

Microwave-assisted Encapsulation of Blue Pea Flower (*Clitoria ternatea*) Colourant: Maltodextrin Concentration, Power, and Time

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Blue pea flower (BPF) is one of the natural anthocyanin colourant sources in the world used in food and cosmetic products. Natural colourants are unstable and thus must be encapsulated. The BPF colourant could retain its colour during storage via microwave-assisted encapsulation. This study evaluated the effect of maltodextrin concentration, microwave power, and encapsulation time on the stability of encapsulated BPF colourant. The colourant was extracted from the BPF via microwave-assisted extraction at 770 W for 1 min. For encapsulation, the concentration of the coating material, maltodextrin, was varied from 20 % to 50 %, a microwave power from (550 to 1100) W, and an encapsulation time from (6 to 8) min. The quality of the encapsulated BPF colourant powder was characterised by total anthocyanin content (TAC), the colour parameter index b^* value, and the water activity value. A TAC of 27.03 mg/L, a colour parameter index b^* value of -9 , a water activity of 0.4941, and an encapsulation efficiency of 73.24 % were obtained when the microwave-assisted encapsulation process was performed using 40 % MD at 770 W for 7 min. With pH adjustment, the blue BPF colourant can be turned into other pigments. The findings of this study can be used as a future reference for developing BPF colourant powder using the microwave-assisted method.

1. Introduction

With the modernisation of the food industry, innovations of various synthetic colourants have been achieved to deliver brighter, attractive, cheaper, and more preferable colourants to customers. However, the public has become concerned about the safety of these synthetic colourants, as these colourants may cause allergic reactions and carcinogenic effects (Amchova et al., 2015) as well as food intolerance in children (Feketea and Tsabouri, 2017). Due to these health concerns, more natural colourant sources have been investigated for their potential in food and cosmetic applications. A natural colourant is a natural pigment that is extracted from organic or natural resources and is regarded as safe, organic, and non-toxic to humans. Of the existing natural colourants in the world, anthocyanin is the most commonly used in the food and cosmetics industry. Anthocyanin is a water-soluble pigment that can appear blue, red or purple, depending on the pH of the environment (Bondre et al., 2012). Anthocyanins can be extracted from flowers (Hamzah et al., 2013), vegetables, and fruits (Zaidel et al., 2015), as well as tubers (Mohd Marsin et al., 2016).

Clitoria ternatea, commonly known as “Butterfly Pea”, “Blue Pea”, or “Bunga Telang”, is a perennial climber belonging to the family of Leguminosae originating from Southeast Asia. It contains ternatins—a group of (poly) acylated anthocyanins—in its petals (Tantituvanont et al., 2008). The bright blue colour in the blue pea flower (BPF) is attributed to the main anthocyanin known as delphinidin glycoside (Terahara et al., 1998). This blue colourant has been used as ingredients in hair-dye products, cosmetic products, as natural colourants in the food industry and as a natural pH indicator in the pharmaceutical industry. The deep blue colour of BPF signifies a large amount of anthocyanins. The limited usage of plant natural colourants is due to its instability and tendency to degrade when exposed to pH, light, and temperature. Since natural colourants are commonly unstable and can easily degrade, encapsulation is the best alternative to protect their pigments. Encapsulation

is a method in which small particles of solid, liquid, or gas are coated with a coating material or agent to form a microcapsule that can protect entrapment against degradation reactions and loss of volatility (Chranioti and Tzia, 2013).

The microwave-assisted technique is known as a green technology that utilises microwave radiation to generate volumetric heating via stimulating the molecular motion and spin of liquid molecules using a constant dipole. Microwave-assisted encapsulation is regarded as an economical method for preserving the natural colourant, promoting good quality and easy handling, and contributing to low water activity end-products. This technique can rapidly and uniformly heat and encapsulate materials. Various studies have been performed using microwave-assisted technique to encapsulate natural colourants from dragon fruit (Zaidel et al., 2015) and purple sweet potato (Mohd Nawi et al., 2015), extraction of natural colourant from hibiscus (Mohd Jusoh et al., 2017), lavender (Farzaneh and Carvalho, 2017), and blackberry (Perez-Grijalva et al., 2018). Elsewhere, the microwave-assisted technique has also been used to refine (Mostafa et al., 2019) and reduce iron oxidation (Gomez et al., 2019). Currently, there are no investigations into the microwave-assisted encapsulation of *Clitoria ternatea* colourant.

