

Microencapsulation of vitamin E based on spray-dried Pickering emulsions synergistically stabilized by gelatin and bacterial nanofibrils

Jie Zhao¹, Tanghui Liu¹, Dong Zhang¹, Chang Su¹, Hai Chi^{1,2}, Hongrui Chen^{1,2}, Jie Tang^{1,2}, Xingzhong Zhang^{1,2} ✉

¹ School of Food and Bioengineering, Xihua University, Chengdu 610039, China

² Food Microbiology Key Laboratory of Sichuan Province, Xihua University, Chengdu 610039, China

✉ Address correspondence to Xingzhong Zhang, 1220220037@mail.xhu.edu.cn

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Abstract: Microencapsulation via spray drying can effectively enhance the processing performance and stability of vitamin E (VE), but it is still challenging to produce high VE-loaded microcapsules with low level of surface oil. Herein, we presented a facile method to prepare emulsions synergistically stabilized by gelatin and bacterial cellulose nanofibers (BCNFs), and fabricating highly loaded and stable VE microcapsules through spray drying. The emulsifying performance was significantly improved by increasing the gelatin content from 3% to 7% (*m/m*). In addition, as the content of BCNFs increased, the droplet sizes of these emulsions became smaller, and their physical stability also improved. After spray drying, the high VE-loaded microcapsules with an integrated spherical structure and uniform size of 10–50 μm had been successfully prepared. With the addition of 0.2% (*m/m*) BCNFs, the microencapsulation yield of VE microcapsules significantly increased to 81.8%, the VE oil loading rate of microcapsules was the highest at 46.51%, and the surface oil content of microencapsulation reduced from 43.2% to 37.1%. The spray-dried VE microcapsules with 0.2%–0.3% (*m/m*) BCNFs possessed a denser formation and improved the flowability of the powder as well as the encapsulation performance of the microcapsules. This work provided a new strategy for preparing microcapsules with low surface oil content and high VE-loaded content using BCNFs.

Keywords: microcapsule; spray drying; emulsion; vitamin E; bacterial cellulose nanofibrils

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1 Introduction

Vitamin E (VE) is a natural fat-soluble nutrient that possesses various active functions such as antioxidation, anti-cancer, and enhancement of immunity. It has been widely applied in numerous fields including food, cosmetics, and pharmaceuticals^[1]. However, natural VE with poor water solubility is prone to degradation when exposed to oxygen or ultraviolet irradiation, which imposes significant limitations on its application and storage^[2]. To address this issue, microencapsulation technology is regarded as an ideal strategy. Microencapsulation is the process of coating micron-sized materials in a polymeric shell to protect active compounds from adverse environmental conditions^[3–4]. Therefore, microencapsulation is a highly promising method that can effectively enhance the stability and processing performance of VE. Various microencapsulation approaches have been reported, including inclusion complexation, fluidized-bed coating, complex coacervation, spray drying and spray chilling^[5]. Especially, spray drying as the commonly used technique for encapsulation of lipid-soluble vitamins, is simple, low cost and suitable for industrial production^[6]. During microencapsulation, the VE as the core material is usually emulsified and trapped within the core of an oil-in-water (O/W) emulsion system by various polymers as both stabilizers and wall materials. The properties of microcapsule powders depend on size of the droplets, the interface structure and the stability of the emulsion, so it is a crucial step to prepare stable emulsions prior to spray drying^[7–8].

However, the appearance of surface oil on microcapsule powders commonly occurs during spray drying, resulting in poor flowability, reduced solubility, low encapsulation efficiency and accelerated lipid oxidation^[9–10]. Recent studies have demonstrated that modifying emulsion is a prospective method to reduce surface oil by moderating the segregation between the lipid and aqueous phase during atomization^[11]. It is worth noting that some polysaccharides with good wettability can adsorb onto the oil-water interfacial layer to further improve the stability of the emulsion during the spray drying process due to environmental factors such as thermal stress and mechanical stress^[12–13]. Foerster et al.^[14] reported that adding 0.3%–0.4% (*m/m*) of λ -carrageenan to milk emulsions could reduce the fat coverage on powder surface during spray drying. The adsorption of λ -carrageenan on the fat globule membranes improved the stability of the emulsion, and also increased the extensional viscosity, and thus inhibited the occurrence of fat perforation, and at the same time reduced the amount of powder surface fat through the disintegration process. Moreover, the application of low molecular weights octenyl succinic anhydride (OSA) modified starches to stabilize emulsions in the spray-drying VE nanoencapsulation had been investigated. The OSA modified starches could enhance the emulsions stability and improved VE retention rates during storage^[15]. In brief, introducing certain polysaccharides to modify the emulsion for spray drying would be an effective and feasible method to reduce the surface oil formation on spray-dried microcapsules. Cellulose, as the richest natural polymer with excellent amphiphilic property, is a good candidate

for emulsion stabilization. However, little research work had been conducted on the utilization of cellulose materials for high loaded spray-drying VE microencapsulation.

