

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2024

Volume 28, Pages 167-174

ICBASSET 2024: International Conference on Basic Sciences, Engineering and Technology

Extraction of Anthocyanin Pigments from the Peel of Dragon Fruit for Food Coloring

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Abstract: Kitchen waste is a source of methane gas which strongly contributes to the greenhouse effect (Lee et al., 2017). One of the organic waste that comes from the kitchen is fruit peel with a large percentage reaching (25-60%) (Nirmal et al., 2023). To increase waste reduction efforts can be done by converting food waste into natural dyes by extraction. This can be a solution to consumer concerns about the insecurity of synthetic dyes that are carcinogenic if consumed excessively. Natural colorants that have the potential to be extracted include anthocyanins. Dragon fruit skin contains a rich source of anthocyanin pigments. The use of natural dyes extracted from dragon fruit peel is suitable with the Sustainable Development Goals (SDGs), this context can be an interesting learning project and grow an understanding of environmental responsibility through direct learning experiences (Andersson et al., 2013). This research aims to identify the optimal method of anthocyanin extraction from dragon fruit peels which can be applied to Education Sustainable Development (ESD) orientated learning. The research method carried out is a laboratory experimental method with qualitative analysis. The parameters observed were the effect of solvent, temperature, pH, and mass on the absorbance of dragon fruit peel extract. Anthocyanin extraction from dragon fruit skin using maceration method. Based on the observation data, the best treatment is the sample with 10% citric acid solvent. Anthocyanins are most optimally stored at a low temperature of 10°C, with an acidic pH of Ph 4 and with a mass: solvent ratio of 250: 100.

Keywords: Natural dye, Anthocyanin, Waste fruit peel

Introduction

The household kitchen is a significant contributor to organic waste. Kitchen waste is a source of methane gas release contributing strongly to the greenhouse effect (Lee et al., 2017). According to Nirmal et al. (2023), one of the organic waste coming from the kitchen is fruit peels with a high percentage of up to (25-60%). Although composting is a suitable option to solve organic waste, there is still a lot of organic waste, especially fruit peel waste, that is thrown away, causing pollution to the environment, and causing bad smells. So, innovative techniques and processes are needed to convert fruit peels, which are usually a waste, into high-quality materials that have a variety of applications (Nirmal et al., 2023., Pathak et al., 2017). According to Dudziak et al. (2022), to improve waste reduction and conform with circular economy principles, it is possible to convert food waste into natural dyes by extraction. This can be a solution to concerns among consumers regarding the unsafety of synthetic dyes (Singh et al., 2023). Most synthetic dyes can cause various effects if their use is more than the threshold, and the long-term use of synthetic dyes is harmful to humans because they are carcinogenic. In addition, synthetic dyes are bad for the environment because they pollute water and soil (Kwartiningsih et al.,

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- Selection and peer-review under responsibility of the Organizing Committee of the Conference

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2016). The limitations on the use of synthetic dyes cause an increased interest in research on natural dyes. Natural dyes are safe to use in the long term are safe for health and environmentally friendly and have the potential to replace synthetic dyes. Natural dyes with potential to be extracted include anthocyanins (Almajid et al., 2021).

Anthocyanins are red, purple, and blue pigments commonly found in plants (Widyasanti et al., 2021). Anthocyanins are classified as pigments called flavonoids. Flavonoid class compounds include polar compounds and can be extracted with solvents that are polar as well. Acidic conditions will affect the extraction results. The more acidic conditions will cause more anthocyanin pigments to be extracted, this is due to the more vacuole cell walls being broken so that more anthocyanin pigments are extracted (Ariviani, 2010). Anthocyanins can replace the use of synthetic dyes rhodamine B, carmoisin, and amaranth as a red dye in food products. JEFCA (*Joint FAO/WHO Expert Committee on Food Additives*) has stated that extracts containing anthocyanins have low toxicity effects. In addition to their role as food colorants, anthocyanins are also believed to contribute to biological systems, including their ability to attract free radicals and the ability to inhibit the initiation stage of chemical reactions that cause carcinogenesis (Almajid et al., 2021).

