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Stability and encapsulation efficiency of sulforaphane microencapsulated by spray drying

Yuanfeng Wu[a,b,*], Ligen Zou[c], Jianwei Mao[a,b,*], Jun Huang[a,b], Shiwang Liu[a,b]

[a] School of Biological and Chemical Engineering, Zhejiang University of Science and Technology, Hangzhou 310023, Zhejiang, China
[b] Zhejiang Provincial Key Lab for Chem & Bio Processing Technology of Farm Produces, Hangzhou 310023, Zhejiang, China
[c] Hangzhou Academy of Agricultural Sciences, Hanzhou 310024, China

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ABSTRACT

Sulforaphane (SF) has received much attention because of its anticarcinogenic, antioxidant and anti-inflammatory properties, but it is quite unstable. Microencapsulation is one way to improve its stability. The aim of this work was to produce microcapsules containing sulforaphane using a spray drying technique. The effects of different wall materials, inlet air temperature and core to wall ratio on the SF stability, encapsulation efficiency, encapsulation yield, moisture content and SF content were determined. The results indicated that optimal encapsulation conditions for SF were: maltodextrin for the wall material, 170 °C for the inlet air temperature and 1:20 for the core/wall ratio. Characterization study showed that the microcapsules had a regular spherical shape. The stability of SF in spray dried microcapsules was greatly enhanced compared with that of free SF.

1. Introduction

Sulforaphane (4-methylsulfinybutyl isothiocyanate, SF), derived from glucoraphanin (4-methylsulfinybutyl glucosinolate), has received much attention over the last 20 years due to its anticarcinogenic (Dashwood & Ho, 2007; Johnson, 2002; Liang & Yuan, 2012), antioxidant (Gaona-Gaona et al., 2011; Guerrero-Beltrán, Calderón-Oliver, Pedraza-Chaverri, & Chirino, 2012), and anti-inflammatory properties (Heiss, Herhaus, Klimo, Bartsch, & Gerhäuser, 2001; Mirmiran, Bahadoran, Hosseinpanah, Keyzad, & Azizi, 2012). SF has thus been identified as a promising chemo-preventive agent, illustrating the potential clinical usefulness of diet-derived substances.

Even though SF shows great promise as an anticancer agent, the compound is susceptible to oxidants, light and heat, resulting in deterioration upon exposure to such factors (Wu, Liang, Yuan, Wang, & Yan, 2010). Free SF therefore must be protected from chemical damage prior to its industrial application. Microencapsulation is one way to improve its stability.

Microencapsulation is a technique by which small droplets of liquid or solid particles are coated with a thin film of protective biomaterial (Sheu & Rosenberg, 1995). The microcapsule is designed to protect the encapsulated material from factors such as oxygen, moisture, light, etc., which may cause its deterioration (Rosenberg, Kopelman, & Talmon, 1985). Microencapsulation has become an attractive approach to transform liquid food flavorings into stable and free-flowing powders, which are easy to handle and incorporate into dry food systems (Bhandari, Dumoulin, Richard, Noleau, & Lebert, 1992). Various encapsulation methods have been reported (Bhandari et al., 1992; Sheu & Rosenberg, 1995). Among them, spray drying is the most popular method of forming microparticles because it is easy to industrialize and allows for continuous production (Su et al., 2008). In addition, the operation costs of spray drying are 30–50 times cheaper than freeze-drying (Desobry, Netto, & Labuza, 1997).

The stability of the core material is influenced to a great extent by the wall materials used (Anwar & Kunz, 2011). Depending on the core material and the characteristics desired in the final product, wall materials can be selected from a wide variety of natural and synthetic polymers. Since almost all spray drying processes in the food industry are carried out from aqueous feed formulation, the wall material must be soluble in water at an acceptable level (Gouin, 2004). The microencapsulation of food ingredients is often achieved using biopolymers such as maltodextrin (MD), gum arabic (GA), κ-carrageenan (CG), β-cyclodextrin (CD), etc. (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). MD is a hydrolyzed

* Corresponding author at: Zhejiang Provincial Key Lab for Chem & Bio Processing Technology of Farm Produces, Hangzhou 310023, Zhejiang, China.
Tel.: +86 571 85070375; fax: +86 571 85070378.
E-mail addresses: wyfhz@126.com (Y. Wu), jgszlg@163.com (L. Zou), zjhzmjw@163.com (J. Mao), hjunlzr@163.com (J. Huang), shiwangliu@163.com (S. Liu).

