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EFFECT OF FEED FLOWRATES ON THE PHYSICAL PROPERTIES AND ANTIOXIDANT OF MAHKOTA DEWA (*PHALERIA MACROCARPA*) ENCAPSULATED POWDER

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ABSTRACT: This paper presents an experimental study on the encapsulation of Mahkota Dewa extracts by maltodextrin using spray drying. The bioactive compound from dried Mahkota Dewa was obtained using a subcritical water extraction process prior to a spray drying process. The effect of feed flow rate (485 to 2115 ml/h) was investigated using one-factor-at-a-time (OFAT). It was observed that the mean particle size increase varied from 3.55 to 8.38 µm when the feed flow rate increased from 485 to 2115 ml/h. Moisture content increased 4.88 to 6.83% as the feed flow rate increased from 485 to 2115 ml/h, whereas the antioxidant activity increased slightly from 90.48 to 91.65%. The findings from this study showed that decrease in feed flow rate reduces antioxidant activity, moisture content, and particle size.

ABSTRAK: Kertas kerja ini adalah berkenaan kajian eksperimen melalui proses pengkapsulan ekstrak buah Mahkota Dewa dengan menggunakan maltodekstrin melalui teknik pengeringan semburan. Sebatian bioaktif dari buah Mahkota Dewa kering diperoleh melalui teknik pengekstrakan air subkritikal sebelum proses pengeringan semburan. Kesan kadar aliran masuk (485 hingga 2115 ml/jam) dikaji menggunakan konsep satu-faktor-pada-satu-masa (OFAT). Dapatan kajian mendapati bahawa purata saiz zarah meningkat dari 3.55 kepada 8.38 µm ketika kadar aliran masuk meningkat dari 485 sehingga 2115 ml/jam. Kandungan kelembapan meningkat dari 4.88 kepada 6.83% dengan kenaikan kadar aliran masuk dari 485 hingga 2115 ml/jam, sedangkan aktiviti antioksida meningkat sedikit dari 90.48 hingga 91.65%. Dapatan kajian menunjukkan bahawa penurunan kadar aliran masuk berkurang melalui aktiviti antioksida, kandungan kelembapan dan saiz zarah.

KEYWORDS: *Mahkota Dewa; spray drying; antioxidant; maltodextrin*

1. INTRODUCTION

Mahkota Dewa (*Phaleria Macrocarpa (Scheff.) Boerl)*, which is a member of the *Thymelaeaceae* family, is an important herbal species originating from Papua New Guinea Island (Irian Jaya), Indonesia [1]. The plant is quite popular among South East Asian peoples and is traditionally used in local folk medicine as complementary alternative medicine for several diseases such as cancer, hypertension, and diabetes mellitus. The major parts of the plant namely the stem, leaves, egg shell of the seeds, and fruits are mostly enriched in

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bioactive compounds such as alkaloids, flavanoids, mangiferin, polyphenols, and saponins [2,3]. In particular, Mahkota Dewa fruit has been related to antioxidant activity and hence, the use of this fruit can be suggested as a strategy to control hyperglycemia problems in diabetic patients. Several studies also have been performed to understand and prove other biological properties related to Mahkota Dewa fruit, such as its anti-carcinogenic, anti-hypertensive, anti-tumour, anti-viral, anti-bacterial, anti-fungal, and anti-inflammatory properties [1]. Previous researchers reported the extraction of bioactive compounds from Mahkota Dewa fruits and leaves using conventional and nonconventional extraction techniques such as maceration [4], microwave [5], subcritical water [6,7], and supercritical carbon dioxide [8]. This is because obtaining the targeted bioactive compounds naturally present in plants depends on the extraction techniques employed.

