International Journal of Technology 15(5) 1282-1291 (2024)
Received November 2022 / Revised December 2022 / Accepted January 2023

International Journal of Technology

http://ijtech.eng.ui.ac.id

Effect of Extraction and Spray Drying Temperatures on The Bioactive Materials Content in Red Dragon Fruit Skin

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Abstract. This study aimed to investigate the effect of extraction and spray drying temperatures on the yield of bioactive materials during the production of dragon fruit skin extract powder as a natural food colorant. Extraction temperature was varied between 25 and 48 °C, and spray drying was conducted at 140, 170, and 190 °C. Moreover, the effects of spray drying aids were also evaluated. At extraction temperatures below 50 °C, higher temperatures resulted in a higher yield of bioactive materials and a significant increase in antioxidant activity. Regarding spray drying, the use of higher air inlet temperatures resulted in a peaking trend of yield from spray drying. However, employing high air inlet temperatures (above 170 °C) for spray drying may result in a decrease in the recoveries of bioactive materials due to the higher degradation rates. Finally, the addition of maltodextrin as a carrier agent in spray drying could enhance the powder yield.

Keywords: Antioxidant; Dragon fruit skin; Food colorants; Spray drying; Thermal sensitivity

1. Introduction

Food antioxidants and colorants are two types of food additives that are commonly applied to enhance the properties of food, such as its appearance, taste, shelf life, or other qualities. Food antioxidants are used to enhance the shelf life of food, specifically by maintaining nutritional quality and avoiding undesirable changes in color, flavor, and texture Shofinita *et al.* (2021). Moreover, antioxidants could prevent chronic diseases caused by free radicals, such as cancer, brain dysfunction, and heart disease (Krisanti *et al.*, 2020). To this date, food antioxidants and food colorants that are commonly used are synthetic based. However, there are several concerns regarding the use of synthetic-based food additives due to their potentially detrimental effects on human health and well-being. Synthetic-based food additives could alter food's vitamin contents and cause allergies such as diarrhea, skin irritation, stomach disorders, or increased body heat (Kamal and Fawzia, 2018). Therefore, these concerns encourage the exploration to discover natural-based food antioxidants and colorants that are presumably safer and also have health-promoting properties by containing functional food ingredients (Brauch, 2016).

Dragon fruit skin is one of the potential sources of food antioxidants and colorants

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doi: 10.14716/ijtech.v15i5.6040

(Madane *et al.,* 2019; Cunha *et al.,* 2018). In the food industry, dragon fruit is a raw material for juice production, which leaves the dragon fruit skin as waste. Previous studies reported that dragon fruit skin contains bioactive compounds, such as phenolic compounds, antioxidant compounds, pectin, and betacyanin pigments (Madane *et al.,* 2019; Nguyen and Pirak, 2019; Priatni and Pradita, 2015). Hence, the utilization of fruit skin to produce valuable products could become a pathway to lower its environmental impact and add further profit for fruit farmers and the juice industry (Harimawan *et al.,* 2024).

The production of food antioxidants and colorants from dragon fruit can be done in two stages: extraction and drying. Extraction is carried out to obtain the bioactive materials, while spray drying is carried out to convert the extract into powder. Solid-liquid extraction of bioactive materials is an important step that affects the yield of the production process. Some variables that affect the extraction yield are the type of solvent, temperature, and solvent-to-solid ratio (Widiputri *et al.,* 2020).

Drying of the extracts is also considered a vital step during the production of food additives due to the thermal susceptibility of the bioactive materials (Shofinita *et al.,* 2023). Spray drying has been considered a suitable method for drying food ingredients, including those sensitive to thermal processing (Darniadi *et al.,* 2019). During spray drying, the drying process occurs rapidly, with particle temperatures usually below 100 °C. Some studies have reported that the recovery of bioactive materials, such as phenolic compounds, antioxidant compounds, and betacyanins, during spray drying is relatively high (Delia *et al.,* 2019; Shofinita and Langrish, 2016; Tze *et al.,* 2012). However, the stickiness of the powder may occur during the spray drying of bioactive materials due to the low glass transition temperature (Sormoli and Langrish, 2016). The addition of drying aids in the spray drying feed may reduce the stickiness, hence increasing the spray drying yields. Maltodextrin is used as a drying aid due to its good biocompatibility, wide availability, and high glass transition temperature. It also easily dissolves in cold water (Sahlan *et al.,* 2019). Other drying aids include gum Arabic and whey protein isolate (Handojo *et al.,* 2022; Shofinita *et al.,* 2015).

