



Original Article

Improved green coffee oil antioxidant activity for cosmetical purpose by spray drying microencapsulation



Anna B.F.L. Nosari^a, Juliana F. Lima^b, Osvaldo A. Serra^b, Luis Alexandre P. Freitas^{a,*}

^a Faculdade de Ciências Farmacêuticas de Ribeirão Preto, Universidade de São Paulo, Ribeirão Preto, SP, Brazil

^b Faculdade de Filosofia Ciências e Letras de Ribeirão Preto, Universidade de São Paulo, Ribeirão Preto, SP, Brazil

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ABSTRACT

The oil extracted by cold pressing unroasted coffee beans, known as green coffee oil, has been widely used for cosmetic purposes. The objective of this work was to prepare and characterize microcapsules containing green coffee oil and to verify its antioxidant activity under the effect of light, heat and oxygen. The encapsulating material was arabic gum and the microcapsules were obtained by spray drying an oil-in-water emulsion containing green coffee oil. The characterization of the microcapsules was performed by laser diffraction, scanning electron microscopy, differential scanning calorimetry and the antioxidant activity. The antioxidant activity was determined by a modified active oxygen method with light irradiation, heating and oxygen flux. The microparticles were effectively produced by the proposed spray drying method, which resulted in green coffee oil loads of 10 and 30%. The morphological evaluation of microcapsules showed spherical shape with smooth and non-porous surfaces, demonstrating the adequacy of arabic gum as encapsulating material. Calorimetric analysis of individual components and microcapsules with 10 and 30% green coffee oil showed diminished degradation temperatures and enthalpy, suggesting a possible interaction between arabic gum and green coffee oil. The antioxidant activities for pure green coffee oil and its microcapsules with loads of 10 and 30% showed high activity when compared to the reference antioxidant α -tocopherol. Microcapsules containing 10 and 30% of oil showed 7-fold and 3-fold increase in antioxidant activity when compared to pure green coffee oil. The new method for antioxidant activity determination proposed here, which applies heat, light and oxygen simultaneously, suggests a high improvement in encapsulated green coffee oil when compared to this active alone. The results showed herein indicate a promising industrial application of this microencapsulated green coffee oil.

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Introduction

Products derived from coffee (*Coffea arabica* L., Rubiaceae) have been long used by mankind as beverages, foods and cosmetics. Most recently, the oil extracted by cold pressing the unroasted beans of coffee was introduced to the cosmetic market with great impact. This so called green coffee oil, GCO, has been studied for its activity on the skin health (Pereda, 2009; Pereda et al., 2009; Savian et al., 2011; Wagemaker et al., 2012; Chiari et al., 2014). This vegetable oil presents a unique composition and previous studies showed an expressive antioxidant activity against lipid peroxidation (Kroyer et al., 1989).

The GCO showed a dose dependent stimulation of collagen, elastin and glycosaminoglycans synthesis by fibroblasts *in vitro* (Pereda et al., 2009) besides an increased release of growth factors, TGF- β 1 and GM-CSF. Pereda et al. (2009) also found AQP-3mRNA expression 6.6 fold higher in the presence of GCO, indicating a protective effect of this oil on physiological balance of the skin. Pereda et al. (2009) also concluded that the GCO is effective against cellulitis. Although cosmetic formulations containing the GCO showed low antioxidant and antimicrobial activities *in vitro* (Wagemaker et al., 2012) there was also observed lack of toxicity *in vitro* and in clinical evaluation (Wagemaker et al., 2013). Those effects of GCO on the skin health may probably be related to its lipid fraction rich in triacylglycerols, sterols and tocopherols, as well as diterpenes of the kaurene family (Speer and Kolling-Speer, 2006), which have been previously connected to benefic actions to the skin (Nakayama et al., 2003). However, the most studied dermatological application of GCO is certainly as a photoprotection aid (Savian et al.,

* Corresponding author.

E-mail: lapdfrei@usp.br (L.A.P. Freitas).

2011; Chiari et al., 2014). A non-ionic O/W emulsion containing 3% (w/w) GCO was proposed as a topic formulation for photoprotection (Savian et al., 2011). Recently, a study of GCO as an additive to sunscreen formulation containing ethylhexylmethoxycinnamate showed a synergistic effect of this oil by increasing the sun protection factor, SPF, by 20% as compared to synthetic sunscreen alone (Chiari et al., 2014).

One of the drawbacks for the cosmetic application of vegetable oils or fats is their lipid oxidative stability (Ramalho and Jorge, 2006) since the unsaturated waxy acids may undergo photo oxidation, thermal oxidation, auto oxidation and enzymatic oxidation. GCO photo oxidation may be a limiting factor especially for sunscreen applications and the use of synthetic antioxidants are subject to many formulation and regulatory aspects of topical administration. Microencapsulation is an effective way to protect these materials, as well as other components, like the diterpenes, against lipid oxidation (Pu et al., 2011; Jimenez et al., 2006) and other environmental factors. According to Costa et al. (2007) many studies have demonstrated the use of microparticles to reduce toxicity and increase the efficiency of active substances.

There are many techniques that can be applied for the production of microparticles, including the spray drying, spray cooling and fluidized bed (Pu et al., 2011). Among the many techniques, the spray drying has caught attention for plant extracts and oils (Jafari et al., 2008; Couto et al., 2013a,b; Peixoto and Freitas, 2013; Porto et al., 2013) due to its many advantages (Oliveira and Petrovick, 2010). There are also many materials that can be used as encapsulating agents such as gums, waxes and polymers (Oliveira and Petrovick, 2010; Couto et al., 2013a,b; Peixoto and Freitas, 2013; Porto et al., 2013). Arabic gum is noted for presenting excellent emulsifying properties and is widely used for the retention and protection of oil (Jimenez et al., 2006; Jafari et al., 2008). They are widely used for controlled release of active and have good stability in variations of pH and moisture levels in addition to being biocompatible (Jafari et al., 2008; Ranjha et al., 2010).