Other works also reported that the conversion of the liquid extracts to powdered form product is preferred in industry for several reasons. For example, the product in powdered form is easier to handle and has been reported to show better stability, longer shelf-life and more convenience for oral consumption [9]. Polyphenols are an example of the bioactive compounds extracted from the plant parts that are very susceptible to an oxidizing environment. Spray drying is an established method used to convert the liquid extracts to powdered form product and well known in food industry due to its flexibility, low cost and large scale applicability [10]. However, the operation of spray drying that requires high operating temperature, up to 215 °C, may cause a thermal degradation issue to the extracts and poor quality of the powdered product produced [9]. One such method to overcome the issue is encapsulation of the extracts using a suitable carrier agent or polymer. In this situation, the carrier agent acts as a coating wall and provides a physical barrier between the extracts and the external environment. Maltodextrin is frequently employed as a carrier agent in the spray drying of food products. It is a low-cost polysaccharide with a neutral scent and flavour that can diminish powder hygroscopicity if larger doses of the agent are used [11]. According to Raja et al. [12], maltodextrin with dextrose equivalent (DE) of 10 to 20 is appropriate for use as a carrier agent.

Previous studies have described the operating conditions and carrier agents used during spray drying for plants such as *Moringa stenopetala* [13] and saffron [14]. A review on the encapsulation technology application in other plants or food products was also reported by Ray et al. [15]. To our knowledge, no work reported the conversion of Mahkota Dewa extracts to powdered form. Based on this consideration, this study was performed to investigate the effect of different feed flow rates during the spray drying process on the physical properties and antioxidant activity of Mahkota Dewa spray-dried extract. The maltodextrin DE10 was utilised as a carrier agent. The powdered forms of the products obtained were all assessed in terms of antioxidant activity, moisture content, particle size distribution, and physical appearance.

2. MATERIALS AND METHOD

2.1 Materials and Chemicals

The commercial dried Mahkota Dewa fruits were purchased from Ethno Resources Sdn. Bhd, Selangor, Malaysia. The samples were ground to an average size of 520 μm using RETSCH GmbH dry grinder (Germany). The deionized water used for the subcritical extraction process was prepared using Milli-Q, Ultrapure Water Purification System (Massachusetts, USA). The food grade maltodextrin DE10, obtained from San Soon Seng Food Industries Sdn Bhd, Selangor, Malaysia, was used as the carrier agent for the spray drying process. The methanol (99.9 wt% purity) and 1,1-diphenyl-2-picrylhydrazyl

(DPPH), purchased from Merck Sdn. Bhd. (Selangor, Malaysia), were used in the analysis. All compounds were used without any further purification process.

2.2 Extraction of Mahkota Dewa

The Mahkota Dewa was extracted using a subcritical water extraction method described in previous work [7]. About 60 g of ground dried Mahkota Dewa were weighed and mixed with one liter of deionized water in a beaker. The mixture was stirred and placed in a laboratory pressure reactor system (Buchiglasuster Kiloclave, Switzerland) at 106 °C for 5 h to perform the extraction. After that, the vessel was cooled down to room temperature using cooling water. The cooling water was supplied by a Stuart recirculating cooler RE300RC (Staffordshire, UK). The collected extract was filtered to remove the residual solids using Whatman filter paper and kept at -4 °C in a refrigerator until further used in the spray drying process.

2.3 Encapsulation using Spray Drying

Maltodextrin DE10 was dispersed directly into the extract solution and stirred at 1500 rpm using a magnetic stirrer to prepare 20% w/v of spray drying stock solution. The solution was continually stirred and pumped into a laboratory spray-dryer (Lab Plant SD06A, UK) using a peristaltic pump. The inlet air temperature, dry air velocity, and liquid flow rate were set to 200 °C, 3.9 m/s, and 2115 ml/h, respectively. The spray dried powder was collected in a chamber glass bottle, sealed, and kept in the refrigerator at -4 °C until used for analysis. The same procedures were carried out with liquid flow rates of 485, 900, 1305, and 1665 ml/h. The inlet air temperature and dry air velocity were set at 200 °C and 3.9 m/s, respectively. The experiments were carried out in triplicate.

2.4 Characterization Methods

2.4.1 Moisture Content

The moisture content of the sample was measured using an A&D MS-70 moisture analyser. The sample was weighed and placed on the moisture analyser pan at a weight of around one gram. Once the reading became steady, the moisture content shown in percentage value was recorded.

