DOI: <https://doi.org/10.5327/fst.00119>

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Effect of three drying methods on physicochemical properties of powdered butterfly pea flower extract (*Clitoria ternatea L***.)**

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Abstract

The butterfly pea flower (*Clitoria ternatea L*.) is a flower that can be grown as a medicinal plant because it contains anthocyanins, which have antioxidant activity. Anthocyanins are compounds that play a role in giving red, purple, and blue colors to petals and fruits. The purpose of this study was to determine the effect of three methods of spray, vacuum, and freeze drying on the physicochemical properties of powdered butterfly pea extract and determine the appropriate type of drying to produce powdered butterfly pea extract as a natural dye. The method used in this study was a randomized block design (RBD) with three treatment levels and four repetitions. The treatments used included drying using a vacuum, spraying, and freezing. The analysis carried out was a physicochemical analysis, which included water content, antioxidant activity, total anthocyanin, dissolution time, solubility, color intensity, and hygroscopicity level. The results showed that the vacuum-, spray-, and freeze-drying methods had a significant effect on water content, antioxidant activity, dissolving time, solubility, color, and hygroscopicity but did not have a significant effect on total anthocyanin. Freeze drying is the best treatment on the parameters of water content, antioxidant activity, and color intensity. Meanwhile, for the solubility level and dissolution time parameters, the best results were in the spray-drying treatment. Vacuum drying is the best treatment on the parameters of total anthocyanin and hygroscopicity.

Keywords: butterfly pea; spray drying; vacuum drying; freeze drying; natural dyes.

Practical Application: This manuscript is relevant for relevant to the food and beverage industry, especially food and beverage products with the addition of natural coloring as an ingredient to make the product more attractive. This research also contains drying methods that can be used to dry food products, especially those containing anthocyanin compounds.

1 INTRODUCTION

The butterfly pea flower (*Clitoria ternatea L*.), a flower obtained from a widely known medicinal plant, is used to treat various diseases such as fever, phlegm removal, and chronic bronchitis because the butterfly pea plant contains anthocyanins, which have antioxidant activity (Suarna, 2005). Butterfly pea flowers can improve the quality attributes of food color, so they have advantages that are quite beneficial for the food industry (Makasana et al., 2017). Indonesia has very rich natural resources, including various types of plants. However, there is a lack of public knowledge about plants that can be used as natural medicines, and they have not been used properly, one of which is the butterfly pea flower (*C. ternatea* L.) that has antioxidant, antibacterial, anticancer, anti-inflammatory, analgesic, antipyretic, and antidiabetic properties (Purba, 2020).

In Indonesia, butterfly pea flowers are generally used as a food coloring agent or directly boiled to be used as herbal medicine, so they are not yet popular among the public to make further products. Until now, research on the development of butterfly peas has not been carried out because many do not know the benefits of butterfly peas. The use of butterfly pea flowers in the food sector has been carried out in several countries. The blue color of the butterfly pea flower has been used as a blue dye for glutinous rice in Malaysia. Butterfly pea flowers are also eaten as a vegetable in Kerala (India) and the Philippines (Lee et al., 2011).

The use of butterfly pea flowers in Indonesia has traditionally been used as a natural food coloring agent for candies, popsicles, and some cosmetic ingredients. In several countries, such as India, China, Central America, and South America, butterfly pea flowers are also used as a natural food coloring agent for rice (Mohamed & Taha, 2011). Generally, butterfly pea flowers are prepared by soaking, boiling, or brewing them as tea (Mukherjee et al., 2008).

2 National Research and Innovation Agency, Jakarta, Indonesia.

Received 7 Oct, 2023.

Accepted 6 Aug., 2023.

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According to Makasana et al. (2017), butterfly pea flowers are purple and contain anthocyanin compounds with good stability to produce the blue and red colors of butterfly pea flowers. Extraction is a way to get anthocyanins in butterfly pea flowers, a process of separating a sample or solvent component. Anthocyanins are compounds that are polar, so they can be extracted optimally with equally polar solvents (Tetti, 2014). Anthocyanins are natural pigments derived from water-soluble flavonoids, which produce blue, purple, magenta, red, and orange colors in most plant species (Miguel, 2011).

