



Original Research Article

Encapsulation of soybean extract using spray drying

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ABSTRACT

Soybean (*Glycine max* L. Merrill) is recognized as a health food due to its many health-promoting bioactive compounds. Phenolic compounds are the main bioactive compounds in soybean because their antioxidant properties. Soybean extract is recommended as an ingredient of many pharmaceutical products. However, the rather poor solubility of soybean extracts and their proneness to degradation/oxidation during storage may reduce the nutritional value of the extracts. A convenient way to improve the solubility and extend the shelf-life of an extract is encapsulation. Among various encapsulation techniques, spray drying is the most widely and typically used technique. The objective of this work is to study the effects of encapsulation via spray drying on various properties of encapsulated soybean extract. The studied spray drying parameters are inlet drying temperature (130-170 °C) as well as the type of encapsulating material (maltodextrin, gum arabic and β -cyclodextrin). The ratio between soybean extract and encapsulating material is 30:70. The properties to be analyzed are moisture content, total phenolic compounds content, encapsulation yield, encapsulation efficiency, hygroscopicity, particle size distribution and microstructure of encapsulated particles. The results showed that both type of wall material and inlet temperature significantly influenced the properties of soybean extract encapsulated powder. Gum arabic as the wall material and an inlet temperature of 170 °C, were the suitable conditions for encapsulation, produced the best encapsulated powder properties.

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INTRODUCTION

Soybean has been used as beneficial dietary and nutritive materials due to its high quality proteins and many other functional substances like phenolic compounds such as isoflavones which are the main phenolic compounds in soybean (Zhang *et al.*, 2003). Phenolic compounds have various biological activities like antioxidative, antimutagenic activities, antibacterial, atherosclerosis, coronary heart disease, or anticancer effects (Chung *et al.*, 2011). Therefore, soybean has received increasing attention in recent years by researchers and soybean extract is recommended as an ingredient of many pharmaceutical products (Mesa *et al.*, 2008).

On the other hand, one of the major problems associated with the nutritional value of the soybean extracts is their high susceptibility to oxidative deterioration during storage. Thus, there is a need to protect these active components in order to make them more stable during handling, processing and storage (Augustin *et al.*, 2006).

Encapsulation, a convenient way to protect the active components, is a process by which solids, liquids or even gases may be enclosed in microscopic particles formation of thin (Agnihotri *et al.*, 2012). Spray drying is a process widely used for encapsulation of active components because it is not only low-cost commercial process but also high quality. Both wall material selection and inlet drying temperature can affect the process efficiency and the encapsulated product stability. A successful encapsulation must result in a powder with maximum encapsulation yield, encapsulation efficiency and retention of the active compounds.

Several studies have been shown that the quantification of phenolic compounds may depend on the type of soy food and its processing (Chung *et al.*, 2011). The temperature, during drying and extraction, affects the activity and stability of compounds due to chemical and enzymatic degradation, losses by volatilization or thermal decomposition. These latter factors have been suggested to be the main mechanism causing the reduction in phenolic content (Moure *et al.*, 2001). Furthermore, the type of wall material also affects the quantification of active compounds due to interaction between active compounds and wall material (Georgetti *et al.*, 2008). Maltodextrin is a hydrolyzed starch commonly used as wall material in the encapsulation of food ingredients (Gharsallaoui *et al.*, 2007). It offers advantages such as relatively low cost, low viscosity at high solids concentrations and good protection against oxidation. Gum arabic is one of the most common wall materials used in encapsulation by spray drying. It presents many desirable characteristics to be a good encapsulating agent (high solubility, low viscosity and good emulsifying properties). β -cyclodextrin is a family of cyclic oligosaccharides. It presents many advantages such as low viscosity and increase bioavailability and stability of active compounds (Chordiya and Senthilkumaran, 2012).

The objective of this work is to evaluate the potential of difference type of encapsulating material (maltodextrin, gum arabic and β -cyclodextrin) and difference inlet drying temperature (130-170°C). The properties to be analyzed are moisture content, total phenolic compounds content, encapsulation yield, encapsulation efficiency, hygroscopicity, particle size distribution and microstructure of encapsulated particles.