A suitable and functional encapsulating agent is required to form more stable encapsulated end-products that can easily solubilise in a mixture. Maltodextrin is one of the encapsulating agents that have proven essential for protecting anthocyanin. The addition of maltodextrin could produce a high-solubility product that has low-moisture content and low hygroscopicity (de Souza et al., 2015). Maltodextrin addition could also enhance the stability, colour properties, and solubility of the encapsulated material in water (Celli et al., 2016). Although maltodextrin is an inexpensive material, it is still an effective encapsulating agent. Several studies have investigated the encapsulation of BPF colourant using maltodextrin. Hamzah et al. (2013) studied the effect of maltodextrin and the mixture of maltodextrin and Arabic gum on the quality of vacuum-dried encapsulated blue pea colourant. Hariadi et al. (2018) studied the effect of different concentrations of maltodextrin addition on the quality of encapsulated blue pea colourant that had been freeze-dried and vacuum-dried. Yet, none have investigated the microwave-assisted encapsulation of BPF colourant incorporated with maltodextrin. This study chose microwave-assisted encapsulation to encapsulate and dry the BPF colourant since this method requires a short processing time.

The first objective of this study is to evaluate the effect of maltodextrin concentration, microwave power, and encapsulation time on the characteristics of the blue pigment of microwave-assisted encapsulated BPF. The second objective is to evaluate the effect of pH on the colour variation of BPF.

2. Materials and methods

Dried blue pea flower (BPF) was obtained from Herbal Remedies, a dry flower wholesaler in Malaysia. Analytical-grade maltodextrin dextrose (MD) (Dextrose Equivalent: 4.0-7.0; Sigma Aldrich, US) was purchased from VNK Enterprise Sdn. Bhd., a local chemical supplier in Taman Universiti, Johor.

2.1 Colourant extraction

The colourant was extracted following the method of Mohd Nawi et al. (2015) with slight modifications. Dried blue pea flower was weighed at a 1:20 ratio (g/mL) to distilled water. The mixture was exposed to microwave extraction at 70 % Power (P) (770W) using a domestic microwave oven (R-397J (S), Sharp, Malaysia) at 1 min extraction time. The extract was filtered using a sieve. The filtered extracts were then centrifuged at 10,000 rpm for 10 min to remove precipitant. The extract was put in a universal bottle and kept frozen at -4°C until further analysis.

2.2 Colourant encapsulation

Maltodextrin (MD) solution was prepared in different percentages (20 %, 30 %, 40 %, and 50 %) by mixing 10 g of MD in 40 g distilled water, 17.14 g MD in 40 g distilled water, 26.67 g MD in 40 g distilled water, and 50 g MD in 50 g distilled water. To dissolve MD, the distilled water was heated to 70°C . MD was added slowly into the heated distilled water. Then, the mixture was stirred using a magnetic stirrer until MD completely dissolved and a clear solution was formed.

The BPF extract and maltodextrin solution in the ratio of 5:1 (20 mL anthocyanin extract with 4 mL MD solution) were mixed thoroughly using a magnetic stirrer in a beaker for 20 min. The mixture was then poured into a 90 mm diameter glass Petri dish, encapsulated, and dried in a microwave oven at different microwave oven power; 50 % P, 70 % P, and 100 % P (550 W, 770 W, and 1100 W) for (6, 7, and 8) min, respectively. An encapsulation time ranging from 6–8 min was chosen based on the acceptable conditions for the encapsulation of dried BPF powder obtained from a preliminary test. The dried encapsulated BPF powder was scraped from the Petri dish, ground, and then stored in a glass bottle at room temperature for further analysis.

2.3 Total Anthocyanin Content

The total anthocyanin content (TAC) was determined using a pH-differential method described by Sutharut and Sudarat (2012). The extracted solution of 1 mL was transferred into a volumetric flask and marked up to 10 mL with two different buffer systems: a potassium chloride buffer, pH 1.0; and a sodium acetate buffer, pH 4.5. For powder analysis, 0.5 g of the sample was diluted to 5 mL of the two different buffers. The absorbance was measured at 520 nm and 700 nm using a spectrophotometer (T70/T80 UV-vis Spectrophotometer, PG Instrument, UK). Distilled water was used as a blank. The absorbance difference between the pH 1.0 and pH 4.5 samples was then calculated using Eq(1).

$$\text{Absorbance, } A = (A_{520 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}1} - (A_{520 \text{ nm}} - A_{700 \text{ nm}})_{\text{pH}4.5} \quad (1)$$

The total anthocyanin content was calculated as cyanidin-3-glucoside (mg/L) according to Eq(2).

$$\text{Monomeric anthocyanin pigment (mg/L)} = \frac{A \times \text{MW} \times \text{DF} \times 1000}{\epsilon \times 1} \quad (2)$$

where MW (molecular weight) = 449.2 g/mol for cyanidin-3-glucoside; DF = dilution factor; 1 = path length in cm; and ϵ = 26,900 molar extinction coefficient in L/mol/cm for cyanidin-3-glucoside.

