Microencapsulation of plum (*Prunus salicina* Lindl.) phenolics by spray drying technology and storage stability

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Abstract

To improve the stability of the phenolic extracts from plum fruit (*Prunus salicina* Lindl.), the microencapsulation conditions of spray drying were optimized by the response surface method. The Box-Behnken experimental results indicated the optimal conditions involved an inlet air temperature of 142.8 °C, a core material content of 23.7% and a feed solids content of 11.7%. The maximum microencapsulating efficiency was 87.7% at optimal conditions. Further, the physicochemical properties of the microcapsule powders were improved overall due to the addition of the coating agents. There were no statistically significant differences in phenolic content of the obtained microcapsules for the first 40 days of storage at 25 °C in dark condition ($p > 0.05$), and the retention rate of total phenol remained above 85% after 60 days. Microcapsules can be potentially developed as a source of natural pigment or functional food based on the advantages of rich phenolic compounds and red color.

Keywords: *Prunus salicina* Lindl.; microencapsulation; phenolics; spray drying; storage stability; physicochemical properties.

Practical Application: Microencapsules of plum phenolics can be used as a source of natural pigment or functional food.

1 Introduction

Plum (*Prunus salicina* Lindl.) is one of the important fruits in the temperate zone. This fruit is widely cultivated in China, Japan, Spain, Chile, the United States, and other countries. Plum fruit presents high levels of phenolic compounds and anthocyanins, as well as strong antioxidant capacities (Bobrich et al., 2014; Lozano et al., 2009). Furthermore, plum polyphenols can inhibit colorectal aberrant crypt foci formation (Banerjee et al., 2016) and xanthine oxidase activities (Li et al., 2016). However, phenolic compounds are unstable and susceptible to oxidation, polymerization, condensation reaction when exposed to light, oxygen, or high temperatures, thus undermining their biological activity.

Microcapsule technology utilizes natural or synthetic polymeric materials to encapsulate solid, liquid, and even gaseous substances to form minute particles with semipermeable or sealed capsules. Microencapsulation is used for protecting a bioactive compound from light, oxygen, or other unfavorable conditions and improving stability (Gonçalves et al., 2017). Therefore, microencapsulation methods have been performed to store phenolic compounds extracted from vegetables or fruits (Çam et al., 2014; Paini et al., 2015).

Spray drying, a common industrial encapsulation technique, has been presented as an efficient method for protecting bioactive compounds from possible physical and chemical interactions with the external environment, thus, allowing a longer shelf life and wider industrial application. Moreover, spray drying provides rapid evaporation of water and maintains the low temperature in the particles (Busch et al., 2017). Several studies were performed using spray drying as microencapsulation technology for phenolic extracts from different plant sources, for example, phenolic

compounds of olive pomace (Paini et al., 2015) and jaboticaba peel extracts (Silva et al., 2013). However, microencapsulation of phenolic extracts from plum using spray drying has not been performed yet.

The objective of this work was to obtain the microencapsulation powder of phenolic extract from plum fruit by optimizing the spray drying parameters. The physicochemical characteristics and storage stability of powders were determined after encapsulation.

2 Materials and methods

2.1 Plant materials

The plum of *Prunus salicina* Lindl. cv. Furong was collected from Yongtai County of Fujian Province, China. After picking and cleaning, the plums pits were removed and the pulps were dried to a moisture content of less than 15% with hot air at 50 °C. The dried pulps were milled and sieved (0.5 and 2.0 mm particle size) then stored at -20 °C refrigerator in the dark.

2.2 Chemicals

Gallic acid was purchased from Shanghai Yuanye Biotechnology Co., Ltd.. Maltodextrin (dextrose equivalence 15) was sourced from Tang Ye (Tianjin) Food Co., Ltd., China. Beta-cyclodextrin was obtained from Shanghai Chemical Reagent Co., Ltd, China. Arabic gum was purchased from Shanghai Quan Wang Biological Technology Co., Ltd.. All other organic solvents used in the study were analytical grade.