MATERIALS AND METHODS

Chemicals

Maltodextrin and gum arabic (food grade) were purchased from Labsystem (Bangkok, Thailand). Beta-cyclodextrin (food grade) was purchased from CTD Holdings (Florida, USA) Ethanol and methanol were obtained from Lab-Scan Analytical Sciences (Bangkok, Thailand). Folin-Ciocalteu reagent was purchased from Carlo Erba (Milan, Italy). Gallic acid was obtained from Sigma-Aldrich (St. Louis, MO). Sodium carbonate was purchased from Ajax Finechem (Sydney, Australia).

Soybean extraction

Soybean samples of Sojo 4 was cleaned and crushed to smaller size. The soybean was extracted in 70% ethanol (ratio between soybean and ethanol solution is 1 g: 10 mL) at room temperature for 24 hours. The supernatant was then separated from the residue by filtration using Whatman no. 1 filter paper. After that, the soybean extract was centrifuged at 9000 rpm for 15 min, evaporated by rotary evaporator and flushed by nitrogen gas. The solid content in the final soybean extract solution was 35.81 % (w/w).

Spray drying of soybean extract

Aqua solution of wall material (maltodextrin, gum arabic and β -cyclodextrin) at 3% (w/v) was mixed with soybean extract solution at solid mass ratio of 70:30 and then homogenized by homogenizer at 9000 rpm for 10 min. The resulting mixture was then spray dried using spray dryer with co-current flow. The drying chamber had a diameter of 30 cm and a height of 55 cm. The feed flow rate was kept at 200 mL/h. The air velocity of the drying air was fixed at 26 m/s. After spray drying, the encapsulated powder was collected through a high efficiency cyclone in a glass container, transferred to a glass vial and stored in clear vacuum bag at -20 °C.

Moisture content

The moisture content of encapsulated powder was determined using AOAC method 984.25 (AOAC, 2000). One g of encapsulated powder was dried at 105 °C until no change in mass was detected.

$$\text{Moisture content (\% d.b.)} = \frac{W_1 - W_2}{W_2} \times 100$$

where: W_1 = weight (g) of sample before drying

W_2 = weight (g) of sample after drying

Total phenolic compounds content

For total phenolic compounds content of particles, 0.02 g of encapsulated powder was dissolved in 10 mL deionized water. It was prepared as sample solution for determination of total phenolic compounds content of particles.

For total phenolic compounds content at surface of particles, 0.12 g of encapsulated powder was dispersed in 10 mL methanol and shaken by shaker for 30 min. The supernatant was then separated from centrifuge at 9000 rpm for 30 min. It was prepared as sample solution for determination of total phenolic compounds content at surface of particles.

The total phenolic compounds content of the encapsulated powder was determined by the Folin-Ciocalteu method (Borkatay *et*

al., 2013). A 2.5 mL of the sample solution was mixed with 2.5 ml of 10% Folin-Ciocalteu's reagent in deionized water. The mixture was vortexed and after 2 min, 2 mL of 7.5% Na₂CO₃ was added. The mixture was incubated for 2 hours in the dark at room temperature and absorbance by UV-Vis spectrophotometer was measured at 765 nm against blank. Deionized water was used as blank for of total phenolic compounds content of particles determination but methanol was used as blank of total phenolic compounds content at surface of particles determination. Gallic acid was used as standard for preparing the calibration curve. The phenolic content in sample solution was expressed in term of gallic acid equivalent (µg of GA/g of dry powder).

