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Increasing the Yield of Powder and Bioactive Materials during Extraction and Spray Drying of Dragon Fruit Skin Extracts

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Highlights:

- High spray drying yield of dragon fruit skin extract powder was obtained.
- Maltodextrin and WPI were found to be able to significantly increase the spray drying yield.
- Spray-dried product could achieve high recovery of TPC and betacyanin in the extracts.

Abstract. One potential utilization of dragon fruit skin is to produce bioactive materials as natural antioxidants and colorants for the food industry by extraction and spray drying. This study investigated the quality (total phenolic compounds/TPC, betacyanin and betaxanthin contents, and antioxidant activity) of the extracts and spray-dried products, and the quantity (powder yield) obtained by the use of different types and amounts of spray drying agents. Two drying agents were introduced during spray drying, i.e. maltodextrin and whey protein isolate (WPI). The result showed that a lower extraction solvent to solid ratio may result in a lower yield of TPC, betacyanin and betaxanthin contents, and also in antioxidant activity of the dragon fruit skin extract. In addition, maltodextrin and WPI were found to be able to significantly increase the yield from spray drying. The highest yield (72.7 ± 8.4%) was obtained with the use of 40% maltodextrin as drying agent, while the control yielded 9.5 ± 1.8%. Furthermore, it was found that the spray-dried product could recover more than 90% of the TPC and betacyanin in the extracts, which indicates that spray drying may be suitable for heat-sensitive materials.

Keywords: *antioxidant; betaxanthin; betacyanin; extraction; food colorants.*

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1 Introduction

Dragon fruit is a tropical fruit that is rich in bioactive materials. In the food industry, dragon fruit is usually processed into juice, which leaves the skin as waste. Previous studies have reported that dragon fruit skin is rich in bioactive materials, such as antioxidants, phenolic compounds, and betacyanin [1]. Further processing of dragon fruit skin into more valuable products is expected to reduce agricultural waste and also to give more profit to the dragon fruit farmer and the juice industry. One potential utilization of dragon fruit skin is to produce natural colorants for the food industry by extraction. Betalains, including betacyanin and betaxanthin, are responsible for the natural colorants in dragon fruit skin. In addition to their color properties, these pigments are also beneficial for human health due to their anti-inflammatory, antioxidant, inhibitory effects [2]. A previous study has reported the possibility of synthetic colorants to be hyper-allergenic and carcinogenic, and can also cause toxicological problems [3]. This may cause the current trend of colorant application in the food industry to move towards natural raw material [4].

The first stage of producing natural pigment from fruit wastes is extraction. Previous studies have reported methods for the extraction of bioactive materials from fruit wastes, such as maceration, Soxhlet extraction, microwave-assisted extraction, ultrasound-assisted extraction, and supercritical fluid extraction [5-7]. However, bioactive materials in the form of extract usually have limited storage life. Thus, further drying of the extract can be applied in order to prolong its shelf life and decrease the storage and transportation costs.

One of the methods that can be applied for drying of bioactive extracts is spray drying. During spray drying, evaporation of the moisture occurs in a short time with the temperature of the product usually below 100 °C. Thus, the use of this method is expected to avoid the thermal degradation of bioactive materials, which can occur during drying processes [8]. However, one of the problems in spray drying of bioactive materials is the stickiness of the powder, which occurs in relation to the glass transition temperature of the material. Drying agents are usually added to the spray drying feed in order to manipulate the glass transition temperature, hence increasing the powder yield. Several carrier agents have been reported to be used in drying of bioactive materials, such as maltodextrin, gum arabic, and whey protein isolate [5,9,10].

In this study, bioactive materials from dragon fruit skin waste were produced by extraction and spray drying. Extraction is an important step during the production of food additives because it determines the yield of bioactive materials that can be obtained from the plant waste. Thus, the extraction yield is discussed first in this paper, particularly regarding the extraction solvent to solid ratio as one of the most important parameters in extraction [8]. Regarding spray drying of dragon fruit skin extracts, the use of a carrier agent in the spray drying feed has not been fully explored yet. In order to fill this knowledge gap, this study investigated the yield of powder and bioactive materials from extraction and spray drying of dragon fruit skin extracts.

2 Materials and Methodology

2.1 Materials

Red dragon fruit (*Hylocereus polyrhizus*) was purchased in Bandung, Indonesia. Demineralized water was used as solvent. Several chemicals were used for analysis, including Folin-Ciocalteu's reagent (Merck), DPPH (2,2-diphenyl-1-picrylhydrazyl) (Sigma Aldrich), and Na_2CO_3 (Bratachem).

