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Research note

Encapsulation of rose hip seed oil into fibrous zein films for ambient and on demand food preservation *via* coaxial electrospinning

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ABSTRACT

Several active encapsulation technologies using polymeric matrix systems for food preservation have been developed in the last decade. In this study, the microencapsulation of rose hip seed oil (REO) in to a zein prolamine (ZP) fiber matrix *via* the coaxial electrospinning technique is studied. The morphological features of core-sheath REO encapsulated ZP fibers were modulated through process parameters (e.g. active and polymer solution concentration, applied voltage and media flow rate). The ZP solution concentration (outer needle) was found to be critical for REO loading capacity (LC) and encapsulation efficiency (EE). The optimal LC and EE of REO in ZP matrix was 12.24% and 90.16%, respectively, and were achieved using a ZP concentration of 35 w/v%. Food packaging potential was evaluated using peeled and segmented fruits (bananas and cumquats). The findings demonstrate a facile packaging route to improve food sustainability and reduce waste.

1. Introduction

In the food industry, perishable food products are easily spoiled causing waste and economic loss; and also increase the risk of food borne illness (Chung et al., 2003; Gram et al., 2002). Thus, it is imperative to preserve food and extend product shelf life. Essential oils are volatile, naturally occurring aromatic compounds extracted from plant sources (Bakkali et al., 2008). These form part of the plants natural defense mechanisms providing crucial anti-bacterial, anti-viral and anti-fungal properties. Furthermore, they have been used extensively in the cosmetic, pharmaceutical and food industries (Andoğan et al., 2002; Ghayempour and Mortazavi, 2014; Yi et al., 2007).

Botanical extracts comprise numerous chemical moieties and molecules of biological interest. For example, the relative Vitamin C content in *rose hips* exceeds the quantities found in citrus fruits. In addition *rose hips* are abundant in carotenoids, organic acids, sugars and numerous essential oils (Yi et al., 2007). Oil extracts from *rose hip* seeds are an ample source of unsaturated fatty acids. Several phytochemicals, tocopherols, sterols, and varied concentrations of carotenoids have also been detected in rose hip seed oil (REO) (Grajzer et al., 2015; Zlatanov, 1999). Utilization of the oil has demonstrated positive outcomes for the treatment of contact dermatitis through anti-conflict mechanisms (Szentmihályi et al., 2002). It's combative action against oxidative stress is a key feature in providing its antioxidant property (Grajzer et al., 2015).

Most essential oils readily undergo an oxidation process on exposure to air or ultraviolet light. In addition, their highly volatile nature limits their applications as antioxidant materials in various dietary, sanitary and medical fields. Numerous studies have demonstrated the significance of encapsulation methodologies as effective platforms to increase essential oil stability and bioactivity (Quispe-Condori et al., 2011; Ziani et al., 2012). However, the method and material (encapsulant) used impacts these properties and also the type of structure formed. For example, the spray-drying technique has been used to fabricate chitosan encapsulated orange oil microparticles for use as a detergent (Li et al., 2013). *Cuminum cyminum*, *Menthe piperita* and *thyme* essential oils have been encapsulated in chitosan-caffeic acid, chitosan-cinnamic acid and chitosan-benzoic acid nanogel, respectively, *via* the sonication process to enhance their antimicrobial activity against *aspergillus flavus* (Beyki et al., 2014; Khalili et al., 2015; Zhaveh et al., 2015).

Electrospinning (ES) is a maturing encapsulation technique that facilitates the production of polymeric fibers on the nanometer and

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micrometer scales (Bhardwaj and Kundu, 2010). Fiber surface morphology and size distribution are modulated using process parameters, such as polymer concentration, applied voltage, solution flow rate and collector-ejector nozzle distance (Agarwal et al., 2013; Sill and von Recum, 2008). The successful encapsulation of cinnamon essential oil and β-cyclodextrin in polylactic acid polymer matrix, *via* the single needle ES process, has been demonstrated yielding fibrous nano-films for antimicrobial packaging (Wen et al., 2016a). The utilization of other polymeric matrix systems (e.g. polyvinyl alcohol) for similar bioactive constituents has also been shown (Wen et al., 2016b). More recently, ES processes have focused on two or more co-flowing phases (through coaxial needle geometry) with the potential to yield core-shell microstructures ideally suited for applications requiring encapsulation (Koo et al., 2014; Pereira et al., 2014). When compared to the single needle ES method, the coaxial ES approach provides greater control on encapsulant layering, morphology, bioactive loading capacity and retention e.g. premature release (McCann et al., 2005; Moroni et al., 2006). However, the encapsulation of rose hip seed oil in polymeric core-shell fiber (*via* coaxial ES) is yet to be explored in detail.

Zein prolamine (ZP) is a biodegradable and hydrophobic protein extracted from corn maize (Neo et al., 2012). Due to its intrinsic low hydrophilicity, exceptional film forming, high thermal resistance and oxygen barrier properties; ZP has been widely explored for numerous biological and biomedical applications (e.g. scaffolds and matrix materials for drug delivery systems (Jiang et al., 2012; Lai and Guo, 2011)). Several studies utilizing ZP have also focused on food applications. For example, a blend of gallic acid and ZP has been used to prepare sub-micron structured antioxidants films (Neo et al., 2013). β-carotene (serving as a colorant and an antioxidant) has also been embedded into a ZP fiber matrix system (Fernandez et al., 2009).