Gelatin as a water-soluble protein is frequently used for microencapsulation in terms of its excellent biocompatibility, biodegradability, emulsifying performance and film formation ability. Polyvinylpyrrolidone (PVP) as the wall material can improve the film-forming performance, and maltodextrin can protect encapsulated ingredients from oxidation and enhance the microencapsulation efficiency of the core material^[16–17]. In our previous works, bacterial cellulose nanofibers (BCNFs) had been certified as a Pickering stabilizer to fabricate O/W emulsions, which could be irreversibly adsorbed at oil-water interfaces to form a physical barrier and prevent flocculation or coalescence of oil droplets^[18]. Besides, it was reported edible oil powder was successfully prepared by combining spray drying with a Pickering emulsion stabilized by BCNFs and soy protein isolate, and the oil powder with appropriate content of BCNFs had better physical properties and excellent digestion resistance^[19].

This work was designed to fabricate high VE-loaded spray-dried microcapsules with low surface oil by preparing stable emulsion stabilized by BCNFs and gelatin. The effects of gelatin or BCNFs on the morphology and stability of the resultant emulsions had been studied. Furthermore, the size distribution, morphology, flowability, bulk density and moisture content of the VE microcapsules after spray drying were compared for different BCNFs contents. The microencapsulation performance and confocal laser scanning microscopy (CLSM) observation had been carried out to explore the reduction of surface oil. This research would provide a simple and low-cost method to prepare the improved microencapsulation performance of VE oil.

2 Materials and methods

2.1 Materials

Bacterial cellulose (BC) was supported by Yeguo Food Co., Ltd. (Haikou, China), and BCNFs dispersion was obtained by the method reported in our previous work^[20]. In detail, rinse BC with deionized water until the pH value reaches 7. Then, use the disintegrator (IKA-T25, IKA Instruments Ltd., Germany) to disintegrate. Subsequently, conduct 20 times homogenization treatments at a pressure of 600 bar using a high-pressure homogenizer (AH-1500, ATS, Canada) to obtain BCNFs dispersion. Gelatin (type B, 240 g Bloom) and PVP (K29-32, 58 000 Da) were obtained from Aladdin Reagent Co., Ltd. (Shanghai, China). Maltodextrin with dextrose equivalent value of 16.5–19.5 was purchased from Aladdin Reagent Co., Ltd. (Shanghai, China). The food-grade VE oil (> 98%) was purchased from Zhejiang NHU Co., Ltd., and the main composition of VE oil was VE acetate. Other reagents with analytical grade were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Deionized water was used in the experiment.

2.2 Preparation of VE-loaded emulsions and microcapsules

The preparation method of the continuous phase is as follows: mixing gelatin (3%, 4%, 5%, 6%, 7%, *m/m*), BCNFs dispersion solution (0%, 0.2%, 0.3%, 0.4%, *m/m*), maltodextrin (15%, *m/m*) and PVP (0.2%, *m/m*) with deionized water at 50 °C for 30 min. The 22 g VE oil were added into 78 g continuous phase aqueous solution at an oil-to-water ratio of 22:78 (*m/m*). The prepared

matrix was first subjected to a 4 min pre-homogenized at a rotational speed of 16 000 r/min by a high shear mixer (IKA-T25, Germany), then followed a 10 times homogenization with a high-pressure homogenizer (AH-2010, ATS, Canada) at 30 MPa.

Finally, the homogenized emulsion was transformed into microcapsules through a spray dryer (YC100, Pilotech Instrument and Equipment Co., Ltd., Shanghai, China). The inlet and outlet temperatures of the spray drying process were maintained at (180 ± 5) and (90 ± 5) °C respectively, and the feeding rate of emulsion was kept at a 6 mL/min.

2.3 Characterizations of prepared emulsions

The droplet size distributions and average sizes of the droplets in the prepared emulsions were measured using a laser particle size analyzer (Mastersizer 2000, Malvern, UK). The mean droplet diameter was determined based on the surface average diameter ($D_{(3,2)}$), the formula is as follows^[21]:

$$D_{(3,2)} = \frac{\sum N_i D_i^3}{\sum N_i D_i^2} \quad (1)$$

Where, N_i represents the number of the droplets with the diameter D_i (μm).

Moreover, the morphology of prepared emulsions was visualized using an optical microscopy (Shunyu CX40, Sunny Optical Technology (Group) Co., Ltd., Ningbo, China). The freshly prepared emulsions were placed on glass slide and covered with a cover slip, and then observed through a 20× objective lens.

