arabic [15–17]. Gum arabic is an effective encapsulation material because it has several properties such as high solubility in water [18], no taste and odor [19], and forms a solution with low viscosity [20]. According to Ozkan and Bilek (2014), gum arabic is a coating material that can be used to protect flavors and dyes and is a good coating material because it has stable emulsion properties and good volatile retention [21]. Gum arabic can be used singly or in combination with other encapsulants in the production of pigment extract powders [22–24].

Encapsulation can be done by freeze drying. Freeze drying is a drying method using the freezing method in which this tool dries the material by sublimation, so that heat sensitive food ingredients (flavor components and natural dyes) can be maintained for oxidation stability [25,26].

The objective of this study was to determine the effect of the type of encapsulant on the physical and chemical characteristics of anthocyanin extract powder from red dragon fruit.

2. Materials and methods

2.1. Materials

Red dragon fruits were obtained from dragon fruit farmers in Soppeng Regency, South Sulawesi Province. Other materials used were 96% ethanol, distilled water, citric acid, maltodextrin (DE 10-12), gum arabic, potassium chloride (KCl) buffer, sodium acetate buffer (CH₃CO₂Na), and DPPH (2,2-diphenyl-1-picrylhydrazyl) compound.

2.2. Preparation and extraction anthocyanin

Extraction of red dragon fruit anthocyanin pigments was carried out by maceration method, as much as 100 g of red dragon fruit mashed with a blender (Philips) for 1 m then added 96% ethanol solvent added with citric acid (C₆H₈O₇) (Merck) 15% with the ratio of ingredients to solvents namely 1:4 (w/v). Maceration was carried out at room temperature for 24 h with modification [27]. Then, the extract was filtered with a vacuum filter (Whatman paper No. 4) to separate the dregs from the filtrate. The filtrate obtained was concentrated using a rotary vacuum evaporator (Stuart RE300) at 40 °C until a thick extract was obtained.

2.3. Encapsulation of anthocyanin pigment extract from red dragon fruit

The encapsulation process of anthocyanin extracts used the freeze drying method with the addition of maltodextrin and gum arabic as coating material with the formulations shown in table 1. Coating material according to the formulation were dissolved using distilled water and heated using a hot plate magnetic stirrer (Thermo Scientific CIMAREC) at a temperature of 40°C for ±30 m until a suspension is formed. Furthermore, the coating material suspension was added to the anthocyanin extract with a ratio between the extract and the coating material suspension, namely 1:3 (v/v) [15,2]. The anthocyanin extract mixture with fillers was poured into a petri dish then frozen in a freezer at -40°C for 24 h, then dried using a freeze dryer (ALPHA 1-2 LD plus) at -50°C with a pressure of 0.036 psi to formed powder [28].
Table 1. Anthocyanin extract formulation with coating material.

| Treatment | Coating material | Extract to coating material ratio (v/v) |
|-----------|------------------|-----------------------------------------|
| P1        | Maltodextrin 20%  | Gum Arabic 0% 1:3                       |
| P2        | 0%               | Gum Arabic 20% 1:3                      |
| P3        | 10%              | Gum Arabic 10% 1:3                      |

2.4. Analysis

2.4.1. Water content [29]. The water content of anthocyanin extracts was analyzed using the thermogravimetric method. The evaporating dish was dried in an oven (Heraeus Instruments) at 105 °C for 15 m and cooled in a desiccator for 10 m, then weighed. A total of 3 g of sample was put into a cup and dried in an oven at 105 °C for 6 h. The evaporating dish was removed and cooled in a desiccator for 15 m and weighed. The evaporating dish was put back in the oven until a constant weight was obtained. The calculation of water content was based on a wet basis.

\[
\text{Water content (\% wb)} = \frac{W - (W_1 - W_2)}{W_1 - W_2} \times 100\% \tag{1}
\]

