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Production of Natural Dye from Butterfly Pea Flower (*Clitoria Ternatea T.*) Extract Using Citric Acid Solvent from Bilimbi (*Averrhoa bilimbi*)

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ABSTRACT

Natural dyes extracted from butterfly pea flowers (Clitoria ternatea L.) hold significant potential as alternatives to synthetic dyes in the food and textile industries. This study aims to evaluate the effectiveness of citric acid isolated from bilimbi (Averrhoa bilimbi L.) as a solvent for extracting anthocyanin pigments from butterfly pea flowers. Ultrasonic extraction was conducted using different solvents (citric acid and ethanol) and varying extraction durations. The results showed that the extract yield ranged from 2.6% to 5.5%, with an average of 3.99%. Ethanol produced a higher yield than citric acid; however, citric acid significantly reduced the extract's pH to a range of 1–6, potentially enhancing anthocyanin pigment stability. Additionally, extraction time positively influenced yield up to an optimal point, after which degradation of active compounds led to a decline. These findings suggest that bilimbi-derived citric acid has potential as an eco-friendly solvent for natural dye production, though further optimization is needed to enhance extraction efficiency and color stability.

Keywords: Anthocyanin, bilimbi, butterfly pea flower, citric acid, extraction

1. INTRODUCTION

Butterfly pea flower (*Clitoria ternatea L.*) is a type of flower from the Fabaceae (legume) family or the Papilionaceae subfamily [1]. This flower contains various chemical compounds such as tannins, carbohydrates, saponins, triterpenoids, phenols, flavonoids, flavonol glycosides, proteins, alkaloids, anthraquinones, anthocyanins, cardiac glycosides, stigmast-4-ene 6-dione, essential oils, and steroids [2].

Flavonoids are polyphenolic compounds with biological activities such as antioxidant, antidiabetic, antibacterial, anti-cholesterol, antiviral, antihyperlipidemic, anti-inflammatory, and anticancer properties[3].

Flavonoids are polar compounds, meaning they dissolve well in polar solvents such as ethanol, butanol, methanol, acetone, dimethyl sulfoxide, dimethylformamide, and water [4]. The extraction of secondary metabolites from butterfly pea flowers can be achieved through various extraction methods. One such method is ultrasonic extraction, which utilizes ultrasonic waves— acoustic waves with frequencies above 16-20 kHz. During the extraction process, ultrasonic waves break down cell walls, releasing the intracellular contents into the surrounding medium.

Several factors influence the efficiency of ultrasonic extraction, particularly temperature and extraction time. Margaretta et al. (2011) [5] reported that temperature increases during extraction must be carefully controlled. High extraction temperatures and excessively prolonged extraction times exceeding the optimal threshold may lead to the degradation of chemical compounds in the solution due to oxidation. Bioactive compounds, such as flavonoids, are sensitive to high temperatures above 50°C, which can alter their structure and reduce extract yield.

Indonesia is a tropical country rich in diverse natural food resources. Its humid climate allows various plants, especially fruit-bearing species, to grow abundantly. One of the most widely cultivated fruits in Indonesia is *Averrhoa bilimbi L.*, commonly known as bilimbi. This fruit is often used as a culinary ingredient, primarily to add a sour taste to dishes. Additionally, it has long been utilized in traditional medicine to treat ailments [6] such as whooping cough, digestive disorders, high blood pressure, canker sores, acne, tooth decay, paralysis, bleeding gums, and rectal inflammation [7-8].

Bilimbi fruit is rich in flavonoids, vitamin C, saponins, tannins, calcium, potassium, and various organic acids such as acetic acid, citric acid, formic acid, lactic acid, and oxalic acid [9]. Among these, citric acid is particularly significant due to its numerous benefits. Citric acid is a weak organic acid that is highly soluble in water. It is naturally found in the leaves and fruits of plants belonging to the Citrus genus. This compound serves as a natural and effective preservative and is widely used in industries such as textiles, cosmetics, and pharmaceuticals. In the food industry, citric acid is commonly used as an acidulant to enhance the sourness of foods and beverages.

