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Microencapsulation of Sacha Inchi (*Plukenetia huayllabambana*) Oil by Spray Drying with Camu Camu (*Myrciaria dubia* (H.B.K.) Mc Vaugh) and Mango (*Mangifera indica*) Skins †

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Abstract: Sacha inchi (*Plukenetia huayllabambana*) oil was microencapsulated by spray drying with gum arabic and with extracts of camu camu (*Myrciaria dubia* (HBK) Mc Vaugh) and mango (*Mangifera indica*) skins, obtained by assisted microwave. The physicochemical characteristics, such as moisture content, encapsulation efficiency, particle size, morphology, fatty acid composition and oxidative stability, were evaluated in order to select the best formulation for the development of functional foods. The most important results indicate that the microcapsules formulated with extracts of the fruit skins provide greater protection to sacha inchi oil (*P. huayllabambana*) against oxidation compared to commercial antioxidant BHT (Butylated Hydroxytoluene), resulting in a slight loss of ω-3 fatty acids.

Keywords: antioxidants; microencapsulation; assisted microwave extraction; oxidative stability; *Plukenetia huayllabambana*

1. Introduction

The camu camu (*Myrciaria dubia* (H.B.K.) Mc Vaugh) and mango (*Mangifera indica*) skins shown high antioxidant activity. Camu camu is a low-growing shrub found throughout the Amazon rainforest of Peru, Colombia, Venezuela and Brazil. The camu camu fruit is mainly consumed after being processed into juices, concentrates, and for the production of vitamin C capsules. As a result, a great volume of residue of seeds, skins and pulp that represent around 40% of the fruit in weight, are generated [1]. The importance of antioxidants is crucial for health, due to its ability to neutralize free radicals, which contain one or more unpaired electrons, being responsible for many degenerative diseases. Sacha inchi *Plukenetia huayllabambana* grows in the province of Rodríguez de Mendoza, Department of Amazonas-Peru, its oil contains a higher percentage of ω-3 (55.62 to 60.42% α-linolenic acid) [2]. However, an existing problem is that, due to its chemical structure, ω-3 acids have a high susceptibility to oxidation. A technology that emerges as an alternative to delay or inhibit its deterioration is microencapsulation [3], which consists in the preparation of an oil-in-water emulsion,

containing encapsulating agents (or carriers, such as gums, fibers, proteins or carbohydrates) and their subsequent drying. This process aims to protect the poly-unsaturated fatty acids (PUFAs) from environmental factors, such as light, air or humidity. The aim of this work was to compare physicochemical characteristics and the oxidative stability of sacha inchi oil microcapsules elaborated with gum arabic and different mixtures of camu camu and mango skin extracts, in order to select the best formulation, which will allow for obtaining functional foods once the production of the microcapsules has escalated.

2. Materials and Methods

2.1. Raw Material

Cold pressed sacha inchi oil from the ecotype *P. huayllabambana* was obtained in the laboratory of *Centro de Estudios e Innovación del Alimento Funcional* (CEIAF) from the Universidad de Lima (Peru) and kept at 4 °C. Additionally, the camu camu and mango skins were washed and dried by infrared dryer (IRC DI8, Spain) at 40 °C, then ground into the food shredder (Grindomix GM200/Restch) and kept at −5 °C in polyethylene bags prior to phenolic extraction. The arabic gum (AG) was the agent encapsulant (wall material) due its versatility, good solubility, low viscosity at high concentrations and very good emulsifying properties.

2.2. Microwave-Assisted Extraction (MAE) of Polyphenols

The extraction of polyphenols from camu camu and mango dried skins were performed using a microwave oven (CW-2000, China) with ethanol:water. The optimal MAE conditions were previously determined. The resulting extracts were evaporated at 30 °C using a rotary evaporator (Büchi rotavapor R100 Labortechnic AG Switzerland).

2.3. Samples Proposed

A total amount of five samples to be analyzed was proposed, making mixtures of camu camu skin (CCS) extract, mango skin extract (MSE) with gum arabic (GA) and sacha inchi (*P. huayllabambana)* (SIPH) oil (Table 1).

Table 1. Samples of sacha inchi, *Plukenetia huayllabambana* oil microcapsules (SIPH) elaborated.

SIPH Oil Microencapsulated
SIPH + GA
SIPH + GA + CCSE[a] (220 ppm)
SIPH + GA + MSE[b] (220 ppm)
SIPH + GA + CCSE (110 ppm) + MSE (110 ppm)
SIPH + GA + BHT[c] (200 ppm)

[a] Camu camu skin extract, [b] Mango skin extract, [c] Commercial antioxidant.

