

O7-2 Effect of the different wall materials on the physical characteristics and stability of coffee oil microcapsules produced by spray drying.**Prata, A.S.¹, Frascareli E. C.¹, Silva V. M.¹ and Hubinger, M. D.^{*#1}**¹ School of Food Engineering, PO Box 6121, University of Campinas - Campinas, Brazil
#mhub@fea.unicamp.br**INTRODUCTION AND OBJECTIVES**

Brazil is the first largest coffee producer in the world and the coffee oil represents an important industry byproduct, which has several different applications. In the food industry it may be employed to enhance the flavor potential of the coffee beverage and may also be added to candies, cakes and puddings as flavoring agent.

Coffee oil is produced by mechanical extraction of roasted coffee grains. The roasted coffee oil is composed by a lipidic fraction and a typical fraction of coffee aroma volatiles and is rich in pigments that provide it a dark brown aspect. Since the exposure of the oil to the atmospheric air causes loss of aroma and lipid oxidation, the use of microencapsulation technique has been proposed to avoid such degradation processes.

The microencapsulation process consists in involving solid, liquid or gaseous components in a wall material in order to form microcapsules that may release their contents in specific conditions (Rosenberg 1990). The microencapsulation by spray drying is the most commonly used in the food industry. This process is cost-effective, flexible and produces good quality particles. The retention of flavor compounds during the spray-drying process depends on the solids content of the feed material, on the characteristics of the flavors compounds (molecular weight, vapor pressure and concentration), on the type and molecular weight of the wall materials (Reineccius 1988).

The aim of this work was to study the effect of the use of different wall materials, gum Arabic (100%), a mixture of protein: maltodextrin 10 DE (75:25, 50:50, 25:75) and whey protein isolate (100%), on the physical characteristics and stability of coffee oil microcapsules produced by spray drying. For this purpose it was determined some physical properties as: encapsulation efficiency, oil retention, moisture content, hygroscopicity, wettability and mean diameter particle. The critical storage conditions were determined by the sorption isotherms and the glass transition temperature of the microcapsules, storage at different relative humidity values.

MATERIALS AND METHODS**Materials**

Roasted coffee oil supplied by Companhia Cacique de Café Solúvel (Barueri, Brazil), gum Arabic Instangum®

by Colloids Naturels (Sao Paulo, Brazil), maltodextrin 10 DE by Corn Products (Mogi-Guaçu, Brazil) and whey protein isolate by Alibra (Campinas, Brazil).

Process

Before the spray drying process, carrier material was added in the concentration of 30% (w/w) to water, with magnetic stirring, until complete dissolution, at room temperature. Then 15% of oil (in relation to solids) were added and the emulsion was mixed using a Turrax, at 14000 rpm for 5 min. Spray drying process was performed in a laboratory scale spray dryer Labmaq MSD1 (Ribeirao Preto, Brazil), with a 1.2 mm diameter nozzle and main spray chamber of 500 mm x 150 mm. The emulsion (at 25°C) was fed into the chamber through a peristaltic pump, the feed flow rate used was of 0.8 kg/h, the drying air flow rate was 36 m³/h, the compressor air pressure was 0.25 MPa and the compressor air flow rate was of 2.4 m³/h. Inlet air temperature was set at 170°C and outlet air temperature varied of 105 ± 5°C, for each experiment. The microcapsules were characterized by encapsulation efficiency (EE), given by Equation 1, oil retention (OR), given by Equation 2, moisture content (AOAC 1997), hygroscopicity (Cai et al. 2000), wettability (Fuchs et al. 2006) and mean diameter particle (De Brouckere diameter, D_{4,3}). Then the samples were storage at different relative humidity to determine the critical storage conditions.

$$EE (\%) = \frac{(Oil_{Total} - Oil_{Surface})}{Oil_{Total}} \cdot 100 \quad \text{Equation (1)}$$

$$OR (\%) = \frac{Oil_{Total}}{Oil_{Initial}} \cdot 100 \quad \text{Equation (2)}$$

Sorption isotherms

Sorption isotherms using 1 g of sample and were determined by the gravimetric method. Eight saturated salt solutions were prepared (LiCl, CH₃COOK, MgCl₂, K₂CO₃, Mg(NO₃)₂, KI, NaCl and KCl) in order to provide relative humidity values of 11.3%, 22.6%, 32.8%, 43.2%, 52.9%, 68.9%, 75.3% and 84.3%, at 25°C temperature, respectively (Greenspan 1977).