Temperature is an important variable in both extraction and spray drying processes. However, the effects of extraction and spray drying temperatures on the quality and quantity of dragon fruit peel extract and powder have not been explored before. Thus, this study aimed to investigate the effects of extraction and spray drying temperatures, as well as spray drying aids during the production of dragon fruit extract powder. The total phenolic compounds, antioxidant activity, and betacyanin contents of the extracts were characterized and quantified using the spectrophotometry method. In addition, the recovery of solid materials during spray drying was also assessed in correlation with the inlet temperatures and different types of spray drying aid.

2. Methods

2.1. Materials

Materials that were used included Folin-Ciocalteu's reagent (Merck, produced in Germany), DPPH (2,2-diphenyl-1-picrylhydrazyl) (Sigma Aldrich, produced in Germany), and Na_2CO_3 (Bratachem, produced in Indonesia).

2.2. Extraction

Red dragon fruit (*Hylocereus polyrhizus*) was obtained from a local supermarket in Bandung, Indonesia. The extraction of dragon fruit extract was carried out by maceration. Maceration provides a simple extraction process at 25 °C and 1 atm, thus preventing the degradation of materials (Rasul, 2018). Dragon fruit was firstly washed, cut, peeled, and then the skin was crushed by a food processor to produce dragon fruit skin pulp. The extraction was then carried out with a solvent-to-solid ratio of 1:1 and an extraction time of 150 minutes. The extraction temperature varied between 25 and 48 °C. These operating conditions are chosen based on previous research by Shofinita *et al.* (2021), where a 1:1 ratio provided a high extraction rate and yield without requiring high energy. Furthermore, in 150 minutes, the system has reached equilibrium. The dragon fruit skin extracts were filtered using filter paper and then kept in the fridge for the further spray drying process.

2.3. Spray drying of the dragon fruit skin extract

Spray dryer feed was prepared as a control (without any carrier agent) and with the addition of maltodextrin as a carrier agent. For the variation with the carrier agent, the extracts were prepared by adding the carrier agent at concentrations of between 20-40% of the total solid content of the extracts. Spray drying was carried out by a laboratory-scale spray dryer (Procept 4M8-TriX Spray Dryer, Belgium). The spray drying of dragon fruit skin extract was then carried out at the following conditions: inlet temperatures 140, 170, and 190 °C, feed flow rate 4 mL/min, and inlet air flow rate 0.4 L/min. The powder in the collecting vessel was weighed and compared with the solid content in the liquid feed in order to determine the spray drying yield.

2.4. Characterization and quantification of total phenolic compounds (TPC)

Extract (0.1 mL) was mixed with 0.2 mL of demineralized water and 1.5 mL of Folin Ciocalteu's reagent. The mixture was kept for 3 minutes, and then 1.2 mL of 7.5% Na_2CO_3 was added to the container (Shofinita and Langrish, 2016). The mixture was then kept in the dark for 30 minutes, and the absorbance of the sample was measured using spectrophotometry with a wavelength of 765 nm. The gallic acid solution was used as a calibration standard, and the TPC of the extract was stated as mg gallic acid equivalent (GAE)/g material.

2.5. Characterization and quantification of antioxidant activity

Extracts were diluted to different concentrations by using ethanol (Mardiah *et al.*, 2022; Shofinita and Langrish, 2016). Extracts (2 mL) were then mixed with 6×10^{-5} M DPPH solution in ethanol (2 mL). The mixture was then placed in a dark room for 30 minutes, and then the absorbance was measured using a spectrophotometer at a wavelength of 517 nm. DPPH solution in ethanol was used as a control solution. The inhibition percentage (% inhibition) of the extract was calculated using Equation 1, where $A_{control}$ is the absorbance of the control solution, and A_{sample} is the absorbance of the extract.

$$\%_{inhibition} = \frac{A_{control} - A_{sample}}{A_{control}} \times 100\% \quad (1)$$

The scavenging concentration of each sample at 50% (SC_{50}) was also determined.

