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# Optimization of nanoemulsions processed by high-pressure homogenization to protect a bioactive extract of jackfruit (*Artocarpus heterophyllus* Lam)

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ABSTRACT

The present work investigates the development of an eco-friendly nanoemulsion to maintain the bioactivity of a rich in carotenoids extract from jackfruit (*Artocarpus heterophyllus* Lam) pulp. Firstly, the influence of the sucrose monostearate (SMS) content (0.5–2%, w/w), miglyol content (5–20%, w/w) and homogenization pressure (400–800 bar) on the droplet size distribution, rheology and stability of emulsions processed by high-pressure homogenization was studied. Except two formulations containing the highest SMS content and processed at the lowest pressure, stable fluid submicron emulsions were obtained. Then, a jackfruit pulp extract was incorporated in the oily phase of the selected emulsion (5% miglyol plus 0.5% SMS processed twice in a high-pressure homogenizer at 800 bar). The antioxidant activity of the jackfruit extract loaded in the selected emulsion stored at 4 °C has exhibited a longer stability compared to the same extract only dissolved in miglyol and stored in same conditions.


## 1. Introduction

Native to the Southwestern part of India, *Artocarpus heterophyllus* Lam, known as jackfruit tree, was recently introduced and grown in the Mexico's Nayarit state to satisfy the growing world demand observed in recent years for this exotic fruit. Currently, most of the jackfruit production in Mexico is intended for export to USA as fresh or with limited processing (Kader & Siddiq, 2012). However, it would be attractive to develop applications from jackfruit pulp with a higher value added and taking advantage of its functional properties. The keen interest of consumers, food manufacturers and researchers is mainly due to the presence in jackfruit pulp of a wide range of biochemical compounds, recognized for their important functions and physiological actions in human health (Baliga, Shivashankara, Haniadka, Dsouza, & Bhat, 2011; Swami, Thakor, Haldankar, & Kalse, 2012). Among the bioactive compounds from jackfruit pulp, eighteen carotenoids have been identified and quantified, including all-*trans*-lutein, all-*trans*-β-carotene, all-*trans*-neoxanthin, 9-*cis*-violaxanthin and 9-*cis*-neoxanthin (de Faria, de Rosso, & Mercadante, 2009). Besides, some extracts from jackfruit pulp, rich in bioactive molecules, have shown promising biological activities (Jagtap, Panaskar, & Bapat, 2010; Ruiz-Montañez et al., 2015). Particularly, Ruiz-Montañez et al. (2015) have evaluated the antimutagenic and antiproliferative activity of several fractions of jackfruit pulp extract and highlighted a fraction, extracted with hexane and partitioned with methanol, exhibiting a high biological activity. Particularly, this fraction was characterized by a high content of carotenoids suggesting potential applications in pharmaceutical, food or cosmetics areas.

Carotenoids are a large family of pigmented fat-soluble compounds. They are known to have functional benefits on human health reducing the incidence of certain cancers and eye diseases, and probably playing a role in the prevention of cardiovascular and metabolic disorders (Johnson, 2010; Rao & Rao, 2007). However, carotenoids are sensitive to light, oxygen and/or heat and most of them display a very low aqueous solubility and a poor bioavailability as crystalline form (Bradley & Min, 1992; Fernández-García et al., 2012). Nanoencapsulation constitutes a promising strategy to preserve their properties over time, during processing and improve their bioavailability (Gutiérrez et al., 2013; Joye, Davidov-Pardo, & McClements, 2014). Among the various nanoparticle-based systems suitable for encapsulation of bioactive compounds, oil-in-water (O/W) nanoemulsions have been studied in the food, pharmaceutical and cosmetic industries to encapsulate lipophilic molecules (Cornacchia & Roos, 2011; Troncoso, Aguilera, & McClements, 2012; Yuan, Gao, Zhao, & Mao, 2008). O/W nanoemulsions are thermodynamically unstable systems formed with submicron oil droplets ($d < 200$ nm) dispersed within an aqueous continuous phase, each oil droplet being surrounded by a protective coating of emulsifier molecules (Acosta, 2009; Mason, Wilking, Meleson, Chang, & Graves, 2006). High Pressure

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Homogenization (HPH) is one of the high-energy mechanical methods to process O/W nanoemulsions, particularly suitable for continuous production of finely dispersed emulsions (Dumay et al., 2013; Gutiérrez et al., 2013; Troncoso et al., 2012). The stability, physicochemical properties and functional performance of nanoemulsion-based delivery systems can be controlled by modifying the composition and preparation conditions to produce emulsions with different particle size distributions and/or interfacial properties (Lesmes & McClements, 2009; McClements, 2010).

The nature and concentration of the surfactant are important elements for the formulation of stable nanoemulsions. In recent years, there has been a growing interest in using sucrose ester (SE) as emulsifier agent. These non-ionic, food-grade, surface-active agents are biocompatible, with an excellent biodegradability and a low toxicity (Savic, Tamburic, & Savic, 2010; Szuts & Szabó-Révész, 2012). They are considered as environmentally friendly agents since they are produced from natural products (sucrose and vegetable oil). Several studies have shown that SE can be used alone or as co-surfactant to stabilize oil-in-water nanoemulsions (Choi, Kim, Cho, Hwang, & Kim, 2009; Henry, Fryer, Frith, & Norton, 2009; Klang, Matsko, Raupach, El-Hagin, & Valenta, 2011). Rao and McClements (2011) have processed by high-pressure homogenization a mix of 10% (w/w) lemon oil plus various sucrose monopalmitate content (1–20%, w/w) dispersed in 10 mM sodium dihydrogenophosphate buffer (pH 7). They obtained stable nanoemulsions at low surfactant-to-oil ratios (1% sucrose monopalmitate) during storage at 5 °C and 23 °C up to one month but exhibited extensive particle growth/aggregation at pH lower than 6 and higher than 7 and coalescence during storage at 40 °C.

