Preparation and Characterization of Emulsion-based Peony Seed Oil Microcapsule

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Abstract: Microcapsules were constructed with starch sodium octenyl succinate (SSOS), β -cyclodextrin (β -CD), and pectin walls and peony seed oil cores. A rheological phenomenon occurred in which the emulsion initially behaved like a shear-thickening fluid and then a shear-thinning fluid within a shear range. The emulsion exhibited good stability under low amplitude stress; however, as amplitude increased the concentration of pectin played an important role in maintaining the stability of the emulsion system. The optimum embedding yield of peony seed oil (92.5%) was achieved with a ratio of 70% SSOS, 22.5% β -CD, and 7.5% pectin. This ratio produced 4.521 μ m particles with the lowest surface-oil content (2.60%) and moisture content (1.76%). The peony seed oil microcapsules were spherical with smooth surfaces and a synchronous thermogravimetric analysis showed they possessed good thermal stability. Encapsulation increased the induction period to 5-7 times that of unencapsulated peony seed oil.

Key words: embedding rate, β -cyclodextrin, rheology, pectin, emulsion stability, shear rate

1 Introduction

The peony is a flowering plant of genus *Paeonia* with many general and medical uses. The peony is native to Europe, Western North America, and Asia, including China and can have a seed oil content of 40%^{1,2}. Peony seed oil has many potential applications, including as an agent to reduce the risk of eye disease and eye fatigue. This potential is due to its high percentage (>90%) of unsaturated fatty acids (UFAs) of which 45% are α -linolenic acids (ALAs)³. ALA can be converted to eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) in humans and is used in the growth and development of brain cells. ALA and its derivatives suppress thromboxane, which is implicated in cardiovascular diseases. Due to a wide range of potentially beneficial applications, peony seed oil was approved as a new food resource by the State Administration for Market Regulation, formerly the China State Food and Drug Administration, in 2011⁴. Although high in UFAs, peony seed oil is low in natural antioxidants, which makes it highly susceptible to oxidation leading to a reduction in nutrients and flavour and the production of free radicals which may negatively impact health⁵. Therefore, an effective method of preventing oxidation is necessary⁶⁻⁸.

Microencapsulation is commonly used to protect functional substances from the external environment. The ma-

terials used for the walls of the microcapsules affect encapsulation efficiency, physicochemical properties, and the storage stability of the encapsulated product^{9,13}. Soy lecithin is a good emulsifier and can improve microcapsule morphology¹⁰. Microencapsulation of ALA-containing linseed oil using whey protein concentrate and maltodextrin as wall materials achieved embedding rates of 90%¹¹. Walnut oil microcapsules constructed from soybean protein isolate and maltodextrin significantly improved the oxidation stability of walnut oil, although microencapsulation efficiency was less than that of linseed oil¹². Selection of appropriate wall materials is important for controlling the microencapsulation efficiency, physicochemical properties, and the storage stability of microencapsulated powders. Starch sodium octenyl succinate (SSOS) possesses hydrophilic and lipophilic properties and is used in various food and cosmetic products as a stabilizer for oil-in-water emulsions. SSOS possess good emulsion stability, however, does not adequately protect the core material. This problem is overcome by combining SSOS and other substances. SSOS has been combined with other substances to successfully embed blackcurrant oil and lemon oil in a microcapsule core^{14,15}. As a dextrin molecule having a hydrophilic appearance and a hydrophobic internal structure, β -cyclodextrin can form an inclusion complex with a guest molecule, re-

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sulting in a change in the physicochemical properties of the guest, which is widely used in the pharmaceutical field to improve the bioavailability of guest molecules^{16, 17}. The precursor of the microcapsule powder is a microcapsule emulsion. Emulsion stability is an important criterion for the selection of microcapsule wall materials. The properties of the microcapsule emulsion are affected by both the external environment (pH, ion concentration, shearing, etc.) and intrinsic factors (wall material, core material, emulsifier, etc.). Besides, different metal ions had significant effects on the stability of O/W emulsions¹⁸. Pectin, a complex polysaccharide rich in galacturonic acid, possesses emulsifying and emulsion-stabilizing properties which makes it ideal for use as a microcapsule wall material^{19–21}. The techniques used for microencapsulation, including spray-drying, also influence the efficiency of microencapsulation by contributing to variations in size, viscosity, and the rheological properties of the emulsion.

