



Preparation and properties of cinnamon-thyme-ginger composite essential oil nanocapsules

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ABSTRACT

Essential oils (EO) as one of natural antimicrobials possess excellent antibacterial, antifungal and antioxidant properties. However, their main components are easy to be oxidated and deteriorated when EO was exposed to the oxygen, light and heat. In this study, cinnamon-thyme-ginger composite essential oil nanocapsules (CEO-NPs) were prepared with chitosan as the wall via ionic gelification reaction. The effect of the mass ratio of chitosan (CS) to tripolyphosphate (TPP), composite essential oils (CEO) and surfactant concentration and pH on the properties of the CEO-NPs were investigated in detail. The morphology and structures of CEO-NPs were measured by dynamic light scattering (DLS), fourier transformation infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), gas chromatography–mass spectrometry (GC–MS) and ultraviolet spectrum (UV). Meanwhile, the antibacterial property of CEO-NPs was determined by inhibition zone method. CEO-NPs with the size of 215 nm and zeta potential of 25.12 mv were obtained when the mass ratio of CS/TPP, CEO and fatty alcohol polyoxyethylene ether-9 (AEO-9) concentration and pH were 8:1, 1.8 g/L, 0.7 g/L and 5.26. FTIR demonstrated that CEO has been encapsulated into NPs. TGA showed that the thermal stability of CEO-NPs was improved compared with CEO and the loading capacity of CEO was 13.4%. GC–MS displayed that more than 90% ingredients of CEO had been encapsulated into NPs. UV indicated that CEO-NPs had the excellent sustained-release property. Furthermore, CEO-NPs showed a long-lasting antibacterial activity against *Escherichia coli*, *Bacillus subtilis* and *Staphylococcus aureus*. In all, CEO-NPs can be used as a potential long-term natural preservative.

1. Introduction

Recently, natural antimicrobials have attracted more attention due to the increased consumer awareness on the safety (Benjemaa et al., 2018). Essential oils (EO) are aromatic natural oily liquids obtained from flowers, leaves, fruits, stems and other part of plants, which can be prepared by steam distillation, fermentation, or by expression (Baran et al., 2007; Benelli and Pavela, 2018; Burt, 2004; Tiziana Baratta et al., 1998) and exhibit excellent antibacterial activities, antifungal and antioxidant properties. For example, cinnamon oil obtained from foliages and barks showed the antioxidant and antimicrobial activities because of the high eugenol and cinnamic aldehyde content (Yildirim et al., 2017). Thyme oil isolated from herb thyme had the strong antimicrobial activity, which could replace synthetic fungicides (Vilaplana et al., 2018; Ryu et al., 2018). Ginger oil produced by fresh ginger exhibited antibacterial and antioxidant properties (Nile and Park, 2015). However, EO is the mixture of volatile compounds including terpenes, aromatic hydrocarbons, esters, phenols and other natural substances. When EO is exposed to the environment of oxygen, light

and heat, most ingredients can be easily oxidated and deteriorated (Perdones et al., 2012; Carvalho et al., 2016). Encapsulation technology provided an effective approach to keep the stabilization of EO and prevent the loss of volatile ingredients (Campelo et al., 2017; Wang et al., 2018; Calvo et al., 2012). Natural polysaccharides including chitosan (Tan et al., 2018), maltodextrin (Medina-Torres et al., 2016), cyclodextrin (da Rosa et al., 2013) and gum arabic (Binsi et al., 2017) have been widely used to encapsulate EO due to their non-toxic, biodegradability and biocompatibility (Xiao and Grinstaff, 2017; Olejnik et al., 2016; Tabart et al., 2012; Sotelo-Boyás et al., 2017; Cota-Arriola et al., 2013). Cinnamon EO was encapsulated with β -cyclodextrin by inclusion complexation method and showed the high antifungal activity against *Botrytis* sp (Munhuweyi et al., 2018). Simon-Brown et al. (2016) reported that ginger EO microcapsule (the particle size: 8.2–15.3 μ m) was prepared with maltodextrin (MD) and/or gum arabic (GA) as the walls by spray drying and exhibited effective antioxidant activity to prolong food shelf-life.

