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Nano-emulsification of orange peel essential oil using sonication and native gums

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ABSTRACT

Essential oils are widely used in food and pharmaceutical industries where they encounter major concerns more likely insolubility and instability. Therefore, using the response surface methodology, the influence of ultrasonication conditions as well as native gums on the mean droplets diameter (*Z*-average value), polydispersity index (PDI), and viscosity of the orange peel essential oil (OPEO) nanoemulsions were evaluated. In addition, the flow behavior and stability of selected nanoemulsions was assessed during storage at different temperatures. Results showed that the optimum conditions for producing OPEO nanoemulsions (12.68 nm) were determined as 94% (sonication amplitude), 138 s (sonication time) and 37 °C (process temperature). Moreover, the soluble fraction of Persian and tragacanth gums at low concentration showed significant effect on stability, particle size, and rheology. In addition, the flow behavior of produced nanoemulsions was Newtonian, and the effect of storage conditions (time and temperature) on the *Z*-average value was highly significant ($P < 0.0001$).

1. Introduction

Nowadays, due to increased level of knowledge, most consumers prefer foods without synthetic chemicals. Accordingly, the use of natural aromatic compounds and flavors such as essential oils in food products is of utmost importance (Teixeira, Andrade, Farina, & Rocha-Leao, 2004). Orange peel essential oil (OPEO) is among the most common important essential oils used in food, cosmetics, and pharmaceutical industries. The OPEO normally includes limonene (94%), myrcene (2%), linalool (0.5%), octanal (0.4%), decanal (0.4%), neral (0.1%), geraniol (0.1%) and some others (Ashurst, 1999). Moreover, the most constitutional components of OPEO such as aldehydes (octanal, decanal), alcohols (linalool), esters and terpenoids (limonene) are low molecular weight compounds (Fisher & Scott, 1997), making the essential oil more volatile. In addition, most of these compounds are highly lipophilic, therefore they have lower solubility in aqueous and aqueous sugar-containing beverages due to lower hydrogen bonding (Ashurst, 1999).

The popularity of OPEO is due to its pleasant aromatic scent as well as facilitating its acceptance by the individuals to benefit from its therapeutic properties (Duke, Bogenschutz-Godwin, Cellier, & Duke, 2002).

Due to their particular aromas and low costs, the use of such compounds has always been considered by food industries, but their applications have been associated with problems such as lack of compatibility and solubility in most food environments, volatility and instability during processing and storage. Therefore, finding some methods to increase the stability of these essential oils in food environments as well as its controlled release when needed is a turning point in the production, trade and use of aromatic compounds and flavors in foodstuffs and other non-food products. In this regard, the emulsion technology seems to be one of the most essential processes to enhance their solubility, nanocapsulation, and protection (Peter & Given, 2009).

Nanoemulsions are a group of emulsions that have droplet sizes mostly smaller than 100 nm, and have been used for many industrial applications due to high kinetic stability, low viscosity, and high transparency, which are not formed spontaneously (McClements, 2005; Solans, Izquierdo, Nolla, Azemar, & Garcia-Celma, 2005).

In emulsion systems, properties such as stability, rheology, appearance, color, and texture depend on the size of emulsion

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droplets and particle size distribution (Gutierrez et al., 2008; Tadros, Izquierdo, Esquena, & Solans, 2004). The characteristics of emulsions are also dependent upon the techniques used to prepare the emulsion and emulsification process. There are also many techniques for producing nanoemulsions, each of which with its own advantages and disadvantages generating emulsions with different properties (Gutierrez et al., 2008; Qian & McClements, 2011; Silva, Cerqueira, & Vicente, 2012; Tadros et al., 2004).

As mentioned earlier, nanoemulsions are unbalanced systems which are not formed spontaneously. Therefore, some kinds of energy (mechanical or chemical) are required for their formation (Gutierrez et al., 2008; Silva et al., 2012; Solans et al., 2005; Tadros et al., 2004). As a result, their optimization would be achievable through optimizing the composition and variables related to the preparation methods and conditions. In addition, the ultimate objective in optimizing is often to reach the minimum droplet size, minimum polydispersity, and maximum stability (Gutierrez et al., 2008; Tadros et al., 2004).

In the food industry, nanoemulsions are usually produced using high-energy emulsification methods, such as high pressure homogenization, microfluidization, and high intensity ultrasonication (Rao & McClements, 2011a). Among these, the use of ultrasonication to produce nanoemulsions is a recent development (Li & Chiang, 2012). Lower energy and surfactant consumption, smaller droplet size, lower polydispersity, and higher stability of nanoemulsions are among the major advantages of this technique over the other methods (Kentish et al., 2008; Li & Chiang, 2012; Tadros et al., 2004). It should be noted that in the production of nanoemulsions using ultrasonication, some variables such as amplitude, sonication time, as well as temperature can be effective upon the characteristics of nanoemulsions (Kaltsa, Michon, Yanniotis, & Mandala, 2013; Kentish et al., 2008; Li & Chiang, 2012; Mirmajidi Hashtjin & Abbasi, 2014).

