DESENVOLVIMENTO DE PARTÍCULAS VEGANAS PARA USO EM COSMÉTICOS

Palavras-Chave: goma de cajueiro, óleo essencial, coacervação complexa

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INTRODUCTION:

Cosmetic formulations are designed to externally act in the human body, by cleaning, protecting or changing its appearance. They may be grouped into 04 categories considering the main part of the body: skin care, body care, hair care, and personal care products. Currently, cosmetics have extremely high performance in delivering therapeutic effects and sensory aspects such as uniformity of color and texture. The growing demand of the consumers by natural products, clean labels, and no-animal tests products impose many challenges on the cosmetic industry, due to the difficulty of obtaining the same results with organic compounds.

Most bioactive compounds, for example, are susceptible to degradation when in contact with oxygen, light, temperature, and moisture, and may present low stability under storage conditions. On the other hand, cosmetics are known for presenting shelf lives of 1 year at room temperature. In skincare products, bioactive compounds are explored to provide antioxidant (Marangoni and Moura 2011; Zhuang et al. 2022), antimicrobial (against acne, for example)(Yang et al. 2018; Esmael et al. 2020), or healing (Agwa et al. 2022; Miastkowska et al. 2023) properties. In this context, essential oils, a complex liquid mixture with a lot of volatile and odoriferous compounds, predominantly from aromatic plants, have various biological activities and have been explored in the development of cosmeceuticals of natural origin (Carvalho et al. 2016).

Microencapsulation has been considered one of the most effective techniques to allow the incorporation of bioactive compounds in complex formulations, contributing to the stability during storage and fabrication, improving the mixture, avoiding the interaction or reaction with other compounds, providing the controlled-release delivery and improving the handling of the new ingredients (Carvalho et al. 2016).

Among the possible techniques, complex coacervation allows the stabilization of an emulsion containing the active material through a polymeric wall electrostatically complexed.The hydrogel-like structure formed might to protect the oily material in the cosmetic formulation. The goal of this project is to explore two polymer combinations for encapsulation of an essential oil mixture, aiming to be applied in topic cosmetic formulations.

Objective:

To microencapsulate the essential oil mixture using the complex coacervation method and define the best wall material for the mixture.

MATERIALS AND METHODS:

The materials used in this article was cashew gum variety A. occidentale from EMBRAPA (CG - Pacajus, Brazil), Arabic Gum from Colloid Naturals (AG, Brazil), and Soy protein isolate (SPI) from Allibra were employed as wall materials. Essential oil from Mellaleuca was provided by Misture Perfumaria (Campinas, Brazil). All other reagents were of analytical grade, and deionized water was used in all experiments.

The first step was to determine the Zeta Potential of wall material solutions as a function of pH. The SPI, CG, and AG powder were dissolved in ultra-pure water (3% w/w) the day before the experiment. The pH of the solution was then adjusted in a range of pH from 3.0 to. 8.0. These solutions were stirred for 5 min and then an aliquot was diluted 1:10 in the acidic or alkalinized water at the same pH of the solution, and analyzed in Zetasizer equipment (Malvern, UK).

After that, the influence of stoichiometry between polymers was determined. Based on the zeta potential curves, 3 ratios between SPI and GA or CG were prepared at 0.5%, w/v. The stock solutions were mixed at volumetric ratios and kept under stirring in a jacketed Becker with recirculating water at 50°C. The pH of the solution was modified by adding HCl or NaOH (0.1 M) to get on determined pH. An aliquot of 2 mL was submitted to turbidimetric evaluation in UV-Vis spectrophotometer at 590 nm. All measurements were performed after equilibrating the coexisting phases for 15 min after pH adjustment.

The same solutions were centrifuged at 9000 *g* for 10 min and the coacervates were harvested and dried to constant weight at 105°C. The coacervate yield was calculated as the precipitated mass collected in relation to the total solid mass employed.

To prepare the microparticles, the optimum conditions of interaction determined by zeta potential and turbidimetric assays were employed. Diluted conditions are required to prepare coacervates, and then, 2.5% of total solid was adopted. Then, at defined volumetric ratio and pH for coacervation, the oil phase (25 %) was emulsified in a determined volume of SPI (2.5%, w/v) at 14,000 rpm in a T-18 homogenizer for 3 min. To this emulsion, a determined volume of CG solution (5%, w/v) was then added. The temperature was kept at 50°C. Ultrapure water (400 mL) was added and then, the adjustment of the pH of the mixture. The system was cooled for 30 min to a temperature of 10°C. The system was allowed to settle in the refrigerator for 16 h. After this time, the water phase was drained and the process yield was determined. This step was repeated for at least 5 times with soybean oil in order to standardize procedures. The moisture content of the settled microparticles was determined using a vacuum oven technique for 24 h, at 80°C, in triplicate. The moisture content is the mass of water lost during drying in relation to the total moist particle analyzed.

The yield of the process was calculated at this moment, calculated as the percent of dry material precipitated in relation to the initial dry mass (considering the moisture content of the polymers)

The mean particle size was determined using a laser Light Scattering (Malvern, UK) with sample suspension unit. Microparticles were suspended in deionized water.

Morphology of the moist particles was observed using an optical microscope, coupled with a digital camera, and registered using Image Pro Plus 4.0 Software.