2.4.2 Antioxidant Activity

The antioxidant activities were determined using Tecan Infinite M200 Pro (Switzerland) microplate reader with Magellan software at a wavelength of 517 nm. One milligram of spray dried powder was dissolved in 10 ml of deionized water to make a sample solution. Methanol was used to make a DPPH radical solution at a concentration of one mg/ml. Both solutions were combined and incubated in the dark for 20 min before measuring its absorbance. The DPPH in methanol and ascorbic acid were used as a standard and blank, respectively. The percentage of inhibition was calculated by using Eq. (1):

$$Inhibition\,activity, \% = \left[1 - \left(\frac{A_1}{A_0}\right)\right] \times 100 \tag{1}$$

where A_1 is the absorbance of the sample and A_0 is the absorbance of the control [7].

2.4.3 Particle Size Distributions

A laser diffraction particle size analyser (Malvern 2000 Mastersizer, Malvern Instruments Co., Worcestershire, UK) was used to analyse the particle size distributions. The analyzer was equipped with a sample dispersion unit to ensure that particles were distributed to the measuring region during the analysis. The particle size distribution was recorded in volume weighted mean.

2.4.4 Morphology

The morphological images were captured using a field emission scanning electron microscope (TM3030 plus, Hitachi High-Technologies Corporation, Japan) at 1e3 kV with a magnification of 1000x. The samples were put on a sample stump made of a double-sided adhesive carbon tape. A Sputter Coater (Quorum Technologies Q300TD, Ltd., United Kingdom) was used to sputter gold onto the samples.

3. RESULTS AND DISCUSSION

3.1 Moisture Content

Moisture content is an important property that determines the flowability, stickiness, and storage stability of microencapsulated powder. It was reported that the powder produced by spray-drying with moisture contents less than 5% is considered safe from microbiological activity and can be stored for long period [16]. The influence of different feed flow rates on moisture content of the spray dried powder is shown in Fig. 1.

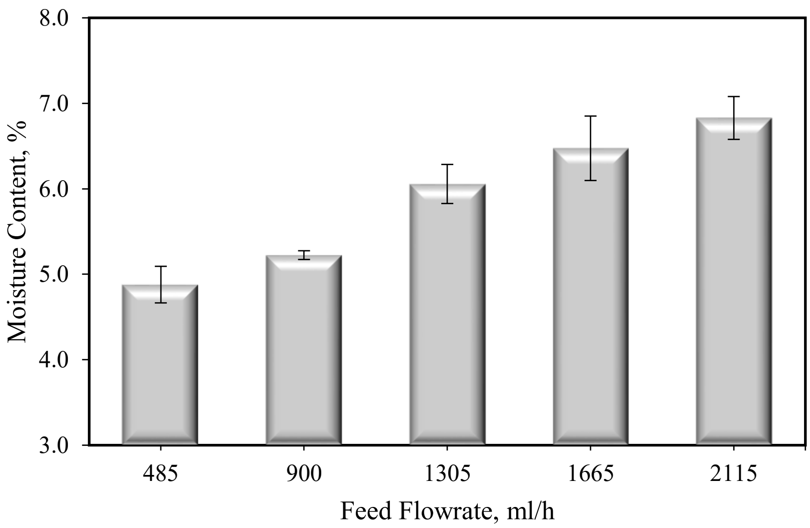


Fig. 1: Moisture content of spray dried powder at different feed flow rates with 0.224 average standard deviation.

As can be seen, the moisture content increased with the increased of feed flow rates pumped into the spray dryer. The lowest moisture content of 4.88 % was achieved at a feed flow rate of 485 ml/h, whereas the highest moisture content of 6.83 % was achieved at a feed flow rate of 2115 ml/h. This result is expected since higher feed flow rates may reduce the contact time between feed samples and drying air. Besides that, the droplets with larger surface area, which were produced at higher feed flow rates, reduce the heat and mass transfer efficiency, hence reducing the water removal during the spray drying process [17]. The result obtained shows similar trends with those reported by Chegini and Ghobadian [18] who studied the effect of feed flow rates on moisture content and bulk density of orange juice powder. Hong and Choi [19] also reported that the powder moisture content increased with the increased of sample pumping rate and inlet air temperature. Braga et al. [20] reported that a feed flow rate of 300 – 900 ml/h used for spray drying of pineapple mint juice showed significant effect on the powder moisture content, where powder with lower moisture content was obtained when the lower feed flow rate was used. Additionally, other work reported that dripping inside the spray drying chamber was observed at high feed flow rates [21]. However, the phenomenon was not observed for the range of feed flow rates used in this work.