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2.3 Extraction of phenolics

Phenolics of plums were extracted with 61% ethanol by applying previously optimized conditions at a ultrasonic power of 420W, and an extraction temperature of 59 °C for 47 min (Li et al., 2016). Twenty grams of plum samples were placed in a 1 L beaker and mixed with 200 mL of 61% ethanol (w/v). The extraction process was performed in an ultrasonic bath (KQ-600DV, 40 kHz, Kunshan Ultrasonic Instrument Co. Jiangsu, China). After ultrasonic extraction, the mixture was filtered (Whatman No. 1 paper). The filtrate was condensed to 17% solid content with a vacuum rotary evaporator (Senco-GG17, Shanghai Shenke Science and Technology Co., Ltd., China) at 0.09 MPa, 45 °C. The phenolic extracts of plum were stored in -20 °C for further use.

2.4 Encapsulation of propolis by spray drying

In previous studies, the effects of different coating agents and their combinations on the microencapsulation efficiency of plum phenolics were discussed. Finally spray drying of phenolic extracts was performed using maltodextrins, beta-cyclodextrin and arabic gum as coating agents. The ratio of the coating agents was maltodextrin: beta-cyclodextrin: arabic gum = 7: 2: 1 (w/w/w). The microencapsulation process was performed using a laboratory-scale spray dryer (Shiyuan Mini Spray Dryer SY6000, China) under the following operational conditions: atomization pressure 90 MPa, feed flow rate 550 mL/h, air flow rate 330 m³/h, and inlet-air temperature ranged from 110 °C to 150 °C. Under these conditions the outlet-air temperatures ranged from 88 °C to 94 °C. The spray drying powders were packed with aluminum foil bags and stored at -20 °C for further use.

2.5 Experimental design

On the basis of the previous single factor test, a response surface method (RSM) was used to evaluate the influence of three independent variables on the microencapsulating efficiency of total phenolics. The independent variables were air inlet temperature (°C, X_1), Core content (% , X_2) and Feed solids concentration (% , X_3). The experimental design for the Box–Behnken Design was performed in random order and consisted of 17 combinations including five replicates at central point (Table 1). In order to obtain the regression coefficients, an analysis of variance (ANOVA) was carried out using the Design-Expert.V8.0.6.1 software (State-Ease Inc., Minneapolis, MN, USA). Experimental data were fitted to a second-order polynomial model where multiple regression analysis and ANOVA were used to determine fitness of the model and optimal conditions for investigated responses. All of the treatments were performed in triplicate.

2.6 Determination of total phenolic content

Total phenolic content was evaluated using the Folin–Ciocalteu assay (Li et al., 2016). An amount of 100 mg of phenolic powder was accurately weighed and dissolved in 5 ml deionized water. Then the mixture was agitated using a vortex shaker (QL-861, Haimen Qilinbell Instrument Manufacturing Co., Ltd, China) for 1 min and filtered with microfilter (0.45 µm). 2.5 mL diluted Folin–Ciocalteu reagent (10%, v/v) was added to 200 µL of filtrate

in a capped glass tube. After 2 min of incubation in the dark at room temperature, 2 mL of aqueous sodium carbonate (7.5%, w/v) was added to the mixture. And then it was made up 10 ml by adding distilled water. After gentle vibration, the mixture was placed in a water bath at 50 °C for 30 min and then rapidly cooled down to room temperature (25 °C). Absorbance was measured at 765 nm using a UV-vis spectrophotometer (756P, Shanghai Spectrum Instruments Co., Ltd., China). Total phenolic content of spray-dried phenolic powder was expressed as mg GAE (gallic acid equivalent) per gram of dried weight.

