P-054 **Coffee oil microencapsulation using spray dryer**

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INTRODUCTION AND OBJECTIVES

Coffee is one of the main agricultural crops and one of the most traded commodities in the planet (Naidu 2008). Brazil is the largest coffee producer in the world and has the second largest consumer's market (Silva 2008). Coffee oil is produced by the mechanical extraction of roasted coffee grains and it is composed of a lipidic fraction and a typical fraction of coffee flavor volatiles.
Since the exposure of the oil to the atmospheric air cause loss of flavor and lipid oxidization, the use of microencapsulation technique has been proposed to avoid such degradation processes and enhance the stability of the material (Soottitantawat 2005).
The objective of this work was to evaluate the encapsulation efficiency and physico-chemical characteristics of coffee oil microcapsules produced with different concentrations of maltodextrin and isolated soy protein as wall materials.

MATERIALS AND METHODS

The roasted coffee oil was supplied by Companhia Cacique de Café Solúvel (Barueri/Brazil). The wall materials used were maltodextrin DE 20 (MD), supplied by Corn Products (Mogi Guaçu/Brazil) and isolated soy protein (ISP), with 89% of protein, supplied by Alibra Ingredientes Ltda (Campinas/Brazil).
The carries solutions (30% w/w) were prepared by dispersing dried powders, until complete dissolution. The studied proportions of maltodextrin:ISP were 25:75, 50:50 and 75:25. Coffee oil was added to the hydrated wall material (15% w/w of matrix solids) and the emulsion was formed by the use of a rotor-stator homogenizer (Ultra Turrax Ika, T18 Model, Staufen, Germany) operating at 14000 rpm for 5 minutes.
Emulsions were spray-dried in a laboratorial dryer (mini spray dryer, Labmaq, MSD 1.0 model, Ribeirão Preto, Brazil) equipped with a dual fluid nozzle of 1.2 mm of diameter. The feed flow rate was 0.8 L/h.
Emulsion droplet size distributions were measured using an optical microscope (Carl Zeiss, model MF-AKS 24 x 36 Expomet, Zeiss, Germany) connected to a digital camera (Pentax V20, 8.0 Megapixel). The average droplet size was characterized in terms of the average diameter d_{43}, defined by equation 1,where n_i is the number of droplets of diameter d_i.

$$d_{32} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \qquad (1)$$

The microcapsules moisture content was determined gravimetrically by drying 1g of sample in a vacuum oven at 70º C, until constant weight (AOAC, 1997).
The encapsulation efficiency was determined by the fraction of the encapsulated oil over the total quantity of oil (Equation 2).

$$\text{Encapsulation Efficiency} = \frac{(Oil_{Total} - Oil_{Surface})}{Oil_{Total}} \cdot 100 \qquad (2)$$

The non-encapsulated oil ($oil_{surface}$) was determined by the difference of the mass before and after the extraction with hexane (Bae 2008). The total oil (oil_{total}) was determined gravimetrically by ether extraction, according to Rose-Gottlieb method (Bradley 1993).
The particle size distribution was measured with a Laser Scattering Spectrometer Mastersizer (model MAM 5005, Malven Instruments Ltda., Worcestershire, United Kingdom). The particle size was expressed as the mean volumetric size $D_{4,3}$ (De Brouckere mean diameter), which is the mean diameter of a sphere with the same volume.
To observe particles morphology, powders were attached to scanning electron microscope (SEM) stubs using double adhesive tape, coated with 3–5 mA gold/palladium under vacuum, and examined with a scanning electron microscope (Leica, model LEO440i, Cambridge, England).
Variance analysis (ANOVA), using the software STATISTICA 5.0 (StatSoft, Inc., Tulsa, OK, USA), were used to determine statistically significant differences between the different studied treatments. The analysis of means was performed using the Tukey procedure at $p < 0.05$.