Encapsulation yield (EY)

The encapsulation yield was calculated from total phenolic compounds (TPC) content as the ratio of the total phenolic compounds content of particles after spray drying to the total phenolic compounds content before spray drying (Paramera *et al.*, 2011).

$$\%EY = \frac{\text{TPC content after spray drying}}{\text{TPC content before spray drying}} \times 100$$

Encapsulation efficiency (EE)

The encapsulation efficiency was calculated from phenolic content as the ratio of the inside phenolic content (total phenolic content minus surface phenolic content) to the total phenolic content before spray drying (Paramera *et al.*, 2011). Where the surface phenolic content is the quantity of phenolic compound around the encapsulating powder.

$$\%EE = \frac{\text{the inside TPC content of particles}}{\text{TPC content before spray drying}} \times 100$$

where the inside TPC content is the total TPC content minus the surface TPC content

Hygroscopicity

About 1 g of powder was spread evenly on Petri dishes (9 cm diameter) to allow for a high surface area between humid air and powder. Samples of each powder in the dishes were placed under 76% relative humidity using saturated NaCl solution at room temperature. A 15 min interval was selected to get hygroscopicity value. The gain in weight of the samples was considerably lower after 135 min. Although hygroscopicity is based on the equilibrium moisture content, to compare hygroscopicities, the weight increase per g of powder solids after being subjected to the atmosphere with relative humidity of 76% for 135 min was determined (Goula *et al.*, 2004).

Particle size distribution

Particle mean diameter was measured using a laser particle size distribution analyzer, Mastersizer 3000 (Malvern Instruments, Malvern, UK). A small sample was suspended in methanol. The particle size was expressed as D₄₃, the volume weighted mean diameter.

Scanning electron microscopy (SEM)

The morphology of the spray-dried particles was visualized using scanning electron microscopy (SEM) (JEOL JSM-6480LV, Japan) at 15

kV. Samples were mounted on self-adhesive carbon sticky tape and gold coated before imaging.

Statistical analysis

Statistical analysis was done according to SPSS 22.0 software program. Analysis of variance was performed by the one-way ANOVA procedure. Duncan's multiple range tests were used to determine significant differences between the means. Mean differences were considered significant at the $P < 0.05$ level.

RESULTS AND DISCUSSION

Effect of wall material on powder properties

The encapsulated powders were spray dried with the same inlet temperature (consider inlet temperature of 170 °C). The 3 wall materials tested were: maltodextrin, gum arabic and β-cyclodextrin. The moisture content, total phenolic content, encapsulation yield, encapsulation efficiency and hygroscopicity of encapsulated powder prepared with different wall materials are shown in Table 1.

The successful encapsulation of nutraceutical extracts should result in an encapsulated powder with maximum retention of the core material inside the particles (Jafari *et al.*, 2008). According to Table 1, the encapsulation efficiency of samples was significantly by the type of wall material used. The EE and EY values varied from 54.40% to 79.94% and 77.93% to 96.33%, respectively, among them β-cyclodextrin gave the lowest EE and EY values. Gum arabic gave the highest EE value due to its maximum retention of soybean extract inside the particles. Maltodextrin gave the highest EY value due to its highest total phenolic compounds content but it did not show the highest EE due to its high total phenolic compounds content at surface.

Hygroscopicities of powders were varied from 0.0731 to 0.1272 g /g (Table 1). It can be seen that gum arabic give the lowest hygroscopicity. It was inferred that gum arabic had the lowest hygroscopicity and maltodextrin had the highest hygroscopicity

Effect of inlet drying temperature on powder properties

When the inlet temperature increased from 130 to 170 °C, the EE values were varied from 79.94% to 80.41%, and remained almost unchanged for gum arabic.

The moisture content of the spray dried powder decreased with increasing inlet air temperature (Table 1). This was because the higher the inlet air temperature, the greater the rate of heat transfer to the particle, providing a greater driving force for moisture evaporation. Consequently, powders with reduced moisture content are formed. This result is consistent with other findings (Goula and Kazakis, 2004)

The microcapsules prepared at inlet temperatures of 130 to 170 °C, showed similar encapsulation efficiency even though the moisture content varied from 0.32% to 2.04% (dry basis). This study suggests that encapsulation efficiency is not influenced by moisture content of the encapsulated powder. This finding is in agreement with Wu *et al.* (2014) who state that the microcapsules prepared at inlet temperatures of 150 °C to 230 °C showed SF stability is not influenced by moisture content of the microcapsules.