Where:
- \( W \) = Weight of sample before drying (g)
- \( W_1 \) = Weight of sample and dry evaporating dish (g)
- \( W_2 \) = Empty evaporating dish weight (g)

2.4.2. Gravimetric method solubility [30]. Solubility measurement was carried out to measure the solubility level of the powder produced by calculating based on the gravimetric method, which is the highest residual weight on Whatman filter paper no.42. A total of 1 g of powdered dye preparation was dissolved in 150 mL of distilled water and filtered using Whatman No. filter paper, 42 with the help of a vacuum pump. Before use, the filter paper was dried in an oven at 105 °C for 30 m and weighed. After filtering, the filter paper and the residue were dried in an oven at 105 °C for 3 h, cooled with a desiccator and weighed. Drying was carried out until a constant weight is obtained.

\[
\text{Solubility} = 100 - \frac{a - b}{(100 - \text{water content (\%)} \times c} \times 100 \tag{2}
\]

Where:
- \( a \) = filter paper weight and residue (g)
- \( b \) = filter paper weight (g)
- \( c \) = sample weight used (g)

2.4.3. The color intensity using the spectrophotometer method [31]. A buffer solution of citric acid or dibasic sodium phosphate pH 3 was prepared as much as 200 mL by way of 159 mL of a 2.1% citric acid solution mixed with 41 mL of 0.16% dibasic sodium phosphate solution. Then, the pH was adjusted to pH 3 using a citric acid solution or a dibasic sodium phosphate solution. The maximum wavelength of the solution was measured by weighing 20 mg of the sample, then diluting it in a 25 ml volumetric flask using a buffer solution of citric acid - dibasic sodium phosphate pH 3, then measuring the absorption so that the measured absorbance was 0.2-0.7. The other sample was then measured for its absorbance (A) on a cuvette with a thickness of 1 cm using a buffer solution of citric acid - dibasic sodium phosphate pH 3 at a predetermined maximum wavelength so that the measured absorbance was 0.2-0.7. Citric acid buffer solution - dibasic sodium phosphate pH 3 was used as the blank.
2.4.4. *Total anthocyanin content* [32]. Total anthocyanin content was analyzed using the differential pH method. A total of 1 g of anthocyanin extract powder was put into 2 different test tubes. The first test tube was added with a solution of potassium chloride (KCl) (0.025 M) pH 1.0 until the volume became 10 mL. The second test tube was added with sodium acetate (CH$_3$CO$_2$Na) buffer solution (0.4 M) pH 4.5 to volume to 10 mL. The absorbance of the two pH treatments was measured with a UV-Vis spectrophotometer (Genesys 10S, Thermo Scientific) at wavelengths of 510 nm and 700 nm after being allowed to stand for 15 m. The absorbance value was calculated by the formula:

$$A = [(A_{510nm} - A_{700nm})_{pH\, 1.0} - (A_{510nm} - A_{700nm})_{pH\, 4.5}]$$

(3)

The anthocyanin concentration was calculated as cyanidine 3-glucoside using the equation:

$$\text{Total anthocyanin (mg/L)} = \frac{A \times MW \times DF \times 1000}{\epsilon \times 1}$$

(4)

A  = (A$_{510nm}$ - A$_{700nm}$)$_{pH\, 1.0}$ - (A$_{510nm}$ - A$_{700nm}$)$_{pH\, 4.5}$

MW  = Molecular Weight (449.2 g/mol for cyanidin-3-glucoside)

DF  = Dilution factor

$\epsilon$  = Molar extinction coefficient (26,900 L/mol/cm for cyanidin-3-glucoside)

l  = Pathlength (1 cm)