Especially, chitosan (CS) obtained from deacetylation of chitin, as the second most abundant polysaccharide showed the excellent

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synergism antimicrobial effect in combination with EOs (Gharsallaoui et al., 2007; Ali and Ahmed, 2018; Xu et al., 2017). Patchouli oil CS microcapsules with diameter in the range of 1–20 μm were prepared via complex coacervation method and displayed high antibacterial activity against *Staphylococcus aureus* and *Escherichia coli* in cotton fabrics (Liu et al., 2013). In addition, Velmurugan et al. (2017) prepared orange and lavender EO CS nanoparticles with the size of 213.6 and 273.8 nm and then applied them in leather finishing against *B. cereus*, *B. subtilis*, *Aspergillus fumigatus*, *Macrophomina phaseolina*. In our previous study, CS nanoparticles loaded with cinnamon EO (CE-NPs) with three sizes of 112 nm, 215 nm and 527 nm were prepared by ionic gelification reaction. Then these CE-NPs were applied in the package preservation of the chilled pork. CE-NPs with 527 nm led to a significant decrease of microbial growth, pH, peroxide value, 2-thiobarbituric acid and sensory scores of the pork than the other treatments at 4 °C during 15 day (Hu et al., 2015).

Up to now, few studies about the encapsulation of composite essential oil (CEO) with CS as wall material have been reported. In this study, cinnamon-thyme-ginger composite essential oil nanocapsules (CEO-NPs) with CS as the wall were prepared successfully via ionic gelification method. The influence of the mass ratio of CS to TPP, CEO and surfactant content and pH on the particle size and zeta potential of CEO-NPs were investigated in detail. Then the morphology and structure of CEO-NPs were characterized by DLS, FT-IR, TGA, GC–MS and UV. Finally, the antibacterial activity of CEO-NPs against *Escherichia coli*, *Bacillus subtilis* and *Staphylococcus aureus* were studied.

2. Materials and methods

2.1. Materials

Chitosan (CS, average molecular weight = 150000) was obtained from Sigma-Aldrich, USA. Sodium tripolyphosphate (TPP), fatty alcohol polyoxyethylene ether-9 (AEO-9), glacial acetic acid (HAC) and sodium hydroxide (NaOH) were supplied by Shanghai National Chemical Reagent Co., Ltd., China. Cinnamon, thyme and ginger essential oil were purchased from ZhengzhouXomolon Food Flavor Co., Ltd., China. All chemicals were used as received without any further purification. Deionized water was used for all experiments.

2.2. Preparation of CEO-NPs

CEO-NPs were similarly obtained according to the previously described method (Xiao et al., 2014a,b; Hu et al., 2015). Typically, CEO was obtained by blending three EOs (the mass ratio of cinnamon EO, thyme EO and ginger EO: 4:3:3). CEO (0.09 g) was mixed with AEO-9 (0.035 g) under 1000 rpm stirring for 5 min at 25 °C. Then the mixture was added into 15 mL TPP aqueous solution to form O/W emulsion under the homogenization for 10 min. After that, 35 mL CS aqueous solution was added into the above emulsion to keep the weight ratio of CS/TPP 8/1 with a stirring speed of 400 rpm at 30 °C. Meanwhile, the pH of the emulsion was controlled to 5.26 with 1 mol/L sodium hydroxide aqueous solution and the reaction was kept for 2 h. Finally, CEO-NPs were purified by the centrifugation at 15 000 rpm for 30 min at 5 °C. CS-NPs were also prepared as the above process without CEO.

2.3. CEO-NPs characterization

2.3.1. Particle size and zeta potential

The particle size, polydispersity index (PDI) and zeta potential of CEO-NPs in three replicates were measured by the Malvern Zetasizer Nano ZS (Malvern Instruments, Worcestershire, UK). Each sample was determined using a solid state He-Ne laser of 633.0 nm at 25 °C with an angle detection of 90 °C.