Many high-performance oils/fats are enhanced, including extensions of shelf-life, through encapsulation. However, studies on the preparation of peony seed oil microcapsules are lacking. We constructed microcapsules using SSOS, β -cyclodextrin (β -CD), and pectin as wall materials to embed a peony seed oil core and determined the ideal ratio of β -CD and pectin required to produce the highest yield of peony seed oil.

2 Materials and Methods

2.1 Preparation of the oil-in-water emulsion

In this study, the emulsion system was divided into two parts, deionized water and solids, and deionized water as a solvent throughout the emulsion system (65% w/w). The solid content is 35%. Equal amounts of soy lecithin (Aladdin Reagent Company, Shanghai, China) and polyglycerol esters of fatty acids (PEFAs) (2% of the solid content w/w) (Huibo Food Additive Co., Ltd., Shanghai, China) were dissolved in deionized water. This represented the aqueous phase of the emulsion system. SSOS (Deqing Sanfu Food Co., Ltd., Zhejiang, China), β -CD (Henan Hua Yue Chemical Products Co., Ltd., Henan, China), and pectin (Wanlida Biotechnology Co., Ltd.) were added to the aqueous phase and stabilized in a 55°C water bath (HH-4, Wincom Company Ltd., Shanghai, China) for 1 h. Four different ratios of SSOS, β -CD, and pectin represented the four test groups: A (70% SSOS, 30% β -CD, 0% pectin), B (70% SSOS, 25% β -CD, 5% pectin), C (70% SSOS, 22.5% β -CD, 7.5% pectin), and D (70% SSOS, 20% β -CD, 10% pectin) (Table 1). Peony seed oil (35% of the solid content) (Yaoshun Peony Biological Technology Co., Ltd., Heze, Shandong, China) was added and the mixtures were vigorously stirred at 60°C for 10 min and then emulsified in a high-shear emulsifier (FA-25, FLUKO, China) at 13,000

Table 1 Different proportions of wall materials.

	A	B	C	D
SSOS	70%	70%	70%	70%
β -CD	30%	25%	22.5%	20%
pectin	–	5%	7.5%	10%

rpm for 3 min, followed by high-pressure homogenization at 40 MPa and twice the number of homogenizations. All chemicals used were of reagent grade and all solutions were prepared using distilled water.

2.2 Morphology and particle size of the emulsion

The particle size of the emulsion was analysed by laser particle size analyser (MS-2000, Malvern, UK) with a laser intensity of $80 \pm 0.5\%$ and laser opacity of $12.5 \pm 0.5\%$. A sample of the emulsion was uniformly drawn into deionized water at a speed of 2000 rpm for uniform dispersion. The size of the emulsion droplets is recorded by the volume average diameter of the droplets ($d_{4,3} = \sum n_i d_i^4 / \sum n_i d_i^3$), where n_i is the number of particles with diameter d_i ²².

The morphology of the emulsion droplets produced by each test group was observed using an optical microscope (Fluorescence microscope 80i, Japan) and images were produced.

2.3 Rheological measurements

Samples were measured using a rheometer (DHR-3, Waters Instruments, USA) with a cone plate radius of 20 mm and 1000 mm gap height. The samples were subjected to strain sweep testing at a temperature of 25°C, frequency of 1 Hz, and strain amplitudes ranging from 0.1% to 100%. The measurements of the emulsion viscosity were performed at a shear rate of 1 s^{-1} to 100 s^{-1} .