Based on previous research findings, this study aims to isolate citric acid from bilimbi fruit using calcium chloride ($CaCl_2$) as the extraction medium. The use of $CaCl_2$ in this study was chosen due to its ease of application, availability, and the fact that it does not require specialized equipment, unlike more complex methods such as Soxhlet extraction and Supported Liquid Membrane (SLM) techniques.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used were bilimbi (Averrhoa bilimbi), butterfly pea flowers (*Clitoria ternatea*), NaOH 10%, NaOH 2 M, $CaCl_2$ 5%, HCl 2 M, H_2SO_4 2 M, ethanol 70%, distilled water, filter paper, and universal indicator. The equipments used include standard beakers, erlenmeyer flasks, measuring cylinders, a heating device, a glass stirrer, a glass funnel, and measuring cylinders.

2.2. Research Procedure

The star fruit was thoroughly washed and processed using a juicer. The result of the juicer was filtered to obtain an extraction. In this study, about 400 mL and 500 mL of fruit juice was used. The isolation of citric acid, firstly the starfruit juice was poured into a beaker. The fruit juice was added to NaOH 10% solution gradually until the pH reached 7-8 then filtered with filter paper. The filtrate was added with 10% $CaCl_2$ solution as much as 50 mL per 100 mL of fruit juice. The solution was stirred for 15 minutes using a magnetic stirrer, heated to boiling, and filtered with filter paper. The precipitate was collected and washed with hot water, followed by the addition of 5 mL of 2 M HCl per 100 mL of fruit juice. Next, NaOH 2 M was added dropwise until the pH reached 7.5. The solution was then boiled and filtered again with filter paper. The precipitate formed was collected and dried at room temperature. After drying, the precipitate was weighed and added with 2 M H_2SO_4. The solution was then stirred for ± 30 minute and filtered. Subsequently, the filtrate was concentrated by heating. The concentrate solution was left at room

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temperature until the formation of citric acid crystals. The citric acid crystals were filtered, dried at room temperature and weighed.

Preparation of butterfly pea (*Clitoria ternatea*) flower extract started by washing the dried butterfly pea flowers and removing the damaged parts. About 50 g of dried butterfly pea flowers was boiled in 500 ml of clean water (or a mixture of 250 ml of water and 250 ml of 70% ethanol) over low heat for 15-30 minutes, stirring occasionally to avoid vigorous boiling. Afterwards, filter the hot solution using a sieve or cheesecloth and squeeze the pulp to ensure that all the extract was removed. And finally, the final pH of the solution is measured. The experiment was repeated by using citric acid solvent.

3. RESULTS AND DISCUSSION

3.1. Effect of Extraction Time and Solvent Type on Butterfly Pea Flower Extract Yield

This study examined the yield of butterfly pea flower extract, finding that it ranged between 2.6% and 5.5%, with an average of 3.99%. This variation was notably influenced by two main factors: extraction time and the type of solvent used.

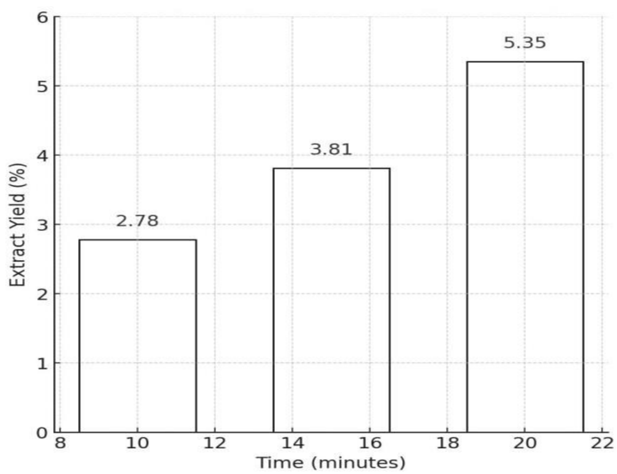

Figure 1. Draw graphs of yield and extraction time

Figure 1 depicts a strong positive correlation between extraction time and yield up to an optimal point. Beyond this point, the yield tended to decrease, likely due to the degradation of the active components in the butterfly pea flower caused by prolonged exposure to the solvent or environmental factors such as temperature and light