2.2 Extraction

The red dragon fruit was purchased from a local supermarket in Bandung. For extraction, the dragon fruit was washed, wiped to dry, and peeled, and the skin was chopped in a food chopper (Kirin, Indonesia) to produce dragon fruit skin in the form of slurry. Extraction was then carried out by maceration at room temperature (25-30 °C) using water as solvent. The solvent to solid ratio was varied between 1 and 3, while the extraction time was varied between 10 and 200 minutes. The dragon fruit skin extracts were filtered using filter paper (Whatman filter paper No. 41, Sigma Aldrich) and then kept in the fridge (4 °C) for the further spray drying process.

2.3 Spray Drying

Dragon fruit skin extracts that were produced at a solvent to solid ratio of 1 and an extraction time of 240 minutes were used as the feed for the spray drying experiments. A lower solvent to solid ratio was used in order to avoid low-concentration extracts, which may require higher energy for spray drying. The variation during spray drying was: control (without carrier agent) and with carrier agent (WPI and maltodextrin at concentrations between 20 and 40%). For the spray drying feed with carrier agent, the extract was prepared by adding the carrier agent at concentrations between 20 and 40% of the total solid content of the extract. Spray drying of the dragon fruit skin extract was then carried out at the following conditions: inlet temperature 100 °C, feed flow rate 150 mL/h, and inlet air flow rate 0.45 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 Total Phenolic Compounds (TPC)

Extract (0.1 mL), demineralized water (0.2 mL), and Folin Ciocalteu's reagent (1.5 mL) were mixed in a container. The mixture was allowed to stand for 3 minutes, after which 1.2 ml of 7.5% Na_2CO_3 was added to the container. The mixture was then kept in the dark for 30 minutes and the absorbance of the sample was measured using spectrophotometry (Smart Spectro Spectrophotometer, USA) with a wavelength of 765 nm. Gallic acid solution was used as calibration standard; the TPC of the extract is stated as mg gallic acid equivalent (GAE)/g sample. In addition, the TPC recovery during spray drying was determined by comparing the TPC of the extracts and TPC of the powder obtained after the drying process.

2.5 Antioxidant Activity

Extracts with several concentrations were prepared by using ethanol. 2 mL of the extract solution was mixed with 2 mL of 6 x 10^{-5} M DPPH solution in ethanol. The mixture was allowed to stand in a dark room for 30 minutes, after which the absorbance was measured using a spectrophotometer (Smart Spectro Spectrophotometer, USA) at a wavelength of 517 nm. DPPH solution in ethanol was used as control solution. The inhibition percentage (% inhibition) of the extract was calculated by using the following equation, where $A_{control}$ is the absorbance of the control solution and A_{sample} is the absorbance of the extract:

$$\%\,\boldsymbol{inhibition} = \frac{\boldsymbol{A_{control} - A_{sample}}}{\boldsymbol{A_{control}}}\ \boldsymbol{x\ 100}\% \quad (1)$$

2.6 Betacyanin and Betaxanthin Content

Liquid extract (0.1 mL) was prepared and then diluted 50 times with demineralized water. As blank solution, demineralized water was prepared. The absorbance of the diluted sample was measured using a spectrophotometer at wavelengths of 535 nm and 477 nm for quantification of betacyanin and betaxanthin, respectively. The amount of betalain can be calculated with the following equation:

$$Betacyanin\ or\ betaxanthin\ content\ \left(\frac{mg}{g\ material}\right) = \frac{A\ x\ DF\ x\ Mr\ x\ V_d}{\varepsilon\ x\ L\ x\ w_d} \quad (2)$$

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 atenuation coefficient [60000 L/(mol.cm) for betacyanin and 48000 L/(mol.cm) for betaxanthin]; L = Cuvette length (1 cm); and W_d = dragon fruit skin mass (g).

2.7 Morphology of the Powder

The powder morphology was studied using a Hitachi SU3500 scanning electron microscope (Hitachi High Technologies America, Inc.). A small amount of the powders was placed on a carbon tape mounted on an aluminum stab and was coated with gold. The operating voltage of the instrument was set to 10 kV for all the samples.

2.8 Statistical Analysis

The data in this study were obtained from two replicates for each experiment and are presented as mean ± standard deviation. For statistical analysis, differences were tested for significance by using the ANOVA method using significance level $P \leqslant 0.05$.