2.4 Microencapsulation by spray-drying

The emulsion was spray-dried using a small spray dryer (B-290, BUCHI, Switzerland). Inlet air temperatures were $160 \pm 5^\circ\text{C}$ and outlet air temperatures were $90 \pm 5^\circ\text{C}$. The feed pump rate was set at 15 rpm and the aspirator rate was 80%. The spray-dried microcapsule powders were removed from the cyclone and stored in a sealed bag at 4°C.

2.5 Determination of encapsulation efficiency

To determine the surface oil content a powder with a mass (m_0) of 4.0 g was weighed in a triangular flask and immersed in 40 mL of 30–60°C petroleum ether (Guangzhou Chemical Reagent Co. Ltd., Guangdong, China) and gently agitated for 1 min and immediately filtered with a G₃ core funnel. The filter residue was washed with 25 mL of petroleum ether for 40 s and filtered, and then the filtrate was transferred to a conical flask (m_1). The petroleum ether

was recovered and dried at 65°C to a constant weight (m_2) and surface oil content (SOC) was calculated:

$$SOC(\%) = \frac{m_2 - m_1}{m_0} \times 100 \quad (1)$$

Peony seed oil content was measured by accurately weighted 4.0 g microcapsules and extracted by soxhlet extraction^{23, 24)}. Ether was selected as the extractant and extraction time was 6 h. After extraction, the solvent was evaporated completely and weighted to obtain the total quantity of oil extracted by ether. Encapsulation efficiency (EE%) was calculated as follows:

$$EE\% = 1 - \frac{SOC}{total\ oil\ content} \times 100 \quad (2)$$

2.6 Determination of moisture content

Determination of water content of peony seed oil microcapsule powder by thermogravimetric weight loss method. The measurement of the moisture content was slightly modified by referring to the method of Sani *et al.*²⁵⁾.

2.7 Tungsten filament Scanning Electron Microscope

A sample of microcapsule powder was applied to conductive tape, sprayed with gold at a pressure of 9.0 Pa for 120 s, and observed using a tungsten wire scanning electron microscope (JSM-6490LV, Japan).

2.8 Fourier-transform infrared spectroscopy

The wall materials, peony seed oil, and microcapsule powder samples were analyzed by attenuated total reflectance infrared spectroscopy using a Fourier Transform Infrared (FT-IR) Spectrometer (Nicolet 67, Thermo Nicolet, USA). The FT-IR spectrum of the samples was measured with spectral ranges from 400 cm^{-1} to 4000 cm^{-1} .

2.9 Synchronous thermogravimetric analysis

Mass loss of the powder sample was observed after placing the sample in the sample pan of the thermogravimetric analyser (STA449F5, Germany) at temperatures between 25 to 700°C and a heating rate of 10°C/min. Testing was performed under a constant N_2 flow rate. Draw the thermogravimetric loss curve corresponding to different samples.

2.10 Induction period

Oxidative stability of the peony seed oil sample (5.00 g) and microcapsule sample (14.28 g) was measured using an OXITEST Oxidation Test Reactor (Velp Scientifica, Italy). Oxidative stability testing was performed under an oxygen pressure of 6 bars at 90°C. The induction period of the sample was calculated from the oxidation curve.

2.11 Statistical analysis

All experiments were repeated three times, and the presented data is calculated from the mean and standard deviation.

Fig. 1 Microstructure and particle size distribution of peony seed oil microcapsule emulsions.

ANOVA analysis was employed to analyze the significant difference at a significance level of 0.05.