2.6. Characterization and quantification of betacyanin content

The extract (0.1 mL) was diluted using demineralized water (Shofinita *et al.*, 2020). The absorbance of the diluted sample was then measured by a spectrophotometer at a wavelength of 535 nm. The amount of betacyanin was calculated using Equation 2 below:

$$BC\left({}^{mg}/_{g\ material}\right) = \frac{A \times DF \times Mr \times V_d}{\varepsilon \times L \times w_d} \quad (2)$$

where:
A = absorbance value
DF = dilution factor
Mr = molecular weight (550 g/mol for betacyanin and 339 g/mol for betaxanthin)
V_d = solution volume (mL)

ε = molar attenuation coefficient [60000 L/(mol . cm) for betacyanin and 48000 L/(mol . cm) for betaxanthin]
L = cuvette length (1 cm)
W_d = dragon fruit skin mass (g)

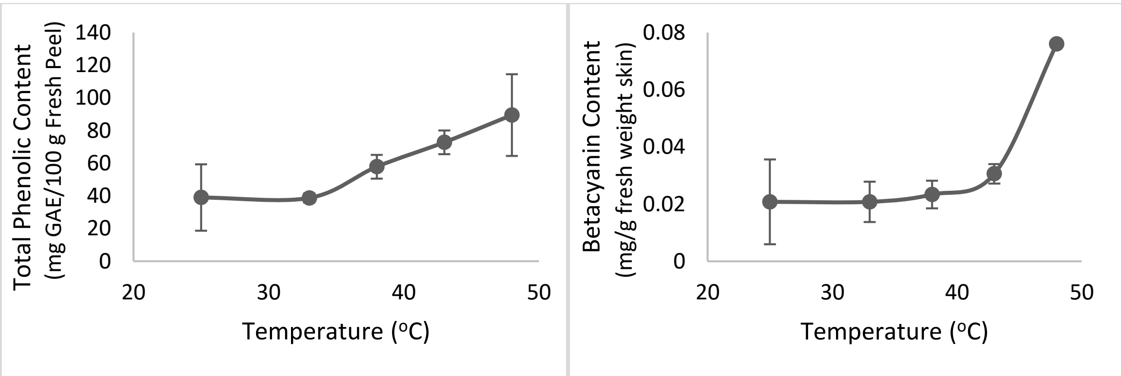
2.7. Statistical analysis

Data in this study were obtained from three replicates for each experiment and are presented as means ± standard deviation. For statistical analysis, differences were tested for significance using the ANOVA method, with a significance level of $P \leqslant 0.05$.

3. Results and Discussion

3.1. Effect of extraction temperature on the yield of bioactive materials

Figure 1a shows the effect of extraction temperature on the TPC of the dragon fruit skin extracts. The result shows that there was a significant increase in the TPC of the extract obtained at a higher extraction temperature (p-value < 0.05). A higher extraction temperature may cause an increase in the solubility of phenolic compounds in the solvent, thus increasing the extraction yield. In addition, an increase in the extraction temperature may increase the extraction rate. This trend is also reported by previous studies (Spigno and De Faveri, 2007; Liyana-Pathirana and Shahidi, 2005) during the extraction of phenolic compounds from wheat and grape stalks and marc. However, another study also reported a possibility of phenolic degradation at higher temperatures, particularly above 60 °C (Cacace and Mazza, 2003).



(a) (b)

Figure 1 Effect of extraction temperature on (a) the total phenolic compounds and (b) betacyanin content of the dragon fruit skin extracts

The TPC obtained in this study was between 39-89 mg/100 g fresh weight dragon fruit peel. Som *et al.* (2019) reported the TPC of dragon fruit peel extracts was between 18.89 and 48.15 mg/100 g, which is similar to the values found in this study. Figure 1b shows the effect of extraction temperature on the betacyanin content of the dragon fruit skin extracts. The result indicates that increasing the extraction temperature may affect the betacyanin yield significantly (p-value < 0.05). A previous study has also reported that higher temperatures are favorable for the extraction of betacyanin from red beetroot (De Azeredo *et al.*, 2009).

The values of betacyanin found in this study were between 0.021 and 0.076 mg/g of fresh dragon fruit skin extract. These values are slightly higher than the value reported previously regarding the betacyanin content of dragon fruit skin extracts obtained at room temperature, which was 0.015 mg betacyanin/g dragon fruit skin, which may be attributed to the differences in the dragon fruit source, genetics, and maturity (Harivaindaran, Rebecca, and Chandran, 2008).

