P-036	**Characterization of micro particles of oregano essential oil produced by spray drying**	

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INTRODUCTION AND OBJECTIVES

In the food industry the use of microencapsulation to protect, isolate or control the release of a given substance is of growing interest. Converting a liquid into a powder allows alternative use of ingredients. One of the largest food applications is the encapsulation of flavors (Schrooyen 2001). Among several encapsulation techniques, spray drying is the most common one. Dry solid particles are obtained by hot air drying of liquid droplets (solution, emulsion or suspension) produced at the top of the drying chamber (Turchiuli 2005). Essential oils are slightly soluble in water and impart to the water their odor and taste. They contain terpenes, alcohols, esters, aldehydes, ketones, phenols, ethers, and other minor compounds (Parris 2005). These compounds are frequently used in the food industry as flavoring. Oregano (*Origanum vulgare*) essential oil has a broad spectrum of biological activities, including growth inhibition observed against bacteria and fungi and antioxidant activity (Sahin 2004). In this study we investigated the characteristics of the oregano essential oil produced by spray drying process using response surface methodology.

MATERIAL AND METHODS

Maltodextrin Maltogil DE10 (Cargil®, Brazil), arabic gum (Colloides Natureis Brasil, Brazil) and modified starch (Capsul®, National Starch Food Innovation, Brazil) were used as wall materials. The core material used was oregano essential oil (Ferquima, Brazil). For all treatments the same emulsion was used. The emulsion, oil-in-water type, was comprised of the aqueous phase, a solution of maltodextrin, Capsul and arabic gum and the oil phase consisting of oregano essential oil. The emulsion was prepared by mixing 1 % oregano essential oil, 9 % wall materials (1 % modified starch, 36,25 % arabic gum and 12,75 % moltodextrin) and 90 % distilled water. Wall materials proportion was determinate in previous studies. The previously prepared emulsions were dried in a spray dryer (LABMAQ, model MSD 1.0, Brazil) equipped with a one-fluid pneumatic atomizer. Inlet air temperatures (AT) and feed rates (FR) were optimized using response surface methodology, according to Table 1. Outlet temperature and yield were measure for each treatment. Outlet temperature and yield were measure for each treatment.

Water activity (aw) was measured by direct lecture in digital device AQUA-LAB, CX-2 model (Decagon Devices Inc., US), with controlled temperature of 25 ± 0,5°C.

Table 1 – Experimental design for the spray drying tests

	Coded variables		Process variables	
Assay	X1	X2	Inlet air temperature (°C)	Feed rate (L/min)
1	+1	+1	180	0,90
2	+1	-1	180	0,60
3	-1	+1	140	0,90
4	-1	-1	140	0,60
5	0	-1,41	160	0,54
6	0	+1,41	160	0,96
7	-1,41	0	132	0,75
8	+1,41	0	188	0,75
9	0	0	160	0,75
10	0	0	160	0,75
11	0	0	160	0,75

Moisture determination was done by gravimetric method, on temperature of 105 ºC, until reach constant weight (AOAC, 1998). For hygroscopicity analysis 2 g of were weigh, transferred to a petri dishe and put inside a glass desiccators, at 25 °C, containing saturated solution of Na_2SO_4 (81 % relative humidity). One week after, samples were weighed and hygroscopicity was expressed as g of water/100 g of dry solids (Cai 2000). For determination of bulk density, 2 g of powder were transferred to a 10 ml graduated cylinder. Packed bulk density was calculated from the weight of powder contained in the cylinder after being tapped by hand until constant volume (Goula 2004). Solubility was determinate by method described by Eastman (1984), modified by Cano-Chauca (2005), where 25 ml of distilled water were transferred to a beaker and submitted to 2500 rpm agitation in Ultraturrax homogeneizer. One gram of powder (dry basis) were gently added to the water and the agitation was maintained for 5 minutes. The solution was transferred to a tube and centrifuged for 5 minutes at 2600 rpm. One aliquot (20 ml) of supernatant was transferred to a petri dish and dried for 5 hours at 105 ºC. The percent (%) of solubility was calculated by weight difference. Particle morphology was evaluated by scanning electron microscopy (SEM). Powders were attached to a double-sided adhesive tape mounted on SEM stubs with 1 cm diameter and 1 cm height, coated with gold under vacuum and examined with a MEV 1430 VP - LEO scanning electron microscope (Electron Microscopy Ltd., Cambridge, England). SEM was operated at 20 kV with magnifications of 900 to 1200x

