Encapsulation of oil in Ca-alginate microcapsules by inverse gelation technique.

Martins E., Renard D.*, Davy J., Marquis M. and Poncelet D.*
ONIRIS/INRA UR BIA, Nantes, France (evandrombi@yahoo.com.br)

INTRODUCTION AND OBJECTIVE

The oil encapsulation has been used in different industry sectors as agriculture, pharmaceuticals, foods, cosmetics and fragrance (Abang et al. 2012). This process is advantageous for various reasons, such as for conversion of liquid to solid form to facilitate handling, transportation or incorporation into other components, taste/smell masking, protection from evaporation or oxidation, and controlled-release applications.

A promising technique for oil encapsulation in Ca-alginate capsules by inverse gelation has been proposed by Abang et al. (2012). This method consists of emulsifying calcium chloride solution in oil then dropped into an alginate solution to produce aqueous-core calcium alginate capsules. This technique allows the production of capsules with diameters around 3.2 mm, though this size can be inappropriate for application in industrial products. As this size is a limiting factor for applications of these capsules in industry, the objective of this study is to propose a new process of oil encapsulation based on double emulsion in Ca-alginate microcapsules by the inverse gelation method.

MATERIALS AND METHODS

Sodium alginate powder Algogel 3001 was kindly donated by Cargill (France). Calcium chloride powder ($CaCl_2$. $2H_2O$) (Panreac Quimica Sau, Spain) and sunflower cooking oil (Associated Oil Packers, France) were used to prepare the emulsions. All other chemicals of analytical grade were obtained from Sigma Aldrich (France).

Preparation of alginate solution

Ten gram of alginate powder was dissolved in 1 L of demineralised water. A surfactant (Tween 85) at 0.5% v/v was then added to the alginate solution.

Preparation of primary emulsion and double emulsion

For the preparation of a primary emulsion, 100 mL of sunflower oil containing 0.5 mL of Tween 85, 0.5 mL of Span 85 and 0.01 g of Sudan red, were stirred using a high shear mixer (Ultra-Turrax T25, IKA, Germany) at 13 500 rpm during 30 s. Thirty mililiters of calcium chloride solution was then added slowly and a new shear mixing at 13 500 rpm for 3 min was performed (Figure 1A).

For the production of a double emulsion, 10 mL of primary emulsion was dispersed in 100 mL of sunflower oil using a paddle stirrer (Eurostar digital, IKA, Germany) at 500 rpm for 2 min (Figure 1B)

Experimental set-up

Hundred mililiters of double emulsion was added into 400 mL of alginate solution (Figure 1C). The alginate solution was stirred at 350–400 rpm with an 80 mm long wedge-shaped magnetic barrel. After 15 min of curing at ambient temperature (20 ± 2 °C), the wet capsules were washed with demineralised water to remove excess alginate and to prevent the capsules from sticking each other.

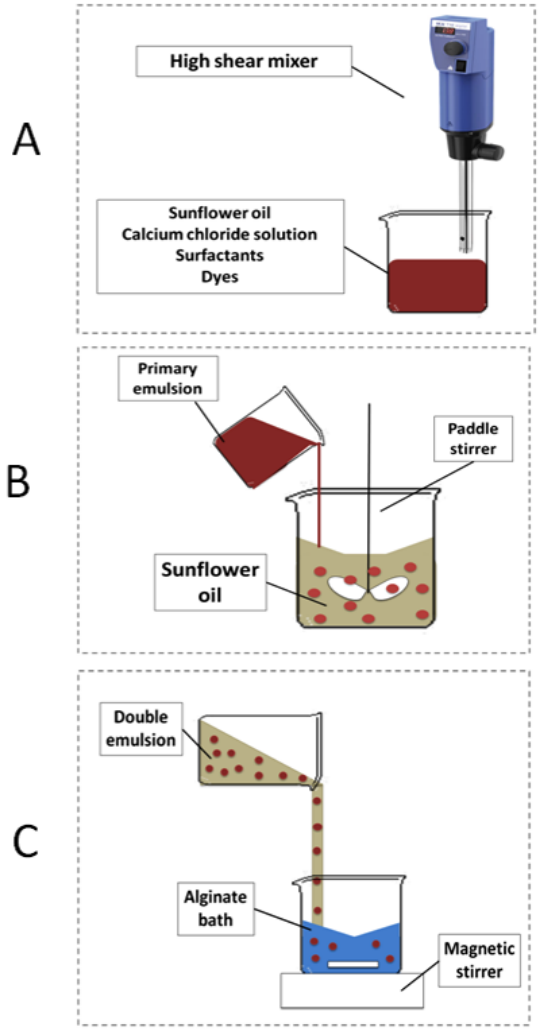

Figure 1. Diagram of the experimental set-up. Production of primary emulsion (A), double emulsion (B) and microcapsules (C).