According to Tetti (2014) and Angriani (2019), the extraction method commonly used in anthocyanin extraction is maceration. Besides being simple, this method is one of the most widely used and can avoid damage to the anthocyanin compounds found in butterfly pea flowers. The use of polar solvents, such as polar ethanol, can produce the most anthocyanins in butterfly pea flowers (Pham et al., 2019).

The drying methods used to make the butterfly pea flower extract into a natural dye powder are spray, vacuum, and freezing. The advantage is that it can be adjusted to the temperature and does not depend on the weather (Duan et al., 2010). The freeze-drying method is the most effective method for drying substances that are susceptible to heat. The working principle is to freeze the material and reduce the pressure around it, which causes the sublimation of water particles into the gas phase (Ozkan & Bilek, 2014). The vacuum-drying method is short because the partial pressure of water vapor is lower than atmospheric pressure. This method has the principle of lowering the partial pressure of water vapor in the air in the drying chamber (Asgar et al., 2013). The spray-drying method is a drying method that is widely used in the food industry and capable of producing products in powder form (Siddick & Ganesh, 2013).

Natural dyes are generally in concentrated form, but dyes in concentrated form have several drawbacks that the natural dye extracts produced must be used immediately, have low stability, and have a short shelf life (Tama et al., 2014). The production of powder products is carried out by adding maltodextrin fillers to increase the volume and weight of the powder produced and accelerating drying (Gonnissen et al., 2008).

2 MATERIALS, TOOLS, AND RESEARCH METHODS

2.1 Ingredients

The materials used are butterfly pea flower, which is the raw material, DPP, distilled water, ethanol PA, HCl (hydrochloric acid), sodium acetate trihydrate, potassium chloride, ammonium chloride, KCl buffer, and Na-acetate buffer, which is the material for analysis.

2.2 Tools

The tools used include a spray dryer, a vacuum dryer, a freeze dryer, a desiccator, dark vials, a measuring flask, a cuvette, a micropipette, a glass beaker, a funnel, muslin cloth, a centrifugation tube, mortar and pestle, aluminum foil, a colorimeter, Whatman filter paper No. 42, and a centrifugation machine.

2.3 Research methods

This study used a randomized block design (RBD) with three treatment levels and four replications. The factor to be studied is the drying method of powdered butterfly pea flower extract, which consists of three methods: spray drying, vacuum drying, and freeze drying. The resulting data were analyzed using variance to obtain an estimator of the error variance and determine whether or not there was a treatment effect. If there was a significant difference between the treatments, the test was continued with the physicochemical properties test. The results of the various analyses were then F-tested to determine the level of difference in each treatment; if it turned out that the calculated F was greater than the F table, then it was continued with Duncan's multiple range test (DMRT) at the 5% level.

3 RESULTS AND DISCUSSION

3.1 Moisture content

The results of the water content analysis of powdered butterfly pea flower extract are presented in Table 1.

In the three treatments, the drying method of powdered butterfly pea flower extract on water content showed that treatment A (vacuum) was not significantly different from treatment B (spray), but significantly different from treatment C (freeze). Treatment C (freeze) has the best results with a percentage of the water content of 2.53%. It is suspected that freeze-dried food will last longer because this method removes more water content from food than the drying methods A (vacuum) and B (spray) to minimize damage to food ingredients by microorganisms and enzymes. Strengthened by Maulina et al. (2012), freeze drying can retain a water content of up to 1%, so that the dried natural products become more stable and meet the requirements for making pharmaceutical preparations from natural ingredients whose content must be less than 10%. Based on SNI (01-7085-2005) regarding Simplicia powder, the water content for Simplicia is a maximum of 10%. The results showed that all treatments were by SNI.

3.2 Antioxidant Activity (IC50)

The results of the antioxidant activity analysis of powdered butterfly pea flower extract are presented in Table 2.

In the treatment of the three drying methods of powdered butterfly pea flower extract on antioxidant activity, it was shown that in treatments A (vacuum), B (spray), and C (freeze), there were significant differences between the treatments. Treatments A (spray), B (vacuum), and C (freeze) had an antioxidant activity of 97.21, 64.27, and 60.45 mg/L, respectively. The powders

Table 1. The moisture content of powdered butterfly pea flower extract

Water content (%wb)
3.36 ^b
3.03 ^b
2.53 ^a

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

produced in this study as a whole were included in the category of strong antioxidant activity.