Dragon fruit skin is the most discarded part of the fruit processing process. The peel component is about 20% of the dragon fruit (Widyasanti et al., 2021). Dragon fruit skin contains a rich source of anthocyanin pigments (Carrillo et al., 2022). The process of extracting anthocyanin pigments from dragon fruit skin is quite easy to do. One method that can be used is maceration, a process where the peel is soaked in a solvent to release the color (Vinha et al., 2018). The maceration method was chosen because the damage factor of the active substance is smaller. This is because the maceration method does not use heat, which can damage the active substances drawn. The main emphasis of this method is the availability of sufficient contact time between the solvent and the extracted tissue (Hanum, 2000). Although simple in this maceration method, it is necessary to know the factors that affect the extraction of anthocyanin compounds from dragon fruit peels such as the type of solvent, extraction temperature, extraction pH, material size, and the ratio of the amount of material to solvent (Sudarmi et al., 2015). This maceration technique is very easy to adapt for learning purposes (Dewi et al., 2020). By including maceration in school learning, students can gain practical experience in processing fruit peel waste into natural dyes (Ingrath et al., 2015).

The use of natural dyes extracted from dragon fruit peels aligns with several key aspects of the Sustainable Development Goals (SDGs) with the 12th Goal, Responsible Consumption, and Production, prioritizing minimizing waste production and optimizing resource utilization (Wang et al., 2019). Reusing fruit peels as a colorant directly aligns with this goal by turning waste into a valuable product (Otles & Kartal, 2018). In addition, SDG Goal 4, Quality Education, advocates integrating sustainability principles into the education curriculum (Treaty, 2003). This context can be an engaging learning project and foster an understanding of environmental responsibility through hands-on learning experiences (Andersson et al., 2013).

Based on the description above, it is important to identify the most optimal way of extracting anthocyanins from dragon fruit peels which can later be applied to Education Sustainable Development (ESD) with a focus on waste utilization to make useful products. So, it is expected that students can develop an understanding of the importance of protecting the environment and managing fruit peel waste.

Method

The research method conducted was a laboratory experimental method with qualitative analysis. This research was conducted using 3 different solvent treatments, aquades, 10% citric acid, and 10% acetic acid. Qualitative determination of anthocyanins was carried out with phytochemical tests and maximum wavelength measurements. The parameters observed were the effect of solvent, temperature, pH, and mass on the absorbance of dragon fruit peel extract.

Instruments and Materials

The instruments used are a stirring rod, bottle, blender, glass funnel, beaker, measuring cup, hot plate, watch glass, Whatman filter paper, cuvette, volumetric flask, fume hood, pH meter, dropper pipette, measuring pipette, knife, propped, tube rack, centrifuge, metal spatial/spatula, UV-Vis spectrophotometer, test tube, centrifuge tube, thermometer, and analytical balance. The materials used were glacial acetic acid (CH_3COOH), hydrochloric acid (HCl), citric acid ($\text{C}_6\text{H}_8\text{O}_7$), Sodium Hydroxide (NaOH) distilled water, pH 4 buffer solution,

pH 5 buffer solution, pH 7 buffer solution, pH 10 buffer solution, pH 11 buffer solution, and red dragon fruit peel.

Research Steps

This research consists of three steps, the first step is the preparation of raw materials, the second step is maceration extraction, and the third step is quality testing and data analysis.

Raw Material Preparation

Samples of red dragon fruit were washed first then peeled and cleaned to separate the pulp from the skin and wet sorting was carried out by separating the green or brown skin and the remaining pulp contained in the inner skin of the dragon fruit. Then washing and draining are done. Next, the peel is cut into small pieces and blended until it becomes smooth to expand the surface area of the sample until it is shaped like porridge.