3 Results and Discussion

3.1 Extraction of Bioactive Materials from Dragon Fruit Skin

3.1.1 Total Phenolic Compounds (TPC)

Figure 1 shows the result of the TPC in the dragon fruit skin extract as a function of solvent to solid ratio and extraction time. Figure 1 shows that increasing the extraction time may cause an increase in the TPC of the extracts. Initially, a high gradient of TPC concentration could be observed, particularly below 100 minutes. However, this gradient decreased at extraction time above 240 minutes as the driving force of the mass transfer decreased and the extraction approached equilibrium condition.

It can also be seen in Figure 1 that an increase in the solvent to solid ratio may increase the TPC of the extracts. As previously reported, during the extraction of phenolic compounds from orange peel, a higher solvent to solid ratio provides a higher gradient of phenolics concentration between the solid and the bulk of the solvent [8]. This may result in an increase of the extraction rate and the extraction yield [11]. However, a higher solvent to solid ratio may result in a lower concentration of extract, which may require higher energy for spray drying [8]. The TPC in the dragon fruit extracts found in this study were between 50 and 250 mg GAE/100 g fresh skin. The values found in this study were higher than the TPC values in dragon fruit skin extracts previously reported [12], i.e. 28.16 mg GAE/ 100 g fresh skin. This difference may be due to the difference in the source, genetic factors, and maturity of the dragon fruit. Different sources of fruit are expected to have different nutritional values and morphologies. In addition, Nurliyana, *et al.* [12] dried the dragon fruit skin as a pre-treatment before extraction, in comparison with the use of fresh dragon fruit skin in this study.

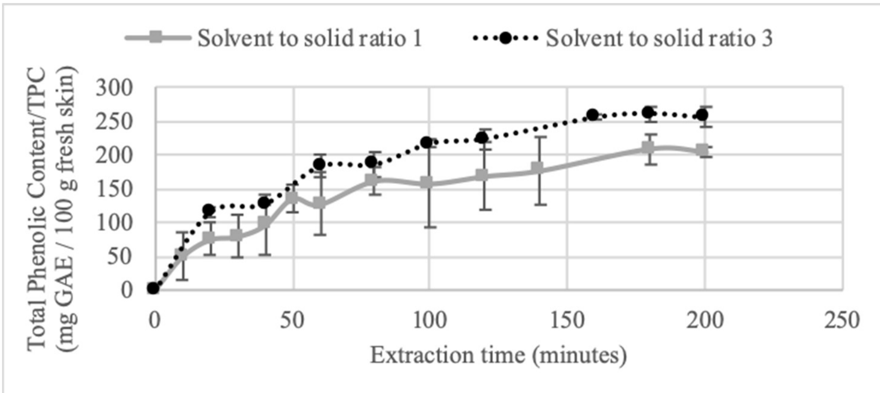

Figure 1 Total phenolic compounds (TPC) of dragon fruit skin extracts.

A previous study found that drying may destroy some phenolic compounds [13] and thus decrease their concentration in the extracts [14]. In addition, Nurliyana, *et al.* [12] used the Soxhlet method with ethanol as solvent, in comparison with the maceration method using water in this study. The type of solvent used affects the extraction yield significantly. In addition, in the Soxhlet method, the extraction temperature is the same as the boiling point of the solvent; in comparison, room temperature was used in this study. A higher extraction temperature may also result in a lower extraction yield due to the thermal degradation of phenolic compounds.

3.1.2 Antioxidant Activity

The antioxidant activities of the extracts in this study are represented by inhibition percentage (% inhibition), which was determined at an extract concentration of 40 mg extract/mL. Figure 2 shows the effect of different solvent to solid ratios on the % inhibition of the extracts.

A higher inhibition percentage represents higher antioxidant activity of the material. Figure 2 shows that a higher solvent to solid ratio may result in a higher inhibition percentage and therefore higher antioxidant activity of the material. A higher solvent to solid ratio may result in a higher gradient concentration of antioxidant compounds between the solvent and the solid, which may enhance the diffusion rate [11]. This result regarding antioxidant activity had the same trend as the total phenolic compounds discussed in the previous section. The presence of phenolic compounds may result in the inhibition effect of radicals due to their ability to donate electrons and antioxidant activity. A previous study also found a correlation between the total phenolic compounds and the antioxidant activity of the extracts [15].