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starch commonly used as a wall material in microencapsulation of food ingredients. It offers advantages such as relatively low cost, neutral aroma and taste, high water solubility (up to 75%), low viscosity at high solid concentrations, and it provides good protection against oxidation (Turchiuli et al., 2005). In addition, MD possesses multitude of other functions including bulking and film formation, binding of flavor and fat compounds, and reduction of oxygen permeability of wall matrix (Sansone et al., 2011). GA, another common wall material, has been used to encapsulate lipids because of its efficient emulsifying properties (Kenyon, 1995). In the microencapsulation by spray drying, CG is also favored as wall material because of its pseudo plastic properties (Krishnaiah, Sarbatly, & Nithyanandam, 2012).

The microencapsulation of SF by spray drying has previously been investigated by Do, Pai, Rizvi, and D'Souza (2010). In their work, SF was spray dried using bovine serum albumin cross-linked chemically by glutaraldehyde as the wall material. The resulting microparticles were spherical in shape, with most of the microspheres about 2 μm in diameter. The microcapsule is a potential delivery system and can attain an effective therapeutic effect (Do et al., 2010). To increase the stability of SF, the inclusion complex of SF with hydroxypropyl-β-cyclodextrin was also prepared (Wu et al., 2010; Wu, Mao, Mei, & Liu, 2013). To the best of our knowledge, there are no other available publications on the stabilization of SF.

The objective of the present work was to evaluate the effect of different wall materials, inlet temperatures and core/wall ratios on the spray drying of SF and also to find the best spray drying process parameters to create a powdered SF. The microcapsules were characterized for SF stability, encapsulation efficiency (EE), moisture content, SF content, encapsulation yield (EY), FTIR and morphology.

2. Materials and methods

2.1. Chemicals

Broccoli seed was purchased from Taizhou Academy of Agricultural Science. Maltodextrin, gum arabic, κ-carrageenan and β-cyclodextrin were purchased from Sangon Biotech Co., Ltd. (Shanghai, China). Methanol (Tedia, USA) was HPLC grade. Distilled water was used throughout the entire study. SF (purity >98%) was purchased from Toronto Research Chemicals Inc. (Toronto, Canada) and was stored below 0 °C. And all other reagents were of analytical reagent grade and were purchased from Huadong Medicine Co., Ltd. (Hangzhou, China).

2.2. Extract methods

50 g of broccoli seeds were ground in a Chinese herbal medicine grinder to produce seed meal, the seed meal was subsequently de-fatted with 400 ml hexane in an incubator shaker for 3 h, after which the residual seed meal was allowed to dry in a fume hood. De-fatted seed meal was mixed with 150 ml potassium phosphate buffer (0.05 mol/L, pH 5.8) and 200 ml ethyl acetate, the resulting mixture was agitated for 4 h and then 20 g sodium chloride were added and mixed thoroughly. The ethyl acetate layer was filtered and the residual paste was extracted two times with equal volumes of ethyl acetate, which was combined and dried in a vacuum rotary evaporator. The residue was then dissolved by deionized water.

2.3. Spray drying of SF

Wall materials were dissolved in distilled water to form an aqueous solution, and then SF sample was dripped into the aqueous solution with stirring. A SD-06 spray dryer (LabPlant, United Kingdom) was used to convert the solution into encapsulated powder. The inlet temperature was set at 150–230 °C and outlet temperature was 85 °C. Each jar was changed every 10 min to avoid prolonged exposure to heat, and the jar was immediately immersed in an ice-water bath.

2.4. Characterization of SF microcapsules

2.4.1. Encapsulation yield (EY)

The encapsulation yield was calculated as the ratio of the mass of the microcapsules obtained at the end of the process to the mass of the initial substances added including SF and wall material (Su et al., 2008).

2.4.2. Encapsulation efficiency (EE)

The encapsulation efficiency was calculated as the ratio between the mass of SF in the final product and its initial mass to be encapsulated. About 500 mg exactly weighed microcapsule sample was dissolved in distilled water to form homogeneous solution. Then SF was extracted two times with equal volumes of dichloromethane, which was combined and dried in a vacuum rotary evaporator. The residue was dissolved by methanol and filtered through a 0.45 μm membrane. The total SF in the solution was determined by HPLC.