2.4.5. *DPPH radical scavenging activity* [33]. The antioxidant activity of anthocyanin extracts powder from red dragon fruit was determined using the DPPH method (2,2-diphenyl-1-picrylhydrazil). Anthocyanin extract powder samples were added to 2 mL of 0.1 mM DPPH solution. The mixture was homogenized and incubated at room temperature for 30 m in a dark place. The absorbance was measured using a UV-Vis spectrophotometer (Genesys 10S, Thermo Scientific) at a wavelength of 517 nm. The same treatment was also carried out for a blank solution consisting of 2 mL DPPH 0.1 mM (DPPH solution containing no test material). Absorbance measurement results analyzed the percentage of antioxidant activity using the formula:

$$\% \text{ inhibition} = \frac{\text{Absorbance(control)} - \text{Absorbance(sample)}}{\text{Absorbance(control)}} \times 100\%$$

(5)

2.5. *Statistic analysis*

The experimental design used was a Completely Randomized Design (CRD) with one factor, namely the concentration of citric acid. The data obtained were analyzed using the SPSS version 25 application, with analysis using ANOVA to determine whether there were differences in treatment at the level of $\alpha=0.01$ followed by the Duncan Multiple Range Test (DMRT) at the same $\alpha$ level.

3. Results and discussion

3.1. *Water content*

Figure 1 shows the results of the analysis of the various water content of the anthocyanin powder produced from the three treatments showing a significant difference ($\alpha=0.01$). The highest water content of anthocyanin powder was obtained in the addition of gum arabic encapsulant treatment of 10.66% (db) and the lowest water content was the addition of maltodextrin encapsulant of 5.96% (db).
The difference in moisture content in anthocyanin powder products can be related to the viscosity of the coating emulsion. High viscosity will cause the water content of the encapsulation product to be high. According to Gardjito et al., (2006) maltodextrin has a lower molecular weight of about (<4000) and has a simpler molecular structure so that water can easily be evaporated during the drying process [34]. While the molecular weight of gum arabic is greater (± 500,000) and has a more complex molecular structure, which causes the bond with water molecules to become stronger, so during the drying process, water molecules are difficult to evaporate because they require greater evaporation energy.

3.2. Solubility

Figure 2 shows the results of the analysis of the various levels of solubility of anthocyanin powders resulting from the three treatments showing a significant difference (α=0.01). The highest solubility of anthocyanin powder was obtained in the addition of maltodextrin encapsulant treatment of 94.00% and the lowest solubility yield was with the addition of gum arabic encapsulant of 89.23%.

The high solubility of anthocyanin powders using maltodextrin encapsulants is because maltodextrin has properties that can dissolve completely in cold water, so that it can release active ingredients appropriately in certain applications, besides that according to Srihari et al., (2010), maltodextrin can undergo rapid dispersion and have water-soluble properties [35]. Maltodextrin is the result of hydrolysis of starch with a chain length of 5-10 glucose molecule units. Glucose has a free hydroxyl (OH) group so that when dissolved in water it is able to form hydrogen bonds with water. Another factor that affects the solubility rate of maltodextrin in water is the value of dextrose
equivalency (DE). According to Badarudin (2006) the higher the DE value of maltodextrin, the better the solubility rate [36].

3.3. Color intensity
Color intensity shows the strength of the color present in the dye when applied in the product [37]. The results of the analysis of the color intensity variance can be seen in Figure 3 which shows a significant difference (α=0.01) between the three treatments. The highest color intensity measurement results in anthocyanin powder were obtained in the addition of maltodextrin encapsulant treatment of 0.304 and the lowest color intensity measurement results with the addition of gum arabic encapsulant was 0.266. According to Hamzah et al (2013), the use of maltodextrin as an encapsulant in the anthocyanin extract powder of telang flower has a higher color intensity than gum Arabic [15]. According to Putri et al., (2019) the encapsulation process of flavium cation compounds in anthocyanins can form complexes with carbohydrates in maltodextrin so that it can bind more anthocyanin pigments [38].