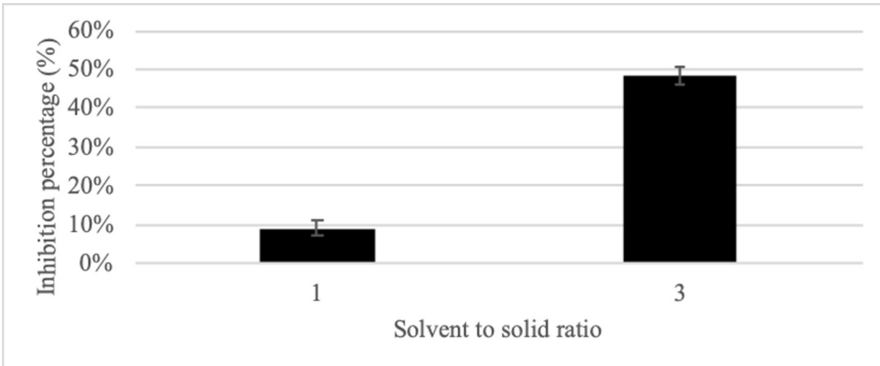

Figure 2 Inhibition percentage at 40 mg extract/mL of dragon fruit skin extract at different extraction solvent to solid ratios (extraction time: 200 minutes).

3.1.3 Betacyanin and Betaxanthin Contents

Betacyanin and betaxanthin belong to the betalain pigments. Betacyanin is responsible for a reddish to violet color, while betaxanthin is responsible for a yellow to orange color. The betacyanin and betaxanthin contents of the dragon skin extracts are important for quantifying the natural pigments. Figures 3 and 4 show the betacyanin and betaxanthin contents of the dragon fruit skin extracts, respectively. This result also shows the same trend with total phenolic compounds and antioxidant activity. As for the natural pigments, a higher solvent to solid ratio may result in higher betacyanin and betaxanthin contents.

A previous study [16] found that dragon fruit skin extracts had 5 to 25 mg/L betacyanin, or equal to around 0.015 to 0.9 mg betacyanin /g dragon fruit skin. The total betacyanin content found in this study was between 0.01 and 0.04 mg/g dragon fruit skin, which is similar to that from [16] during extraction at room temperatures (0.015 mg betacyanin/g dragon fruit skin). Ref. [16] also found that the total betacyanin content increased at higher temperature.

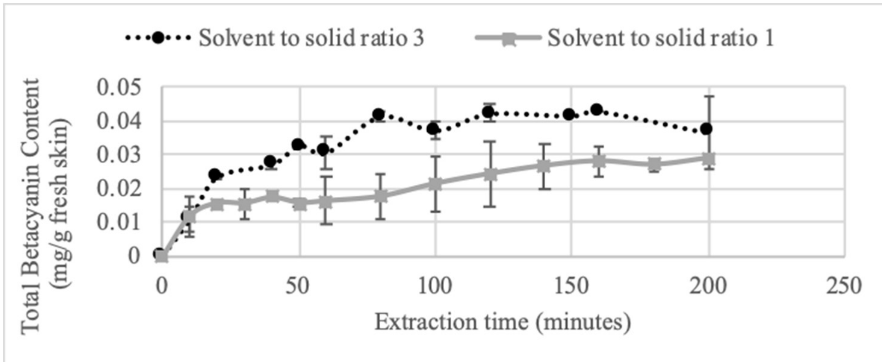

Figure 3 Total betacyanin content of dragon fruit skin extracts.

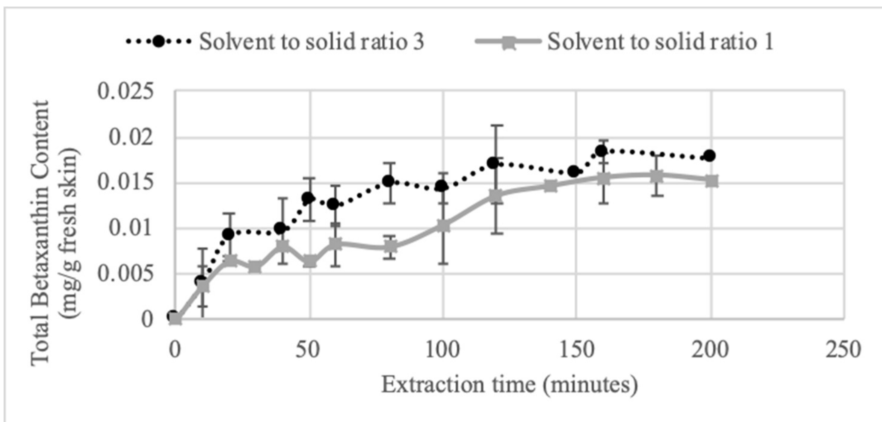

Figure 4 Total betaxanthin content of dragon fruit skin extracts.

