Microencapsulation of xylitol by double emulsion followed by complex coacervation: effect of homogeneization rate on rheological behaviour of emulsions and morphology of microcapsules

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INTRODUCTION AND OBJECTIVE

Xylitol is an important sugar substitute with interesting physical and chemical properties which make it a high value compound for pharmaceutical, odontological and food industries (Misra et al. 2011).

Microcapsules produced by coacervation are water-insoluble and heat-resistant, possessing excellent controlled-release characteristics based on mechanical stress, temperature and sustained release (Dong et al. 2011). Few articles deal with influencing parameters of the coacervation itself (Mathieu et al. 2006). The controlling release property of coacervate microcapsules is determined on the structure, the particle size, the loading, the cross-linking degree and dispersing medium (Dong et al. 2011).

The rheology behaviour depends on many parameters, such as shear rate, solids loading, particle size, and surface potential, and can be measured by monitoring changes in flow behaviours in response to an applied stress (or strain) (Lu and Kessler 2006). However, a clear understanding of the correlation between microstructure and bulk rheological properties is still lacking (Caggioni et al. 2007).
The aim this work was a
nalyse effect of homogeneization rate on rheological behaviour of emulsions and morphology of microcapsules.

MATERIALS AND METHODS

Primary emulsion (water-in-oil) was prepared using corn oil, 70% solution of xylitol and polyglycerol polyricinoleate (PGPR 90), as the surfactant, homogenised at three homogenization rate: 4.000, 8.000 and 12.000 rpm for 4 minutes. Double emulsion (water-oil-water) was prepared with primary emulsion, adding gelatine (Gelita South America, Brazil) (0.25mg/mL) homogenised at 10.000 rpm for 3 minutes at 50^{o}C. Gum Arabic solution (Dinâmica Química Contemporânea Ltda., Brazil) (0.25mg/mL) was added drop wise into the double emulsion, maintaining the temperature at 50°C. The pH value was adjusted to 4.0 using citric acid in order to induce the complex coacervation. The coacervates were stirred in an ice bath until it reached a temperature of 10°C and maintained in the refrigerator (7^{o}C) for 24h to complete particle precipitation. Particle size analysis of double emulsion and coacervate were performed using Shimadzu SALD-201V laser diffraction particle analyser (Kyoto, Japan) and primary emulsion using Zeta Particle Size (BTC – Brookhaven instrument Corporation, New York, USA). The microcapsules were characterized using Zeiss Microscope (Carl Zeiss, New York). Rheological measurements were performed with a rheometer (AR 2000, TA Instruments, New Castle, Delawale, USA) using a standard-size double concentric cylinders geometry with 17.50mm-outside rotor, 16.00mm inside rotor, 15.10mm stator, a gap of 2000, a temperature 25^{o}C (primary emulsion) and 50^{o}C (double emulsion). In all cases, replicate samples were analysed in triplicate. The data obtained were analysed statistically by the analysis of variance (ANOVA) and Tukey's test using version 9.1.3 of the SAS (Statistical Analysis System) statistical program (SAS, 1995). The results were considered to be statistically significant when $\alpha \leq 0.05$.

RESULTS AND DISCUSSION

Morphology

The structure of coacervate microcapsules was significantly influenced by homogenization rate during emulsification process (Figure 1). Low homogenization rate produced large droplets which were not completely encapsulated. High rates produced smaller microcapsules with wall material defined. Yeo et al. (2005) observed that particle morphology depended on the homogenization rate and mononuclear microcapsules prepared with a lower homogenization rate release the interior core material more quickly than multinuclear microcapsules produced by high homogenization rate.

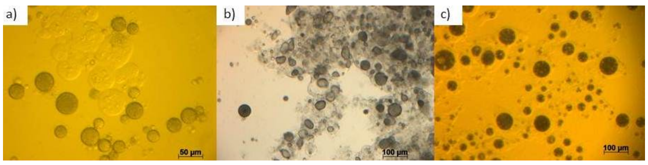

Figure 1 : Optical microscopy image of coacervates microcapsules with homogenization rate of 4000rpm (a); 8000 rpm (b) and 12000 rpm (c)

Particle Size

Variation of the homogenization rate did not bring about a significant difference in mean particle size of simple emulsion and double emulsion, but in coacervate there was significant difference (Figure 2). Homogenization rate of 12000 rpm showed particles sizes smaller than 100µm, which is desirable in food applications.