The main objective of the present investigation was firstly to optimize the process of an environmentally friendly submicron emulsion by high-pressure homogenization. For that purpose, a screening of the influence of the homogenization pressure, content of oil and sucrose ester on the emulsion characteristics (size distribution of oil droplets, viscosity and stability during storage at 4 °C and 20 °C) was carried out. Then, the O/W submicron emulsion displaying a high stability over time and a suitable viscosity was selected to encapsulate the rich in carotenoids jackfruit extract and evaluate the protection of its antioxidant activity during storage at 4 °C up to eight weeks.

## 2. Materials and methods

### 2.1. Solvents and reagents

Methanol, potassium persulfate, ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) and Trolox ((±)-6-Hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid) were from Sigma-Aldrich (Saint-Quentin Fallavier, France). Hexane was purchased from VWR International (Fontenay sous Bois, France). Miglyol 812 was from Sasol (Witten, Germany). Sucrose monostearate (SE, HLB 15) was purchased from Stéarinerie Dubois (Boulogne Billancourt, France). Milli-Q water (Millipore®) was used to prepare all emulsions and solutions during the study.

### 2.2. Raw material

Jackfruits (*Artocarpus heterophyllus* Lam) were manually harvested in orchards in the state of Nayarit (Mexico) at commercial maturity (16°Brix). Fruits were subsequently sorted and washed before peeling and then the pulp was recovered. Pulp samples were immediately frozen at −70 °C and then freeze-dried using a FreeZone 4.5 freeze dryer (Labconco, Kansas City, Missouri, U.S.). The freeze-dried pulp was then stored at −20 °C.

### 2.3. Preparation of jackfruit pulp extract

A two-step process of extraction was carried out as described previously by Ruiz-Montañez et al. (2015). All the process was performed in darkness to avoid degradation of sensitive compounds. A 50 g sample of freeze-dried jackfruit pulp was extracted with three parts of hexane (w/v). The mixture was sonicated for 30 min at a constant frequency of 42 kHz at 20 °C in a sonicator (Bransonic 220, Branson). The mixture was then filtered through Whatman No. 1 filter paper under vacuum and the filtrate was evaporated under reduced pressure at 40 °C to dryness. Then, a partition step was carried out by dissolving the previous dry extract in methanol-hexane (2:3 v/v) under sonication for 30 min at 20 °C. The immiscible phases were separated in a separating funnel for 3 h at 4 °C. The methanolic extract was then evaporated under reduced pressure at 40 °C and redissolved in methanol before drying under nitrogen. The final dry extract was stored in amber vial at −20 °C until use.

### 2.4. Emulsion preparation

Sucrose monostearate (SMS) dispersions were prepared by dissolving different amounts of SMS in milli-Q water by gentle magnetic stirring for 30 min at ambient temperature. Dispersions were then stored overnight at 4 °C. Oil-in-water (O/W) emulsions were prepared using Miglyol (density 0.940 g/ml, viscosity 28 cP) as lipophilic dispersed phase and SMS dispersion as continuous phase.

Table 1 synthesizes the miglyol and SMS concentrations and the homogenization pressure applied to prepare the nine emulsion formulations tested in the screening step according to a full factorial design completed with centerpoint replication. Emulsion preparation involves two subsequent steps. Firstly, pre-emulsion (or coarse emulsion) was prepared for the different formulations (Table 2) by mixing the SMS solution with miglyol, both equilibrated at 24 °C, using a Silverson emulsifier (model L4RT, Silverson Machines LTD, Chesman, UK) at 5000 rpm for 15 min. As an increase by 4 °C of the coarse emulsion temperature was observed at the end of mixing (final temperature of pre-emulsions $\approx 26 \pm 1$ °C), coarse emulsion was then equilibrated at 24 °C before the following step. Then, pre-emulsion was processed using a high-pressure homogenizer (HPH) (Niro Panda 2K, GEA, France) at different pressure levels (Table 1). The HP-homogenizer was equipped with a high-pressure valve (HP-valve) and a low-pressure valve (LP-valve). In this study, the pressure at the LP-valve was adjusted at 10% of the HP-valve pressure and the processing pressure (P) corresponds to the sum of the pressures at the both valves. The flow rate was ~4 l/h at 800 bar. A cooling device was placed just after the outlet of the HP-homogenizer to limit the increase of the emulsion temperature. To produce the emulsion loaded jackfruit extract, a final jackfruit dry extract prepared as described above was dissolved in miglyol (0.01%, w/w) and gentle stirred few minutes just before processing emulsion taking care to always keep sample in darkness.

### 2.5. Emulsion stability by centrifugation

The stability of emulsions processed by HPH was evaluated by centrifugation of 10 ml samples for 5 min at 2000 ×*g* at ambient temperature (Centrifuge B4i, Thermo Electron).

**Table 1**
The experimental and coded levels of the variable parameters in the screening study: oil content, sucrose monostearate content and homogenization pressure level.

| | | Applied levels | | |
|---|---|---|---|---|
| Variables | Symbol | −1 | 0 | +1 |
| Oil content (%, w/w) | O | 5 | 12.5 | 20 |
| SMS content (%, w/w) | SMS | 0.5 | 1.25 | 2 |
| Pressure homogenization (bar) | P | 400 | 600 | 800 |