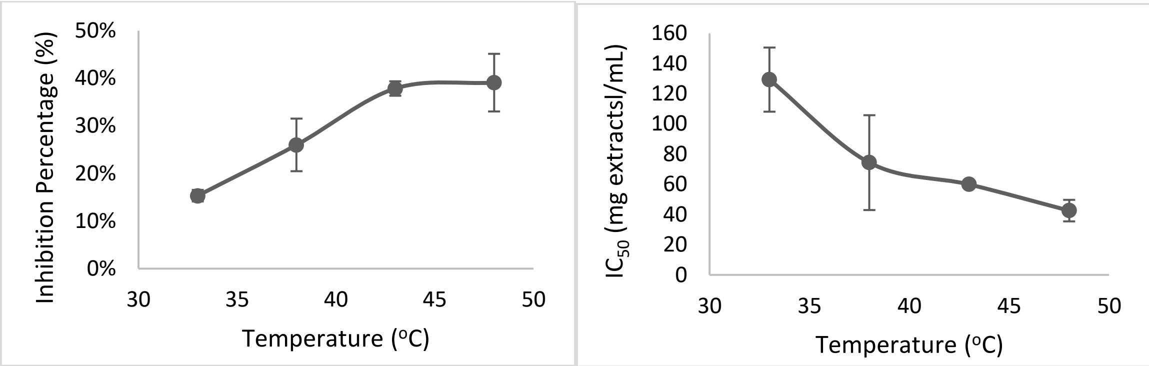


Figure 2 Effect of extraction temperature on the antioxidant activity of the dragon fruit skin extracts: (a) Inhibition percentage; (b) IC_{50} at a concentration of 40 mg extract/mL

Figure 2 shows the antioxidant activities of the dragon fruit skin extracts obtained at different temperatures, which are stated as IC_{50} and inhibition percentage at an extract concentration of 40 mg/mL in Figures 2a and 2b, respectively. IC_{50} represents the sample concentration required to reduce the radical activity of DPPH by 50%. Thus, a lower IC_{50} means that the sample has a higher concentration of antioxidants. The result in Figure 2a indicates that, at a higher extraction temperature, there was a significant decrease in IC_{50}, which obtained a higher yield of antioxidant compounds. This trend is also similar to the results presented in Figure 2b, where at the same extract concentrations, the extract obtained at a higher temperature could achieve a higher inhibition percentage. The extraction temperatures used in this study were all below 50 °C. A previous study reported a decrease in the antioxidant activity of plant extracts at temperatures above 60°C (Liyana-Pathirana and Shahidi, 2005).

The results in Figures 2a and 2b have the same trend as the yield of TPC, as shown in Figure 1a. Increasing the extraction temperature may increase the solubility and the diffusion coefficients of the antioxidant compounds, hence increasing the extraction yield. Some previous studies have also reported positive correlations between TPC and antioxidant activity obtained during the extraction of fruit and vegetable parts (Skotti *et al.*, 2014; Lagha-Benamrouche and Madani, 2013). The values of IC_{50} found in this study were between 42.6 and 129.3 mg extracts/mL, or equal to 0.27-0.41 mg DM/mL. These values are within the same range of IC_{50} of dragon fruit extract previously reported, which were between 0.26-0.82 mg/mL (Lourith and Kanlayavattanakul, 2013).

3.2. Effect of spray drying temperature on the yield of powder and bioactive materials

3.2.1. Powder recovery (yield) from spray drying

Figure 3 shows the effect of spray drying temperature on the powder recovery (yield) from spray drying. The result shows that a higher inlet temperature may result in a higher powder recovery from spray drying, as shown in the spray drying yield obtained at inlet temperatures of 140 and 170 °C. Higher temperatures decreased the moisture content of the material; thus, the powder became less sticky. In addition, a previous study reported that the yield from spray drying could be correlated to the glass transition temperature of the powder (Shofinita and Langrish, 2016). Lower moisture content corresponds to a higher glass transition temperature of the powder. Thus, the overall difference between the particle and glass-transition temperature decreases, which results in a less sticky powder and a higher powder recovery.