3.2 Spray Drying of the Dragon Fruit Skin Extract

3.2.1 Powder Yield from Spray Drying

Powder yield, or powder recovery, represents the amount of product obtained during spray drying. The powder yield was calculated as the amount of powder collected in the collecting vessel. The result of the spray drying of the dragon fruit skin extract is shown in Figure 5. It was shown that the use of WPI and maltodextrin increased the yield of the powder significantly. The addition of WPI could increase the powder yield up to 44.4 ± 2.9 %, while the addition of maltodextrin could increase the powder yield up to 72.7 ± 8.4 %. The yield obtained by adding 40% maltodextrin can be considered to be relatively high, as a previous study [17] mentioned that a yield between 60% and 80% in a laboratory-scale spray dryer can be considered to be relatively good and may indicate a much higher yield is possible with an industrial scale spray dryer. The use of additional maltodextrin may increase the production cost of the material due to the additional raw material requirement, but the productivity of the process may also increase. Further economic analysis can be done in order to optimize the process.

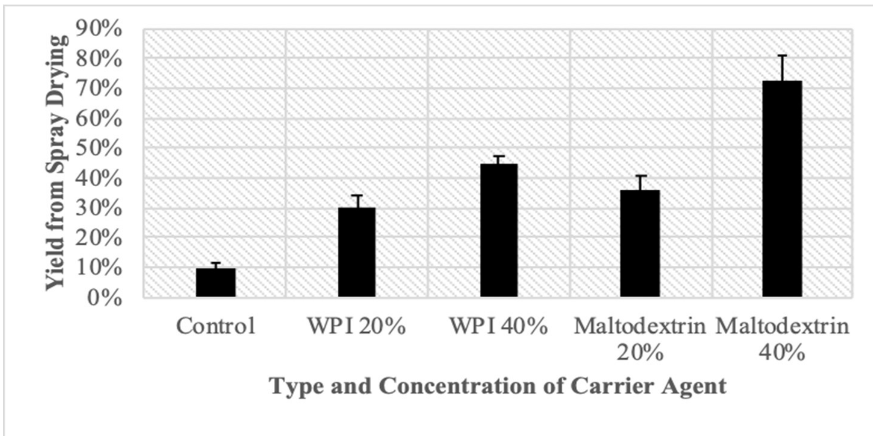

Figure 5 Powder yield from spray drying of dragon fruit skin extracts.

For the variation of control (without addition of a drying aid), a very low powder yield was achieved (9.5 $\pm$ 1.8%). The low powder yields in a laboratory scale spray dryer can be due to the high deposition of very wet particles on the drying chamber walls [18]. This stickiness phenomenon during spray drying may be affected by the composition of the dragon fruit skin extracts that were used as the spray dryer feed. A high content of sugars in the spray dryer feed, for example, may result in adhesive and cohesive effects during spray drying. Several components in fruit juices or extracts that are known to be difficult to spray dry are citric acid, fructose, glucose, and sucrose [19,20].

The stickiness during spray drying may be correlated with the glass transition temperature of the material. Typical sugar-rich materials may have very low glass transition temperatures. As a consequence, their molecular mobility is high, particularly when the particle temperature is more than 20 °C above the glass transition temperature [19].

In order to increase the powder yield from spray drying of sugar-rich materials, the addition of a drying agent is commonly applied, which is expected to reduce this stickiness. One commonly used drying aid for spray drying is maltodextrin.

Figure 5 shows that increasing the maltodextrin concentration in the spray drying feed significantly increased the spray drying yield. The same trend was observed in Ref. [21] regarding the spray drying of mulberry juice with maltodextrin as drying aid. Maltodextrin is a high molecular weight material that has a relatively high glass transition temperature. This characteristic helps to reduce the stickiness of dragon fruit extract during spray drying.

Figure 5 shows that an increase in the concentration of WPI added to the feed increased the spray drying yield. WPI, which has protein as main component, may interact with sugar, such as sucrose, so non-sticky particles may be formed. During drying, some proteins form glassy surface films rapidly [22]. Furthermore, these films have high glass transition temperatures, which can reduce the cohesive stickiness between particles and the stickiness between the particles and the drying chamber walls.