RESULTS AND DISCUSSION

The droplet mean diameter size of all emulsions was between 3.85 and 4.02 µm (Table 1). Changes in the carrier solutions composition did not result in statistically significant differences ($p > 0.05$) in the droplets diameter. With the increase of protein concentration on the carries solutions, it was possible to observe a reduction in the droplets diameter size dispersion.
The carrier composition had a significant effect ($p < 0.05$) on the microparticles diameter size (Table 1). Carries solutions with higher MD concentrations resulted in the production of larger particles. Also, a higher concentration of surface oil (Table 2) was observed in particles containing higher concentrations of MD. The production of larger particles occurs probably because of the ag-

glomeration of particles as a result of the oil present on particles surface.

Table 1 – Emulsion droplet and microparticles mean diameter.

	Emulsion droplet	Microparticles
25% MD and 75% ISP	4.02 ± 0.29[a]	8.63 ± 0.72[a]
50% MD and 50% ISP	4.05 ± 0.29[a]	13.42 ± 0.26[b]
75% MD and 25% ISP	3.85 ± 0.51[a]	13.27 ± 0.21[b]

*Means with the same letter, in the same column, did not differ significantly at $p < 0.05$

The moisture content of different microparticles was significantly influenced ($p < 0.05$) by the carrier solution composition (Table 2). Highest values of moisture content were obtained for particles encapsulated high concentration of ISP. Bhandari (1992) studying the relation between powder moisture content and emulsion viscosity observed that the highest the emulsion viscosity, the highest is the particle moisture content. This explains the results obtained in the present work, since emulsions with higher IPS concentration were more viscous.

Table 2 – Particles moisture content, surface oil and encapsulation efficiency.

	Moisture content (%)	**Surface oil (%)**	**Encapsulation efficiency (%)**
25% MD and 75% ISP	5,30 ± 0,13[a]	1,77 ± 0,06[a]	86,33
50% MD and 50% ISP	4,30 ± 0,17[b]	2,28 ± 0,07[b]	82,72
75% MD and 25% ISP	1,89 ± 0,08[c]	2,05 ± 0,06[c]	79,87

*Means with the same letter, in the same column, did not differ significantly at $p < 0.05$

Higher values of encapsulation efficiency were obtained when carries solutions with higher concentration of ISP were used (Table 2). Encapsulation efficiency can be associated with carrier solutions emulsifying properties since IPS was used with the objective to incorporate an emulsifier agent to MD. Similar values for encapsulation efficiency were obtained by Bylaite (2001), that studied the encapsulation of caraway essential oil using protein-based matrices.

The SEM micrographs show that the obtained particles presented similar shape and various sizes, which is a normal characteristic of particles produced by spray drying. Besides being observed the presence of some microparticles with smooth surface, most of them present a rough surface, with the presence of some dents. According to Krishnan (2005) the characteristic of spherical and smooth surface show the suitability of the mixture for encapsulation. Yet Ré (1998) associates the presence of dents to the shrinkage of the droplets during the early stages of the drying process.

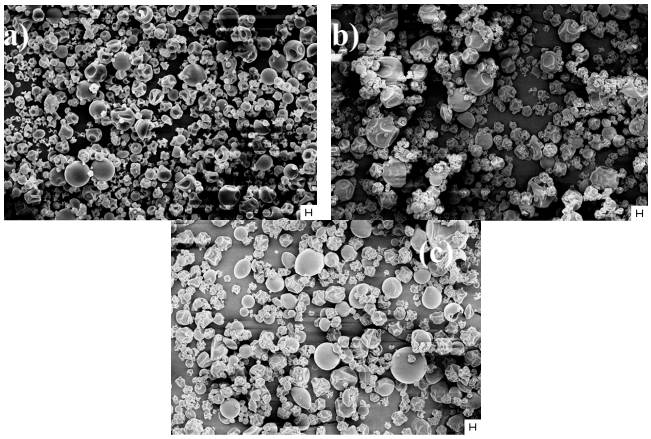

Figure 1 - Scanning electron microscopy of the microcapsules: (a) 25% MD and 75% ISP; (b) 50% MD and 50% ISP; (c) 75% MD and 25% ISP. Bar = 3μm.

CONCLUSIONS

Microencapsulation of coffee oil using maltodextrin + IPS as carrier solutions is a suitable process to obtain powdered flavors. Less surface oil and high encapsulation efficiency were obtained when carries solutions with higher concentrations of IPS were used.

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