2.4.3. Scanning electron microscopy

Microcapsule powders were deposited on double-coated carbon conductive tape previously adhered to SEM aluminum stubs. The microcapsules samples were then sputter coated with a thin gold layer, and analyzed in a HITACHI S-4700 operated at 15 kV. The inner structure was studied embedding the capsules in resin. After dried, the blocks were fractured with a razor blade and the fragments obtained were deposited on SEM stubs.

2.4.4. Fourier transform infrared spectroscopy (FTIR)

The molecular structure changes in maltodextrin, pure SF and spray dried microparticle powder were characterized by FTIR analysis. FTIR spectra were obtained with a VERTEX 70 FTIR spectrometer (Bruker) using a transformation of 20 scans with a spectral resolution of 4 cm^{-1}. FTIR spectra were collected in the mid-infrared region between 4000 and 500 cm^{-1}. Spectra were acquired using an air-background correction, and the same spectra were also obtained using the software.

2.4.5. Stability of SF in microcapsules

The microcapsules were placed in a plastic tube (3 ml total volume, Thermo Fisher Scientific Inc., USA) and was covered with aluminum foil. The well capped tubes were placed in a thermostatic water bath (Beijing Era Beili Centrifuge Co. Ltd., China). The heating temperature was 90 °C. After 5 h, the tube was taken from the water bath and rapidly cooled by plunging into an ice water bath. The retention percentage, which was defined as the ratio between the content of SF that retained in the microcapsule after 5 h and the original content of SF in the microcapsule, was used to evaluate the stability of SF in microcapsules.

2.4.6. Storage stability of SF microcapsules

The samples of each wall materials were placed in plastic tubes and were stored at temperatures of 35 °C. Quantification was conducted for 28 days, every 7 days a tube of each sample was opened, the retention percentage was used to evaluate the storage stability of SF microcapsules.

Table 1
The moisture content, SF content, EY and EE of the microcapsules with different wall materials.

No.	Microcapsules	Moisture content (%)	SF content (%)	EY (%)	EE (%)	Retention percentage (%)[a]
1	MD	5.3 ± 0.3	3.4 ± 0.2	58.1 ± 0.8	39.1 ± 2.6	76.6 ± 3.9
2	GA	3.4 ± 0.1	3.0 ± 0.1	69.2 ± 1.4	39.8 ± 1.5	43.3 ± 1.6
3	CG	2.2 ± 0.06	2.0 ± 0.1	34.3 ± 3.2	12.6 ± 0.6	42.2 ± 2.0
4	MD/GA (25:75)	2.4 ± 0.2	2.8 ± 0.02	63.2 ± 3.9	34.0 ± 3.5	62.2 ± 1.4
5	GA/β-CD (2:5)	1.2 ± 0.1	3.1 ± 0.04	51.8 ± 4.6	29.4 ± 2.9	69.7 ± 2.8

[a] The retention percentage of SF was determined after heating at 90 °C for 5 h.

2.5. HPLC

The content of SF was analyzed on a Waters e2695 HPLC system by the method of Wu et al. (2013). The column employed in our experiment was a ZORBAX Eclipse XDB-C_{18} (4.6 mm × 250 mm, 5 μm). The mobile phase consisted of 20% methanol in water, changing linearly over 10 min to 60% methanol, then raising to 100% in 2 min and maintained 2 min to purge the column. Column oven temperature was set at 25 °C, the flow rate was 1.0 ml/min, and 10 μl samples were injected into the column. SF was detected by Waters 2489 detector at the wavelength of 241 nm.

2.6. Statistical analysis

All experiments were done in triplicates, the results were expressed as mean values. The errors of experimental data from the mean values were expressed as standard deviation and illustrated as error bars.