3.2.2 Recovery of Bioactive Materials from Spray Drying

Figure 6 shows the effect of spray drying on the recovery of bioactive materials, including TPC and betacyanin. Figure 6 shows that spray drying may not affect the TPC of the material significantly, as the TPC recovery for all variations can be considered to be high (more than 95%). High recovery of phenolic compounds during spray drying of other materials such as orange peel extracts has been reported previously [15].

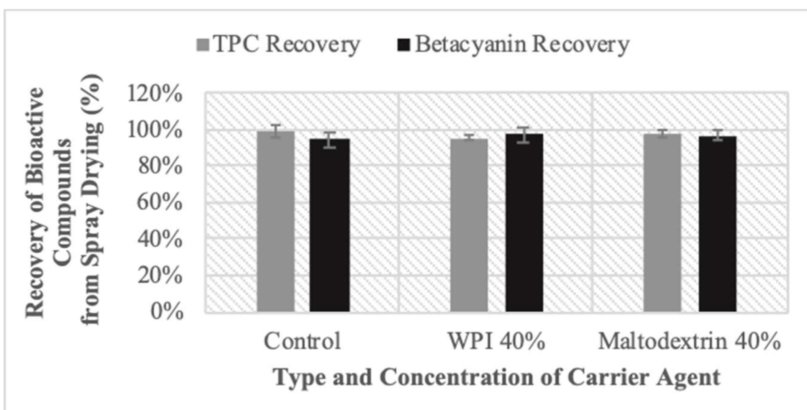

Figure 6 Recovery of bioactive materials from spray drying of dragon fruit extracts.

Figure 6 shows that for all variations of spray drying aids and control, high recoveries (more than 90%) of betacyanin were achieved. High recovery of betacyanin during spray drying has been reported previously related to spray drying of bioactive compounds from cactus pears [23]. In addition, Delia, *et al.* [24] found 72.4 to 98.8% of betalain retention during spray drying of *Escontria chiotilla* and *Stenocereus queretaroensis* fruits, which is in the range of betacyanin recoveries found in this study. This may be due to the rapid drying process of spray drying, so extensive degradation of betacyanin did not occur. During heat treatment processes like spray drying, some chemical reactions may take a place, such as the Maillard reaction and oxidation [5].

3.2.3 Morphology of the Spray Dried Powder

Figure 7 shows the surface morphology of the spray-dried powder with 40% maltodextrin as carrier agent, which had a relatively high powder yield compared with the other variations. From Figure 7 it may seem that the powders initially had a spherical form and then formed liquid bridges between each other by utilizing the available moisture or absorbing moisture from the environment. This morphology in the spray-dried powder has also been found previously in materials that contain a high amount of sugar such as fruit and vegetable extracts, for example in spray-dried orange peel extract powder [25]. This phenomenon of creating liquid bridges between the particles of spray-dried powder may be linked to the tendency of agglomerating or caking in food powders. The caking of food powders may occur due to wetting of the particle surface.

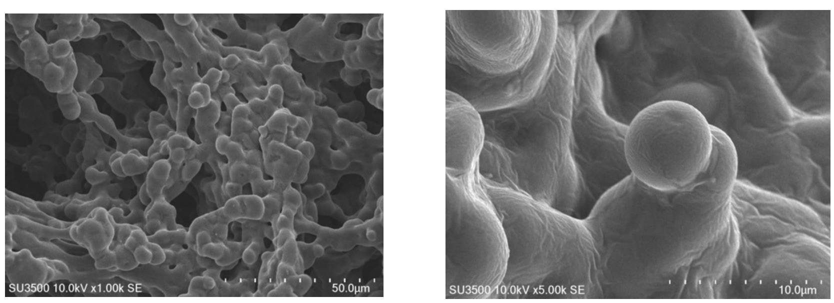

Figure 7 Morphology of the particle obtained from spray drying of dragon fruit extract with 40% maltodextrin as drying aid.

4 Conclusions

It was found in this study that a higher extraction solvent to solid ratio may result in a higher yield of TPC, betacyanin and betaxanthin contents, and also antioxidant activity of the dragon fruit skin extracts. Maltodextrin and WPI were found to be able to significantly increase the powder yield from spray drying, with the highest powder yield (72.7 $\pm$ 8.4 %) obtained from the use of 40% maltodextrin as drying aid, in comparison with 9.5 $\pm$ 1.8% for the control (without carrier agent). Furthermore, it was found that the spray-dried product had more than 90% of the TPC in the extracts, which indicates that spray drying may be suitable for heat-sensitive materials, such as bioactive materials in the dragon fruit skin extracts.

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