



Extraction of Anthocyanins from Dragon Fruit Peel Using Solvent Extraction Method

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(Submitted: 2 April 2024; Revised: 12 June 2024; Accepted: 12 June 2024)

Abstract. Dragon fruit skin contains 26.4587 mg/L anthocyanins. Anthocyanins have benefits such as natural coloring agents in the food sector and are used as an alternative to synthetic dyes which are of course also safer for health. The purpose of this study was to determine the effect of extraction variables (time, solvent concentration, and particle size) on the anthocyanin content of dragon fruit skin from the extraction results with the Solvent Extraction method. The definition of Solvent Extraction is the separation of materials from a solid or liquid with the help of a solvent. The extraction process starts from the agglomeration of the extract with the solvent then contact occurs between the material and the solvent so that on the flat plane of the interface of the extraction material and the solvent there is mass deposition by diffusion. The extraction process starts with 25 grams of dragon fruit peel powder with a variety of particle sizes (30, 60, and 80 mesh) then put into an Erlenmeyer tube. Then, the citric acid solution with various concentrations (0.1 M; 0.2 M; and 0.3 M) was added as much as 250 ml. After that, the Erlenmeyer was placed on a stirrer to stir for (90, 120, and 150 minutes). After that, the extraction results were filtered using filter paper to produce a filtrate. Then the filtrate was precipitated to obtain anthocyanin extract. After that, it was analyzed using the spectrophotometric method to calculate anthocyanin content. In this study, the best results were obtained at 11.439 mg/L in conditions without repetition. The optimum conditions of extraction were obtained at a particle size of 60 mesh, a time of 150 minutes, and a solvent concentration of 0.5 M citric acid.

Keywords: *Dragon Fruit Peel, Anthocyanins, Solvent Extraction*

1. Introduction

Red dragon fruit (*Hylocereus polyrhizus*) also called pitaya fruit is a plant that comes from dry tropical climates. This fruit comes from Mexico, the red dragon fruit skin weighs 30-35% of the weight of the whole fruit which is only discarded as waste, so it can cause environmental pollution [1]. There are several types of dragon fruit, in this study using the type

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of red fleshy dragon fruit (*Hylocereus polyrhizus*). Red dragon fruit itself is one of the fruits that has potential as a natural colorant in food because it contains anthocyanin pigments. Red dragon fruit skin can usually be processed to be used as food products, as a base for cosmetics, and as natural dyes [2].

The content in the dragon fruit skin includes betalain compounds, anthocyanins, vitamin C, vitamin E, vitamin A, alkaloids, terpenoids, flavonoids, thiamine, niacin, pyridoxine, cobalamin, phenolics, carotene, and Phyto albumin [3]. Dragon fruit that has red flesh (*Hylocereus costaricensis*) has potential as a source of anthocyanins. Anthocyanins can be used as an alternative to synthetic dyes. In addition, it can also be used as an active ingredient in making cosmetics, because anthocyanins also act as antioxidants [4].

Anthocyanins are one of the pigments found in plants that have the potential to be used as food coloring and can replace synthetic dyes. Apart from being used as a dye, anthocyanins are also included in flavonoid compounds which have a function as natural antioxidants [5]. Apart from being a colorant, anthocyanins are antioxidants that are good for the body, including reducing the risk of degenerative diseases, such as cancer, and heart disease [6].

Antioxidants sourced from anthocyanins function as absorbers or traps where the molecules can react to free radicals and neutralize free radicals. Excessive oxidation reactions in our body can cause the formation of highly active free radicals that damage the structure and function of cells in our body [7]. Consuming foods that contain antioxidants such as anthocyanins can help the body's defense system return to normal [8].

The reason for choosing dragon fruit skin in this study is to reduce food waste in dragon fruit, especially in Banyuwangi regency which produces 82.544 tons per year. To get economic value, it is necessary to process the dragon fruit. One of them is extracting the substances contained in the dragon fruit skin. Factors that can maximize efficiency and selectivity in the extraction process such as the combination of solvents, temperature, pH, and extraction time.

In general, the definition of Solvent Extraction is the technique of separating one or more ingredients from a solid or liquid with the help of a solvent. So, Solvent Extraction is the transfer of solute between two solvents that do not mix [9]. This method will cause some constituents to move from the first solvent to the second solvent. The advantage of the Solvent Extraction method is the quality of the substance that will be produced because the dissolution technique uses a relatively low temperature so that the denaturation process of anthocyanin substances can be avoided compared to using the Microwave Assisted Extraction method so

that anthocyanin substances are not degraded due to the heating process in the extraction process. There are also fewer solvents used compared to using other methods.