A peaking trend was observed during the spray drying of dragon fruit skin extracts between 140 and 190 °C. This peak trend has also been reported by previous studies, particularly during the spray drying of bioactive extracts (Shofinita and Langrish, 2014; Fang and Bhandari, 2011). Moreover, the use of an inlet temperature above 170 °C may result in a decrease in the spray drying yield when the inlet temperature is increased. At much higher inlet and outlet temperatures, the particle temperature increases at a more rapid rate compared to the glass-transition temperature. This may increase the overall difference between the particle and glass-transition temperature; hence, the powder recovery decreases after a certain inlet temperature.

The result in Figure 3 also indicates that, without any addition of a carrier agent, the powder recovery is very low (7.31 ± 1% for an inlet temperature of 140 °C). A previous study mentioned that in small-scale spray dryers, a high deposition rate of high moisture content particles in the drying chambers occurs because the atomizer is located very close to the dryer walls; thus, the yield is considered to be low compared with the yield of a similar process on an industrial scale (Hanus and Langrish 2007). The low yields from spray drying of juices and extracts may be correlated with the low glass-transition temperatures of some food components (Shofinita, Feng, and Langrish, 2015). Sugars and citric acids have been reported to have low glass-transition temperatures, which cause the materials to be difficult to spray dry. Dragon fruit skin was reported to contain some sugars, such as glucose, maltose, and fructose (0.86 – 4.15%), and also some organic acids, such as oxalic acid, malic acid, succinic acid, and citric acid (0.08 – 0.80%) (Jamilah *et al.*, 2011).

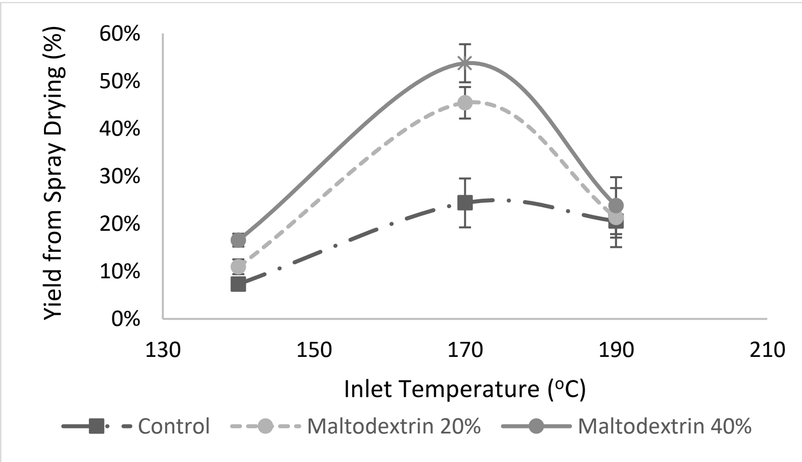
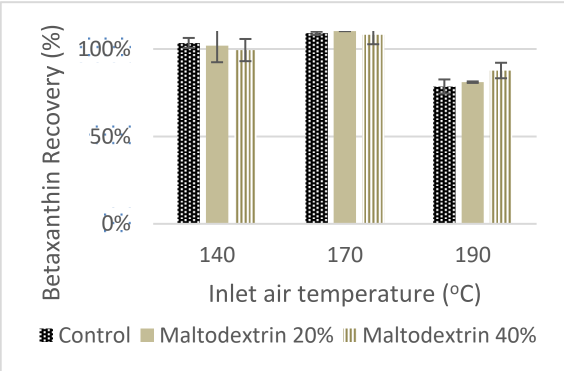


Figure 3 Effect of spray drying temperature on the powder recovery (yield) from spray drying

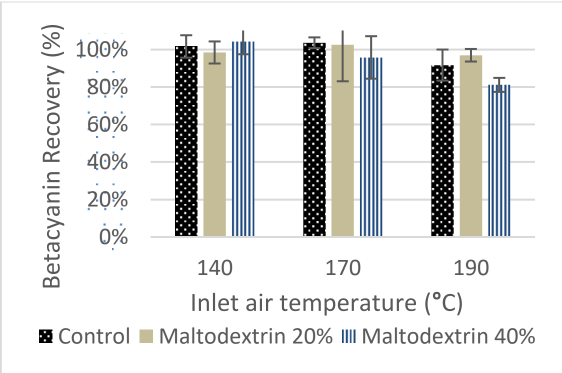
In order to increase the powder recovery from spray drying, maltodextrin was introduced as a carrier agent in this study. Figure 3 shows that at both temperatures, the addition of maltodextrin may increase powder recovery significantly. Maltodextrin is a high molecular weight material that has a relatively high glass transition temperature. Therefore, the addition of maltodextrin in the spray drying feed may increase the glass-transition temperature of the powder and decrease the stickiness of dragon fruit extract during spray drying.