3 Results and Discussion

3.1 Microstructure and particle size distribution of the emulsion

A photograph of a peony seed oil microcapsule is displayed in Fig. 1. Group A, without pectin, produced the largest average droplets, $d_{4,3} = 8.23 \mu\text{m}$, which were unevenly distributed throughout the emulsion and droplet size was bimodally distributed. Group D had the smallest droplet size, 3.65 μm . According to Stoke's law, the smaller the particle sizes of the droplets in the emulsion system, the better the stability of the system. Increased pectin concentration produced smaller more homogeneous droplets. Group C (7.5% pectin) and D (10% pectin) produced homogeneous droplets between 3-4 μm in size. The smaller more evenly distributed droplets may be the result of reduced interfacial tension during emulsification²⁶⁾. The main component of pectin is D-galacturonic acid which increases pectin's reactivity with water due to electronegative oxygen atoms attached to D-galacturonic acid's carboxyl group²⁷⁾. Therefore, as the pectin concentration increased the repulsion between the droplets increased which resulted in a more even droplet distribution. However, as we increased the pectin concentration the viscosity of the emulsion increased, which was not conducive to the preparation of peony seed oil microcapsules (Fig. 4).

3.2 Rheological characterization of emulsions

As shown in Fig. 2, group A exhibited pseudoplastic fluid characteristics with apparent viscosity decreasing with increasing shear rate. Different from group A, with the increase of shear rate, group B, C, D experienced shear-

Fig. 2 The effect of shear rate on the apparent viscosity of the peony seed oil microcapsule emulsions.

thickening and then a shear-thinning. The four groups varied in β -CD and pectin concentration. Group A had the highest β -CD concentration and did not contain pectin. Barnes *et al.* observed a similar phenomenon in the rhinemic experiment of starch suspended in water²⁸. Shear thickening behaviour occurred at lower shear rates and shear thinning behaviour occurred at higher shear rates. Changes in viscosity may be related to the shearing range²⁹. Below pectin's critical shear rate, the rate of pectin agglomeration increased, which increased the pectin concentration of the emulsion droplets. The agglomerated pectin particles at this time, the viscosity changes similarly to the expanded body. Above pectin's critical shear rate, the rate of deformation of agglomerated particles exceeded the rate of formation and the particle concentration of the emulsion was reduced. A dilute polymer solution was formed resulting in viscosity changes resembling that of a pseudoplastic fluid.

The effects of amplitude on the four groups are shown in Fig. 3. Initially, as the concentration of pectin increased, the storage modulus (G') and loss modulus (G'') increased indicating the molecular weight of the pectin in the solution increased, and more energy was stored. The four groups had viscous properties ($G'' > G'$). Under low amplitude stress, each group displayed good stability ($G' > G''$), indicating elastic properties. When the amplitude stress increased, G'' remained unchanged in group A. Group D was the most sensitive to shear stress amplitude, displaying the largest changes in G' and G'' , and groups B and C were minimally affected.

We speculated that a high concentration of pectin (10%) greatly influenced the viscosity of the emulsion system. When the shear stress amplitude exceeded a threshold, the system with the largest concentration of pectin was most affected.

Fig. 3 The effect of amplitude stress on storage modulus and loss modulus in peony seed oil microcapsules.

Fig. 4 Scanning electron micrograph of peony seed oil microcapsules.

3.3 Physicochemical properties of microcapsules

The physicochemical properties of the experimentally prepared peony seed oil microcapsules are shown in Table 2. The moisture content in group A was 2.15% and the moisture content in groups B, C, and D was 1.6%. This difference was not significant ($p > 0.05$). Group A had significantly higher surface oil content (11.52%) than the other groups ($p < 0.05$). Group C had a surface oil content of 2.60%, which was the lowest. The higher surface oil content in group A is due to a lack of pectin. SSOS and β -CD in combination are not effective encapsulation materials (Fig. 4A). Examination of group A revealed significant pores in the microcapsule which resulted in oil loss during the ether extraction process. The encapsulation efficiency of the pectin-containing groups (B, C, and D) was significantly higher, 66.7%, than the non-pectin-containing, group A ($p < 0.05$). A pectin concentration of 7.5% (group C) produced the highest encapsulation efficiency, 92.5%. Higher encapsulation efficiency is speculated to be caused by pectin tightening the cross-linking between SSOS and β -CD, which increased the embedding of peony seed oil.