Extraction of Anthocyanins from Red Dragon Fruit Peel

Dragon fruit peels that have been blended until they are like porridge are weighed and solvents are added (1:2 = material: solvent). The solvents used were distilled water, 10% citric acid, and 10% acetic acid. The next stage was maceration extraction for 3 days. The results of maceration for 3x24 hours (macerate) obtained were filtered using a filter and Whatman filter paper, and then the macerate obtained was centrifuged for 10 minutes (2500 rpm/minute). The supernatant obtained from the centrifuge results in a precipitate-free anthocyanin liquid extract, the anthocyanin liquid extract is stored in a bottle and the filtrate obtained is ready for analysis.

Phytochemical Test

Anthocyanin phytochemical test was conducted according to Harborne, 1996, that is, 3 mL of anthocyanin liquid extract was added 3-5 drops of 2M HCl and then heated at 100°C for 5 minutes. In addition, 3 mL of liquid anthocyanin extract can also be added to 2M NaOH drop by drop and waited for 1 minute while observing the color changes that occur.

Finding the Maximum Wavelength

Each sample was taken with 3 ml of different solvents, and then the wavelength from 200-800nm to find the maximum peak point of anthocyanins using a UV-visible spectrophotometer.

Solvent Variation

Take 3 ml of each sample with different solvents, then measure the absorbance with UV-Vis at the maximum wavelength of each sample with solvent.

Temperature Effect

Based on the absorbance results of the solvent variation, the sample that has the highest absorbance value is tested for temperature stability. Samples were made with temperatures of 10°C, 25°C to 27°C, and 80°C to 100°C and then observed by measuring the temperature using a thermometer, then 2 mL of liquid extract that had the highest anthocyanin content was taken and dissolved to 10 mL with the solvent used, then the solution was put in a test tube and treated at each of these temperatures for 30 minutes. After 30 minutes of treatment, the absorbance was seen using a UV-Vis Spectrophotometer at a predetermined maximum wavelength.

Effect of pH

Based on the absorbance results of the solvent variation, the sample that has the highest absorbance value is tested for pH stability. Measured each buffer solution that has been made by measuring the pH of the buffer solution using a pH meter, then 2 mL of liquid extract that has the highest anthocyanin content is dissolved to 10 mL with each buffer solution pH 4, 5, 7, 10, and 12, then put in a test tube and allowed to stand for 30 minutes. After 30 minutes of treatment, the absorbance was seen using a UV-Vis Spectrophotometer at a predetermined maximum wavelength.

Effect of Mass on Solvent

Based on the absorbance results of the solvent variation, the sample that has the highest absorbance value is tested for stability against mass. Samples were made with 5 variations of mass to solvent, namely 50:100, 100:100, 150:100, 200:100, and 250:100 (mass: solvent). Then all samples were macerated for 24 hours and then tested for absorbance using a UV-Vis Spectrophotometer at a predetermined maximum wavelength.

Results and Discussion

The maceration method was chosen because the damage factor of the active substance is smaller. This is because the maceration method does not use heat which can damage the active substances being extracted. The main emphasis of this method is the availability of sufficient contact time between the solvent and the extracted tissue. Maceration is done by immersing the simplistic powder in a liquid solvent. The solvent will penetrate the cell wall and enter the cell cavity containing the active substance (Hanum, 2000).

The solvents used for this maceration process are distilled aquades, acetic acid, and citric acid with a concentration of 10% each. The solvent was chosen because it is a polar organic acid solvent, the use of inorganic solvents such as HCl is avoided because the anthocyanins obtained from dragon fruit peel extract will be used as a food coloring. The anthocyanins obtained from each type of solvent were then seen for their maximum wavelength and absorbance in each solvent.

The most stable anthocyanin will be tested against the effects of temperature, pH, and mass ratio. The optimum wavelength is sought by measuring colored samples in the range of 490-580 nm with spectrophotometer analysis. The identification of anthocyanin pigments is based on the observation of the maximum absorbance located at a wavelength of 490-580 nm (Harborne, 1996).