2.4 Physicochemical properties of VE microcapsules

2.4.1 Scanning electron microscopy (SEM) observation

These samples were fixed on the metal platform with double-sided conductive adhesive tape, and a layer of gold was deposited on their surfaces by vacuum coating method. Samples were then observed using SEM (S4800, Hitachi, Japan) at an acceleration voltage of 10 kV and 1 000× magnification.

2.4.2 Determination of powder size and zeta potential

The powder size of microcapsules was determined by a Mastersizer 2000 with a Scirocco 2000 dry autosampler. The zeta potential of the microcapsules was determined by using Zetasizer Nano ZS instrument (Malvern, UK), the samples were dispersed in distilled water at a content of 0.1% (*m/m*) then placed in the electrophoretic tank for measurement.

2.4.3 Determination of flowability and bulk density

The flowability of the microcapsules was determined by reported method using the angle of repose^[22]. The prepared microcapsule powder (5 mL) was evenly scattered onto the center of the disc through a funnel, thereby automatically forming a powder mass. The angle between the powder pile and the plane of the disc is called the standing angle. To determine the density of the mass, the microcapsules after spray drying were placed in a graduated cylinder, and then the density of the mass was calculated by dividing the weight (g) of the powder by its volume (cm³).

2.4.4 Determination of moisture content

The moisture content of the microcapsules was determined by the oven drying method. The 0.5 g of the microcapsule sample, calculate the moisture content by dividing the lost weight by the original weight of the microcapsules.

2.4.5 CLSM observation

The distribution of proteins and VE oil inside the microcapsules obtained through emulsion spray drying was studied using a CLSM (FV3000, Olympus, Japan). The microcapsule powders were stained with 10 μL mixed dyes of Nile red (1 mg/mL) and fluorescein isothiocyanate (FITC) (1 mg/mL), then placed on a microscope slide and covered with a cover glass. The micrographs were observed using a CLSM operating in fluorescence mode. The detection wavelength of Nile red was 573–591 nm, and that of FITC was 498–517 nm. The CLSM images of VE microcapsules are shown in the z-stack CLSM scanning microscope images.

2.4.6 Measurement of encapsulation performance

The VE oil weighed precisely was dissolved with absolute ethanol and formulated into 10, 20, 30, 40, 50, 60 mg/mL standard solution, and then tested by a spectrophotometer (UV-1750, Shimadzu corporation, Japan) at wavelength of 285 nm. The relationship between VE content (y) and absorbance (x) was obtained by linear fitting: $y = 10.681x - 0.0087$ ($R^2 = 0.9978$).

The amount of total VE in microcapsules (m_1 , g) was determined as follows: 0.2 g microcapsule powder was dispersed in 80 mL absolute ethanol and sonicated for 20 min (pulsed ultrasound, 5 s for the duration and 5 s for the interval) by the high intensity ultrasonic processor (FS-600N, Shengxi Ltd., Shanghai, China) at a frequency of 20 kHz and 50% amplitude to extract VE. Centrifuge the suspension at $3000 \times g$ for 10 min. Then, take 0.1 mL of the supernatant and dilute it with anhydrous ethanol to 10 mL. This solution will be used to determine the total content of VE by ultraviolet measurement.

The amount of surface VE in microcapsules (m_2 , g) was determined as follows^[23]: 0.2 g of microcapsule powder were washed three times with 10 mL of petroleum ether to extract the surface VE oil. The extracts were pooled, removed petroleum ether by rotary evaporation, and then dissolved with 100 mL absolute ethanol. The solution used to measure the surface VE vibration displacement is achieved through ultraviolet measurement.

The microencapsulation performance could be evaluated by the microencapsulation yield (MEY), microencapsulation efficiency

(MEE), loading rate, and surface oil content which were calculated using the following formulas^[24]:

$$\text{MEY} (\%) = \frac{m_1}{m_0} \times 100 \quad (2)$$

$$\text{MEE} (\%) = \frac{m_1 - m_2}{m_1} \times 100 \quad (3)$$

$$\text{Loading rate} (\%) = \frac{m_1}{m_{\text{Total}}} \times 100 \quad (4)$$

$$\text{Surface oil content} (\%) = \frac{m_2}{m_1} \times 100 \quad (5)$$

Where, m_0 is the total VE amount used to make microcapsules (g), m_{Total} is the total mass of the microcapsules (g).

2.5 Statistical analysis

Statistical analysis was carried out by using the SPSS 13.0 statistical analysis program. Data were subjected to analysis of variance (ANOVA) and Duncan's multiple range tests were used to establish the significance of differences ($P < 0.05$) among the mean values.