Moreover, the type and concentration of the components of emulsions play significant roles in determining their characteristics. Therefore, to improve the physical and rheological properties and stability of nanoemulsions, selecting the type of compounds and their proportions in the formulation is an important matter. Water, oil phase and emulsifier form the basic structure of nanoemulsions (Silva et al., 2012). In this regard, the type and concentration of emulsifier are the most important factors affecting nanoemulsion systems. There are a number of diverse emulsifiers such as Spans and Tweens which just play the role of emulsifier (Marie, Perrier-Cornet, & Gervais, 2002), and some such as gums, modified starches and milk proteins which have both emulsifying and stabilizing roles (Dickinson, 2009; Mohan & Narsimhan, 1997).

On the other hand, because of the willingness and propensity of consumers to reduce food additives in formulated food products, recently the feasibility of replacing additives with natural ingredients has become especially important. In this regard, it has already been reported that native gums (*e.g.* gum tragacanth and Persian gum) can function as emulsifier and emulsion stabilizer (Abbasi & Mohammadi, 2013; Abbasi & Rahimi, 2014; Azarikia & Abbasi, 2010), therefore, they might be able to improve some properties of nanoemulsion systems as well.

Creaming, flocculation and coalescence are the most common examples of emulsion instability during storage. It is known that the presence of hydrocolloids in an emulsion strongly influence emulsion stability (Garti & Leser, 2001; Kaltsa et al., 2013). Meanwhile, the presence of an appropriate amount of hydrocolloids in the emulsions can lead to a reduction in the size of the droplets. This is possibly due to covering more interfacial area, higher rate of surface coverage and lower rate of droplet collisions because of the increase in emulsifier concentration and continuous phase viscosity. All these reasons will lead to a lower re-coalescence and consequently, smaller emulsion droplet size (Jafari, He, & Bhandari, 2007; Qian & McClements, 2011). Preliminary results obtained during course of this investigation showed that a proper concentration of soluble phases of gum tragacanth and Persian gum individually and in combined forms had a favorable effect on certain characteristics such as reducing the mean droplet size and PDI. Consequently, the aims of this study were to investigate the effect of different sonication conditions as well as the presence of native gums on some physical and rheological properties of OPEO nanoemulsions using response surface methodology (RSM).

2. Materials and methods

2.1. Materials

Natural orange peel essential oil (OPEO), without any purification, was supplied by a local manufacturer (Giah Essance, Gorgan, Iran). Tween 80 was purchased from Merck Chemicals Company (Darmstadt, Germany). Persian gum (PG) and gum tragacanth (GT) were bought from a local herbal store. Sodium azide (analytical grade) was purchased from Sigma Chemicals Company (St. Louis, USA). Deionized water (Electrical resistivity = 18 MΩ·cm) was used for the production of nanoemulsions.

2.2. Preparation of gum tragacanth and Persian gum powder

Gum tragacanth ribbons (GT) as well as Persian gum hunks (PG) were pulverized using domestic grinder (Moulinex, France), and after passing through a number of sieves, the powders (mesh size < 60) were collected and kept in a closed container (Abbasi & Mohammadi, 2013; Azarikia & Abbasi, 2010).

2.3. Preparation of PG and GT dispersions

In order to prepare dispersions, powders (PG 3, GT 0.5% w/w) were weighted (Tecator, Swiss) and gradually added to a beaker containing deionized water on a magnetic stirrer (Heidolph-MR 3001, Germany) to obtain uniform dispersions. Sodium azide (0.004 wt %) was added to the dispersions as an antimicrobial agent. Afterwards, to assure fully hydration, the dispersions were incubated in water bath (Kottermann, Germany) for 30 min at 50 °C, and were then kept for 24 h at ambient temperature (Abbasi & Mohammadi, 2013; Azarikia & Abbasi, 2010).

2.4. Separation of soluble and insoluble fractions of PG and GT

At this step, the dispersions were transferred to 50 ml plastic tubes. Then, the soluble and insoluble fractions were separated by centrifugation (Sigma, model K 30-3, Germany) at 20,000 g for 20 min at 25 °C. Next, the fractions were manually separated, and after weighting the phases, the amount of their dry matter was determined by oven (EHRET, model TKL 4105, Germany) at a temperature of 105°C (Abbasi & Mohammadi, 2013; Azarikia & Abbasi, 2010).

2.5. Preparation of nanoemulsions

The oil-in-water (O/W) nanoemulsions were prepared using OPEO (1% w/w), as the oil phase, and mixture of Tween 80 (2% w/w), combined soluble fractions of PG (SFPG) and GT (SFGT) (0.25% w/w) and deionized water (96.75% w/w), as the aqueous phase. So that, formulation of all samples was as follows: 1% OPEO + 2% Tween 80 + 0.25% gum (SFPG:SFGT, with a mixing ratio of 75:25). All emulsions were prepared through a two-stage process. At first, the oil and aqueous phases (total of 100 g) were placed in a glass