3.2.2. Powder recovery (yield) from spray drying

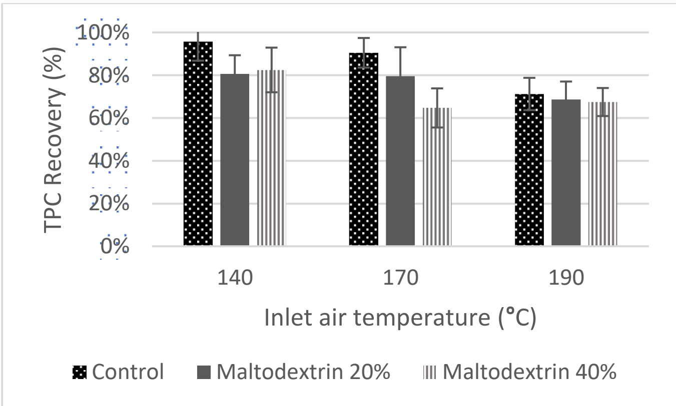
Figures 4a and 4b show the betalain recoveries from spray drying of red dragon fruit skin extract, particularly for betaxanthin and betacyanin recoveries, respectively. As shown in Figures 4a and 4b, high recoveries of betaxanthin and betacyanin (more than 70%) were achieved. Losses of betalain pigments may occur during the spray drying process due to the use of high temperatures. Previous studies have also mentioned the degradation of betalain pigments during spray drying. Saénz *et al.* (2009) found betacyanin recoveries during spray drying of Cactus pear ethanolic extracts to be between 62 and 81%. Cai and Corke (2000) previously reported lower pigment loss (2.77-7.66 %) compared with this study, while Zuanon *et al.* (2019) reported higher betalain loss (54-60 %) compared with this study. In addition, Delia *et al.* (2019) found 72.4-98.8 % of betalain retention during spray drying of *Escontria chiotilla* and *Stenocereus queretaroensis* fruits, which are in the range of betalain recoveries found in this study. Figures 4a and 4b also indicate that the inlet temperatures significantly affect betaxanthin and betacyanin. The use of inlet temperature, particularly above 170 ºC, may result in a significant decrease in betacyanin. A previous study regarding spray drying of betacyanin in *Amaranthus* extract also reported that a higher inlet temperature might result in a higher loss of betacyanin (Tze *et al.*, 2012; Cai and Corke, 2000).



(a)



(b)



(c)

Figure 4 Recoveries of (a) betaxanthin; (b) betacyanin; (c) TPC from spray drying at different inlet air temperatures

Figure 4c shows the amount of TPC recovery from spray drying of red dragon fruit skin extract. TPC recoveries of more than 60% have been found in this study. Figure 4c also shows that a higher temperature may lead to a higher degradation rate of the materials. A previous study has also reported this trend regarding spray drying of phenolic compounds-rich extracts (Shofinita and Langrish, 2016).

4. Conclusions

Up to 50 °C, higher extraction temperatures resulted in a significant increase in the amount of phenolic and betacyanin compounds and an increase in the antioxidant activity of the dragon fruit skin extracts. Regarding spray drying temperatures, the use of higher inlet temperatures may result in a peaking trend of yield from spray drying. However, at temperatures above 170 °C, a decrease in the recoveries of bioactive materials such as betaxanthin and betacyanin might occur due to higher degradation rates. Furthermore, the addition of maltodextrin as a carrier agent in spray drying may enhance the powder yield significantly, up to 54 $\pm$ 4% at an inlet air temperature of 170 °C with the addition of 40% maltodextrin. Based on the results of this research, an appropriate temperature for extraction and spray drying of dragon fruit peel could be chosen, as well as the carrier agent as an aid for the spray drying process, which can be utilized to improve the yield of the desired product.

Acknowledgments

This work was supported by the Faculty of Industrial Technology, Institut Teknologi Bandung (ITB), Indonesia, through The Research, Community Service, and Innovation Program 2022 (Program Penelitian, Pengabdian Masyarakat, dan Inovasi ITB).

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