Influence of homogenization processes on curcumin encapsulation by freeze and spray drying

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INTRODUCTION AND OBJECTIVE

The initial step of the encapsulation process is based on the emulsification of the compound to be encapsulated in the solution containing the wall material. This step has a direct influence on the encapsulation efficiency and properties of the resulting capsules. Generally, emulsification is performed using mechanical homogenization; however the emulsions can also be prepared with the aid of ultrasonic apparatus. The main advantages in the ultrasound application for emulsion preparation are good stability without addition of surfactants and small particles in suspension (Chandrapala 2012).

The objective of this work was to evaluate the influence of ultrasound homogenization on the encapsulation process of turmeric oleoresin using different combinations of modified starch, maltodextrin and gelatin as encapsulating matrices, and different drying processes: freeze and spray drying.

MATERIALS AND METHODS

Turmeric oleoresin OS-50 (Agro-Industrial Olimpia Ltda., Brazil) was the core material. Wall materials included maltodextrin DE 10 (Mor-Rex® 1910, Corn Products, Brazil), modified starch HiCap® 100 (National Starch and Chemical Industrial Ltda, Brazil), and bovine gelatin 240 bloom (Gelita®, Brasil).

Encapsulating matrices were composed by different ratios of modified starch (MS), maltodextrin (MD) and gelatin (GL), according to two binary formulations: (30 g dry MS + 1 g dry GL)/100 g total matter (30MS:1GL) and (26 g dry MD + 0.6 g dry GL)/100 g total matter (26MD:0.6GL). Turmeric oleoresin was added in an amount of 15 g oleoresin/100 g dry wall material. The mixtures were homogenized by two different processes: (a) mixing by using Ultra-Turrax (T25, IKA, German) operating at 18,000 rpm for 10 minutes; or (b) ultrasound homogenization by using an ultrasound probe (Sonic Ruptor 4000, Omni International, USA) at a frequency of 20 KHz for 3 minutes with power output of 210 W. During sonication, sample temperature was kept at 40 °C by a cold water jacket.

The emulsions were dried by freeze drying – with the homogenized emulsions being frozen at -38 ºC for 24 hours and freeze-dried using a freeze-dryer (L101, Liobras, Brazil) at < -40 °C (temperature at the condenser) for 48 hours (pressure < 150 mmHg) - and spray drying – by using a spray dryer (B-290, Büchi, Switzerland) with a spray nozzle diameter of 0.7 mm and the following operating conditions: feed flow rate of 6 mL/min, drying air temperature of 170 ºC and drying air flow of 420 L/h. The air temperature output was 79 ± 2 °C. Dried emulsions were packaged to prevent light incidence and stored over silica gel in desiccators at room temperature.

Total curcumin content was determined in the encapsulated materials by spectrometry following the method described by Chauhan (1999). Moisture content was determined gravimetrically by oven drying at 105 ºC for 6 hours.

Morphology and surface appearance of encapsulated turmeric oleoresin were examined using a Scanning Electron Microscope - SEM (Zeiss, model 960, Germany). Samples were attached to SEM stubs using adhesive tape and coated with gold under vacuum (Sputer Coater, model SCD 050, Brazil). SEM was carried out at 20 kV with work distance of 12 mm. The scanned images were collected digitally using Digital Image Transfer 1.0 (PUC, Brazil).

Data of analytical determinations were subjected to analysis of variance and differences between means were tested by Tukey test at 5 % probability using MINITAB 16 (Minitab Inc., USA).

RESULTS AND DISCUSSION

The use of ultrasound homogenization showed positive results only in spray drying (Figure 1a). Curcumin retention were 63.6 % for formulation 30MS:1GL and 52.2 % for formulation 26MD:0.6GL when using ultrasound homogenization and freeze drying. These values were significantly lower ($p < 0.05$) than those observed for the same freeze-dried formulations prepared by mechanical homogenization. On the other hand, application of ultrasound homogenization combined with spray drying showed significant improvement ($p < 0.05$) in curcumin retention for both formulations, resulting in 92.9 % for sample 30MS:1GL and 94.2 % for sample 26MD:0.6GL. This significant increase was about 28.5 % and 80.8 % for 30MS:1GL and 26MD:0.6GL, respectively, referring to mechanical homogenization.