Table 1 Properties of powders prepared with different wall materials and inlet temperatures.

Wall material	Inlet temperature (°C)	MC (% dry basis)	Total phenolic (μg gallic acid/ g dry solid)	%EY	%EE	Hygroscopicity ($\text{gH}_2\text{O}/\text{g}$ dry solid)	D ₄₃ (μm)
maltodextrin	130	5.44 ± 0.02 ^a	5317.48 ± 33.48 ^b	91.73 ± 1.09 ^b	71.80 ± 0.96 ^b	0.0819 ± 0.0026 ^{bc}	11.74 ± 0.01 ^d
	150	3.29 ± 0.01 ^c	5221.75 ± 20.87 ^{bc}	90.07 ± 0.46 ^{bc}	72.04 ± 0.06 ^b	0.0936 ± 0.0032 ^b	12.73 ± 0.02 ^d
	170	2.92 ± 0.01 ^d	5583.71 ± 40.02 ^a	96.33 ± 1.23 ^a	78.69 ± 1.14 ^a	0.1272 ± 0.0029 ^a	14.60 ± 0.04 ^c
gum arabic	130	2.04 ± 0.02 ^f	5102.10 ± 6.52 ^{cd}	87.32 ± 0.60 ^c	80.41 ± 0.53 ^a	0.0647 ± 0.0010 ^a	15.70 ± 0.08 ^c
	150	1.43 ± 0.01 ^e	5094.10 ± 36.19 ^{cd}	87.17 ± 0.58 ^c	80.13 ± 0.57 ^a	0.0678 ± 0.0004 ^{da}	15.65 ± 0.07 ^c
	170	0.32 ± 0.01 ⁱ	5322.29 ± 53.38 ^b	91.07 ± 0.64 ^b	79.94 ± 0.65 ^a	0.0731 ± 0.0024 ^{cda}	15.44 ± 0.07 ^c
B-cyclodextrin	130	3.75 ± 0.01 ^b	4992.07 ± 6.30 ^{de}	79.51 ± 0.54 ^d	52.47 ± 0.34 ^c	0.0800 ± 0.0001 ^{cd}	15.40 ± 0.38 ^c
	150	2.28 ± 0.01 ^e	4801.23 ± 14.70 ^{ef}	76.47 ± 0.64 ^d	52.59 ± 0.46 ^c	0.0830 ± 0.0010 ^{cd}	34.76 ± 1.13 ^b
	170	1.32 ± 0.01 ^h	4892.64 ± 13.01 ^{ef}	77.93 ± 0.46 ^d	54.40 ± 0.30 ^c	0.0941 ± 0.0028 ^b	38.80 ± 0.68 ^a

Different letters indicate significant difference in the same column between samples at $p \leq 0.05$.

Hygroscopicities of powders were varied from 0.0647 to 0.0731 g/g in case of gum arabic. It increased with decreasing moisture content (Table 1) because the low moisture content powder can absorb moisture than the higher moisture content powder (Sarochwikasit and Tangduangdee, 2011).

Particle size distribution

Particle mean diameters varied from 11.74 to 38.80 μm (Table 1). The encapsulated powder produced from maltodextrin and gum arabic showed monomodal distribution and greater size but β -cyclodextrin shown bimodal distribution (Figure 1) due to the high shear stress during spraying. Spray drying is likely to cause changes in the emulsion structure with some coalescence or fragmentation of oil droplets that may lead to a less efficiency encapsulation (Turchiuli *et al.*, 2014).