According to Molyneux (2004), a compound is considered to have very strong antioxidant activity if the IC50 value is < 50 ppm; strong if the IC50 value is between 50 and 100 ppm; moderate if the IC50 value is between 100 and 150 ppm; and weak if the IC50 value is between 150 and 200 ppm. Based on the statement, it can be said that the lower the IC50 value, the stronger the antioxidant activity of a material.

Treatment C (freeze) showed the best treatment because it had the lowest IC50 value of 60.45 mg/L in the strong antioxidant activity category, which meant that the antioxidant activity was the best. It is suspected that the freeze-drying method has an advantage in maintaining the quality of the drying results. According to Ademiluyi et al. (2018), moringa leaves dried by the freeze-drying method had the highest antioxidant activity compared with other drying methods.

3.3 Total anthocynin

The results of the total anthocyanin analysis of powdered butterfly pea flower extract are presented in Table 3.

Based on Table 3, treatment A (vacuum) is the best method because it has a greater total anthocyanin of 51.84 mg/L. The vacuum-drying method produces the highest total anthocyanins compared with the spray- and freeze-drying methods. This is suspected because, in the vacuum drying process, the operating temperature is quite low, which is around 40–60°C. The drying process under vacuum and at low temperatures has several advantages that it does not damage the texture and appearance of the ingredients and it minimizes the wastage of volatile aromas and active ingredients.

According to Zussiva et al. (2012), a temperature of 60°C resulted in a higher anthocyanin value in butterfly pea flower extract compared with a lower temperature. This shows that high temperatures (more than 60°C) and longer steeping times can reduce the anthocyanin value of butterfly pea herbal tea. According to Markakis (1982) in Zussiva et al. (2012), drying temperatures that are higher than 60°C promote degradation of anthocyanin compounds.

3.4 Color intensity

The results of the color intensity analysis of powdered butterfly pea flower extract are presented in Table 4.

In the treatment of the three drying methods of powdered butterfly pea flower extract, the L* value showed that treatment B (spray) was not significantly different from treatment C (freeze) but significantly different from treatment A (vacuum). The a* value indicates that, for treatment A (vacuum), there is no significant difference with treatment B (spray) and treatment B (spray) has no significant difference with treatment C (freeze), but there is a real difference between treatment A (vacuum) and treatment C (freeze), and the value of b^* shows that there is a significant difference between treatments A (vacuum), B (spray), and C (freeze).

The results of the analysis showed that the highest L^* value of 54.60 was found in treatment C (freeze). The greater the L value, the brighter the color of the powdered butterfly pea extract. The high value in treatment C (freeze) is thought to be due to the low anthocyanin content, so the resulting color is brighter than the other treatments. The highest a* average value found in treatment A (vacuum) was 20.12, and the lowest value found in treatment C (freeze) was 18.00. This shows that treatment C (freeze) tends to be redder than other treatments. The highest average value of b^* found in treatment A (vacuum) was 8.70, and the lowest average value found in treatment C (freeze) was 5.49. This shows treatment C (freeze) has a blue color compared with other treatments. Overall, it shows that the powdered butterfly pea flower extract has a color that tends to be purplish. According to Khuluq et al. (2007), the high pigment content in the extracted material will affect the brightness level.

3.5 Solubility

The results of the solubility analysis of powdered butterfly pea flower extract are presented in Table 5.

In the treatment of the three drying methods of powdered butterfly pea flower extract, the solubility level showed that treatment A (vacuum) was not significantly different from treatment C (freeze), but significantly different from treatment B (spray). From the observed data, it can be seen that the solubility of

Table 2. Antioxidant activity (IC50) of powdered butterfly pea flower extract.

Treatment	$IC_{50} (mg/L)$
$A = vacuum$	97.21°
$B = spray$	64.27 ^b
$C = \text{freeze}$	60.45°

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

Table 3. Total anthocyanin of powdered butterfly pea flower extract.

Treatment	Total anthocyanin (mg/L)
$A = vacuum$	51.84°
$B = spray$	48.15°
$C = \text{freeze}$	46.26 ^a

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

Table 4. Color intensity of powdered butterfly pea flower extract.

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

Table 5. Solubility of powdered butterfly pea flower extract

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

the powder in water ranges from 97.12 to 98.52%. The powder produced in this study as a whole is included in the category of having high solubility in water. That is presumably because the solubility of the butterfly pea flower extract powder is also affected by the addition of maltodextrin.