RESULTS AND DISCUSSION

Results for water activity, moisture and bulk density showed significant diferrence between treatments

($p<0.05$) (Figure 1) while hygroscopicity and solubility showed no significant difference.

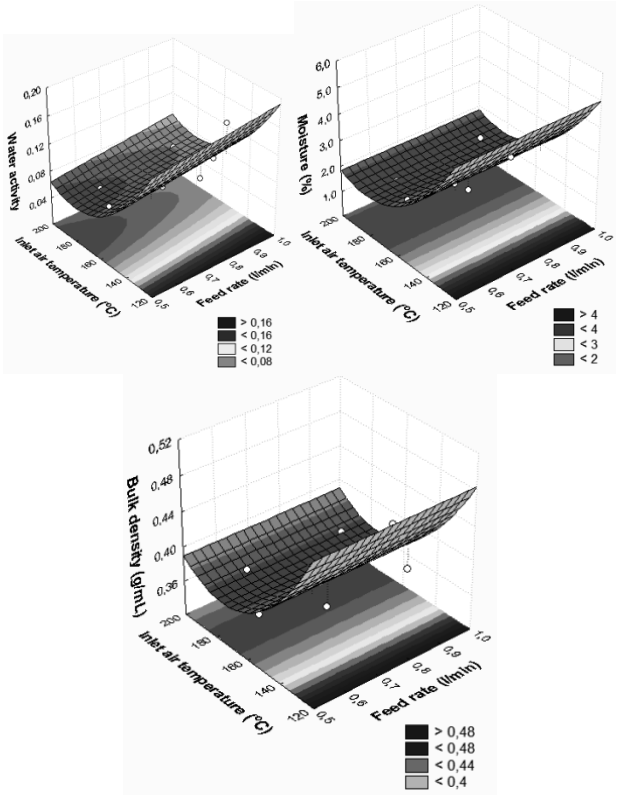

Figure 1 – Surface graphics for water activity, moisture and bulk density related to the variation of inlet air temperature (°C) and feed rate (l/min)

Variation on process of spray drying, based on feed rate (FR) and inlet air temperature (AT) caused variations on water activity and moisture of oregano oil micro particles. Water activity was influenced by AT, indicating that a minimum value can be observed, when AT varies from 165 to 195 °C. Results for aw reached minor values when lowering FR. Moisture was affected only by AT following the same tendency for aw, lower value was observed when temperature varies from 175 to 190 °C. Variation in FR did not show influence over moisture. Bulk density was also influenced only by AT, showing that minimum values can be obtained when temperatures vary from 165 to 185 °C. It is important that bulk density presents lower values based on relation with powder dispersibility that can impact on powder reconstitution properties. Lower water acitivity and moisture values longer can be the shelf life of powders without larger reactions and transformations. The morphology of particles is showed in Figure 2.

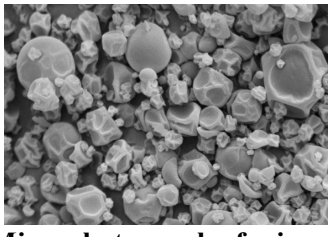

Figure 2 - Microphotograph of micro particles of oregano essential oil

Almost all microcapsules had surface dents. Smaller microcapsules exhibited deep surface dents indicating solidification of the walls prior to onset of expansion. Cracks were not evident in most capsules. Cracks present great influence on loss of volatile compounds of essential oil encapsulated.

CONCLUSIONS

Use o inlet air temperature between 165 and 195 °C showed better results for water activity, moisture and bulk density also evidencing that feed rate did not show great significance on studied parameters.

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