![Figure 3. The color intensity of anthocyanin extract powder.](image)

According to Giusti and Wrolstad (2001), color indicates the visual appearance of the product whereas pigments are chemical compounds that give color [32]. The red color in the powder comes from the anthocyanin pigment. The redder a sample shows the more anthocyanin content in it. The absorbance value of the sample depends on the content of the substance contained in it. The more dye levels, the more molecules that absorb light at a certain wavelength, so the greater the absorbance.

3.4. Total anthocyanin content
The total anthocyanin content in the red dragon anthocyanin pigment extract encapsulation powder is expressed as cyanidin-3-glycoside. The results of the analysis of the total variety of anthocyanins for anthocyanin powder products can be seen in Figure 4 which shows a significant difference (α=0.01) between the three treatments. The analysis results obtained showed that the highest total anthocyanin was obtained in the treatment with the addition of maltodextrin encapsulant, namely 31.17 mgCyE/g, while the lowest total anthocyanin was obtained in treatment with the addition of gum arabic encapsulant, namely 29.16 mgCyE/g.
Total anthocyanins in dye powders with the addition of maltodextrin encapsulants were higher than in dye powders with the addition of gum arabic encapsulants because in the encapsulation process, the flavium cation compounds contained in anthocyanins would form complexes with the carbohydrates used so that they would make anthocyanins more stable [39]. According to Selim et al., (2008) the use of maltodextrin encapsulant provides a much better protective effect compared to gum arabic against the stability of the anthocyanin pigment roselle extract [40].

Total anthocyanins in anthocyanin powders encapsulated using maltodextrin encapsulants are not much different from anthocyanin powders produced using gum arabic encapsulants as well as coating materials for the combination of maltodextrin and gum arabic, this is probably due to the structure of gum arabic which is a highly branched sugar heteropolymer and contains a small amount of protein covalently linked to the carbohydrate chain, so that it acts as a good film-forming agent and can bind to the encapsulated molecule. This makes the flavylum cation in anthocyanins less susceptible to nucleophilic attack by water molecules and enhances the stability of anthocyanins [41].

3.5. DPPH radical scavenging activity
The results of the antioxidant activity test were expressed as % RSA DPPH. The results of analysis of variance of the three treatments showed a significant difference (α=0.01). Figure 5 shows the highest % RSA DPPH found in the treatment with the addition of maltodextrin encapsulant, namely 84.60%, while the lowest % DPPH inhibition power was obtained in anthocyanin powder encapsulated using gum arabic encapsulant material, namely 63.15%.

![Figure 4. Total anthocyanin content of anthocyanin extract powder.](image)

![Figure 5. Antioxidant activity of anthocyanin extract powder.](image)
arabic. There is a decrease in antioxidant activity because according to Aguiar et al., (2017) the antioxidant activity can undergo changes caused by the encapsulation process or the interaction with the encapsulating agent [42]. According to Tazar et al., (2017) the decrease in antioxidant activity in the extract was due to the use of an encapsulant with a high enough concentration which would cause an increase in the total solids in the red dragon fruit coloring powder [43]. The encapsulant material which increases total solids causes a measurable decrease in antioxidant activity.

The results of antioxidant analysis can be seen that there is a relationship between total anthocyanins and antioxidant activity, where the measurement results of total anthocyanin numbers are directly proportional to the results of antioxidant activity. This is because in anthocyanins there are phenolic compounds that have –OH and –OR groups such as flavonoids and phenolic acids which can act as antioxidants that can ward off free radicals [44]. High total anthocyanins can be a source of natural antioxidants that are more effective in warding off various free radicals that cause various diseases [45]. Anthocyanins are secondary metabolites of the flavonoid class of compounds, which are found in plants in the form of polyphenols in large numbers [46].

4. Conclusion

The effect of the type of encapsulant on the physical and chemical characteristics of anthocyanin extract powder using the freeze drying method obtained the best results using maltodextrin encapsulants with the test parameters of water content of 5.96%, solubility of 94.00%, color intensity 0.304, total anthocyanin content 31.17 mgCyE/g, and antioxidant activity 84.60%.

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