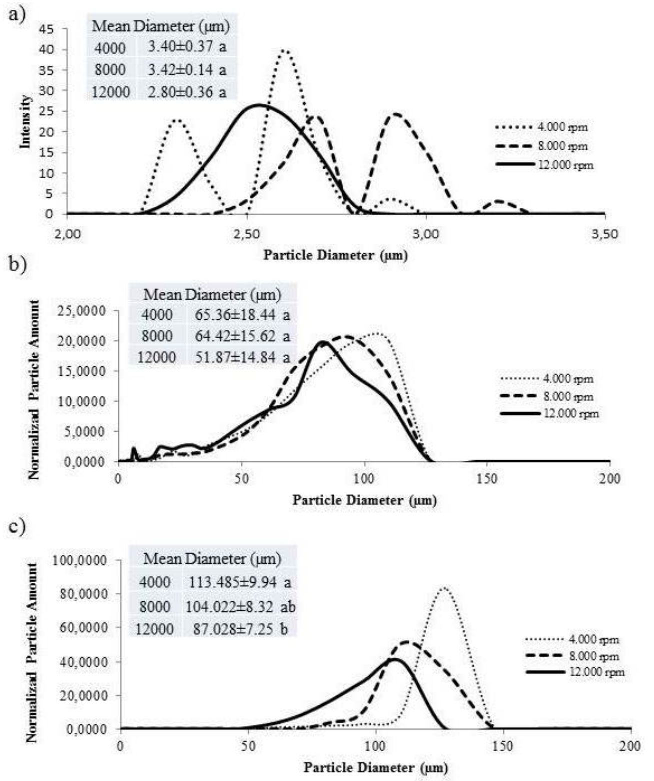

Figure 2 : Particle size distribution of droplets primary emulsion (a), droplets double emulsion (b) and coacervates microcapsules (c)

Rheology

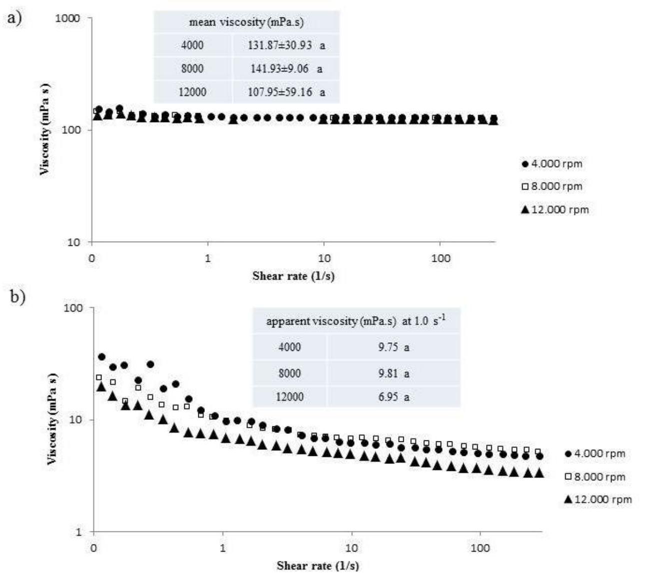

Figure 3 : Viscosity *vs.* shear rate correlation: (a) Primary emulsion, (b) Double emulsion

Primary emulsion showed rate between viscosity and shear rate constant (Figure 3a), being considered Newtonian fluid, as another oils. The plot of the viscosity versus shear rate indicated that all samples of double emulsion exhibit non-Newtonian shear-thinning behaviour, there was a decrease over time (Figure 3b). Homogenization rate of primary emulsion had not influence on viscosity, probably due low concentration of primary emulsion added in aqueous gelatine solution (75mg/mL), which were homogenised in the same speed (10000 rpm).

CONCLUSIONS

Primary emulsion prepared with different speed did not influence in rheological behaviour of double emulsion. However, homogenization rate of 12000 rpm produces xylitol microcapsules with size appropriate (<100µm) and good morphologic characteristics.

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