3.2 Antioxidant Activity

Rezende and co-workers reported that the presence of phenolic compounds contribute to the high antioxidant activities in plant extract [22]. Mahkota Dewa fruit extract has been reported to provide beneficial bioactivity results such as antioxidant effects due to the present of mangiferin, saponin, alkaloid, and polyphenols [10,12]. Figure 2 shows the antioxidant activity in spray dried powder at different feed flow rates. A slight increment of the antioxidant activity was observed when the feed flow rate increased. The highest feed flow rate (2115 ml/h) produced spray dried powder with higher antioxidant activity (91.65%). This is probably caused by an inefficient heat transfer that occurs between the feed solution and hot air inside the spray drying chamber at higher feed flow rate. Phisut [23] stated that the inefficient heat transfer inside the main chamber at high feed flow rates may produce powdered product with low yield but high antioxidant activity.

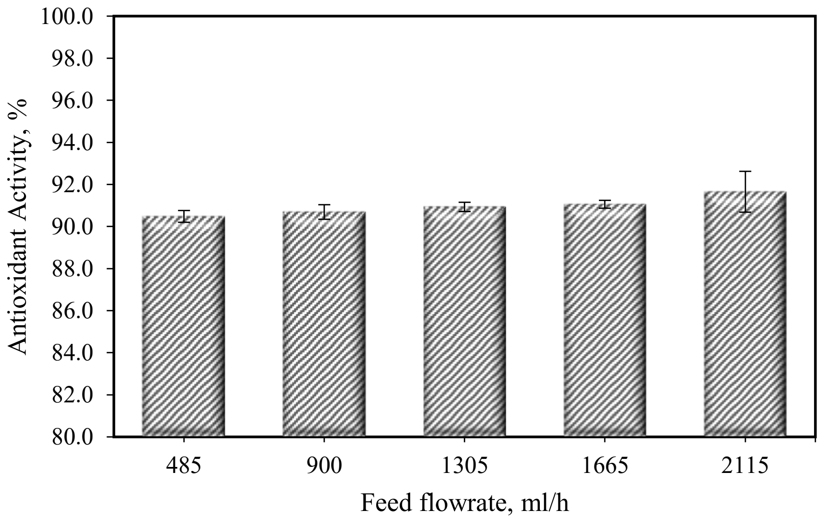


Fig. 2: Antioxidant activity of spray dried powder at different feed flow rates with 0.40 average standard deviation.

3.3 Particle Size Distribution

Figure 3 illustrates the particle size distribution (PSD) of powdered Mahkota Dewa obtained at different feed flow rates. As can be seen, the PSD of the spray dried powder produced are in bimodal shape. The range of PSD for feed flow rates of 485 and 900 ml/h is smaller, which is in between 0.2 to 85 μm. Whereas, for higher feed flow rates, the range of PSD produced is bigger, between 0.9 to 670 μm. The mean diameter and volume weighted mean of spray dried powders produced at different feed flow rates is summarized in Table 1. As shown in the table, the mean diameter of the spray dried power produced is larger at higher feed flow rates. This finding could be related to the high moisture content characteristic of the powdered product produced at high feed flow rates. This is because the powdered product with higher moisture content tends to stick together, thus causing larger particles to form, as shown in Fig. 3. Besides that, bigger droplets obtained at higher feed flow rates may contribute to the findings. Studies by Phisut [23] and Chegini and Ghobadian [18] also reported that larger PSD was produced at higher feed flow rates.