In this research the coaxial ES technique was used to prepare encapsulated fibrous structures for food preservation functions. For the coaxial ES system, REO was selected as the internal medium, while ZP solution was selected as the enveloping medium (sheath material) to encapsulate REO. Surface morphology and surface hydrophobicity of electrospun ZP fibers prepared using various ZP solution concentrations were investigated. The loading capacity (LC) and encapsulation efficiency (EE) were optimized based on solution parameters. Finally, the preservative property of fibrous ZP/REO films was assessed on bananas and cumquat fruit.

2. Materials and methods

2.1. Materials

Zein prolamine (ZP) from corn (Z 3625) was purchased from Sigma Aldrich (St. Louis, Mo., USA) and was used without additional purification. Ethanol and hexane were obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All chemicals used were analytical grade. REO obtained through steam-distillation was purchased from Jinyuan natural flavor Co., Ltd (Jiangxi, China). Deionized water (DI water) was produced using a Millipore Milli-Q Reference ultra-pure water purifier (USA).

2.2. Preparation of zein solutions for electrospinning

25, 30 and 35 *w/v* % ZP solutions were prepared by dissolving 2.5, 3.0 and 3.5 g of ZP powder into 80% (*v/v*, volume of ethanol/(volume of ethanol + volume of DI water)) aqueous ethanol, respectively. The solutions were mechanically stirred (VELP ARE heating magnetic stirrer, Italy) in a flask for 1 h (300 rpm) at the ambient temperature (25 °C) to achieve complete dissolution.

The physical properties of ZP solutions were measured. Solution viscosity was obtained using a viscometer (LVDV-II, Brookfield, USA). Selected solutions were placed into pre-defined stainless steel wells of the viscometer and then measured at 25 °C using S21 spindle at 140 rpm. Solution electrical conductivity was measured at 25 °C using a YSI 3200 electrical conductivity meter (YSI, USA). All characterizations were carried out in triplicate and mean values were obtained.

2.3. Fabrication of coaxial electrospun fibers and films

The ES apparatus consists of a high power voltage supply, precision syringe pumps, a coaxial stainless steel needle and a ring-shaped ground electrode, as shown in Fig. 1a. Individual ZP polymer solutions were loaded into 5 mL plastic syringes. ZP polymer solution was perfused from the syringe (*via* silicon tubing) in to the outer inlet of the coaxial device (flow rates between 0.3 and 0.7 mL/h) using a high-precision programmable syringe pump (KD Scientific KDS100, USA). REO was loaded into a separate syringe and perfused (flow rate fixed at 0.1 mL/h) into the inner needle of the coaxial system. The coaxial system comprised two enveloped needles. The inner needle possessed inner and outer diameters of 0.2 and 0.4 mm, while the outer needle had dimensions of 0.9 and 1.2 mm, respectively. An electric field (between 14 and 16 kV) was applied between the coaxial needle and ground electrode *via* a high voltage power supply (Glassman high voltage Inc. series FC, USA). A ring-shaped electrode with inner and outer diameters of 2.7 and 3.5 cm, respectively, was set directly below the needle exit at a distance of 12 cm. Aluminum foil was used as the fiber collector substrate and was placed directly below the ground electrode. The ES jetting modes were observed using a high-speed camera (Baummer TXG02C, Germany). All experiments were performed at the ambient temperature (25 °C).

2.4. Encapsulated fiber morphology assessment

Optical (OM, Pheonix BMC503-ICCF, China) and field emission scanning electron microscopes (SEM, SU 8000 SEM, Hitachi, Japan) were used to assess the distribution and surface morphology of the electrospun fibers. Prior to SEM analysis, samples were placed on to a metallic stub using double-sided conductive tape and then sputter-coated with a thin layer of gold under vacuum (Ion sputter MC 1000, Hitachi, Japan) for 60 s using a current intensity of 25 mA. All samples were observed at an accelerating voltage of 20 kV. Electron micrographs were analyzed using ImageJ software (National Institute of Health, MD, USA) to obtain mean fiber diameters of various electrospun samples. For each sample, 100 random fiber diameters were assessed and the mean was obtained. All statistical graphs were plotted using Origin software (OrginLab, USA).

2.5. Fourier transform infrared spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used to determine the presence of materials, chemical interactions and material stability of electrospun ZP fibers. The KBr pellet pressing method was used to prepare all samples. Here, 2 mg of pure ZP powder, ZP/REO electrospun fibers and 2 μL of REO were dispersed in 200 mg of KBr medium, individually, by grinding in a mortar. The resulting mixtures were then compressed into transparent pellets (pressure ~20 MPa). The pellets were then scanned with FTIR (IR Affinity 1, Shimadzu, Japan). A resolution of 4 $cm^{-1}$was used and spectra were obtained in the range ~4000 to 400 cm^{-1}. Each spectrum was acquired from 20 scans.