Below is the difference in the results of anthocyanin levels of dragon fruit skin obtained by several methods in the research that has been done:

Table 1 Anthocyanin Yield of Dragon Fruit Peel in Several Methods

Method	Time	Anthocyanin Yield	Reference
Microwave Assisted Extraction (MAE)	6 minutes	25.031 mg/L	[10]
Microwave-Assisted Hydro distillation (MAHD)	4 minutes	52.184 mg/L	[11]
Microwave Assisted Extraction (MAE)	6 minutes	28.11 mg/L	[12]
Maceration	24 hours	8.355 mg/L	[13]
Maceration	72 hours	20.81 mg/L	[14]
Maceration	96 hours	4.73 mg/L	[4]
Ultrasound Assisted Extraction (UAE)	45 minutes	29.640 mg/L	[15]

The purpose of this study is to reduce the level of skin waste produced from dragon fruit and to determine the best variable to extract anthocyanins in dragon fruit skin.

2. Research Method

2.1 Materials

The raw materials in this study used red dragon fruit skin obtained from dragon fruit farmers around Jember Regency that had been dried, 10% Citric Acid, Aquadest, Acetic Acid, and Nitric Acid.

The tools used in this study of dragon fruit peel extraction are a set of tools for cutting, a set of tools for drying, a sieve (30, 60, 80 Mesh), a blender, a UV-Vis spectrophotometer, and a set of extraction tools.

2.2 Sample Preparation

Samples were obtained from red dragon fruit which was taken and sorted to select dragon fruit skin that was still suitable for sample material. The dragon fruit skin was washed first and then cut into small pieces. Then dried using an oven with a temperature of 50°C for 7 hours and obtained a moisture content of 11.08%. To test the water content should not use a temperature of more than 50°C because it can cause damage to the simplicial that will be used. After the drying process is complete, the dried dragon fruit skin is then pulverized using a blender for 5 minutes/10 grams. After that, the grinding results are sieved with particle sizes

(60, 80, and 100 mesh) and then put into plastic clips which can later be used for the extraction process [4].

2.3 Determination of Water Content

The moisture content of fresh dragon fruit peel and dragon fruit peel powder was determined using the oven method. A total of 5 grams of sample was put in a cup and then dried in an oven at 105°C. Drying was carried out until a constant weight was obtained. The determination of water content is calculated with the following equation [4].

$$\text{Water Content} = \frac{\text{initial weight} - \text{final weight}}{\text{initial weight}} \times 100\% \quad (1)$$

2.4 Anthocyanin Extraction

A total of 25 grams of dragon fruit peel powder with various particle sizes (30, 60, and 80 mesh) was wrapped with filter paper and then put into an Erlenmeyer flask. Then, the citric acid solution with various concentrations (0.3 M; 0.4 M; and 0.5 M) was added as much as 250 ml.

After that, the Erlenmeyer was placed on a stirrer to stir for (90, 120, and 150 minutes). After that, the extraction results were filtered using filter paper to produce a filtrate. Then the filtrate was put into a test tube and then centrifuged to get anthocyanin extract. After that, it was analyzed using the spectrophotometric method to calculate the anthocyanin content Essential Oil Composition Analysis

2.5 Anthocyanin Content Analysis

2.5.1 Stages of Making Buffer

The first step is to make an acetate buffer solution (pH 4.5). The sodium acetate buffer solution should be stabilized at room temperature, but the pH should always be checked before use. The spectrophotometer was switched on and allowed to stand for 10 minutes before being used for measurements. The appropriate dilution factor was determined by dissolving 0.01 ml of the sample in a 25 ml volumetric flask with acetate buffer solution (pH 4.5) set to the limit mark and shaken. So that the absorbance of the sample can be obtained compared to the initial volume to obtain the dilution volume. Sodium acetate buffer solution was added to the cuvette, then the cuvette was inserted into the spectrophotometer to be measured at λ 510 and 700 nm so that the spectrophotometer could be zeroed. Each sample was dissolved in sodium acetate buffer solution (pH 4.5) with a predetermined dilution factor. Samples dissolved using buffer

solution were allowed to stand for 5 minutes before measurement [4].

$$\text{Anthocyanins yield} = \frac{A \times BM \times FP \times 1000}{\epsilon \times b} \quad (2)$$

Description:

A : Absorbance

BM : Molecular weight cyaniding 3-glucoside (449.2 g/mol)

FP : Dilution factor

ϵ : Absorbance coefficient 26900 L/mol.cm⁻¹ expressed by cyaniding-3-glucoside

b : thickness cuvette (1 cm)

3. Results and Discussion

Determination of anthocyanin levels or concentrations in red dragon fruit skin has the aim of obtaining maximum anthocyanin levels. Several treatments have been carried out in this study, namely variations in citric acid concentration, variations in particle size, and variations in extraction time, and measured using a UV-vis spectrophotometer.