Glass transition temperature

The glass transition temperature was determined by differential scanning calorimeter, TA-MDSC-2920 (TA Instruments, New Castle, USA) equipped with a mechanical refrigeration system (RCS – refrigerated cooling accessory). 3 mg of microcapsules were placed into differ-

ential scanning calorimeter (DSC) aluminum pans (20 mL) and equilibrated over saturated salt solutions in desiccators at 25°C, for 2 weeks. Samples were heated at 10°C/min from -70 to 120°C and an empty pan was used as reference. Dry helium, 25 mL/min, was used as the purge gas. All analyses were done in triplicate and data were treated by the software Universal Analysis 2.6 (TA Instruments, New Castle, USA).

RESULTS AND DISCUSSION

Characterization of microcapsules is presented in Table 1. The mean error for all the analysis was less than 2.7. Encapsulation efficiency varied from 85.15 to 93.60% and oil retention from 88.40 to 96.18%. It can be seen that increasing the proportion of protein, in the mixture maltodextrin/protein, led to an increase of encapsulation efficiency. According to the results, moisture content varied from 1.30 to 1.79%, wet basis, the microcapsule produced with only gum Arabic, presented the highest value and these produced with only whey protein isolate the lowest value, this is due to the fact that gum Arabic is a wall material more hydrophilic than whey protein isolate. The values of hygroscopicity varied from 10.72 to 17.41 g/100 solids. In the case of mixture of maltodextrin/protein the increase of the quantity of protein led to a reduction of the hygroscopicity. The gum Arabic has in its structure more ramifications with hydrophylic groups and this increase the capacity of adsorb water. In relation to wettability, microcapsules produced with the mixture maltodextrin/protein, which have increased the concentration of maltodextrin had the value of wettability reduced due to higher hydrophilic characteristic of the maltodextrin and lower density of the powders produced with maltodextrin.

The particles diameter varied from 9.29 to 17.95 µm. The microcapsule produced with only gum Arabic presented the highest value of particle diameter due to the highest viscosity of the emulsion. An increase of protein concentration, in the mixture maltodextrin/protein led to an increase of particle diameter of microcapsules.

Critical water activity and moisture content were very high, indicating high physical stability, and varied from 0.62 to 0.86 and 0.11 to 0.18 g water/g dry solids for pure gum Arabic and pure whey protein isolate, respectively. Above these values the microencapsulated coffee oil can collapse and became sticky.

Table 1: Physical Characteristics of Coffee Oil Microcapsules.

	GA	MP ₁	MP ₂	MP ₃	PR
EE (%)	85.2	87.6	88.9	90.7	93.6
OR (%)	96.2	88.4	89.1	94.2	93.1
Moisture (%)	1.8	1.3	1.5	1.4	1.3
Higroscopicity (g/100g)	17.4	10.7	11.3	11.5	13.0
Wettability (min/g)	17.5	22.2	23.2	25.4	28.5
Diameter (µm)	18.0	9.3	11.6	9.7	10.6

Where: GA: 100% gum Arabic, MP₁: 75% Maltodextrin and 25% Whey Protein Isolate, MP₂: 50% Maltodextrin and 50% Whey Protein Isolate, MP₃: 25% Maltodextrin and 75% Whey Protein Isolate, PR: 100% Whey Protein Isolate.

CONCLUSIONS

The results of physical properties of the microcapsules of coffee oil showed that the mixture maltodextrin/protein is a good alternative, for reduction of costs with wall material, considering the high values of encapsulation efficiency, oil retention and wettability and the low values of moisture and hygroscopicity. It was observed a high physical stability of the microcapsules, with high values of critical water activity and moisture content.

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