Observation of emulsions and measurements of microcapsules diameters and thicknesses

A confocal laser-scanning microscope (Nikon, France) with a 4X objective was used to measure the diameters and the membrane thicknesses of the microcapsules. The same equipment was used for observation of the primary emulsion labeled with fluorescein (0.08 g/L).

RESULTS AND DISCUSSION

The first step for the preparation of microcapsules consisted in the production of a primary emulsion containing a source of calcium. This step was a crucial point once the little variations in the protocol could change the properties of primary emulsion.

Surprisingly, analyses by confocal microscopy revealed that the water-in-oil (W/O) primary emulsion became an oil-in-water (O/W) emulsion by catastrophic inversion (Figure 2A). Surfactants, with high and low hydrophilic-lipophilic balance (HBL), were used in this protocol in association with a high speed of shear, thus allowing the phase inversion of the primary emulsion (Scheer et al. 2013). This property allowed the dispersion of the primary emulsion in oil, ensuring the formation of a double emulsion (Figure 2B).

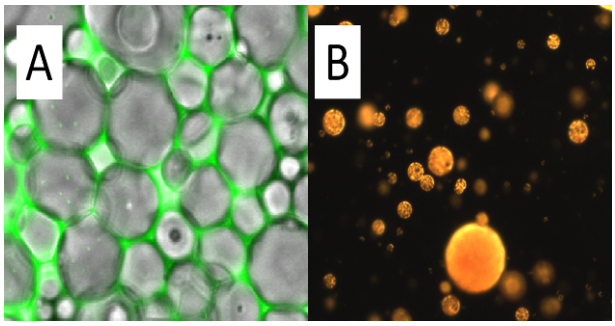

Figure 2. (A) Confocal microscopy of the primary emulsion (Green: Calcium chloride solution; Gray: Sunflower oil). (B) Double emulsion. Droplets of primary emulsion (yellow) dispersed in sunflower oil (black background).

However, during the production of double emulsion, droplets of different sizes were formed, implying the formation of heterogeneous microcapsules in size.

For the production of microcapsules, the double emulsion was then added to the alginate bath. Thanks to the stirring of alginate bath, the droplets of primary emulsion of the double emulsion migrated outside the oil phase where they entered in contact with the alginate solution. During the curing time, the Ca^{2+} diffused through the primary emulsion, leading to the formation of an alginate membrane. At the end of the process (15 min), microcapsules with a Ca-alginate membrane and an oil core were formed (Figure 3).

This new process enabled the production of microcapsules with diameters of ~500 µm (Table 1). In addition, the membrane corresponded on average to 85 % of the volume of microcapsules. However, the membrane thickness was able to vary with the core diameter.

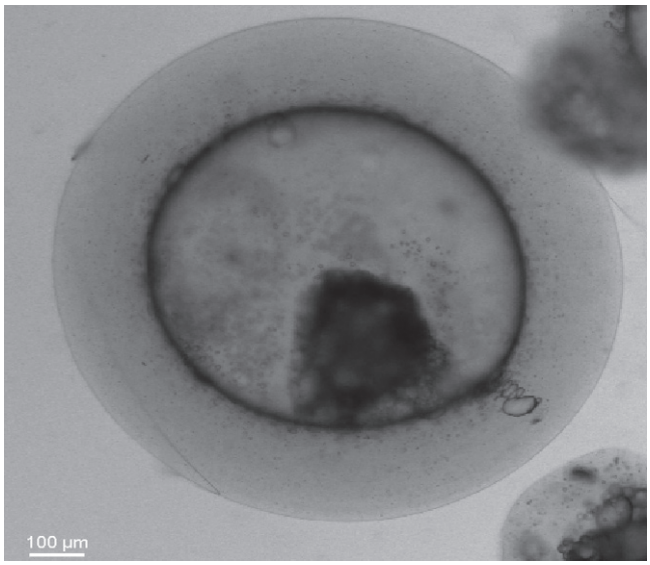

Figure 3. Ca-alginate microcapsules with oil core.

Table 1. Microcapsules characteristics

Parameters	Size (µm)
Microcapsules diameter	513 ± 135
Membrane thickness	121 ± 41
Core diameter	270 ± 94

CONCLUSION

The new process developed allowed the production of Ca-alginate microcapsules by inverse gelation with a considerably higher membrane thickness compare to the classical process. Further studies on physical properties of microcapsules and controlled release of actives will be conducted in order to ensure for these capsules potential industrial applications.

REFERENCES

- Abang et al. (2012) *Effects of process on the encapsulation of oil in ca-alginate capsules using an inverse gelation technique*. Journal of Microencapsulation 1-12.
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