Table 2. Yield Results on Red Dragon Fruit Peel Extraction Treatment

Run	Factors 1 A: Particle size (Mesh)	Factors 2 B: Time (Menit)	Factors 3 C: Concentration (M)	Yield (mg/L)
1	80	90	0.4	11.105
2	60	120	0.4	9.348
3	80	120	0.5	10.353
4	60	120	0.4	9.684
5	60	90	0.3	9.683
6	60	120	0.3	9.683
7	80	90	0.5	9.184
8	30	150	0.4	11.188
9	30	120	0.5	11.105
10	30	90	0.4	10.270
11	30	120	0.3	11.188
12	60	120	0.4	9.573
13	60	150	0.5	11.439
14	60	120	0.4	9.132
15	60	120	0.4	9.278
16	60	150	0.3	9.101

Run	Factors 1 A: Particle size (Mesh)	Factors 2 B: Time (Menit)	Factors 3 C: Concentration (M)	Yield (mg/L)
17	40	150	0.4	9.099

The value of total anthocyanin content was calculated using a UV-Vis spectrophotometer to measure the absorbance value. Absorbance is the ratio of the intensity of the incident light to the intensity of the absorbed light. The highest value of total anthocyanin content was obtained in the sample (60 mesh, 150 minutes, 0.5 M citric acid) which resulted in 11.439 g/L. Meanwhile, the lowest total anthocyanin content value was obtained in the sample (60 mesh, 120 minutes, 0.4 M citric acid) which amounted to 7.348 mg/L.

Table 3. Comparison of Anthocyanin Level Results

Methods	Time	Content	Description
Microwave-Assisted Hydrodistillation (MAHD)	45 minutes	52.184 mg/L	[11]
Solvent Extraction	150 minutes	11.439 mg/L	-

This study shows that the extraction of anthocyanins from red dragon fruit using the solvent extraction method obtained a total anthocyanin content of 11.439 mg/L with optimal conditions of particle size 60 mesh, time 150 minutes, and solvent concentration 0.5 M by extracting according to Table 3.2 obtained with the help of Design Expert. While in some studies, the highest anthocyanin content was achieved using the MAHD method, which was 52.184 mg/L. The difference in results is influenced by the hydro distillation process.

According to Shiddiqi [11] it is said that the hydro distillation process maximizes the separation between the solvent and the extract which causes the concentration of the solution and results in higher anthocyanin content values.

The anthocyanin content obtained from the extraction of red dragon fruit peel depends on the extraction time, particle size, and solvent concentration. These variables are important factors that affect the efficiency of anthocyanin extraction from red dragon fruit skin.

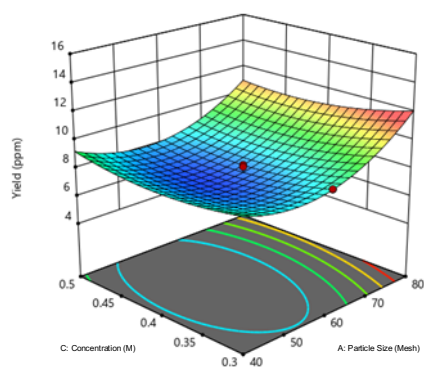


Figure 1. Effect of Concentration & Particle Size Variables on Yield

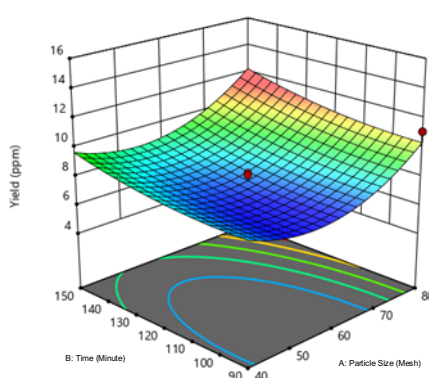


Figure 2. Effect of Time & Particle Size Variables on Yield

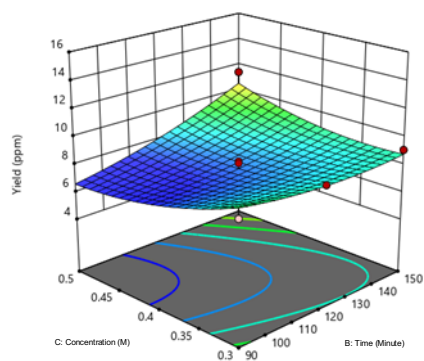


Figure 3. Effect of Concentration & Time Variables on Yield

There are several factors that can affect the increase in anthocyanin levels:

In Figure 1 & Figure 3 particle size in red dragon fruit peel yield also influence anthocyanin yield. This is because the smaller the particle, the ratio between surface area and volume increases so that it can more easily enter the dragon fruit peel powder network. According to Guntarti & Maulida [16] it is said that different particle sizes have different surface areas. The large surface between the simplisa and the solvent will provide a greater

opportunity to extract anthocyanins, which triggers the results of the 60 mesh particle size better than the 80 mesh size. So, anthocyanins will be extracted more if the solvent can interact more widely with simplisa.