3 Results and discussion

3.1 Morphology and droplet size of emulsions

3.1.1 Effect of gelatin content on emulsion morphology and droplet size

The homogeneity and stability of emulsions were closely related to the amount and mass ratio of emulsifiers in the continuous water phase^[25]. Herein, gelatin as an amphiphilic protein played a crucial role in VE-loaded emulsion stabilization. As shown in Figure 1, the obtained emulsions were milky-like, and oil droplet showed a spherical structure with a diameter of approximately 10 μm . At a relatively low content of gelatin, the size of several droplets was more than 10 μm and oil droplets flocculated and aggregated

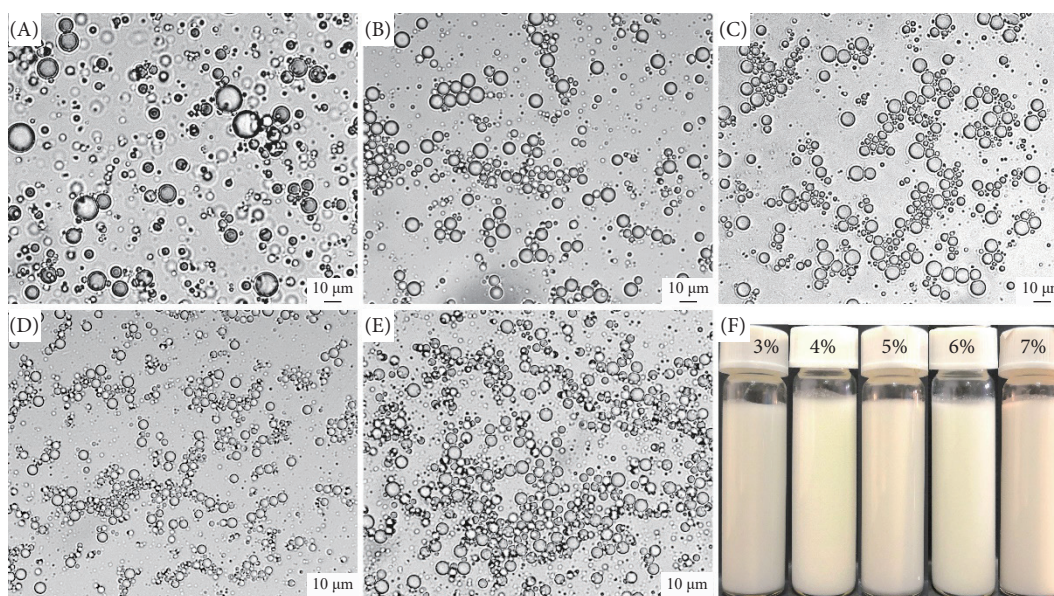


Figure 1 Optical micrographs of VE-loaded emulsions stabilized by gelatin and BCNFs with different gelatin contents. The content of gelatin in water phase was (A) 3%, (B) 4%, (C) 5%, (D) 6%, and (E) 7%, respectively. (F) The digital photos of corresponding emulsions. The content of BCNFs was controlled to 0.2%. The content of BCNFs, maltodextrin, and PVP were fixed values of 0.2%, 15%, and 0.2%, respectively.

seriously. With the increased gelatin content, the droplet size obviously decreased, and size distribution became uniform, which was due to the enhanced adsorption of collagen molecules on the surface of the oil droplets^[26]. This was consistent with the results of previous studies on related systems, as more oil interfaces were covered with gelatin to promote the production of smaller droplets^[27]. As shown in Figure 2A, the emulsions with gelatin content in the range of 4%–6% exhibited unimodal distributions, while the emulsions displayed a bimodal droplet size distribution when the content of gelatin was 3% or 7%. This was ascribed to that too low content of gelatin was not enough to cover the oil-water surface to inhibit the droplet coalescence, while an excess of gelatin molecules in the water phase tended to self-aggregate rather than adsorbed on the oil-water interface^[28]. As the gelatin content increased, the main peak became narrower and shifted to the left. In Figure 2B, the average diameter measurement results of the emulsion are consistent with the results obtained from microscopic observation, which was gradually decreased from 7.8 to 3.9 μm as the gelatin content increased from 3% to 7%, indicating that gelatin could improve the emulsification performance and sufficiently cover the oil-water interface against droplet coalescence^[27]. The reduced emulsion particle size would facilitate the bioavailability of

the encapsulated compound^[29]. According to the droplets size and size distribution, the gelatin of 6% was applied to the following emulsions and microcapsules fabrication.

3.1.2 Effect of BCNFs content on emulsion morphology and droplet size

The influence of BCNFs content on the emulsion properties was investigated. The emulsion contained uniform spherical shape droplets with a size of less than 10 μm was observed by microscopy in Figure 3, and the mean diameter $D_{(3,2)}$ of the emulsion containing 0.2% BCNFs was significantly lower than that of the emulsion without BCNFs in Figure 4B. This was because the increase in the solid content allowed more of the oil surface to be covered, leading the smaller droplet size. As the increase of BCNFs content, the droplet size increased slightly. This might be due to that more amount of BCNFs led to competitive adsorption and inhibited interfacial adsorption of gelatin at the oil-water interface, which reduced emulsifying properties of complexes and consequently increased emulsion droplet size. In addition, the increased BCNFs might promoted the formation of a fibrous network, causing droplet flocculation and a consequent increase in droplet size^[30]. However, the emulsion without BCNFs had a

