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Optimization and preparation of nanocapsules for food applications using two methodologies

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This study investigated preparation of nanocapsules (NCs) containing food-grade ingredients using two experimental designs: a one-factor-at-a-time method (OFATM) and an optimization method (OM). Response surface methodology (RSM) was used to optimize the process. The variables explored were concentration and type of polymer wall, using polycaprolactone (PCL) and polyethylene glycol–polybutylene adipate–polyethylene glycol (PEG–PBA–PEG) (1.0–4.0 mg) polyester triblock copolymer; food oil, using olive and avocado oil (0.5–2.0 mg); solvent, using acetone and ethyl acetate (6–12 ml); and surfactant concentration, using Tween 80 and Tween 60 (1–5 mg). The optimum conditions to obtain NCs were found to be 2.0 mg of PCL and 1.65 mg of PEG–PBA–PEG, olive oil (0.5 and 0.88 mg), acetone (6 and 10.25 ml), and Tween 60 (3.0 and 4.25 mg), with 90.9 and 71.9 nm for OFATM and OM, respectively. This research was conducted to investigate the use of NCs in the manufacture of fruits.

1. Introduction

Food-grade oils have a wide range of application in various commercial industries that manufacture and distribute such products as foods and beverages, nutritional supplements, cleaning agents, and fragrances. These oils are used in vitamins, coloring, and flavoring (Wang, Auty, & Kerry, 2010). Avocado and olive oil in particular, which contain many trace elements, vitamins, and minerals, offer a natural option for detoxification. Globally, more and more people, recognizing the detrimental effects of modern stress, pollution, and processed foods, are searching for natural means of cleansing their systems. Avocado and olive oil are ideal adjuncts to detox programs because of their ability to help the body deal with daily stresses and strains (Esmaeili, Saremnia, Koohian, & Rezazadeh, 2011).

Recently, considerable interest has surrounded the use of NCs as food carriers. The NCs designed for food delivery can be loaded with nanoparticles synthesized from the oils of vegetables and other foods. In general, these fragile compounds are better preserved from biodegradation occurring in a biological medium when they are entrapped in a nanocarrier. In this process, the incorporation of the food should be done during the preparation of the NCs (Vauthier & Bouchemal, 2009). Biocompatible polymers are receiving a lot of interest in the preparation of NCs used in association with biological activity due to their applications in delivery of nutrients and medicines (Donsì, Annunziata, Sessa, & Ferrari, 2011; Harris, Lecumberri, Mateos-Aparicio, Mengíbar, & Heras, 2011).

NCs are fluid-filled sacs in which food materials or drugs are contained by a polymer shell (Shahidi & Han, 1993). NCs synthesized with food-grade oil are widely used for preserving and storing food, serving to enhance the safety of foods by protecting them from external toxins and other hazards. NCs can also prove useful in the formulation of foodstuffs with appealing colors and textures (Esmaeili & Saremnia, 2012). In addition, NCs with different chemical and physical properties make them very attractive systems for many industrial food applications. For example, nanoemulsions can be used as food-oil delivery systems, in cosmetics, in agrochemicals, and as polymerization reaction media (Wang, Li, Zhang, Dong, & Eastoe, 2007). We have recently reported on in vivo and in vitro studies involving preparation of NCs for medical plant delivery using an emulsification–diffusion method (EDM) (Esmaeili & Niknam, 2014). Use of an EDM for encapsulation of food and plant drugs is based on the formulation of an oil-in-water emulsion by creating a homogeneous mixture containing the food or drug, a solvent, and water with another mixture containing a polymer, solvent, stabilizer, and water. When water is added to the emulsion the solvent diffuses into the external phase, and NCs form (Quintanar-Guerrero, Allemann, Fessi, & Doelker, 1998). The use of an EDM to fabricate NCs is a viable means of

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incorporating food-grade materials as part of a harmless and safe nanosystem. NCs containing an oil core can be used to carry anti-bacterial or antioxidant agents, and also can be used for delivery of target drugs requiring capsulation (Lertsutthiwong, Noomun, Jongaroonngamsang, Rojsitthisak, & Nimmannit, 2008). In one such application, Esmaeili, Rahnamoun, and Sharifnia recently investigated NCs prepared with *Crataegus azarolus*, which when used as a drug carrier has hypotensive effects and also acts as a direct and mild heart tonic (Esmaeili, Rahnamoun, & Sharifnia, 2013). The purpose of this research was to investigate the ability of the EDM to process food-grade oils and to find a suitable polymer membrane and food oil for NCs. Two experimental designs, OFATM and OM, were employed. RSM, which is effective in relating responsive and independent variables, was used to optimize the conditions (Baş & Boyacı, 2007; Lee, Yoo, Jun, Ahn, & Oh, 2010). The amount and type of polymer (PEG–PBA–PEG or PCL), oil (avocado or olive), solvent (acetone or ethyl acetate), stabilizer, and surfactant were investigated for each of the two methods employed (Fig. 1).