2.3.2. Chemical structure analysis

The chemical structures of ginger EO, cinnamon EO, thyme EO, CEO-NPs after freeze-drying and CS were determined with a VERTEX 70 FTIR spectrophotometer with ATR accessory (Bruker, Ettlingen, Germany) in the range from 4000 to 600 cm^{-1} .

2.3.3. Determination of CEO loading capacity and thermal stability

In order to obtain the thermal stability and CEO loading capacity of CEO-NPs, CEO, CS-NPs and CEO-NPs after freeze-drying were performed on a Q5000 thermal analyzer (TA Instruments, USA) from 25 °C to 600 °C at 10 °C/min heating rate under nitrogen atmosphere with a flow of 20 mL/min.

2.3.4. Determination of the encapsulated aromatic compounds in CEO-NPs via GC–MS

In order to identify the main ingredients of CEO encapsulated in CEO-NPs, the analysis of cinnamon EO, thyme EO, ginger EO and CEO-NPs after-drying was performed with GC–MS (Agilent Technologies Inc., New York, USA). CEO-NPs were firstly pretreated as the followings. 0.1 g CEO-NPs after freeze-drying was diluted in 5 mL ethanol. The suspension was treated with the ultrasonication at 800 W for 30 min until the shell of CEO-NPs was completely destroyed and CEO was dissolved in the ethanol. Then, the suspension was centrifuged at 13000 rpm for 20 min and the supernatant was collected. After that, 1, 2-Dichlorobenzene of 100 $\mu\text{g/mL}$ as an internal standard (ISTD) was added in the solution to determine the content (%) of CEO ingredients. Meanwhile, each pure aromatic compound in the three EOs was also been determined as its standard.

An Agilent 6890N gas chromatograph with a 5873N mass detection in the range 30–450 mass/charge was used with a HP-INNOWAX polar column (60 m \times 0.25 mm i.d. \times 0.25 μm film, Agilent). The carrier gas was ultrapurified helium at a flow rate of 1.0 mL/min. The injection volume was 0.2 μL with a split ratio of 20:1. The initial column temperature was held at 40 °C for 6 min, programmed to ramp to 100 °C at a rate of 3 °C/min for 2 min, then to 230 °C at a rate of 5 °C/min and held at this temperature for 20 min. The detector ion source temperature was set at 230 °C. Electron impact ionization was performed at electron energy of 70 eV. Identification of the main components of CEO was achieved by comparing mass spectra with those in the NIST08 library.

2.3.5. Sustained-release property of CEO-NPs

In order to study the sustained-release property of CEO-NPs, 1 g CEO-NPs after freeze-drying were placed at 40 °C for 1, 2, 3, 5, 7, 9, 12, 15, 18 days, separately. Then, these CEO-NPs were added into 20 mL dichloromethane. The other extract processes were the same as above. The volatile components in the CEO released from CEO-NPs after different placing times were determined by UV–vis spectrum (UV2100, Unico, Shanghai). (E)-cinnamaldehyde and thymol were selected as the model compounds and determined at 286 nm and 275 nm, respectively.

2.4. Antibacterial experiment

Antibacterial activities of CEO, CS-NPs, CEO-NPs against *Escherichia coli* (ATCC 25922), *Bacillus subtilis* (ATCC 6633) and *Staphylococcus aureus* (ATCC 25923) were carried out by the zone of inhibition method. Bacterial strains were cultured overnight at 37 °C in the nutrient agar plates. The nutrient agar medium in a Petri dish was inoculated with 0.1 mL 10^7 – 10^8 cfu/mL bacteria. The sterile filter paper discs (6 mm diameter) were impregnated with 1 mL CEO and aqueous dispersions of CEO-NPs and CS-NPs (0.2 g/mL) and dried for 5 min by an ultraviolet lamp. Meanwhile, gentamicin sulfate and sterile saline were tested as the positive and negative controls, respectively. The dishes were incubated at 37 °C for 1, 5, 7 and 9 days. The diameter of the inhibition zones around each of the discs was taken as measure of the antimicrobial activity. Each experiment was carried out 6 times and the mean diameter of the inhibition zone was measured.

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