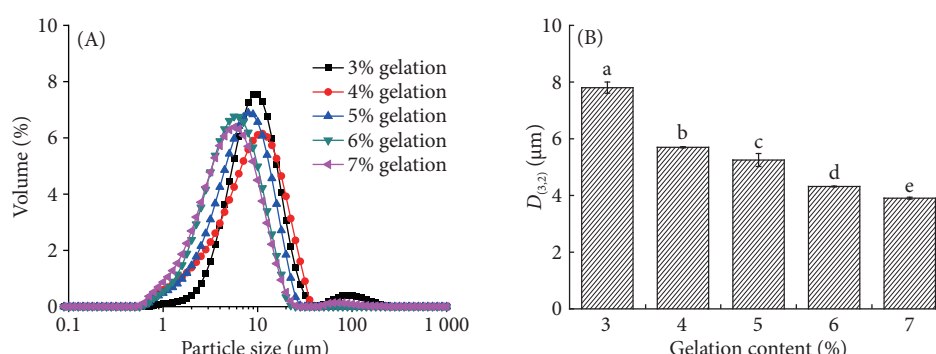


Figure 2 (A) Particle size distribution and (B) droplet size of VE-loaded emulsions stabilized by gelatin and BCNFs with different gelatin contents. The content of BCNFs was controlled to 0.2%. Different lowercase letters indicate significant differences ($P < 0.05$).

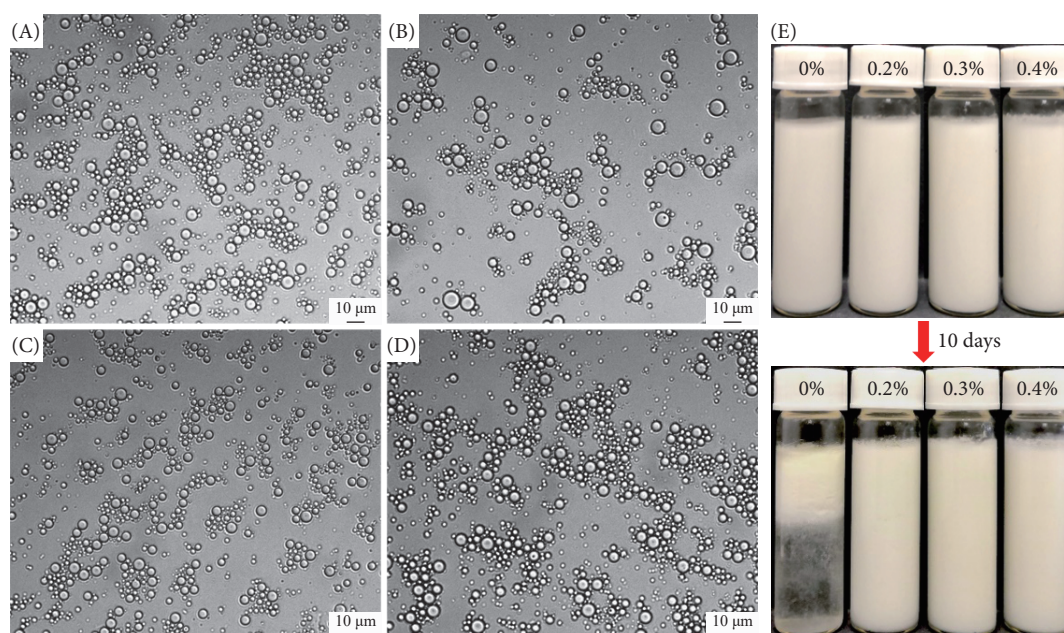


Figure 3 Optical micrographs of VE-loaded emulsions stabilized by gelatin and BCNFs with different BCNFs contents. The content of BCNFs in water phase was (A) 0%, (B) 0.2%, (C) 0.3%, and (D) 0.4%, respectively. (E) The digital photos of corresponding emulsions. The content of gelatin was controlled to 6%.

multimodal distribution (Figure 4A) and evidently creaming occurred for 10 days storage, due to flocculation or depletion effects of the oil droplets. With the addition of BCNFs, the droplets distribution became more homogeneous, and the emulsion remained stable without stratification, suggesting that the addition of BCNFs played a vital role in enhancing the stability of the emulsion. The stabilization mechanism provided by BCNFs as a stabilizer was different from that of gelatin. The BCNFs with high aspect ratio and good wettability could be irreversibly adsorbed at the oil-water interface, thereby forming a three-dimensional network structure, effectively inhibiting the free movement of oil droplets and thus improving the stability of the emulsion^[31–32]. In addition, the interaction force between gelatin and BCNFs improved the particle dispersion in the aqueous phase, thus improving the stability of the emulsion. Considering the various advantages of BCNFs as wall materials, we further explored its impacts on the physical and chemical properties of VE microcapsules.