2.7 Surface phenolic content of microencapsules

Surface phenolic content was determined following the method of Cilek et al. (2012). An amount of 100 mg of microcapsules were dispersed with 5 ml of ethanol for 3 min. The mixture was filtered with microfilter (0.45 µm) and the total phenolic content of the filtrate was determined. The amounts of surface phenolic compounds were determined and quantified with the same method described in total phenolic content section.

2.8 Efficiency and yield of microencapsulation

Microencapsulating efficiency was calculated as the ratio of encapsulated phenolic content to total phenolic content. The microencapsulating yield was the ratio of the mass of the microcapsules obtained at the end of the process to the mass of the initial substances (solids of plum extracts and coating agents). Microencapsulating efficiency and yield of microcapsules were calculated according to Equations (1) and (2), respectively (Cilek et al., 2012; Venil et al., 2016).

Table 1. The experimental design and results for response surface methodology*.

Run	X_1 (°C)	X_2 (%)	X_3 (%)	Microencapsulating efficiency (%)
1	130	20	10	85.7 ± 1.5
2	130	15	15	83.4 ± 2.0
3	130	25	15	85.1 ± 1.8
4	150	20	15	86.4 ± 1.0
5	150	25	10	87.4 ± 1.4
6	110	20	5	77.7 ± 1.4
7	110	20	15	78.3 ± 2.3
8	130	20	10	86.2 ± 1.6
9	150	20	5	83.6 ± 0.7
10	130	20	10	85.0 ± 1.8
11	130	15	5	80.2 ± 1.1
12	130	20	10	85.6 ± 1.7
13	110	15	10	76.4 ± 1.9
14	150	15	10	81.5 ± 2.0
15	130	25	5	84.8 ± 1.6
16	110	25	10	79.0 ± 1.4
17	130	20	10	86.3 ± 1.9

* X_1 = Air inlet temperature; X_2 = Core content; X_3 = Feed solids concentration; Mean values (n = 3) ± S.D.

$$\text{Efficiency (\%)} = \left(1 - \frac{\text{Phenolics on microcapsule surface}}{\text{Total phenolics of microcapsule}} \right) \times 100 \quad (1)$$

$$\text{Yield (\%)} = \frac{\text{Mass of microcapsules (g)}}{\text{Total mass of initial substances (g)}} \times 100 \quad (2)$$

2.9 Physicochemical characterization of powders

Moisture content: Powder was dried at $105 \pm 2^\circ\text{C}$ until a constant weight, and moisture content was calculated based on the loss in weight between before and after drying.

Water activity: The water activity (a_w) values of powders were measured with a water activity measurement device (HD-3A, Wuxi Huake Instrument Co., Ltd., China).

Bulk density: The bulk density of the phenolic powders was determined by pouring approximately 5 g of the powder into a 10 mL graduated cylinder. The volume occupied by the sample was recorded and bulk density was calculated by dividing the mass of the powder by the volume occupied in the cylinder (Paini et al., 2015).

Angle of repose: The angle of repose was determined by pouring the powder from a funnel (dia. 1 cm) to the surface plate. A funnel was clamped with its tip 7 cm above the surface. The tangent of the angle of repose was the ratio of the height of the heap of powder to the semi-diameter of the base of the heap of powder (Kaur et al., 2015).

Color analysis: The color of the samples was carried out with a colorimeter (NS810, Shenzhen 3NH Technology Co., Ltd., China) and reported in CIE L^* , a^* , b^* values. L^* denotes the degree of lightness on 0–100 scale from black to white, a^* is the degree of redness (+) to greenness (–), and b^* is the degree of yellowness (+) to blueness (–) (Simon-Brown et al., 2016). The instrument was standardized by a white tile before the measurements. All analyses were performed in triplicate.

Scanning electron microscopy: Morphological characteristics of the microencapsulated powders were observed using a JSM-6380LV scanning electron microscope (Japan).

2.10 Storage stability

During storage tests, microcapsules and phenolic powder (non-encapsulated) were sealed with aluminum foil bags respectively and were stored in a 25°C incubator for 2 months. The total phenol retention of the samples was analyzed on the days 10, 20, 30, 40, 50 and 60 during storage.