3. Results and discussion

3.1. Effect of technological parameters on moisture content, SF content, EY, EE and SF stability

3.1.1. Effect of wall material

The microcapsules were spray dried with an inlet temperature of 190 °C and a core/wall ratio of 1:10. The five wall materials tested were: MD, GA, CG, MD/GA (25:75, M:M) and GA/β-CD (2:5, M:M). The moisture content, SF content, EY, EE and the retention percentage of SF in the microcapsules 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, Assadpoor, He, & Bhandari, 2008). According to Table 1, the encapsulation efficiency of samples was significantly influenced by the type of wall material used. The EE and EY values varied from 12.6% to 39.8% and 34.3% to 69.2%, respectively, among them CG recorded the lowest EE and EY values. The other materials, MD, GA, MD/GA and GA/β-CD, were deemed suitable for the encapsulation of SF as these materials showed relatively good EY, EE and SF content values. However, compared to other components, EE values reported here were relatively low. SF is easily decomposed when exposed to high temperature (Chiang, Pusateri, & Leitz, 1998) and it is believed that the inlet temperature used in the spray-drier (190 °C) was perhaps one of the reasons for the low efficiency observed.

The main purpose of this study was to improve the stability of SF. As shown in Table 1, SF stability was greatly influenced by the choice of wall material. The retention percentages of SF were significantly greater when encapsulated with MD and to a slightly lesser extent with GA/β-CD compared with other wall materials. Previous work by Kenyon (1995) showed that MD can provide good oxidative stability to encapsulated oil, and was also found to be effective in protecting the carotenoids of paprika oleoresin (Beatus, Raziel, Rosenberg, & Kopelman, 1985). Sansone et al. (2011) found that a two-sided maltodextrin/pectin matrix was effective in stabilizing a phenolic-rich extract, even under harsh storage conditions, and was able to preserve the antioxidant activity of the phenolic-rich extracts. In the study of Ferrari, Marconi Germer, Alvim, and de Aguirre (2013), spray dried blackberry particles produced with maltodextrin were found to have the longest half-life, followed by those produced with a blend of maltodextrin and gum arabic. In contrast, samples produced with gum arabic only had a shorter half-life. In a study evaluating the stability of spray dried Amaranthus bethacyanins at 25 °C for 16 weeks, Cai and Corke (2000) observed that the use of maltodextrin (20 or 25 DE) resulted in the formation of denser and more oxygen-impermeable wall systems, thus provided better storage stability for betacyanin pigments. The biggest problem of MD is its low emulsifying capacity (Carneiro, Tonon, Grosso, & Hubinger, 2013). SF, however, dissolves easily in water (Liang & Yuan, 2012), therefore the emulsifying capacity is not of concern in this study. MD was thus selected as the wall material for our subsequent work.

3.1.2. Effect of inlet temperature

Increasing the inlet temperature from 150 °C to 230 °C while keeping core/wall ratio of 1/10 and using MD as wall material, it was showed that EY decreased with increasing inlet temperature (Table 2). SF is easily decomposed when exposed directly to heat (Chiang et al., 1998). SF content, however, was found to be almost level off with increasing inlet temperature. When the inlet temperature increased from 150 °C to 210 °C, the SF contents were varied from 3.3% to 3.5%, and remained almost unchanged. From this we inferred that the process of spray drying was so fast that the inlet temperature did not influence the SF content.

The moisture content of the spray dried powder decreased with increasing inlet air temperature (Table 2). 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, Adamopoulos, & Kazakis, 2004; Quek, Chok, & Swedlund, 2007).

The microcapsules prepared at inlet temperatures of 150 °C and 230 °C, showed similar SF stability even though the moisture content varied from 7.8% to 4.1%, respectively. The microcapsules prepared at inlet temperatures of 170 °C and 190 °C, however, showed a moderate moisture content, but they showed better SF stability than the other microcapsules. This study suggests that SF stability is not influenced by moisture content of the microcapsules. This finding is in agreement with Anwar and Kunz (2011), who state that higher water content and a_w in a sample do not necessarily correlate with better fish oil stability against oxidation. Bell, Bell, and Glass (2002) also emphasized that the effect of water on food chemical stability in solids is multidimensional and depend on the type of reactions and the physical characteristic of the system, and no single explanation that currently exist on how water affects chemical reactions in reduced-moisture solids.

As shown in Table 2, the highest retention percentage was reached when the inlet temperature was 170 °C and 190 °C. An inlet temperature of 150 °C, however, resulted in the lowest retention

Table 2
The moisture content, SF content, EY and EE of the microcapsules obtained at different inlet temperatures.