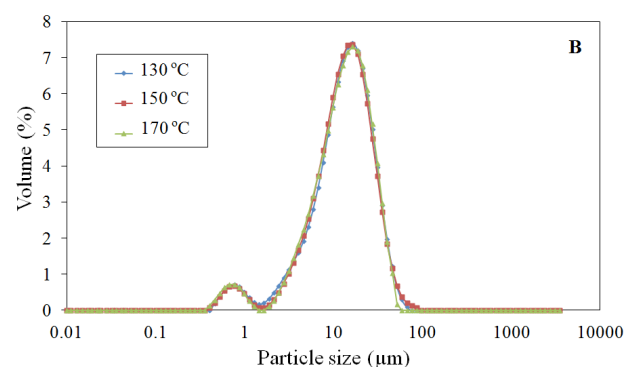
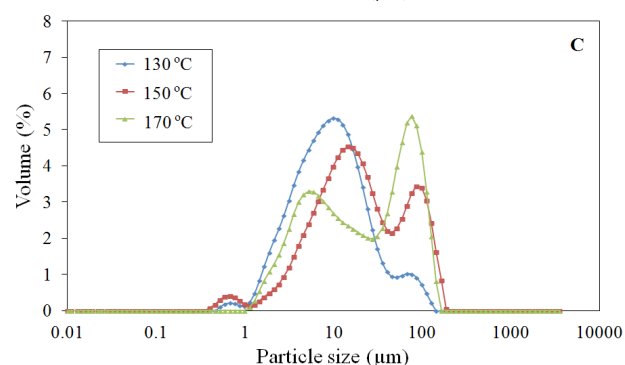
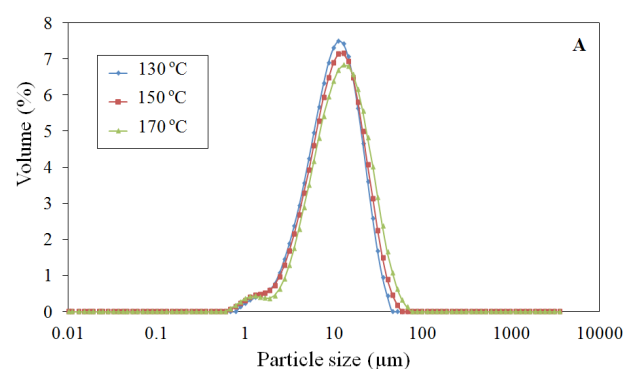


Figure 1 Particle size distribution of powders produced with different wall materials: maltodextrin (A), gum arabic (B) and β -cyclodextrin (C).



Microstructure

Figure 2 shows the SEM microstructures (internal and external) of powder produced with different wall materials (maltodextrin, gum arabic and β -cyclodextrin).

Observing the external morphology particles showed a spherical shape and various size with no apparent cracks or fissures, which is an advantage, since it implies that powders have lower permeability to gases, increasing protection and retention of the active material. Moreover, the variety in size is a typical characteristic of particles produced by spray drying.

Analyzing the internal morphology, all microspheres were hollow and the active material was adhered to the surface as small droplets embedded in the wall materials matrix. It is another characteristic of particles obtained by spray drying. This emptiness is a result of the quick particles expansion during the final stages of drying (Jafari *et al.*, 2008).

From ANOVA (Table 2) the wall material affected significantly moisture content, total phenolic content, encapsulation yield, encapsulation