3 Results and discussion

3.1 Fitting the response surface models

The three factor and three level response surface analysis was designed by selecting the inlet air temperature, the core material content, the feed solids content as independent variables, and the microencapsulating efficiency as the response value. The results of the test and the results were shown in Table 1. By using Design-Expert V8.06 software, the response surface regression analysis of the experimental data was carried out, and the simulation equations of the microencapsulating efficiency and the dependent variables were obtained:

$$Y = 85.77 + 3.44 X_1 + 1.82 X_2 + 0.86 X_3 + 0.81 X_1 X_2 + 0.53 X_1 X_3 - 0.72 X_2 X_3 - 3.3 X_1^2 - 1.41 X_2^2 - 0.98 X_3^2 \quad (3)$$

In order to test the validity of the regression equation, the variance analysis and the model analysis were carried out (ANOVA, Table 2). It could be seen from Table 2 that the $p < 0.0001$ of the model showed that the equation of the two order was extremely significant. Lack of fit ($p = 0.6161 > 0.05$) was not significant, which suggested that the regression equation fit o the test accuracy and small error. The coefficient of determination was $R^2 = 0.9907$,

Table 2. Analysis of variance (ANOVA) for the regression model.

Source	Sum of Squares	DF	Mean Square	F-Value	p-Value
Model	196.0	9	21.8	83.3	< 0.0001
X_1	94.7	1	94.7	362.0	< 0.0001
X_2	26.5	1	26.5	101.5	< 0.0001
X_3	6.0	1	6.0	22.8	0.0020
$X_1 X_2$	2.7	1	2.7	10.2	0.0153
$X_1 X_3$	1.1	1	1.1	4.2	0.0792
$X_2 X_3$	2.1	1	2.1	7.9	0.0263
X_1^2	46.0	1	46.0	175.8	< 0.0001
X_2^2	8.3	1	8.3	31.9	0.0008
X_3^2	4.0	1	4.0	15.4	0.0057
Residual	1.8	7	0.3		
Lack of Fit	0.6	3	0.2	0.7	0.6055
Pure Error	1.2	4	0.3		
Cor Total	197.9	16			
$R^2 = 0.9907$; $R^2_{Adj} = 0.9789$					

X_1 = Air inlet temperature; X_2 = Core content; X_3 = Feed solids concentration; DF = degrees of freedom.

which indicated the regression model fit well and the variation of the response value was 99.07% from the selected variables. The adjustment coefficient of the model was $R^2_{Adj} = 0.9789$, which showed that had excellent predictability and the actual value was close to the predicted value. Therefore, the regression model was feasible, which was suitable to analyze and predict the microencapsulation parameters of spray drying from plum fruit extract.

3.2 Response surface analysis of interaction between different factors

According to the regression equation, the interaction between three factors, including the inlet air temperature, the core material content and the feed solids concentration, was analyzed (Figure 1).

As shown in Figure 1A, the response surface was quite steep, indicating a significant interaction between the inlet air temperature and the core material content. The effect of inlet air temperature on encapsulating efficiency was more than that of core material content, which was consistent with the results of the above ANOVA analysis. It can be seen from Figure 1B that the polyphenol encapsulating efficiency increased firstly and then decreased slowly as the inlet air temperature or the feed solids concentration increased. Figure 1C presented that the interaction between the core content and the feed solids concentration was significant and the core content had a greater effect on the microencapsulating efficiency of phenolic extracts.

3.3 Optimization of microencapsulation conditions and verification of model

The optimum parameters of plum extract microencapsulation were obtained by means of mathematic model. The microencapsulating efficiency was 87.7% when the optimum process parameters of spray drying were as follows: inlet air temperature 142.8 °C, core material 23.7%, and feed solids content 11.7%.