No.	Inlet temperature (°C)	Moisture content (%)	SF content (%)	EY (%)	EE (%)	Retention percentage (%)[a]
1	150	7.8 ± 0.5	3.3 ± 0.3	70.3 ± 2.5	44.5 ± 1.5	51.5 ± 5.1
2	170	5.5 ± 0.1	3.4 ± 0.1	60.1 ± 0.5	38.7 ± 0.8	75.0 ± 1.3
3	190	5.5 ± 0.02	3.5 ± 0.08	59.4 ± 1.2	39.4 ± 2.4	75.5 ± 1.4
4	210	4.9 ± 0.5	3.5 ± 0.2	51.0 ± 3.1	34.3 ± 1.5	63.5 ± 2.4
5	230	4.1 ± 0.1	3.4 ± 0.1	42.2 ± 2.7	27.1 ± 1.8	57.4 ± 0.4

[a] The retention percentage of SF was determined after heating at 90 °C for 5 h.

percentage (51.5%), a value much lower than that of the other temperatures. Low and high inlet temperatures may break the balance between the water evaporation rate and particle formation rate, thus leading to a breakdown in the microcapsule wall system (Shu, Yu, Zhao, & Liu, 2006). We deduced that the stability of SF decreased at both low and high inlet temperatures because of this. Zakarian and King (1982) reported that a high air inlet temperature causes an excessive evaporation and results in cracks in the membrane that induces subsequent premature release and degradation of the encapsulated ingredients. We propose therefore that 170 °C is the most suitable inlet temperature for production of microcapsules.

3.1.3. Effect of core/wall ratio

The microcapsules were spray dried at 170 °C and using MD as wall material. When the core/wall ratio decreased from 1/5 to 1/25, the SF content decreased from 4.5% to 1.3%. EY and EE were approximately 58.3–62.2% and 38.3–39.5%, respectively, remained nearly unchanged (Table 3). However, when the core/wall ratio decreased from 1/5 to 1/20, the stability of SF increased, that is, the retention percentage of SF after heating at 90 °C for 5 h increased from 70.6% to 90.9%, and then proceeded to level off when the core/wall ratio decreased to 1/25 (Table 3). Working with microencapsulation of lycopene with maltodextrin by spray drying, Goula and Adamopoulos (2012) reported that for the higher core material concentration, the thinner shell matrix and higher lycopene concentration near the outer surface of the shell led to greater lycopene loss. We deduced that because of the thicker shell matrix and the lower concentration of SF in the microcapsules, it is more difficult for SF to react with the outside oxygen. Thus 1/20 was the most suitable core/wall ratio for the production of the microcapsules.

3.2. Characterization of SF microcapsules

3.2.1. Appearance

Electron micrographs of spray dried SF microcapsules with different wall materials are illustrated in Fig. 1. The microcapsules was found to have a mean diameter of 2–4 μm. Most of the particles showed little agglomeration, among which the microcapsules containing MD had a rounded outer surface with the formation of a few teeth (Fig. 1). Other particles presented rough surfaces or invaginations (figures not shown). The appearance of teeth or invaginations on the surface is attributed to rapid evaporation of drops of liquid during the drying process in the atomizer (Rosenberg et al., 1985). Bruschi, Cardoso, Lucchesi, and Gremião (2003) reported that the presence of MD that react as plasticizers is important for promoting the formation of spherical and smooth-surfaced microparticles. Similar smooth spheres have been observed in microcapsules of black carrot pigments (*Daucuscarota* L.) with MD (Ersus & Yurdagel, 2007). Microcapsules containing MD as a wall material were also found to present a homogenous distribution of particle diameters (Fig. 2). Microcapsules with smooth surfaces and of homogenous size distributions are considered optimal in the food industrial uses because of their excellent flow properties (Teixeira, Andrade, Farina, & Rocha-Leão, 2004).

Although a small number of microcapsules showed a small amount of cracking, the majority of the external surfaces of spray dried SF microcapsules containing MD showed continuous walls with no fissures or interruptions (Fig. 3a), an attribute that is essential to ensure low gas permeability and better SF protection.