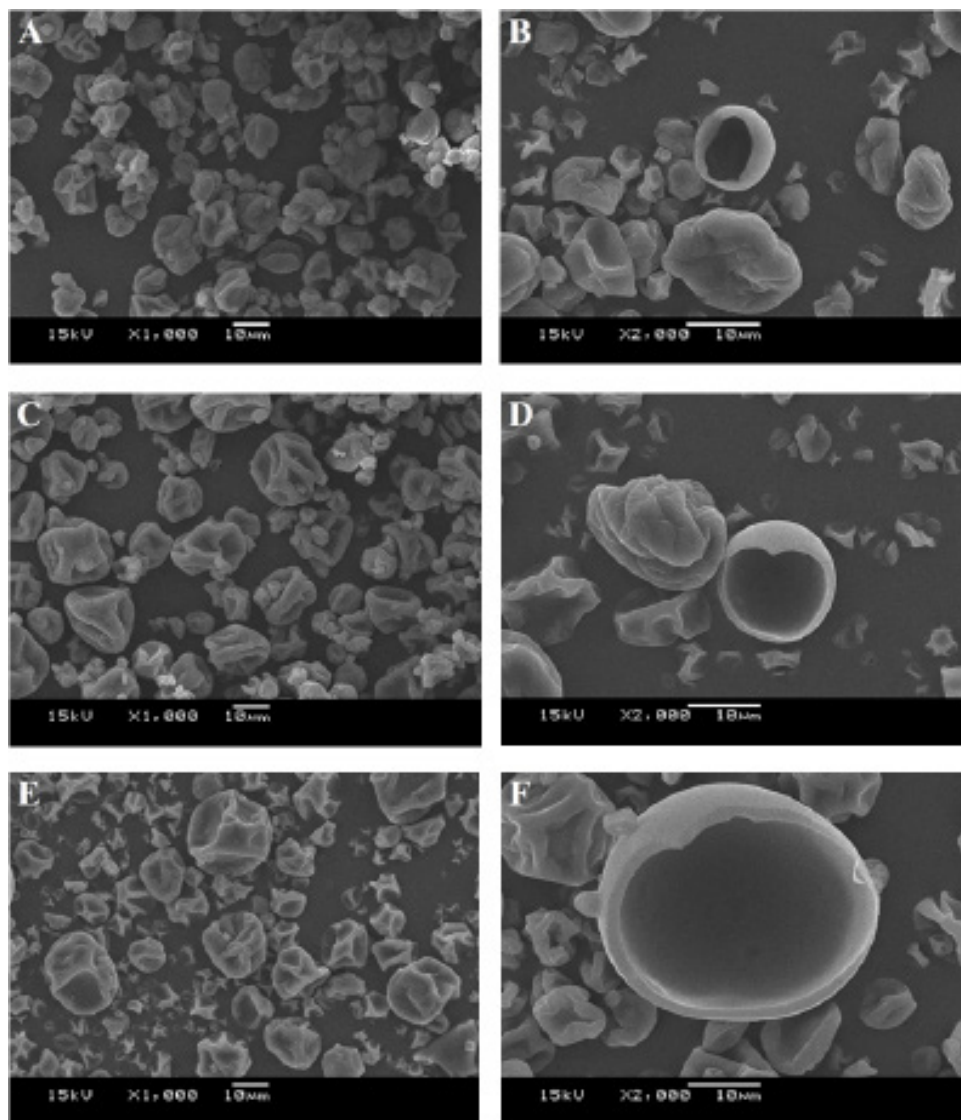


Figure 2 Microstructure of powders produced with different wall materials (maltodextrin (A,B), gum arabic (C,D) and β -cyclodextrin (E,F)) and inlet temperatures of 170 °C.

Table 2 Statistical analysis of effects of encapsulation parameters on properties of powder

Parameters	P values					
	MC	Total phenolic content	%EE	%EY	Hygroscopicity	Size
Wall material (W)	0.000	0.001	0.005	0.001	0.000	0.000
Inlet temperature (T)	0.000	0.061	0.440	0.061	0.001	0.000
W x T	0.366	0.511	0.837	0.511	0.079	0.000

efficiency, hygroscopicity and size of encapsulated powder ($P<0.05$). The inlet drying temperature influenced significantly the moisture content, hygroscopicity and size of encapsulated powder ($P<0.05$).

The interaction of the wall material and inlet drying temperature affected significantly the size of encapsulated powder.

CONCLUSION

The effects of different encapsulation parameters on the properties of encapsulated powder were studied. The wall material and inlet temperature affected the moisture content, total phenolic content, encapsulation yield, encapsulation efficiency hygroscopicity and size of encapsulated powder. Gum arabic as the wall material gave the highest encapsulation efficiency, low moisture content, low hygroscopicity and homogeneous particle size distribution of powder. Higher temperature gave lower moisture content powder. So the suitable conditions for encapsulation were using gum arabic as the wall material and an inlet temperature of 170 °C.

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