2.4. Microencapsulation

The solutions were prepared with GA and distilled water containing 180 g of camu camu and mango skins extracts. Sacha inchi oil was then added at a concentration of 18% with respect to total solids. Emulsions were formed using a Silverson homogenizer L5M-A-England, operating at 9000 rpm for 10 m. The solutions were dried by spray dryer (Büchi B-290-Switzerland). Inlet and outlet air temperature were 140 °C and 70 °C, respectively, and the feed flow rate was 55 mL/min. The dried powders collected were stored in opaque hermetic bags at −5 °C for further analysis.

2.4.1. Moisture Determination

The moisture of the encapsulated samples was determined gravimetrically by drying until constant weight using halogen moisture analyzers (Sartorius MA-30, Germany), operating at 103 ± 2 °C.

2.4.2. Total Phenolic Content (TPC) and Surface Phenolic Content (SPC)

The TPC and SPC of the extract and powder was determined by the Folin–Ciocalteu method [4] with some modifications. The absorbance of the solution was measured at 760 nm using a spectrophotometer (1205 Vis Spectrophotometer UNICO). The results were expressed as μg of equivalent gallic acid (GAE) per gram of microcapsules (powder). All analyzes were done in triplicate. For the determination of the surface phenolic content (SPC), 24 mg of microcapsules were dissolved in 4.5 mL of methanol and stirred using a vortex for 1 min and then filtered through a Whatman filter paper number 2. The surface phenolic content was measured according to the same method described for TPC determination.

The percentage of efficiency of TPC microencapsulation was calculated using the following equation: Percentage Efficiency (%) = [(TPC) − (SPC)/TPC] × 100.

2.4.3. Determination of Antioxidant Activity on DPPH Radical

The determination of antioxidant activity was determined using DPPH as a free radical according the procedures described previously [5]. The absorptions of samples were then detected (Abs 517_{sample}). The percentage inhibition (% I) of free radicals was calculated using the following equation: Percentage Inhibition (% I) = [(Abs_{517} control) − (Abs_{517} sample)/(Abs_{517} control)] × 100.

2.4.4. Fatty Acid Composition

The fatty acid methyl esters (FAMEs) were prepared according to the International Union of Pure and Applied Chemistry, IUPAC [6] and the FAMEs formed were analyzed using a 7890B Agilent gas chromatograph (Agilent Technologies, Santa Clara, CA, USA) equipped with a SP2380 polar capillary column and a flame ionization detector (FID). The injector and detector temperatures were maintained at 225 and 250 °C, respectively. Hydrogen was used as carrier gas at a flow rate of 1.0 mL/min. The oven temperature was set at 165 °C and increased to 230 °C at 3 °C/min maintaining this temperature for 2 minutes. The injection volume was 1 μL.

2.4.5. Particle Size Distribution and Morphology

The particle size distribution was determined by laser diffraction spectroscopy on a Master Sizer Micro equipment (measuring range: 0.3 μm–300 μm). The average diameter of the equivalent volume or D [4,3] was informed. The photomicrographs were analyzed by a FEI scanning electron microscope, model QUANTA 250 FEG, (Hillsboro, OR, USA). Samples were previously gold sputtered with an Edwards Sputter Coater S150B (Crawley, England).

2.5. Oxidative Stability

The thermal analysis was carried out by differential scanning calorimetry (DSC), for the determination of oxidation onset temperature (OOT) according to the ASTM E2009-08 Standard Test Method for Oxidation Onset Temperature of Hydrocarbons by Differential Scanning Calorimetry.

3. Results and Discussion

According to Figure 1 the moisture content was between 3.74 and 4.32% being the highest value for the mixtures of antioxidants extracts, and the lowest values for the mango skin extract. The higher inlet (140 °C) and outlet temperature (80 °C) promoted the drying rate of droplets and resulted in low moisture content [7].