To validate the constructed mathematical models, a spray-drying experiment was carried out at the optimal conditions for three parallel experiments. Considering the actual operation, the optimum parameters of spray drying were modified to the inlet air temperature of 143 °C, the core material content of 23.7% and feed solids content of 11.7%. Under these conditions, the microencapsulating efficiency of the plum extract was 89.3%. The error between the actual value and the predicted value was 1.8%, which indicated that the constructed mathematical model appropriately explained the actual microencapsulation process of the phenolic extract from plum.

3.4 Physicochemical properties of microencapsulated powder

The physicochemical properties of the microcapsules (Figure 2) produced under the optimized spray dryer operating conditions and phenolic powder (non-encapsulated) prepared by spray drying without using any coating agents were detailed in Table 3.

Moisture, a_w and yield

Table 3 indicated that the spray drying yield of phenolic extract significantly increased after microencapsulation, and the moisture content and water activity of the product obviously decreased, which showed that the coating agents improved the yield and quality of the spray dried powder from plum extracts. Similar results were also reported by Peng et al. (2013).

Figure 1. Response surface plots for encapsulating efficiency of phenolic compounds as a function of (A) inlet air temperature to core content (feed solids concentration = 10%); (B) inlet air temperature to feed solids concentration (core content = 20%); (C) core content to feed solids concentration (air inlet temperature = 130 °C).

Figure 2. Microencapsulated powders of phenolic extract prepared by spray drying.

Table 3. Physicochemical properties of microencapsulated powder (MP) and phenolics powder (non-encapsulated) (PP)^a.

Items	Moisture (%)	a_w	Yield (%)	Bulk density (g·mL ⁻¹)	Angle of repose (°)	Color ^b			Total phenol (mg GAE/g)
						L^*	a^*	b^*	
PP	5.4 ± 0.3	0.4 ± 0.1	59.6 ± 2.1	0.6 ± 0.1	55.5 ± 3.6	55.2 ± 1.4	22.3 ± 1.2	3.9 ± 0.2	182.8 ± 4.2
MP	2.8 ± 0.1	0.2 ± 0.1	27.6 ± 1.1	0.5 ± 0.1	46.5 ± 2.1	78.3 ± 1.2	9.0 ± 0.3	1.9 ± 0.1	57.8 ± 1.2

^aData are presented as the mean ± SD (n = 3). MP represents the microencapsulated powder prepared by the above optimum conditions of spray drying, and PP represents phenolic powder (non-encapsulated) prepared by spray drying without using any coating agents; ^bIn the CIE-Lab system, L^* denotes lightness on 0–100 scale from black to white; a^* , (+) red or (–) green; and b^* , (+) yellow or (–) blue.

Bulk density and angle of repose

Bulk density of the spray-dried microparticles is the density of powder when packed or stacked in bulk, and the angles of repose are used to describe the fluidity of powder. Table 3 showed that microencapsulation improved the fluidity of the powder as indicated by the decreased angles of repose and reduces the bulk density. Similar findings were observed by Fazaeli et al. (2012). The bulk density and fluidity of the microcapsule product were related to the nature of the coating material used for embedding (Bhusari et al., 2014).

Color

Color parameters, including L^* , a^* , and b^* values of the spray-dried powder were presented in Table 3. An increase in L^* value was as a result of the addition of the coating agents. This observation was similar to that made by Peng et al. (2013). a^* values and b^* values of microparticles with coating agents were significantly lower than those of the powder without coating agents, thus decreasing the redness and yellowness of powder because of the addition of the coating agents. The observation was in agreement with the result reported by Bhusari et al. (2014). The evaluation of a^* parameters for the microparticles containing plum extract was related to the red color and may be associated

with the content of anthocyanins present in the sample. Similar trend was found by Carvalho et al. (2016). The changes in color parameters were attributed to the addition of coating agents and were associated with variation in phenols and anthocyanins (Peng et al., 2013).

Total phenol

Table 3 showed that both spray-dried powders were rich sources of phenolic compounds. But the microcapsules were lower on the indicator, which might be that the coating agents reduced the concentration of the active ingredient in the microcapsule powder.