2.4 Efficiency of encapsulation

The efficiency of encapsulating BPF colourant was calculated using Eq(3) proposed by Zhang et al. (2007). The encapsulation efficiency (EE) is defined as the ratio of total anthocyanin content in the powder to the anthocyanin content in the extract.

$$\%EE = \frac{\text{Total anthocyanin content in the powder}}{\text{Total anthocyanin content in the extract}} \times 100 \quad (3)$$

2.5 Colour Parameter Index

By using a colour reader (CR 10 Tristimulus, Konica Minolta, Tokyo), the b^* values of the samples (100 to -100) were determined based on the method outlined in Mohd Nawi et al. (2015). A negative b^* value infers a blue hue while a positive b^* value indicates a yellow hue. Each sample was measured in triplicate.

2.6 Water activity

The water activity of the encapsulated BPF powder was determined using a portable water activity meter set (4TE Aqua Lab by Decagon, USA). The method was performed according to Ramos et al. (2013) but with different materials. For each analysis, a 0.3 g average sample weight was placed in a plastic stub until the base was fully covered. The water activity meter took approximately 5 min to analyse each sample. The measurement was conducted at 25 °C.

3. Results

3.1 Effect of maltodextrin concentration

Table 1 shows the effect of maltodextrin (MD) concentration on the TAC and the blue parameter index of the encapsulated BPF colourant.

Table 1: The effect of different maltodextrin concentrations on the total anthocyanin content (TAC) and the blue parameter index (b^) at 770W for 6 min.*

MD	TAC (mg/L)	b^*
20 %	8.35	3
30 %	11.61	1
40 %	18.79	-1
50 %	10.44	-6

In general, different concentrations of MD caused a significant difference in the TAC and the blue parameter index values of the encapsulated BPF colourant. The TAC of the non-encapsulated extract was 30.9 mg/L. In this study, the highest TAC was retained when the BPF colour was encapsulated with 40 % MD. However, increasing the MD concentration to 50 % caused a reduction in TAC. This result is in agreement with that of Hariadi et al. (2018), who found that the increment in maltodextrin concentration could decrease the total anthocyanin content in blue pea powder due to the addition of dried solids in the mixture. A similar result was also obtained by Padzil et al. (2018) whereby an increment in maltodextrin concentration lowered the total monomeric anthocyanin content of purple sweet potato extract. As for the blue parameter index, the best MD

concentration for maintaining blueness (0 to -100) or to lower the value of b^* of the BPF colourant was between 40 % and 50 % MD. This result is slightly different than that of Hariadi et al. (2018).

3.2 Effect of Microwave Power

Table 2 highlights the effect of microwave power on the TAC and the blue parameter index of encapsulated BPF colourant. By applying varied microwave power with constant MD concentration (40 %) and constant encapsulation time (7 min) on the BPF colourant and maltodextrin mixture, the resulting colourant powder showed significantly different characteristics based on the TAC retained. The highest TAC was obtained when encapsulation and drying were performed using 770 W. For 770 W and 1100 W, the TAC retained showed a similar average result for the blue parameter index, indicating a tendency towards blue. There is not much information relating to the effect of power in microwave-assisted encapsulation since the microwave-assisted technique is generally used for pigment extraction. Nevertheless, in microwave heating, a higher power normally produces a higher heating rate. In this study, the high power possibly induced faster encapsulation with the maltodextrin; and thus a higher TAC was retained when the encapsulation was performed at 770 W and 1100 W. At a low power of 550 W, the encapsulation process possibly occurred at a slower rate such that the extract was not fully encapsulated with the maltodextrin and stuck onto the Petri dish as waste. As the power increased, the blue parameter index of the encapsulated BPF colourant tended to 0. Based on the CIE $L^* a^* b^*$ colour index value, the colour parameter of BPF could also be influenced by the L^* and a^* values. Thus, the increment in the b^* value (below 0 value) is acceptable.

Table 2: The effect of different microwave encapsulation power on the total anthocyanin content (TAC) and the blue parameter index (b^) of encapsulated BPF colourant using 40 % MD for 7 min.*