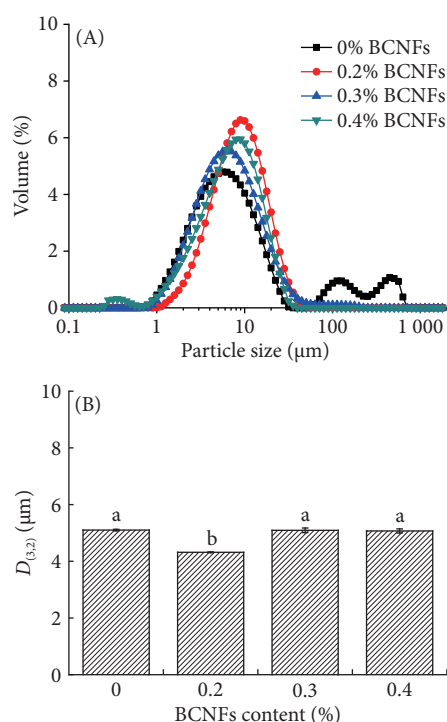


Figure 4 (A) Particle size distribution and (B) droplet size of VE emulsions stabilized by gelatin and BCNFs with different BCNFs contents. The content of gelatin was controlled to 6%. Different lowercase letters indicate significant differences ($P < 0.05$).

3.2 Morphology of VE microcapsules

After spray drying treatment, the prepared VE-loaded emulsions were successfully transformed into white and fine microcapsule powders. As shown in Figure 5, the microcapsule powders showed a spherical structure with smooth and wrinkled surface. Meanwhile, the typical folds and slight cracks of the surface were most likely formed by the dehydration shrinkage of interfacial films assembled with protein and polysaccharide components during the drying and cooling process^[33]. Obviously, without BCNFs, the micrographs of powders tended to agglomerate and appeared some broken structure, which might be attributed to the collapse of emulsion droplets during spray drying and the presence of high levels of VE

oil on the surface. When the content of BCNFs was increased from 0.2% to 0.3%, the powders presented more homogeneous size and had no holes or cracks on the surface, but the powders showed highly agglomeration when the BCNFs content was 0.4%. It was demonstrated that adding 0.2%–0.3% BCNFs improved the morphology of the microcapsules by inhibiting the collapse of VE oil droplets and decreasing powder size. However, the higher content of BCNFs ($\geq 0.4\%$) would result in the agglomeration of microcapsule powders, owing to more extensive hydrogen bonding and interactions between the nanofibers on the microcapsule surface^[34].

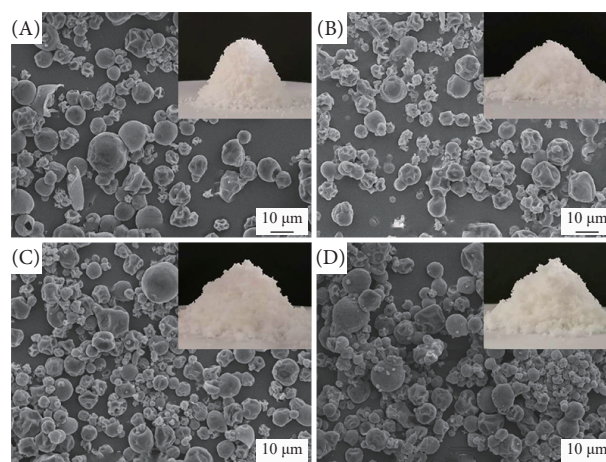


Figure 5 Effect of BCNFs contents on the SEM images of the obtained VE microcapsules (1 000×). The content of BCNFs was (A) 0%, (B) 0.2%, (C) 0.3% and (D) 0.4%, respectively.

3.3 Particle size distribution and zeta potential of microcapsules

As shown in Figure 6A, all dried microcapsules have a broad unimodal distribution within the particle size range of 1–150 μm, and the main distributional proportion was in the range of 10–50 μm. As the content of BCNFs increased, the distribution peak became narrower and shifted to the left, and the content of BCNFs was 0.3%, the size distribution was the most concentrated. Compared with other samples, the distribution peak of the microcapsule powders with the BCNFs content of 0.4% corresponded the lowest size. Moreover, in Figure 6B, the zeta potential of microcapsule powders was negative, with the value of approximately −20 mV, and it does not change much with the content of BCNFs, demonstrating that the BCNFs with weak anionic charges had a slight effect on the surface charge density of microcapsules. Consistent with the size distribution, the average size of microcapsule powders gradually decreased from 28.6 to 13.5 μm with BCNFs content varying from 0.2% to 0.4%, and the average size of microcapsule powders without BCNFs was the largest, indicating more homogeneous microcapsules could be produced with the presence of BCNFs. The decrease in particle size of microcapsule powders was due to the increase in the ratio of shell-core materials. However, the size of microcapsule powders was obviously larger than those of original emulsions. This could be attributed to that the large amount of maltodextrin existed in the continuous phase had sufficiently covered the surface of VE oil droplets and occupied a larger volume with more densely, packed interior during spray drying^[35].