Table 2 Characterization of microcapsules prepared using different ratios of wall materials in the initial emulsions.

Formulation	Moisture content (%)	Surface oil content (%)	Particle size (μm)	Encapsulation efficiency (%)
A	2.15 ± 0.07^a	11.52 ± 0.47^c	8.236 ± 0.06^a	66.7 ± 1.31^c
B	1.55 ± 0.07^b	4.30 ± 0.33^b	5.285 ± 0.64^a	86.5 ± 0.94^b
C	1.76 ± 0.03^b	2.60 ± 0.05^a	4.521 ± 0.05^{bc}	92.5 ± 0.14^a
D	1.64 ± 0.05^b	3.27 ± 0.10^a	3.656 ± 0.04^c	90.2 ± 0.29^a

Data was presented as means $\pm$ standard deviation. Different letters indicate significant difference between samples at $p < 0.05$ after spray-dried.

3.4 Observations using a scanning electron microscopy

The spherical structure produced by all groups is shown in Fig. 4. Group A produced a microcapsule with the most obvious pores resulting in the highest surface oil content during the ether extraction process (Table 2). The addition of pectin to emulsions B, C, and D resulted in smooth microcapsules without obvious pores. β -CD is a cyclic structure and various organic substances can be embedded in its hydrophobic cavity to form a complex³⁰. The smooth shape of the microcapsules formed with β -CD and pectin may be caused by non-covalent bonds forming a clathrate that is more easily supported by the microcapsule. Group C possessed the best microcapsule morphology and a more uniform distribution indicating there is an ideal ratio of SSOS, β -CD, and pectin which would produce the ideal compact spherical shape during spray-dried microencapsulation. However, as the pectin concentration increased to 10% (group D), more particle aggregation occurred.

The size of the emulsion particles determines the size of the spray-dried particles. During spray-drying, atomized droplet size at the inlet varies with viscosity at a constant speed³¹. Group D had the highest pectin concentration, resulting in increased emulsion viscosity, which may result in the accumulation of particles during spray-drying. Group D had increased surface wrinkles and degree of spherical depression and contained the lowest concentration of β -CD. We believe β -CD contributed to a smooth surface.

3.5 Infrared spectroscopy analyses

The infrared spectrum of SSOC, β -CD, and pectin is displayed in Fig. 5a. SSOS and β -CD had their largest absorption peaks at 3293 cm^{-1} and 3299 cm^{-1} , respectively, which represented the stretching vibration peaks of -OH. The absorption peaks for $-\text{CH}_2$ and $-\text{CH}_3$ at 2923 cm^{-1} and 2927 cm^{-1} , respectively, represented stretching vibrations and the peaks at 1637 cm^{-1} and 1652 cm^{-1} represented their respective bending vibrations caused by H_2O . The peaks at 998 cm^{-1} and 1012 cm^{-1} are the results of stretching $\text{C}=\text{O}$ connected by primary hydroxyl groups. A SSOS peak at 758 cm^{-1} represented the type 3 absorption band of D-glucopyranose. The pyran ring of SSOS is indicated by its

characteristic primary and secondary hydroxyl groups^{32, 33}. Pectin contains a D-galacturonic acid backbone. Pectin's infrared spectra revealed a broad vibrational absorption peak at 3291 cm^{-1} resulting from -OH stretch, a peak near 2935 cm^{-1} resulting from -CH stretch, and a peak at 1637 cm^{-1} resulting from the asymmetric stretch of carboxylate ($-\text{COO}$). Peony seed oil had an absorption band between approximately $2800\text{--}3100\text{ cm}^{-1}$ resulting from $\text{C}=\text{C}$ stretching vibration from peony seed oils many conjugated unsaturated double bonds. This also leads to the high instability of peony seed oil. A sharp absorption peak at 1735 cm^{-1} was observed due to $\text{C}=\text{O}$ bond vibration. However, in the B, C, D combination, weak absorption peak intensity between approximately $2800\text{--}3100\text{ cm}^{-1}$ for $\text{C}=\text{C}$ and 1745 cm^{-1} for $\text{C}=\text{O}$ represented significant bond weakening (Fig. 5b). This pattern indicated that the peaks were well covered by the wall materials. The lack of a shift in the absorption spectrum for the four groups indicated that the wall materials and the peony seed oil were combined using physical forces or formed non-covalent bonds.