Fig. 3b shows a fractured MD microcapsule, where its internal structure is visible. As SF can dissolve easily in water, we deduced that SF is homogeneously distributed throughout the wall material. The formation of internal voids in microcapsules may be associated with the evaporation of water from the core of microcapsules during spray drying.

3.2.2. FTIR analysis

FTIR analysis provides information about chemical bonds and the molecular structure of materials. The FTIR spectra of SF, spray dried MD and SF microcapsules (using MD) were recorded. The FTIR

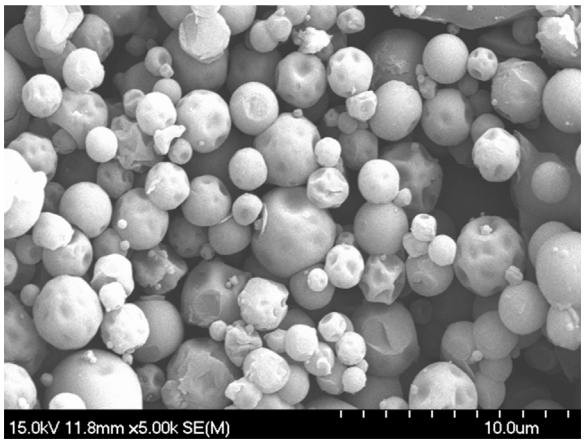

Fig. 1. SEM of spray dried powders of SF microcapsules using maltodextrin as the wall material.

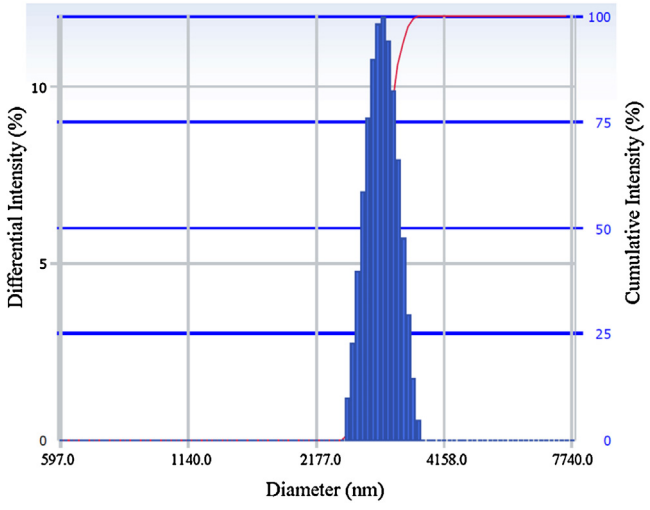

Fig. 2. Particle size distribution curve of spray dried powder. Core/wall ratio was 1:20, inlet temperature was 170 °C and using MD as wall material.

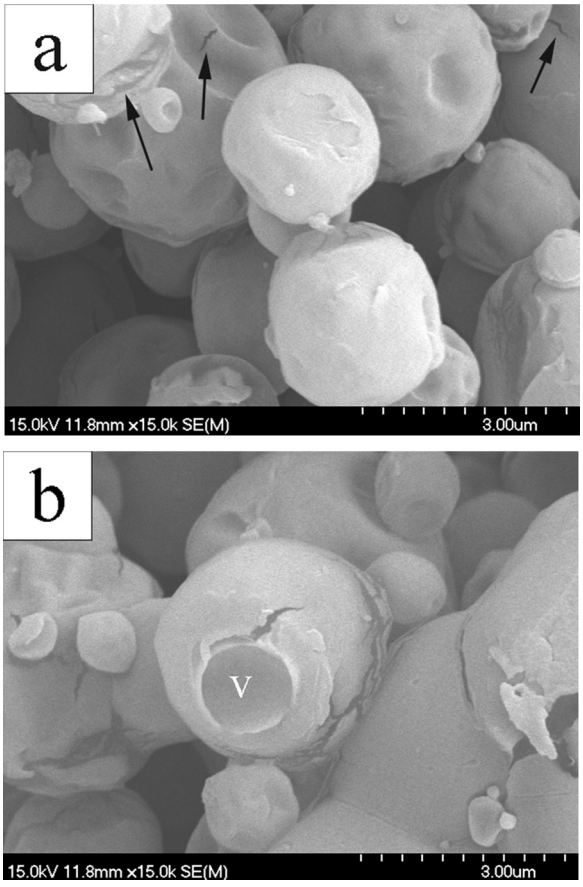

Fig. 3. SEM of external surfaces of spray dried SF microcapsules with maltodextrin (a), a few cracks (arrows) can be seen. (b) SEM images of fractured microcapsules of SF sample embedded in maltodextrin showing the inner structure, voids (V) can be seen.

spectra of all of the above were recorded and compared and are shown in Fig. 4.