The best solubility was in treatment B (spray) because it had the highest solubility of 98.52%. This was alleged because spray drying produced fine particles in the form of powder. Reinforced by Hall (1979), the advantages of using spray drying are that the solubility of the resulting dry material is very good without coming into contact with metal plates, changes in flavor are not so obvious, the resulting particle size is smooth so that it is easily dispersed in water, contact with heat is very short, and operation is easy.

According to Hakim and Chamidah (2013), the solubility of powders is influenced by the type and concentration of fillers or binders used during the drying process. The solubility of the product in powder form is above 95%. Maltodextrin has high solubility properties, low hygroscopic properties, and low browning properties, can inhibit crystallization, and has a strong water-holding capacity (Srihari et al., 2010).

3.6 Soluble time

The results of the soluble time analysis of powdered butterfly pea flower extract are presented in Table 6.

In the three treatments, the drying method of powdered butterfly pea flower extract in terms of dissolving time showed that there was a significant difference between treatments A (vacuum), B (spray), and C (freeze). Treatment B (spray) has the best results with a shorter dissolution time of 145.25 s. It is suspected that the air pressure comes from compressed air using a compressor to suppress fluids or materials that are atomized using a nozzle. The nozzle in the drying process acts as a spraying tool used to atomize the material or make the droplets as small as possible so that the dissolving time is fast.

Reinforced by Hall (1979), the advantages of using spray drying are that the solubility of the dry ingredients produced is very good without contact with metal plates, changes in taste are not so obvious, the resulting particle size is fine so that it is easily dispersed in water, contact with heat is very short, and the operation is easy.

3.7 Hygroscopicity level

The results of the hygroscopicity level analysis of powdered butterfly pea flower extract are presented in Table 7.

In the treatment of the three drying methods of powdered butterfly pea flower extract, the level of hygroscopicity showed that there were significant differences in treatments A (vacuum), B (spray), and C (freeze). Treatments A (vacuum), B (spray), and C (freeze) for powdered butterfly pea flower extract ranged from 11.53% to 12.74%, which means that the material is classified as a hygroscopic material. The degree of hygroscopicity is the final content of the material after being placed in the air with a conditioned RH. Non-hygroscopic products have a

Table 6. Soluble time of powdered butterfly pea flower extract

Treatment	Late time (seconds)
$A = vacuum$	157.75c
$B = spray$	145.25°
$C = \text{freeze}$	$152.75^{\rm b}$

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

Table 7. Hygroscopicity level of powdered butterfly pea flower extract

11.35°
12.13 ^b
12.74c

Superscripts a, b, and c show that there are real differences in the Duncan 5% follow-up test.

hygroscopicity level below 10%. Hygroscopic materials make it easier for the product to bind water from the environment so that the product becomes sticky and can cause changes in moisture content during storage (Schuck et al., 2012).

Powdered butterfly pea extract in treatment C (freeze) has lower levels, and in the level of hygroscopicity, treatment C (freeze) has a higher level of hygroscopicity. This is thought to be an indication that the water content of the product after the drying process is very low and has a high level of hygroscopicity, which is quite high, so that it requires faster handling when taking the product from the dryer and packaging it using moisture-tight packaging to avoid increasing the water content in the product.

The hygroscopicity parameter does not directly determine whether the quality is good or bad. Hygroscopicity is closely related to the stability and shelf life of a product. Products with a high hygroscopicity value will trigger a higher absorption of water vapor. The decline in the quality of food ingredients increases in line with the increase in the water content of food ingredients (Goula & Adamopoulos, 2010). Factors that affect the hygroscopicity value are the surface area of the product, the moisture content of the product, and the components contained therein (Darniadi et al., 2010).

4 CONCLUSION

Based on the research results regarding the effect of three drying methods on the physicochemical properties of powdered butterfly pea flower extract (*C. ternatea* L.), it can be concluded as follows:

- Treatment with vacuum-, spray-, and freeze-drying methods had a significant effect on water content, antioxidant activity, dissolution time, solubility, color, and hygroscopicity but did not have a significant effect on total anthocyanins;
- Freeze drying is the best treatment at the level of moisture content, antioxidant activity, and color intensity. Meanwhile, at the level of solubility and dissolving time, the best results were obtained in the spray-drying treatment. Vacuum drying is the best treatment at the level of total anthocyanin and hygroscopicity.

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