In Figure 1 & Figure 3 the increase in citric acid solvent concentration in this study showed an increase in yield, while the use of citric acid solvents with lower concentrations resulted in lower yields. In this study, the extraction was carried out using a variable concentration of citric acid solvent variation of 0.3 M; 0.4 M; and 0.5 M. In this study, the highest yield was obtained when using 0.5 M citric acid solvent concentration.

This is because the more acidic conditions will cause the anthocyanin pigment to get bigger. This is because the greater the concentration of solvents, the greater the collision between solvents, thus accelerating the reaction process.

In Figure 2 & Figure 3, the extraction time influences the increasing anthocyanin yield, this is because the material has a long interaction with the solvent. The time variable itself is needed for anthocyanin exposure. Where the citric acid solvent takes time to penetrate the sample. According to Winata & Yuniarta [17] it is said that the longer the extraction time, the quantity of extracted compounds will also increase, this is because the opportunity for contact between the material and the solvent is greater.

Analysis of Variance (ANOVA) is a statistical test used to estimate which variable from the data is more dominant based on the relationship between other variables [18]. The purpose of analysis using ANOVA is to test statistical hypotheses and to determine data optimization. A parameter can be said to be significant if the analysis results in a probability value ≤ 0.05 or 5% for the p-value and a lack of fit value for the p-value ≥ 0.05 . Other parameters are the R² value greater than 0.7 and the precision value greater than 4 [19]. The research model produces a p-value of 0.0151, so the research analysis model has a significant effect on the extraction of results. ANOVA analysis results can be seen in Table 4.

Table 4. ANOVA Analysis Results

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	30.19	9	3.35	5.81	0.0151	Significant
A- Particle size	2.04	1	2.04	3.53	0.0025	
B-Time	2.61	1	2.61	4.52	0.0017	
C-Concentration	0.2439	1	0.2439	0.4221	0.0066	
AB	0.0497	1	0.0497	0.0859	0.7779	
AC	0.3617	1	0.3617	0.6260	0.4548	
BC	4.00	1	4.00	6.92	0.0338	
A ²	8.51	1	8.51	14.72	0.0064	
B ²	0.7390	1	0.7390	1.28	0.2954	

Source	Sum of Squares	df	Mean Square	F-value	p-value	
C ²	1.63	1	1.63	2.82	0.1370	
Residual	4.05	7	0.5779			
Lack of Fit	3.44	3	1.15	7.54	0.0602	Not Significant
Pure error	0.6080	4	0.1520			

The analysis results show that the model formed is significant where the f-value is 5.81 and the p-value is $0.0151 < 0.05$. Lack of fit formed $0.0602 > 0.05$. Lack of fit that is not significant states that the test model used is appropriate so that it can explain the problem being studied. The variables of particle size, time, and solvent concentration have a value of less than 0.05, which means that they have a significant effect on yield results.

R-squared (R^2) is the coefficient of determination with a value between 0 and 1. If R^2 is close to 1, then the relationship between one variable and another variable is getting stronger. Conversely, if R^2 is getting smaller, the relationship between one variable and another variable is getting weaker. The results of the fit statistics that have been carried out can be seen in Table 5.

Table 5. Fit Statistic

Std. Dev.	0.7602	R²	0.8819
Mean	9.38	Adjusted R²	0.8653
C.V.%	8.11	Predicted R²	0.6254
		Adeq Precision	6.4520

The R^2 value from the analysis results obtained 0.8819 or 88.19%, so it can be declared appropriate because the value is more than 75%. The adjusted R-value obtained is 0.8653, indicating that there is a significant relationship between the parameters of particle size, time, and solvent concentration on anthocyanin extraction results. The total anthocyanin yield as a response to the extraction parameters in the ANOVA model can be modeled using the following quadratic equation:

$$\text{Yield} = 9.36 + 0.5686 A + 0.4459 B - 0.1442 C - 0.1095 AB - 0.1891 AC + 1.10 BC + 1.07 A^2 - 0.0974 B^2 + 0.1440 C^2$$

where A is the particle size of the simplisa, B is the extraction time, and C is the solvent concentration. The regression equation can be used to determine the response value of the total yield concentration obtained when the particle size, extraction time, and solvent concentration are different.

4. Conclusion

In this study, anthocyanins can be extracted using the Solvent Extraction method. From the experimental results, the highest yields were obtained at 11.439 mg/L. Optimal conditions were achieved when the process parameters were 60 mesh particle size, 0.5 M solvent concentration, and an extraction time of 150 minutes. For further research, it is recommended to use the Microwave-Assisted Hydrodistillation (MAHD) method because the yield of anthocyanins produced is greater than the Solvent Extraction method.

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