RESULTS:

Zeta potential of the biopolymer solutions versus pH curves is presented in Figure 1. The solutions of Arabic gum (AG) had negative zeta potential in the whole range of pH analyzed. The values obtained were similar to those found by other authors (AL-DOAISS et al, 2020), varying from -10 to -20 mV. The initial reduction of zeta potential, observed from pH 3.5 to 5.5, refers to the protonation of groups present in this structure.

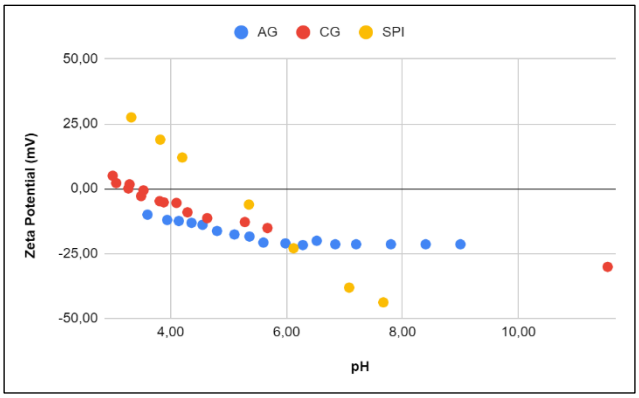

Fig. 4: Variation of zeta potential as pH function of AG, CG and SPI solutions.

With these results, it can be determined the best pH to prepare the combinations for encapsulation. The mixed preparations, CG-SPI and AG-SPI, were tested with oil at pH 4.0.

Based on the zeta potential curves previously present, three ratios of AG-SPI and of CG-SPI were prepared following the method previously described, and the average results are presented on Table 1 and 2 , respectively.

Proportion AG:SPI	Total weight	Wet weight	Dry weight	Absorbance	Moisture	Precipitated
0.8:1	9.8047 g	0.4207 g	0.0313 g	2752	7.4400%	31.9071%
1:1	9.9651 g	0.3749 g	0.0312 g	2686	8.3141%	31.2697%
1.3:1	9.7782 g	0.4141 g	0.0271 g	2408	6.5443%	27.7177%

Table 1: Mean analyticals results of precipitation using AG:SPI solutions.

As presented in Table 1, the best performance was in a 0.8:1 proportion because of the absorbance and precipitated quantity was biggest compared to others.

Proportion	Total weight	Wet weight	Dry weight	Absorbance	Moisture	Precipitated
2.8:1	9.8773 g	0.5083 g	0.0329 g	2174	6.4656%	33.2727%
2.9:1	9.0399 g	0.4697 g	0.0297 g	2229	6.3165%	32.8168%
3:1	9.4290 g	0.5338 g	0.0314 g	2306	5.8886%	33.3340%

Table 2: Mean analyticals results of precipitation using CG:SPI solutions.

As presented in Table 2, the best performance was in a 3:1 proportion because, although the similar results for all the tests, the absorbance and precipitated quantity was biggest compared to others.

Using the best stoichiometry ratio between polymers, ie, 3:1 for CG:SPI, the coacervation was tested for encapsulating soybean oil only for this polymeric pair. The best encapsulation efficiency for soybean oil was 85.13% using the rotation velocity on 14000 rpm during 3.0 minutes.

Then, coacervation was performed by using Mellaleuca oil, varying the homogenization speed. The test parameters and results are presented in Table 3.

Test	Rotation velocity	Time (min)	Dry Matter	Precipitated
A	14000 rpm	2.5	0.51%	44.33%
B	14000 rpm	3.0	0.35%	36.33%
C	14000 rpm	3.5	0.31%	33.67%
D	12000 rpm	3.0	0.68%	66.33%
E	12000 rpm	4.0	0.43%	37.00%
F	13000 rpm	3.0	0.90%	70.29%
G	13000 rpm	3.5	0.84%	78.67%
H	13000 rpm	4.0	1.07%	77.14%

Table 3: Calculated results of oil coacervate using CG:SPI solutions.

The higher encapsulation efficiency (78.67%) was obtained with the lower rotation velocity and greater time. Comparetively, Mellaleuca oil had less encapsulated particles than soybean oil.

To confirm that Mellaleuca oil has been incorporated into particles, a chromatography was made that presents the same characteristic curve as the Mellaleuca oil presented in figure 2.

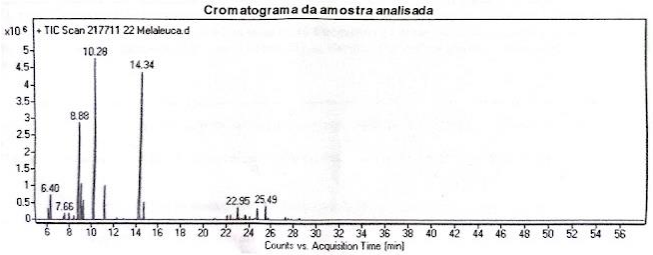

Fig. 2: Mellaleuca oil chromatography.

CONCLUSION:

The best combination of soy protein isolate and cashew gum was 1:3, considering the complex between the polymers, without the presence of oil. For soybean oil encapsulation the method parameters presented was the best scenery but for Mellaleuca oil encapsulation it was necessary to change the parameters to get more encapsulation efficiency. For both oils the encapsulation efficiency presented very good results. The microscopy particles show the encapsulation was successful for the both oils.

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