2. Methods and materials

2.1. Materials

Synthesized PEG–PBA–PEG and PCL (Mw ~ 80 kDa, ρ = 1.147 g/cm^3 at 25 °C) were purchased from Sigma–Aldrich (USA). Tween 60 and Tween 80 (Mw 26 kDa) were obtained from BASF, Mexico. HPLC-grade ethyl acetate (EA) and analytical-grade acetone were supplied by Sigma–Aldrich, France. Avocado oil, olive oil, and pure water were obtained locally.

2.2. Plant material

Avocados were collected in August 2013 from north of Tonekabon, Mazandaran, Iran. Voucher specimens were deposited at the Herbarium of the Research Institute of Forests and Rangelands, Tehran (Code 5563) (Esmaeili & Niknam, 2014).

2.3. NCs synthesized employing OFATM and OM

The NCs were synthesized using EDM. Varying types and quantities of the following experimental components were examined using OFATM and OM respectively: polymers—PCL (1.0–2.5 mg) and PEG–PBA–PEG (0.5–4.0 mg); food oil—olive oil (0.5–2.0 mg) and avocado oil (0.5–2.0 mg); solvent—acetone (6–12 ml) and acetone combined with ethyl acetate (5.5–10.5 ml); surfactant—Tween 80 (1–5 mg) and Tween 60 (2–5 mg). The optimal conditions for synthesizing NCs, under which particles sizes of 90.9 and 71.9 nm were obtained, were determined under the two experimental methods to be PCL (2 mg) and PEG–PBA–PEG (1.65 mg), olive oil (0.5 and 0.88 mg), acetone (6 and 10.25 ml), and Tween 60 (4.25 mg) (Figs. 2a and 2b). In the organic phase, the PCL/PEG–PBA–PEG and olive oil were dissolved in acetone. The resulting organic solution was added drop by drop using a rotary machine set at 400 rpm (50 °C) to distilled water containing 3 mg Tween 80. After exposing the solution to ultrasound, it was diluted with deionized water, and after that acetone was removed under a vacuum and the water was removed by freeze drying. The final sample was then filtered and centrifuged (Esmaeili & Niknam, 2014).

2.4. Screening design for qualitative parameters

Minitab 16 statistical software and particle size (PS) were used to establish the screening design for the qualitative parameters. The variable data were oil (avocado vs. olive), polymer (PCL vs. PEG–PBA–PEG), and solvent (acetone vs. EA) (Tables 1a and 1b). All experiments in the model were repeated at least twice. The graphs of analysis of variance (ANOVA) and normal residuals showed no unusual variations, indicating that errors of measurement did not occur. The evaluation of the ANOVA demonstrated interaction *among the* oil, solvent, and polymer ($P \leqslant 5\%$). The study revealed that the type and proportions of oil, solvent, and polymer used can have significant effects on PS. The screening design identified olive oil, PEG–PBA–PEG, acetone, and Tween 60 as the optimum components for producing a small PS.

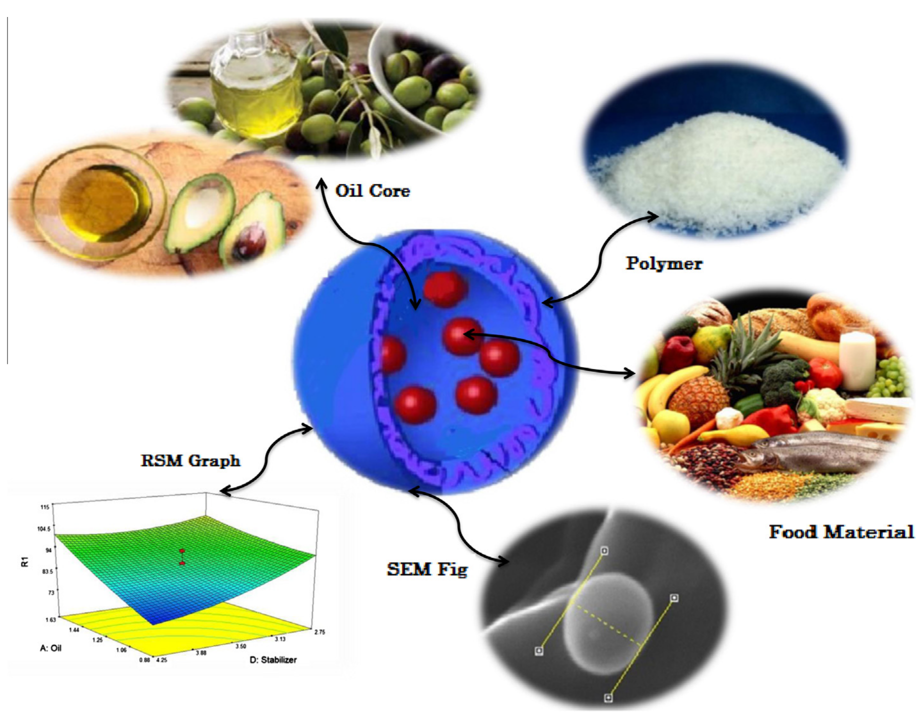

Fig. 1. Schematic formation of nanocapsules.