Morphology of microencapsulated powder

The surface morphology of microcapsules is an important index for the quality of microcapsules. The surface structure of microcapsules not only affects the microencapsulation effect, but also is closely related to physicochemical properties of microcapsules, such as fluidity and dispersibility (Santana et al., 2013; Çam et al., 2014). The surface microstructure observed in the scanning electron microscope of phenolic microcapsules from plums was shown in Figure 3 with a level of magnification of 1,000× and 2,000×. Most of the microencapsulated particles were

Figure 3. Scanning electron microstructure of microencapsulated powder from plum extract. (A) $\times 1000$; (B) $\times 2000$.

spherical shapes on the whole, and particles had a complete surface structure with no cracks or collapses, which suggested that the microencapsulation of powder may be favourable and effective. The surface of the microcapsule particles was wrinkled, which is one of the reasons for the poor fluidity of the microcapsules. This is consistent with other microcapsules prepared by spray drying (Fernandes et al., 2014).

3.5 Storage stability

In order to determine the storage stability of microcapsules as well as to evaluate the protective effects of coating material on phenolics, the microcapsules and phenolic powder (non-encapsulated) prepared by spray drying without using any coating material were stored at 25 °C for 60 days. Figure 4 obviously showed that the phenolic microcapsules were more stable than the phenolic powder (non-encapsulated) during storage. There were no significant differences in the total phenol content of the microcapsules within the first 40 days of storage ($p > 0.05$). And after 60 days storage at 25 °C, the total phenol retention rate of the microcapsules was still greater than 85%. However, 36.75% and statistically significant loss in phenolic content of the phenolic powder (non-encapsulated) was observed after 60 days storage at 25 °C. The results indicated that the film-forming property of coating material and capsule membrane compactness of the microcapsule product were well, which could resist the destructive effect of the adverse environment and reduce the influence of the external conditions on the stability of phenolic compounds.

This was in agreement with the reports of other researchers who had evaluated the effect of microencapsulation by spray drying on phenolics. Santiago et al. (2016) observed that anthocyanin retention in microcapsules produced by spray drying of pomegranate juice was equivalent to 90% and 60% after being stored at 25 °C for 90 and 120 days, respectively. Çam et al. (2014) evaluated the stability of the microcapsules and phenolics powder (non-encapsulated) obtained by spray drying without using any coating material from Pomegranate peel stored at 4 °C for 3 months, these authors observed phenolic microcapsules are more stable than the phenolics powder. Only 18% decrease in anthocyanin content of the microcapsules obtained from

Figure 4. Storage stability of microcapsules (○) and phenolic powder (●). (Values represent the mean $\pm$ standard deviation ($n=3$). Different letters after mean values indicate significant differences among the storage periods of samples ($p < 0.05$).

Guarcinia indica was observed at the end of 12 weeks storage at 25 °C (Nayak & Rastogi, 2010).

4 Conclusions

The microencapsulating conditions of the spray drying from plum fruit (*Prunus salicina* Lindl.) phenolics were optimized by a Box-Behnken experiment design of three variables. Using the response surface method, the optimum conditions of spray drying were the inlet air temperature of 143 °C, the core material content of 23.7% and feed solids content of 11.7%. Although total phenol content of microcapsules decreased, overall the physicochemical properties of the spray-dried powders were improved after adding the coating agents. Phenolic components of the microcapsules did not lose significantly in the first 40 days of storage under the condition of 25 °C dark storage, and the retention rate of total phenol remained above 85% after 60 days. The results clearly indicated that the storage stability of microencapsulated powder was better than that of phenolic powder (non-encapsulated). Therefore, microencapsulated

powder of plum extract can be considered as a potential source of natural pigment or functional food due to the advantages of rich phenolic compounds and red color. Future studies are necessary to investigate the interaction of plum phenolics with other food components.

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