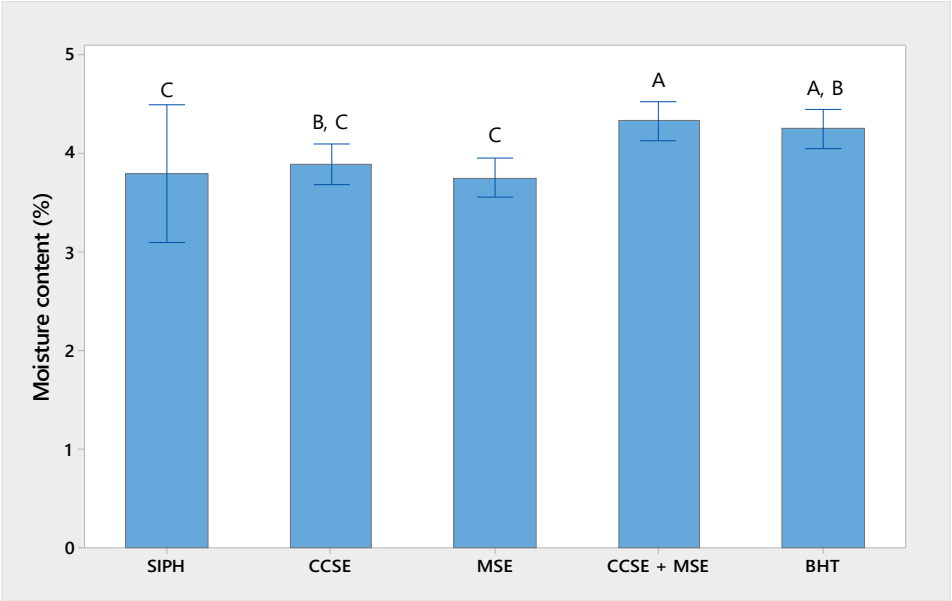

Figure 1. Moisture content (%) of sacha inchi, *P. huayllabambana* oil microencapsulated (SIPH). Different capital letters indicate significant differences ($p < 0.05$).

The total phenolic content (TPC) was found to be between 357.7 and 1677.9 μg GAE g^{-1} powder, and the highest amount of phenolic compound was obtained with camu camu and mango skin extract. There was a statistically significant difference in TPC between them. The encapsulation efficiency ranged from 90.25 to 98.28 %.

The microencapsulates showed the highest antioxidant activity (75.29 I% to 91.76 I%) and a composition very similar to the starting oil with a slight loss of omega-3 (57 vs. 58%) and a slight amount of *trans* fatty acid isomers (0.05–0.09%).

According to the particle size determination (Table 2), microcapsule diameters were between 1.6 and 20.9 μm. All samples showed a monodisperse distribution. The morphological analysis performed by SEM microscopy allowed us to distinguish rounded and concave microcapsules (Figure 2). This was an expected behavior for samples obtained by spray drying [8].

Table 2. D [4,3] values obtained from sacha inchi, *P. huayllabambana* oil microencapsulated (SIPH).

Formulation	D [4,3] μm	Span	Volume Distribution, μm		
			D(v, 0.1)	D(v, 0.5)	D(v, 0.9)
SIPH + GA	2.6 (0.1)	2.0 (6.1)	0.8 (0.1)	2.1 (0.1)	5.1 (0.1)
SIPH + GA + CCSE (220 ppm)	20.9 (1.4)	1.1 (0.1)	1.1 (0.1)	6.2 (0.1)	66.4 (0.2)
SIPH + GA + MSE (220 ppm)	1.6 (0.1)	1.4 (0.1)	0.7 (0.1)	1.4 (0.1)	2.7 (0.2)
SIPH + GA + CCSE (110 ppm) + MSE (110 ppm)	4.0 (1.4)	1.5 (0.1)	0.7 (0.1)	1.4 (0.1)	2.9 (0.2)

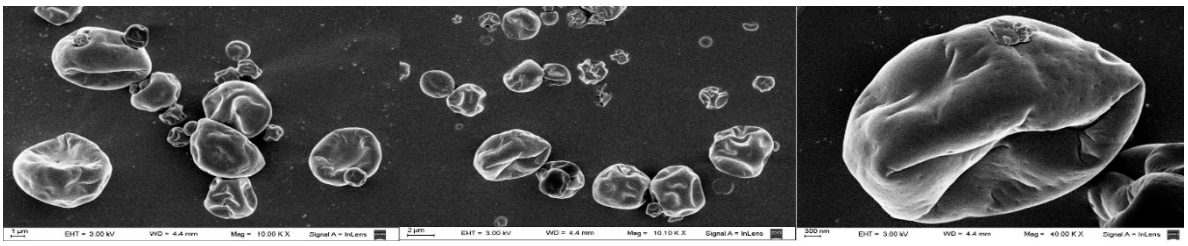

Figure 2. Morphology of microcapsule: SIPH + GA + CCSE (110 ppm) + MSE (110 ppm).

The results obtained for the oxidative stability of microcapsule with naturals antioxidants were to be between 188 to 198 °C and for the commercial antioxidant were 174 °C, thus showed that the

antioxidant extracts from natural origin provided greater protection to sacha inchi oil (*P. huayllabambana*) against oxidation.

4. Conclusions

The microcapsules of sacha inchi (*P. huayllabambana*) oil showed a low percentage of moisture content (3.74 to 4.32%), a high amount of phenolic compound (357.7 to 1677.9 μg GAE g^{-1} power) and a high encapsulation efficiency (90.25 to 98.28 %). The microencapsulates showed high antioxidant activity (75.29 I% to 91.76 I%) with a composition very similar to the starting oil with a slight loss of omega-3 (57 vs. 58%) and a slight amount of *trans* fatty acid isomers (0.05 to 0.09%). The morphological analysis allowed us to distinguish rounded and concave microcapsules, and they vary in their shape due to their chemical composition. The results obtained from OOT showed that the antioxidant extracts from natural origin provide greater protection to sacha inchi oil (*P. huayllabambana*) against oxidation. For this reason, the microcapsules can be used as a natural antioxidant in functional foods or nutraceutical products, with possible health benefits.

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