The spectrum of spray dried MD shows the band at 3420, 2928, 1637, 1413, 1370, 1151, and 1027 cm^{-1}. The characteristic peak at 3420 cm^{-1} due to O—H stretching; 2928 due to sp^3 C—H stretching; 1637 assigned to C=O stretching and 1413 due to $-CH_2$ bending; 1370 due to O—H bending; 1151 and 1027 due to C—O stretching.

The spectrum of SF shows the band at 2181, 2107, 1452, 1350, 1252 and 1025 cm^{-1}. Strong absorbance at 2181 and 2107 cm^{-1} were due to the —N=C=S stretching; the absorbance peak at 1025 and 1252 were due to the S=O, C—N bond; and 1452 and 1350 cm^{-1} were noted for deforamation vibration of C—H from CH_3. Our observations were similar with those similar works for the purified SF (Abassi, Abassi, Yari, Hashemi, & Nafisi, 2013; Wu et al., 2010; Zhang, Talalay, Cho, & Posner, 1992).

The spectrum of spray dried SF microcapsules (1/20 at 170 °C, using MD as wall material) shows the band at 3418, 2927, 2184, 2110, 1635, 1410, 1365, 1152 and 1028 cm^{-1}. The characteristic peak at 3418 cm^{-1} due to O—H stretching; 2110 due to —N=C=S stretching and 1028 assigned to C—O stretching. All the other peaks appeared in SF and MD also appeared in microencapsulated powder, and those characteristic peaks did not show any shifts, indicating that no molecular structure change formed between the SF, the spray dried MD and SF microcapsules. This shows that the SF was retained after spray drying using MD as wall material.

3.3. Storage stability

To study the storage stability of the microcapsules, samples produced with the five different wall materials were stored at 35 °C in plastic tubes. The degradation kinetics of most biological materials follows the first-order reaction:

$$\ln\left(\frac{C}{C_0}\right) = -kt \quad (1)$$

where C is the concentration of SF at time t, C_0 is the initial concentration of SF, and k is the reaction rate constant. The plots of ln C against time were shown in Fig. 5. The distribution of the points shows the plots are approximately linear (R^2 = 0.9092–0.9884), confirming that SF degradation in microcapsules showed a first-order reaction. This observation was similar to that obtained by other researchers who studied the kinetics of SF degradation in solution (Van Eylen, Oey, Hendrickx, & Van Loey, 2007; Wu et al., 2013).

The k values in Fig. 5 showed that for all treatments, the greatest losses were observed for free SF, indicating that the microencapsulated system offers greater protection to the SF. The reason may be the effective microencapsulation of SF within the wall materials, which can effectively avoid the damage from oxygen, light and other influences during storage (Shu et al., 2006). As mentioned