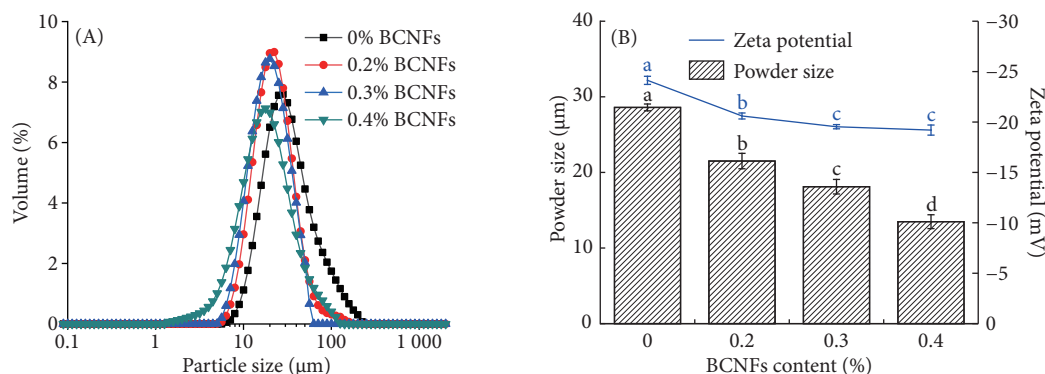


Figure 6 Effect of BCNFs contents on the (A) particle size distribution, (B) powder size and zeta potential of the obtained VE microcapsules. Different lowercase letters indicate significant differences ($P < 0.05$).

3.4 Moisture content, flowability and bulk density of microcapsules

As shown in Table 1, as the content of BCNFs increased, the moisture content in the microcapsules increased slightly from 2.98% to 3.28%, which was due to the excellent hydrophilicity of the cellulose nanofibrils^[18]. The moisture content was maintained at a relatively low level compared to that of the reported VE microcapsules (4.50%–4.98%), which was beneficial in preventing mildewing and lipid oxidation^[36]. The static repose angle revealed the fluidity of the prepared microcapsules. The evaluation criteria are as follows: an angle lower than 30° indicates good fluidity, an angle between 30° and 55° indicates some viscosity and an angle exceeding 55° indicates poor fluidity^[19]. The measured results were 60.0° , 45.2° , 46.6° , and 44.6° , respectively, indicating the flowability of microcapsules could be improved by adding BCNFs. This phenomenon could also be observed in Figure 5. Meanwhile, the bulk density of microcapsules with BCNFs was around 0.17 g/cm^3 , which was significantly lower than the microcapsules without BCNFs (0.191 g/cm^3). These results might be related to the decrease in the content of surface VE oil after the addition of BCNFs, because the addition of BCNFs can inhibit the agglomeration of the powder, thereby improving the fluidity, making the powder looser and having a relatively lower bulk density.

Table 1 Effect of BCNFs contents on the moisture content, flowability (repose angle) and bulk density of the obtained VE microcapsules.

BCNFs content (%)	Moisture content (%)	Repose angle ($^\circ$)	Bulk density (g/cm^3)
0	2.98 ± 0.18^b	60.0 ± 2.1^a	0.191 ± 0.016^a
0.2	3.02 ± 0.17^b	45.2 ± 1.3^b	0.174 ± 0.002^b
0.3	3.21 ± 0.23^a	46.6 ± 1.2^b	0.169 ± 0.006^b
0.4	3.28 ± 0.10^a	44.6 ± 1.2^b	0.171 ± 0.002^b

Note: Different lowercase letters in the same column indicate significant differences ($P < 0.05$).

3.5 Microencapsulation performance of microcapsules

In order to confirm the high VE oil loading rate and decline of surface VE oil by BCNFs on the microcapsules obtained through spray drying, the encapsulation properties of microcapsules with different BCNFs contents were further investigated. As shown in Table 2, with the BCNFs content of 0%, 0.2%, 0.3%, 0.4%, the MEY of the microcapsules was 68.7%, 81.8%, 73.3% and 73.0%, respectively. The MEY of microcapsules with 0.2% BCNFs was significantly higher compared with that of microcapsules without