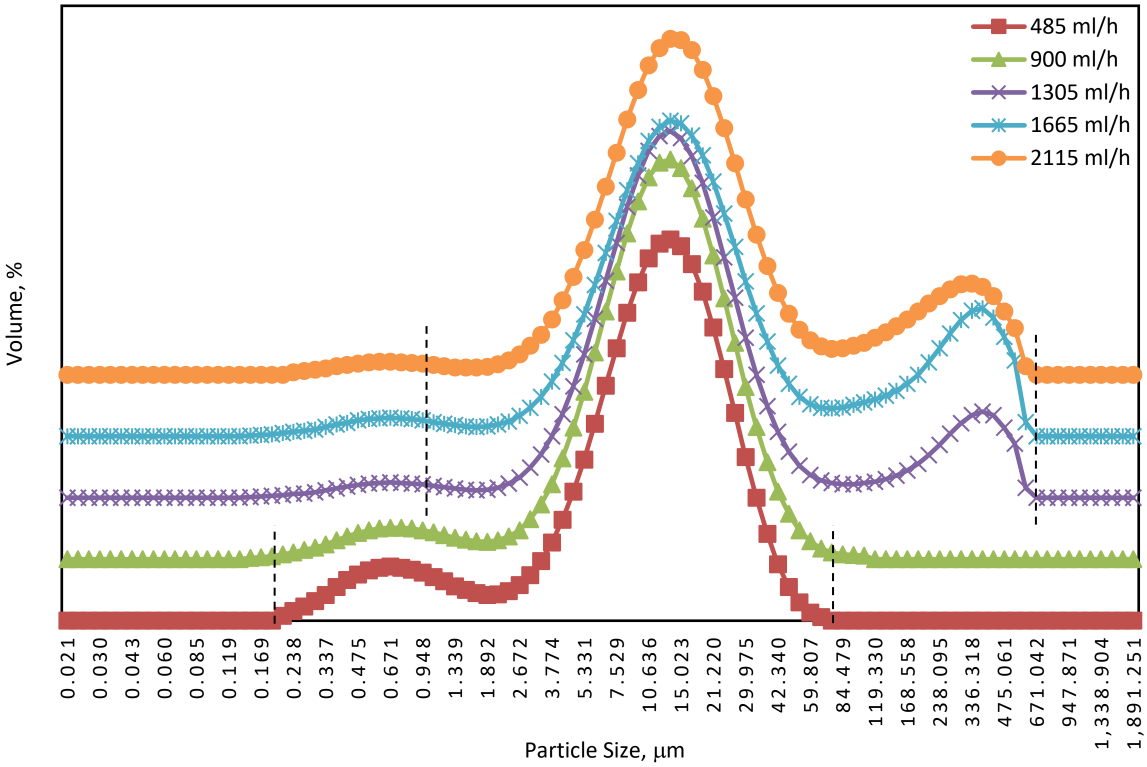


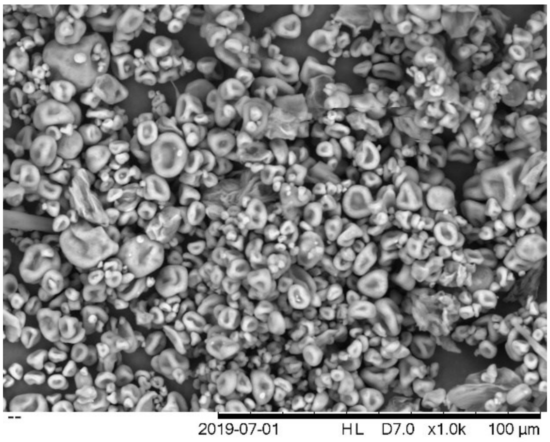
Fig. 3: Particle size distributions of spray dried powder at different feed flow rates.

Table 1: Mean diameter and volume weighted mean of spray dried powders produced at different feed flow rates

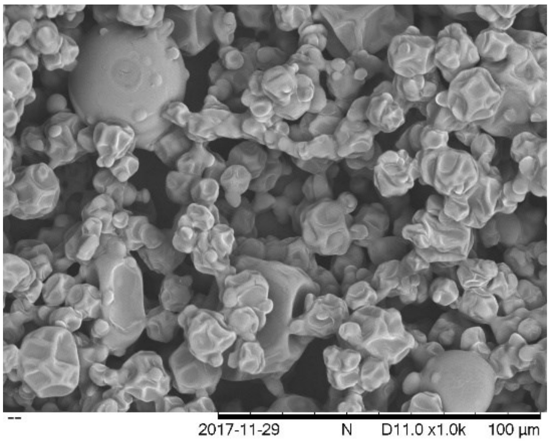
Feed flow rate (ml/h)	**Mean diameter (µm)**	**Volume weighted mean D [4,3] (µm)**
485	3.549	13.157
900	4.670	14.400
1305	7.169	53.484
1665	7.014	74.706
2115	8.376	60.585