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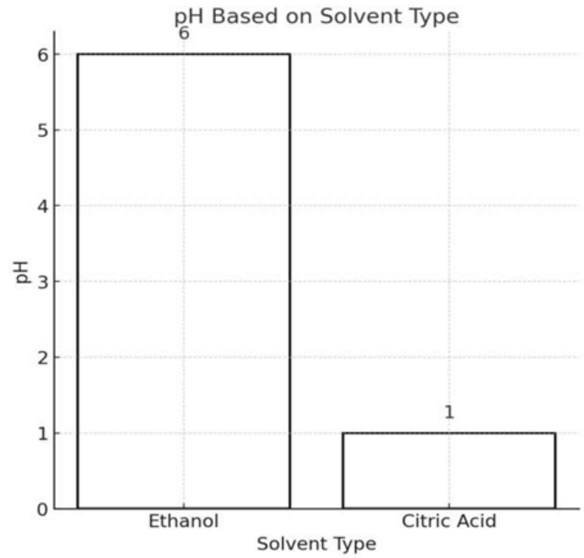

Figure 2. Yield graph & type of solvent

Furthermore, Figure 2 illustrates the type of solvent significantly affected the yield as well. Ethanol demonstrated better performance than citric acid, resulting in a higher yield. This is likely due to differences in polarity and the solvent's ability to extract active compounds from the butterfly pea flower matrix. As an organic solvent, ethanol may be more effective in penetrating cell walls and dissolving the desired components.

3.2 Analysis of pH

The study found that the pH values of the butterfly pea flower extract ranged from 1 to 6. Data analysis showed that extraction time had little effect on the extract's pH. However, the type of solvent, particularly citric acid, had a significant impact on the acidity (pH) of the butterfly pea flower extract.

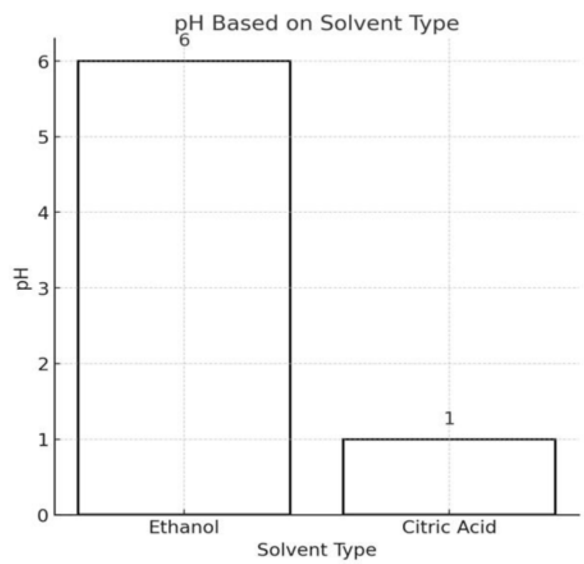

Figure 3. Effect of solvent on pH

As it observed in Figure 3, the effect of solvent on pH showed various ranging from 1 to 6 depending on the

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solvent used. Unlike yield, extraction time did not have a significant effect on pH values. However, the type of solvent demonstrated a clear role. The use of citric acid, being an organic acid, significantly reduced the pH of the extract compared to other solvents. This difference in pH is crucial as it can affect the stability and application of the butterfly pea flower extract. Extreme pH conditions (either highly acidic or basic) can lead to the degradation of active compounds and reduce extract quality.

Therefore, the choice of solvent and the optimization of extraction time should be well-considered in terms of yield and the desired pH value for future applications. Further investigation could explore on identifying active compounds in the extract, examining the effect of extraction temperature, and optimizing extraction methods to enhance the efficiency and quality of butterfly pea flower extract.

4. CONCLUSION

The results of this study indicate that citric acid from bilimbi has potential as a solvent for extracting anthocyanin pigments from butterfly pea flowers, particularly in maintaining color stability. However, its effectiveness in yielding extract remains lower compared to ethanol. This difference is likely due to variations in polarity and the solvent's ability to penetrate cell walls and dissolve active compounds. Additionally, the use of citric acid significantly affects the extract's pH, which tends to be lower compared to other solvents. This condition may help enhance anthocyanin pigment stability, as these compounds are more resistant in acidic environments.

Nevertheless, further research is needed to ensure the color's durability against external factors such as light and temperature. Regarding the extraction process, a positive correlation was observed between extraction time and yield up to an optimal point. Beyond this point, the yield decreased, possibly due to the degradation of active compounds caused by oxidation or excessive heat exposure. Therefore, determining the optimal extraction duration is crucial to ensure that the natural dye produced maintains its best quality without pigment degradation.

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