BCNFs ($P < 0.05$). Meanwhile, the MEE of microcapsules increased from 56.80% to 62.90% as the BCNFs content increased from 0% to 0.2%. The VE oil loading rate in microcapsules with the BCNFs content ranging from 0.2% to 0.4% was 46.51%, 41.59% and 41.34%, and all were higher than that of the microcapsules without BCNFs, which was significantly higher than the previous reported results^[37–38]. The amount of surface oil of microcapsules was reduced from 43.2% to 37.1%, when the BCNFs content was 0.2%. The results revealed that the suitable amount of BCNFs remarkably decreased the surface VE oil content in microcapsules by decreasing droplet size and improving emulsion stability during the spray drying, and thus boosted the microencapsulation performance. The VE acetate yield of microcapsules containing cremophore RH40, Tween-80 and maltodextrin or OSA-modified starches as emulsifiers and wall material by spray drying was 53%–61%, which was lower than the MEY of the core material in this manuscript (68.70%–81.80%)^[12]. Moreover, the CLSM images provided more details about the surface oil and protein distribution in microcapsules. In Figure 7, for the microcapsules with the absence of BCNFs, the signal of VE oil (red) was stronger and almost covered the microcapsule powders surface, the gelatin marked in green was placed on the surface. With the addition of BCNFs, the distributions of surface oil were significantly reduced. The phenomenon observed from CLSM could explain why the encapsulation efficiency had been modified in the existence of BCNFs.

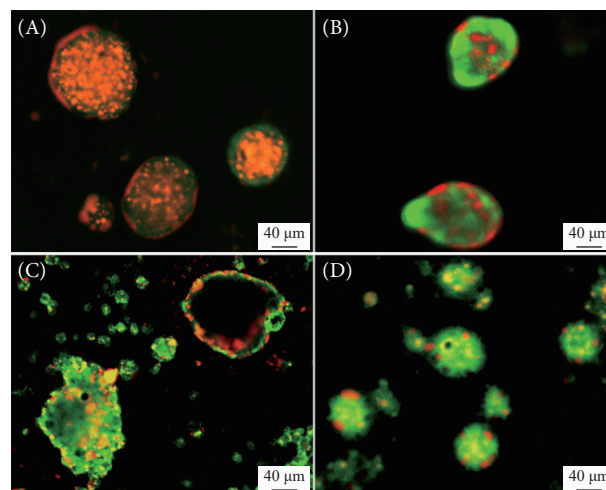


Figure 7 Effect of BCNFs contents on the z-stack CLSM micrographs of the obtained VE microcapsules. The content of BCNFs was (A) 0%, (B) 0.2%, (C) 0.3%, and (D) 0.4%, respectively. Green color located the gelatin protein stained by FITC, and red color located the VE oil stained by Nile red.

Table 2 Effect of BCNFs contents on MEY, MEE, loading rate and surface oil content of the obtained VE microcapsules.

BCNFs content (%)	MEY (%)	MEE (%)	Loading rate (%)	Surface oil content (%)
0	68.70 ± 0.42 ^c	56.80 ± 0.44 ^d	39.22	43.2
0.2	81.80 ± 0.74 ^a	62.90 ± 1.08 ^a	46.51	37.1
0.3	73.30 ± 0.43 ^b	60.00 ± 0.89 ^b	41.59	40.0
0.4	73.00 ± 0.43 ^b	58.40 ± 0.45 ^c	41.34	41.6

Note: Different lowercase letters in the same column indicate significant differences ($P < 0.05$).

4 Conclusions

In this work, the stable O/W emulsions stabilized by BCNFs and gelatin were successfully prepared, and high-loading VE microcapsules were obtained through spray drying. Gelatin played a key role in VE-loaded emulsion stabilization, and the emulsions droplet size obviously decreased and size distribution became uniform with the increase of gelatin content. Meanwhile, after adding BCNFs, the stability of the emulsion during storage for 10 days was significantly improved, effectively preventing the occurrence of stratification. The VE-loaded microcapsule powders presented more homogeneous size in the range of 10–50 μm with the absence of wall holes or cracks on the surface when the content of BCNFs was 0.2%–0.3%. The moisture content of microcapsules was maintained at a relatively low level of $< 3.28\%$ and the powder flowability was improved by BCNFs. In addition, the VE microcapsules in the presence of BCNFs presented improved microencapsulation performance by the high VE loading rate and a reduction of surface oil. This work provided a new pathway for designing high VE-loaded microencapsulation with reduced amount of surface oil by introducing BCNFs.

Conflict of interest

The authors declare no competing financial interest.

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Authors contribution

Jie Zhao: Data curation, formal analysis, software, validation, visualization, writing-original draft. Tanghui Liu: Formal analysis, software, validation, visualization. Dong Zhang: Software, validation, visualization. Chang Su: Software, validation, methodology. Hai Chi: Validation, visualization, methodology, supervision. Hongrui Chen: Validation, visualization, methodology, supervision. Jie Tang: Conceptualization, methodology, writing-review & editing. Xingzhong Zhang: Conceptualization, funding acquisition, methodology, project administration, supervision, writing-review & editing.

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