Phytochemical Test

Phytochemical tests were conducted to identify anthocyanin compounds in the form of color tests using NaOH 2M and HCl 2M reagents. The results obtained from the anthocyanin phytochemical test on the liquid extract of red dragon fruit peel are the same as the literature used, namely according to Harborne, 1996, the positive anthocyanin sample is marked if it is dripped with 2M HCl and heated at 100 °C for 5 minutes, the color remains or does not fade and if the sample is dripped with 2M NaOH drop by drop, there is an initial color change which becomes bluish green, so that the sample of red dragon fruit peel liquid extract with the different solvents used is positive indicating the presence of anthocyanin compounds (Almajid et al., 2021).

Table 1. Results of anthocyanin phytochemical test of dragon fruit peel extract

Test	Result Aquades	Citric acid 10%	Acetic acid 10%	Description
Dropped with 5 drops of 2M HCl and heated at 100°C for 2 minutes)	Fixed color or no fading	Fixed color or no fading	Fixed color or no fading	Positive
Dropped with 5 drops of 2M NaOH and heated at 100°C for 2 minutes)	Color change or fading	Color change or fading	Color change or fading	Positive

Finding the Maximum Wavelength

The measurement results of determining the maximum wavelength using a Uv-Vis Spectrophotometer at a wavelength of 200-800 nm can be seen in Table 2.

Table 2. Results of maximum wavelength determination (λ max)

Maximum wave peak	Solution
530 nm	Aquades
531 nm	Citric acid 10 %
529 nm	Acetic acid 10%

The results of the maximum wavelength (λ max) of the research obtained are still around the λ max of the literature used that the maximum wavelength characteristics of anthocyanins have a range of visible light spectrum areas at 505-535 nm (Almajid et al., 2021). The highest maximum wavelength result was obtained from the extraction of anthocyanins from dragon fruit skin using a citric acid solution. The wavelength obtained was 531 nm.

Effect of Solvent Type

The results of anthocyanin absorbance show that the use of 10% citric acid solvent produces the highest absorbance compared to distilled water and 10% acetic acid solvents. The test results of the solvent-type test on anthocyanins can be seen in Table 3.

Table 3. Results on the effect of solvent type

Solvent	Absorbance
Aquades	0,220
Citric Acid	0,451
Acetid Acid	0,406

The difference in absorbance values produced for every solvent with the addition of types of organic acids is considered to be a result of the difference in the dissociation constant of each type of acid. Citric acid has a higher dissociation constant than acetic acid. The dissociation constants for citric acid and acetic acid are 7.21×10^{-4} and 1.75×10^{-5} , respectively (Almajid et al., 2021). The higher the dissociation constant, the stronger the acid is because the higher the number of hydrogen ions released into the solution. The more acidic situation, especially close to pH 1, will cause anthocyanin pigments to be in the form of colored flavilium or oxonium cations, and absorbance measurements will show a higher amount of anthocyanins and cause the vacuole cell walls to break so that more anthocyanins are extracted (Almajid et al., 2021).

Temperature Effect

The results of the anthocyanin stability test on temperature for 30 minutes can be seen in Table 4.

Table 4. Results on the effect of temperature

Temperature	Absorbance
10°C	0.959
25°C-27°C	0.399
50°C-80°C	0.228

Anthocyanin absorbance was measured at a wavelength of 531 nm. The higher the temperature, the lower the absorbance results produced which indicates that anthocyanins are not stable at higher temperatures. This may be because at high temperatures, anthocyanins are decomposed so the more colour that is degraded, so the lower the absorbance value (Khoo et al., 2017). This is supported by Markakis in Almajid et al. (2021), which states that the decrease in colour stability due to high temperatures is due to the decomposition of anthocyanins from the aglycone form to chalcone (colourless). Anthocyanins are stable when stored at 4-25°C under low light conditions (Vargas et al., 2013).