Power (W)	TAC (mg/L)	b^*
550	9.02	-6
770	27.05	-3
1,100	26.55	-1

3.3 Effect of Encapsulation Time

Table 3 shows the effect of encapsulation time on TAC, blue parameter index, and encapsulation efficiency at constant microwave power (770 W) and maltodextrin concentration (40 % MD). The results show that different encapsulation times gave different characteristics of BPF colourant. The result was also considered in the selection of a suitable condition for developing an optimum BPF colourant powder. The minimum time of 6 min was selected for encapsulation, showing a low TAC retainment compared to 7 min and 8 min encapsulation time under 770 W microwave power. This result is probably due to the insufficient time for the anthocyanin to be fully encapsulated by the 40 % MD in microwave-assisted encapsulation. Hence, the encapsulated BPF powder retained a lower TAC. The blue parameter index became less blue as the encapsulation time increased due to exposure to higher heat treatment, which possibly made the surface slightly darker, resulting in changes to the BPF colour parameter. The highest encapsulation efficiency (EE) of (77.71 %) TAC in the powder was obtained when the encapsulation was conducted for 7 min. A study by Li et al. (2016) showed that the extraction of flavonols increased rapidly as the microwave irradiation time increased, and then decreased after reaching the optimum time. Farzaneh and Carvalho (2017) also mentioned that the extra exposure time of flavonol compounds to microwave irradiation might decompose the monomeric anthocyanins and reduce the extraction efficiency of TAC. On the contrary, Romero-díez et al. (2019) showed that a longer irradiation time increased extraction yield, and, at the same time, increased processing temperature, which could be a factor for higher extraction yield.

Table 3: The effect of different encapsulation times on the total anthocyanin content, the blue parameter index (b^), and the efficiency of encapsulation of encapsulated BPF colourant using 40 % MD at 770 W.*

Time (min)	TAC (mg/L)	b^*	EE %
6	16.68	-11	55.25 %
7	26.80	-10	77.71 %
8	19.59	-7	64.89 %

3.4 Best Conditions for the Development of a Stable BPF Colourant

Based on the tests conducted above, the best condition consisted of 40 % MD as the encapsulating agent, 770 W microwave power, and 7 min encapsulation time. The condition was repeated three times to obtain the TAC, the encapsulation efficiency, and the blue index. The result showed a high TAC of 27.03 ± 1.415 mg/L, a

blue index b^* of -9 ± 0.324 , and an encapsulation efficiency of $73.24 \% \pm 0.051$. Using the best conditions for the BPF colourant encapsulation, the water activity was analysed and found to be 0.4941, less than 0.5, which means that this BPF colourant is unlikely to suffer significant microbiological growth during storage (Gutiérrez et al., 2015). This result is similar to the result found in Mohd Nawi et al. (2015) study of purple sweet potato extract powder encapsulated with maltodextrin, in which microwave-assisted encapsulation was also used and the water activity of the encapsulated product was in the range of 0.5 to 0.52. In comparison, Tze et al. (2012) observed lower water activity in the encapsulation of betacyanin colourant using the spray-dried method.

3.5 Effect of pH on BPF Colourant

BPF contains anthocyanins; thus the extract can change colour at different pH. Figure 1 (a) shows the encapsulated BPF colourant and Figure 1 (b) shows the colour change of the BPF when the encapsulated colourant was mixed with a pH 1 to pH 13 buffer solution. The change in colour confirmed that BPF contains anthocyanins. Figure 1 (b) shows the transition of the blue-coloured solution to a pink colour when it reacted with a pH 1 acidic solution and a purple colour when it reacted with a pH 2 acidic solution. It remained blue at pH 3 to pH 7 and then changed to a green colour when mixed with alkaline solution (pH 8 to pH 13). This result is in agreement with that of Castañeda-Ovando et al. (2009), who found that at pH 1, the flavyium cation (red colour) was the predominant species; hence, the solution turned into a purple and red colour followed by the original colour remaining in the natural base (pH 7). Then, the colour degraded as the pH went above pH 7.

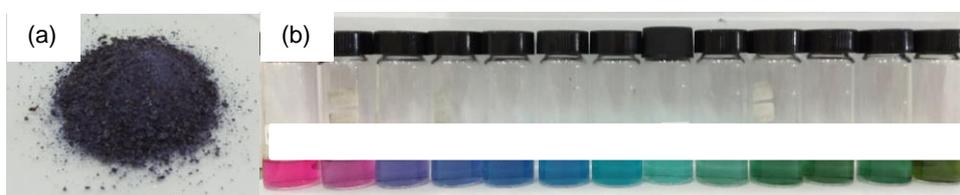


Figure 1: (a) The BPF colourant powder and (b) the anthocyanin from the BPF colourant in different pH buffers

4. Conclusions

From this study, it can be deduced that in the microwave-assisted encapsulation of BPF extract, maltodextrin concentration, microwave power, and encapsulation time played a significant role in producing high quality encapsulated BPF colourant. The best encapsulation condition determined from this study (40 % MD, 770 W and 7 min) produced an encapsulated BPF colourant with high encapsulation efficiency (73.24 %), high TAC (27.03 mg/L), and low water activity value (0.4941), besides better retainment of the blue pigment in the encapsulated product (b^* value -9).

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