3.4 Morphology

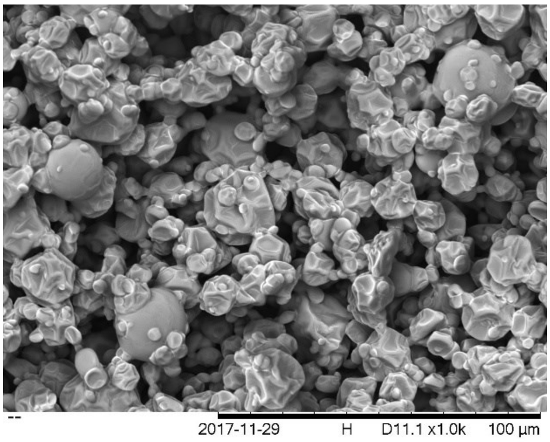
The micrograph images of Mahkota Dewa powder were taken using SEM and shown in Fig. 4. The micrograph images were taken for powdered product obtained with and without maltodextrin DE10 as carrier agent. For experiments with carrier agents, micrograph images were taken for powdered samples obtained using feed flow rates of 485 ml/h and 2115 ml/h. As can be seen in Fig. 4 (a), the particles obtained without maltodextrin are in spherical and irregular forms. No pores were observed on the external surfaces of the powdered product produced but most of it shows shrinking effect. According to Ding et al. [24], the shrinking effect on the particles' surfaces is probably caused by rapid water evaporation during the drying process. Moreover, the shriveled surface, which is a usual characteristic for organic materials produced using spray drying with various sizes, was also observed. As can be seen in Fig. 4 (a), a sample's surface is in the shriveled state and does not attach to other particles. However, the shriveled particles, especially in Fig. 4 (b), were attaching to one another. This is probably related to the moisture content in the particles. Even though some of the particles were shrunk and had a shriveled surface, other particles had a smooth surface.



(a)



(b)



(c)

Fig. 4: SEM micrographs of powdered samples obtained at (a) without maltodextrin DE 10 and feed flow rate of 2115 ml/h, (b) with maltodextrin DE 10 and feed flow rate of 2115 ml/h, and (c) with maltodextrin DE 10 and feed flow rate of 485 ml/h. The inlet dry air temperature and air flow rate were fixed at 200 °C and 3.5 m/s, respectively.

Meanwhile in Fig. 4 (c), some of the particles had shriveled surfaces, while others had smooth surfaces. The bulk of the particles had shriveled and constricted surfaces when the inlet air temperature was low. The number of particles with smooth surfaces rose as the drying temperatures increased. This is due to the differences in drying rates, or higher temperatures, which resulted in faster water evaporation and the formation of a smooth and hard outer layer. Nijdam and Langrish [25] demonstrated the formation of more rigid particles when high temperature was used in the spray drying of milk. According to their findings, vacuole shapes form inside a molecule not long after an outer layer appears, and it expands once the molecule temperature exceeds the local ambient boiling point and the vapor pressure inside the vacuole exceeds the local ambient pressure. When the drying temperature is high enough, moisture evaporates quickly and the outer layer becomes dry and hard, preventing the hollow molecule from flattening as it moves towards colder, dryer areas. However, the outer layer remains fluid and supple for longer when the drying temperature is lower, allowing the empty molecule to flatten and wither as it cools [25].

4. CONCLUSION

The effect of different feed flow rates on Mahkota Dewa extracts encapsulated with maltodextrin using a spray drying technique has been studied. The results show that higher feed flow rates produced powdered product with higher moisture content, antioxidant activity and PSD. However, since the powdered product with low moisture content is desirable, the low feed flow rate of 485 ml/h is recommended in this work. Besides, the antioxidant activity produced at 485 ml/h is quite high, which is more than 90%.

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