Effect of pH

The results of the anthocyanin stability test on pH for 30 minutes can be seen in Table 5.

Table 5. Results on the effect of pH

pH	Absorbance
4	0.468
5	0.245
7	0.163
10	0.124
12	0.123

Anthocyanin absorbance was measured at a wavelength of 513 nm. In pH 4 the acid has a better absorbance value than the buffer pH 5, 7, 10, and 12. The higher the pH given the more unstable the anthocyanin content or the higher the anthocyanin degradation from the red dragon fruit skin. At low pH, anthocyanins turn into platinum cations which are red (Lullung Sampebarra, 2018). The higher the pH, the color of the anthocyanin pigment will change to a colorless chalcone compound (Sukaminah et al., 2007).

Effect of Mass on Solvent

The results of the anthocyanin stability test on mass for 24 hours can be seen in Table 6.

Table 6. Results on the effect of mass

Mass ratio: solvent	Absorbance
50 : 100	0.686
100 : 100	1.045
150 : 100	1.057
200 : 100	1.188
250 : 100	1.599

Based on Table 6 shows the absorbance of anthocyanins measured at a wavelength of 531 nm. The mass ratio of 250:100 has a better absorbance value than the mass ratio of 50:100, 100:100, 150:100, and 200:100. However, the decrease in absorbance does not visually affect the color pigment changes in the extract but affects the amount of anthocyanin extract obtained. The more mass used during extraction, the higher the absorbance value obtained while the less mass used during extraction, the lower the absorbance value obtained. This is because the absorbance value is directly related to the concentration of the solute in the extract phase, which is affected by the amount of mass used during the extraction process. Because the more mass there is in the extract, the higher the mass concentration in the extract (Handayani et al., 2018).

Conclusion

This study discusses the importance of effective and appropriate kitchen waste management, with a focus on the utilization of fruit peel waste as a source of natural dye. One of them is separating anthocyanin pigments from red dragon fruit skin using the maceration method. The maceration method was chosen because the damage factor of the active substance is smaller. This is because the maceration method does not use heat which can damage the active substance being extracted. Based on the observation data in the laboratory, the extraction process using maceration showed the best treatment on samples with 10% citric acid solvent. Anthocyanins are most optimally stored at a low temperature of 10°C, with an acidic pH of Ph 4 and with a mass: solvent ratio of 250:100. The maceration extraction process is a simple method that can be applied in extracting anthocyanin compounds from red dragon fruit skin.

Recommendations

This study suggests paying attention to effective and appropriate kitchen waste management, by utilizing fruit peel waste as a source of natural dye. In addition, this study shows that the topic of separating anthocyanin pigments from dragon fruit peels as a natural dye can be an interesting context to be applied in chemistry learning, especially in the context of Education for Sustainable Development (ESD). Therefore, it is recommended to integrate this topic into chemistry learning to improve students' understanding of chemical separation in natural materials and the importance of developing sustainable and environmentally friendly

natural dyes. In addition, this study also showed that the utilization of anthocyanin pigment extract from dragon fruit skin as a natural dye can contribute to the achievement of SDGs. It is hoped that this research can increase public awareness about the importance of using natural dyes that are environmentally friendly and effective for increasing sustainable consumption.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

* This article was presented as an oral presentation at the International Conference on Basic Sciences, Engineering and Technology (www.icbaset.net) held in Alanya/Turkey on May 02-05, 2024.

* The researchers would like to express their deepest gratitude to the Education Fund Management Institute (LPDP and KEMENAG/Indonesia Endowment Fund for Education) under the Ministry of Finance of the Republic of Indonesia as the sponsor for their master's studies, and the support for this paper and publication

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To cite this article:

Muyassaroh, A., Supriatna, A., & Hernani, H. (2024). Extraction of anthocyanin pigments from the peel of dragon fruit for food coloring. *The Eurasia proceedings of science, technology, engineering & mathematics (EPSTEM)*, 28, 167-174.