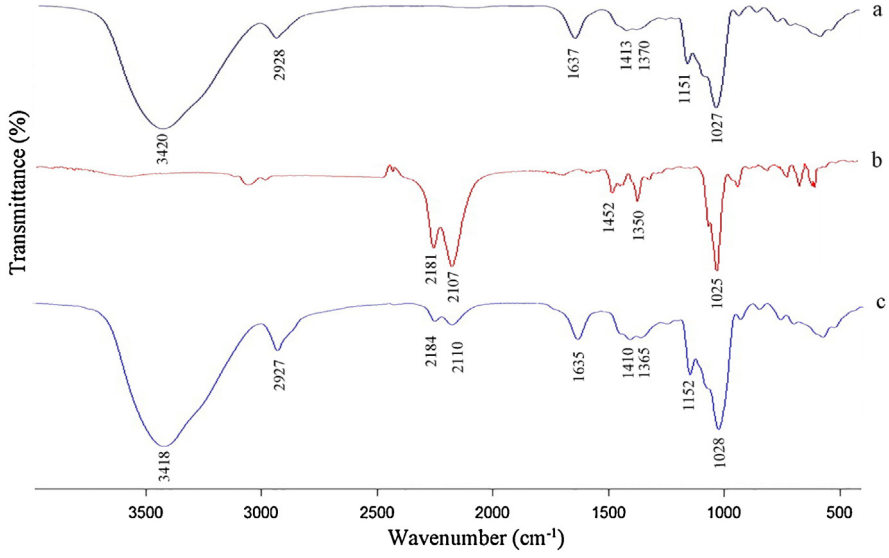

Fig. 4. The FTIR spectra of MD (a), SF (b) and 1:20 at 170 °C spray dried powder using MD as wall material (c).

Table 3
The moisture content, SF content, EY and EE of the microcapsules obtained at different core/wall ratios.

No.	Core/wall ratio	Moisture content (%)	SF content (%)	EY (%)	EE (%)	Retention percentage (%)[a]
1	1/5	5.5 ± 0.2	4.5 ± 0.2	60.4 ± 0.2	38.3 ± 0.6	70.6 ± 8.3
2	1/10	5.3 ± 0.8	3.2 ± 0.6	58.3 ± 1.5	37.3 ± 1.1	76.8 ± 2.1
3	1/15	4.9 ± 0.2	2.2 ± 0.2	61.3 ± 2.1	37.5 ± 1.8	84.4 ± 3.6
4	1/20	4.9 ± 0.6	1.5 ± 0.05	62.2 ± 0.4	37.3 ± 0.2	90.9 ± 1.2
5	1/25	5.2 ± 0.02	1.3 ± 0.01	62.1 ± 1.6	39.5 ± 2.0	88.9 ± 2.8

[a] The retention percentage of SF was determined after heating at 90 °C for 5 h.

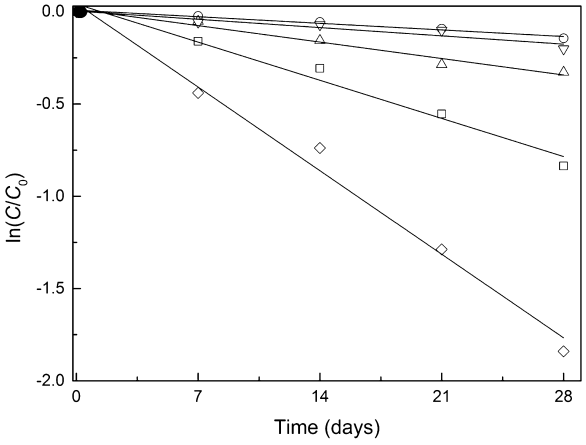

Fig. 5. SF degradation kinetics of spray dried microcapsules produced using different carrier agents: Free SF (◊): ($-0.453x + 0.044$; $R^2 = 0.9884$); GA (□): ($-0.207x + 0.042$; $R^2 = 0.9790$); MD + GA (△): ($-0.089x + 0.0141$; $R^2 = 0.9744$); GA + CD (▽): ($-0.0448x + 0.004$; $R^2 = 0.9092$); and MD (○): ($-0.036x + 0.008$; $R^2 = 0.9795$).

previously, SF stability was greatly influenced by the type of wall materials. As shown in Fig. 5, the retention percentage of SF was significantly greater when microencapsulated with MD and to a slightly lesser extent with GA/β-CD compared with other wall materials.

4. Conclusions

SF microcapsules were successfully prepared using a spray drying method. The type of wall material, the inlet temperature and the core/wall material ratio significantly affected the stability of SF. The optimal conditions for microencapsulation were determined to be MD as the wall material, an inlet temperature of 170 °C and a core/wall ratio of 1:20. Under these conditions, the microencapsulated SF showed good encapsulation yield, encapsulation efficiency and SF stability.

SEM analysis showed the microcapsules had a regular spherical shape with a smooth outer surface. Voids were found in the internal structure of microcapsules, and we deduced that SF was homogeneously distributed throughout the wall material. FTIR analysis showed that the SF was retained after spray drying.

Storage stability analyses were also carried out. The degradation of SF in microcapsules of different wall materials showed a first-order reaction. Compared with free SF, the retention percentage of SF was significantly greater when microencapsulated by MD. This study would be helpful to promote the application of SF.

Acknowledgments

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