The emulsions studied in this work were also evaluated in our laboratory for stability (creaming

index) and particle size by optical microscopy (Ferreira 2012a, b). The ultrasound homogenization increased the stability and reduced the particle size of the emulsions. These results may be related to the enhancement in the curcumin retention in encapsulated materials produced by ultrasound homogenization and spray drying, especially for the maltodextrin/gelatin formulation.

Comparing the homogenization processes, only 26MD:0.6GL samples dried by freeze drying showed significant differences ($p < 0.05$) in moisture content (Figure 1b).

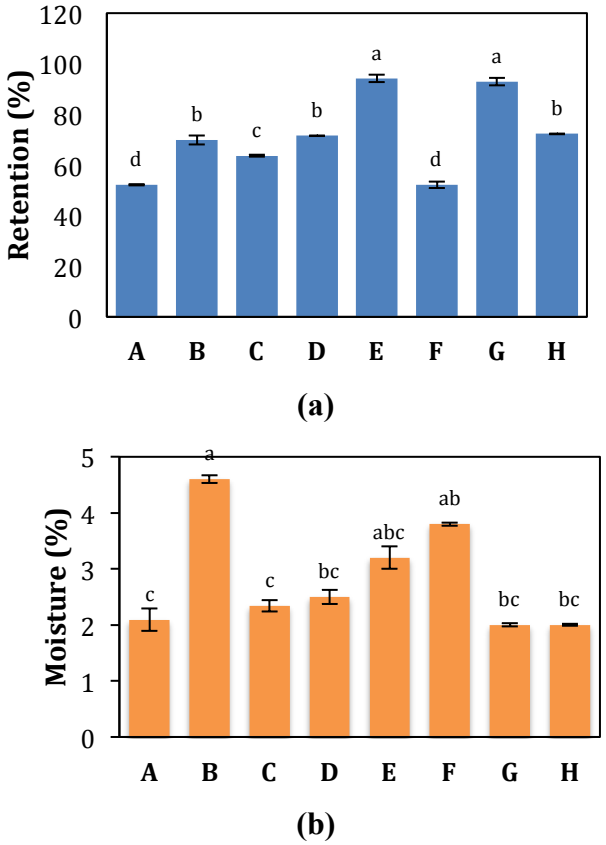

Figure 1: Curcumin retention (a) and moisture (b) in encapsulated turmeric oleoresin: freeze dried 26MD:0.6GL with ultrasound (A) and mechanical homogenization (B); freeze dried 30MS:1GL with ultrasound (C) and mechanical homogenization (D); spray dried 26MD:0.6GL with ultrasound (E) and mechanical homogenization (F); spray dried 30MS:1GL with ultrasound (G) and mechanical homogenization (H).

The morphological analysis of the microcapsules produced by the different homogenization techniques showed their effect in the microstructures of encapsulated materials. When freeze drying was used, the formulation of wall matrices and the homogenization process had little influence on the particle structure. Nevertheless, in the case of spray drying, both the wall material and emulsification method were significant. The microcapsules produced with the maltodextrin/gelatin matrix using ultrasound homogenization showed more spherical and smoother surfaces with fewer concavities and "teeth", as well as a narrower particle size distribution (Figure 2). Another feature of these particle structures was the presence of cracks. Similar behavior was observed in the microcapsules produced with modified starch/gelatin wall matrix. Although some authors (Jafari 2007) have pointed out that surface morphology could be independent from the emulsification method, being affected only by wall material properties and drying conditions, the results obtained in the present work indicate that sonication have affected the microcapsule surface morphology.

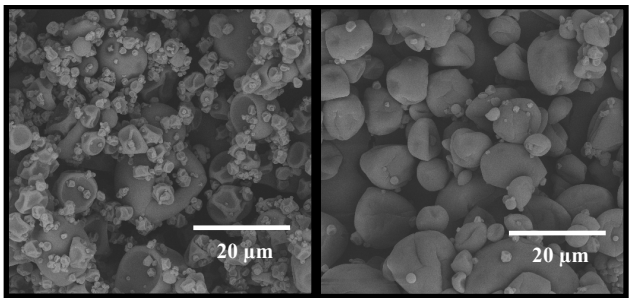

Figure 2: Microphotographs of turmeric oleoresin encapsulated in 26MD:0.6GL matrices using mechanical homogenization (a) and ultrasound homogenization (b) followed by spray drying (1000x of magnification).

CONCLUSIONS

The present work suggested that ultrasound homogenization could be a useful technique to increase efficiency of encapsulation processes of hydrophobic compounds by spray drying.

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