3.6 Synchronous thermogravimetric analysis

Peony seed oil demonstrated a slight quality loss at 313°C , mass loss was maximized at 375°C and underwent complete thermal decomposition at approximately 500°C (Fig. 6). The microcapsules from the four groups showed a three-step heat loss curve³⁴. Mass loss at 100°C indicated water evaporation; between $180\text{--}262^\circ\text{C}$ each group began to show significant mass loss, representing the rupture of the wall materials; mass loss accelerated between $262\text{--}313^\circ\text{C}$ which may represent further rupture; between $313\text{--}500^\circ\text{C}$ the microcapsules lost integrity exposing the peony seed oil core, this was followed by thermal decomposition of all components. The rate of decomposition for peony seed oil was lower in the four microcapsule groups than for unencapsulated peony seed oil, which indicated effective embedding of the oil. Group D possessed the lowest crack rate indicating it provided the best protection to the peony seed oil core.

Fig. 5 Analyses of the infrared spectra of each peony seed oil microcapsule group and their wall materials.

Note: (a) Infrared spectrum of wall material and peony seed oil. (b) Infrared spectrum of peony seed oil microcapsules

3.7 Stability and oxidation resistance measured using induction period

Longer induction periods indicated increased stability and resistance to oxidation. Peony seed oil has a substantially shorter induction period (5.297 h) than many other oils because it contains higher amounts of UFAs, which are

easily oxidized at temperatures $\geq 90^{\circ}\text{C}$. Encapsulation significantly ($p < 0.05$) increased the induction period to 5-7 times that of unencapsulated peony seed oil (Table 3). Between B, C and D, Group B with a lower pectin concentration showed a lower induction period (30.76 h) than the C and D groups ($p < 0.05$). It indicated that the microen-

Fig. 6 Synchronous thermogravimetric loss analyses of peony seed oil and microcapsule powder samples.

Note: a Peony seed oil, b c d e respectively represents group A B C D

Table 3 Induction period for unencapsulated peony seed oil, and encapsulated peony seed oil with varying ratios of wall materials.

Formulation	Induction period (h)
peony seed oil	5.297 ± 0.16^d
A	27.470 ± 0.83^c
B	30.760 ± 0.54^b
C	35.800 ± 0.79^a
D	36.217 ± 0.84^a

Different letters indicate significant difference between samples at $p < 0.05$ after spray-dried.

capsulation of peony seed oil by different ratios of SSOS, β -CD and pectin can significantly prolong the induction period of peony seed oil. The highest induction periods were seen in the microcapsules from group D (36.21 h) and C (35.8 h), respectively. Difference between these groups was not significant ($p > 0.05$).

4 Conclusions

The addition of pectin to SSOS and β -CD resulted in reduced average particle size and a more uniform distribution of droplets in the emulsion. We believe that pectin improved cross-linking of SSOS and β -CD improving stability. As shear rate was increased pectin experienced shear-thickening and then a shear-thinning. This is indicative of a special rheological phenomenon. The addition of pectin does not increase stability appreciably under low amplitude stress; however, as the amplitude increases pectin plays an increasingly important stabilizing role. Group C, with a

ratio of 70% SSOS, 22.5% β -CD, and 7.5% pectin produced microcapsules with the best properties, the moisture content was 1.76%, and the lowest surface oil content was 2.60%. The highest encapsulation efficiency is 92.5%, including a smooth surface; even size, and distribution of particles. Additionally, group C possessed good thermal stability and an induction period of 35.8 h, which is seven times that of unencapsulated peony seed oil (5.297 h).

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