Thus, the objective of this study was to prepare microparticles by the technique of spray drying containing GCO and using AG as the wall forming material. In addition important characteristics of microparticles such as the morphology, thermal behavior and photocatalytic activity were studied. Possible interactions between the GCO and AG were evaluated by differential scanning calorimetry, DSC.

Material and methods

GCO microcapsules preparation

Materials

Arabic gum powder analytical grade batch number 144617 was supplied by Labsynth Ltda (São Paulo, Brazil). The green coffee oil brand name 'Melscreen Coffee' (Chemunion Química Ltda, Sorocaba, Brazil) batch number CN102-0811 was purchased from Distriol Comércio de Insumos Ltda (São Paulo, SP). The DL- α -tocopherol acetate cosmetic grade (Zhejiang Medicine Co, China) batch number 20120615 with 99.6% purity was supplied by Viafarma Ltda (São Paulo, Brazil). The castor oil fatty acid Acros Organics BVBA containing 85% ricinoleic acid (12-hydroxy-oleic acid) was purchased from Janssen Pharmaceutica (Geel, Belgium).

Microencapsulation

AG was dissolved in Milli-Q (EMD Millipore, Billerica, MA, USA) water (1:2 w/w) under magnetic stirring at 250 rpm and at room temperature ($25 \pm 2^\circ\text{C}$) 24 h before the preparation of the emulsion for drying. The emulsions were prepared from the aqueous

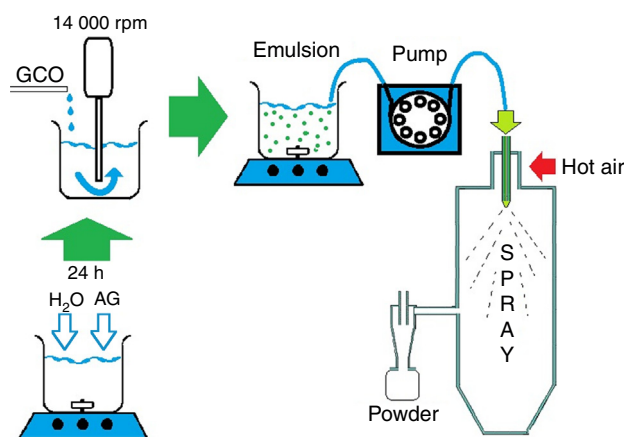


Fig. 1. Sequence of steps during GCO microcapsules preparation.

solution of AG by incorporating GCO in concentrations of 10 and 30% (w/w) relative to AG. The emulsion was then prepared using a high shear homogenizer Turratec Te-102 (Tecnal Ltda, Piracicaba, Brazil) was used at 14,000 rpm for 5 min at room temperature. After the preparation the emulsions were readily spray dried.

The drying process of the emulsion was performed using a laboratory scale spray dryer model MSD 0.5 (Labmaq Ltda, Ribeirão Preto, Brazil). The emulsion was atomized by a pneumatic spray nozzle in the drying chamber and the microparticles were separated by a cyclone and collected in a flask. The following drying conditions were kept constant during the experiments: emulsion feed rate 6 ml/min; drying air flow rate $1.25 \text{ m}^3/\text{min}$; atomization pressure 6 bar, atomizing air flow rate 50 ml/min; inlet and outlet drying air temperature 140 and 100°C , respectively. Fig. 1 depicts the sequence of steps during GCO microcapsules preparation.

Morphology

The microcapsules were poured on a stub and coated with gold in a Bal-Tec sputter coater. Microparticles morphology was observed by Scanning Electron Microscopy, SEM, using a microscope XL30-TMP NO and FEG XL 30 (Phillips Co., Netherland).

Thermal analysis

Samples (5 mg) were placed in aluminum pans and heated to 420°C at a rate of $10^\circ\text{C}/\text{min}$ under a nitrogen flux of 10 ml/min. DSC measurements were performed using a DSC-50 (Shimadzu Corp., Kyoto, Japan).

Photocatalytic activity

The samples were analyzed by an Active Oxygen Method, AOM, adapted from the conductometric technique Rancimat[®] (Lima et al., 2009; Nosari, 2012; Lima and Serra, 2013). The experimental assembly is shown in Fig. 2. Castor oil (3 ml) was placed in the flask 2A (Fig. 2) and 40 mg sample of alpha-tocopherol, GCO or microcapsules was added. This mixture was submitted to constant stirring at 120°C and irradiation of light by a xenon lamp Xenarc D-H4R of 35W (Osram GmbH, Munchen, Germany). The volatile degradation compounds formed during photo-oxidation are dragged by a controlled flux of air to the flask 2B (Figure 2) containing 17 ml of Milli-Q (EMD Millipore, Billerica, MA, USA) water, where the conductivity is measured by a conductimeter model C708 (Analion Ltda, Ribeirão Preto, SP, Brazil). In this method the generation of volatiles species by oxidation under light, airflow and heat was evaluated for castor oil, CsOil, employed as reference, and also for CsOil containing GCO, Vitamin E and microcapsules prepared by spray drying with GCO concentration of 10 and 30% GCO.

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