

## POTENTIAL AND FREQUENCY INFLUENCE ON THE MICROCAPSULES FORMATION BY COEXTRUSION AND GELLING

FERRÁNDIZ Marcela<sup>1</sup>, FRANCO Esther<sup>1</sup>, PASCUAL Mónica<sup>1</sup>, DOLÇA Celia<sup>1</sup>,  
CAPABLANCA Lucía<sup>2</sup>, DIAZ Pablo<sup>2</sup>, LÓPEZ Fernanda<sup>2</sup>

<sup>1</sup> Textile Research Institute (AITECH), Biotechnology Research Group, Plaza Emilio Sala 1, 03801 Alcoi (Alicante), Spain,  
E-mail: [mferrandiz@aitex.es](mailto:mferrandiz@aitex.es)

<sup>2</sup> Universitat Politècnica de València (UPV), Campus de Alcoy, Plaza Ferrándiz y Carbonell 1, 03801 Alcoi (Alicante),  
Spain, E-mail: [maboar@txp.upv.es](mailto:maboar@txp.upv.es)

Corresponding author: Ferrándiz, Marcela, E-mail: [mferrandiz@aitex.es](mailto:mferrandiz@aitex.es)

**Abstract:** Wide varieties of techniques are currently available for encapsulation of active components in food, nutraceutical, pharmaceutical, cosmetics, textile, etc, industries. Coextrusion and gelling technique is a physical encapsulation technique used for the formation of core-shell liquid filled microcapsules. The general microcapsules morphologies are presented as core-shell spheric microcapsules. There are several parameters to control in the process, and their control is outstanding for the correct microcapsules morphology with proper encapsulation of the active ingredient, these parameters are: frequency, potential, nozzle diameters, encapsulating material concentration, cross-linking concentration, flow rates of encapsulation and active materials, etc. Furthermore shell and core solutions must have viscosity, interfacial tension, density, solubility and thermal properties that must be compatibles with the coextrusion process. The versatility of the systems allows for encapsulation of water- or - oil soluble liquids and slurries.

This work is focused on encapsulating an essential oil, rosemary oil, by coextrusion and gelling, therefore is an important study and optimize the process parameters, focusing on potential and frequency.

A large number of experiments have been made in order to know the optimal values, the microcapsules morphology allow us which are the correct values.

A stereomicroscope was used to know the microcapsules morphology and determine the correct values.

**Key words:** Core, Shell, Cross-linking, Droplet, Sodium Alginate, Calcium Chloride

### 1. INTRODUCTION

Coextrusion, also known as annular jet atomization, is the preferred physical technique for the microencapsulation of liquids, slurries, or emulsions to form a core-shell morphology [1]. Coextrusion technology is capable of producing core-shell microcapsules from a few microns to several millimeters in diameters with payloads over 90%. There are three basic steps to consider for the use of coextrusion process: compound droplet formation; shell formation and capsule collection.

Droplet formations based on the successful combination of multiple parameters, such as frequency, potential, nozzle diameters, flow rates of encapsulation and active material, encapsulating material concentration, etc. Once the droplet is formed, the shell must completely harden while maintaining the core-shell morphology. Finally, the microcapsules must be collected with breaking the shell or developing unwanted agglomeration. [2, 3]

The aim of this work is to optimize two parameters of the microencapsulation coextrusion process, potential and frequency, in order to encapsulate an essential oil. The shell material and the active material concentrations, nozzle diameters, cross-linking material have been optimized in a previous research [4, 5, 6].

A stereomicroscope can allow know the microcapsules morphology and then optimize these parameters.

## 2. EXPERIMENTAL

### 2.1 Materials

A low viscosity sodium alginate with a 3,5% of concentration provided by SIGMA ALDRICH was used as shell material. The active material was an essential oil, rosemary oil, provided by Esencias Lozano. In order to observe the oil presence inside the microcapsules we used a natural dye, Verde Cornasol C 0,1% provided by Prochimac.

The cross-linking material, calcium clorhídre 0,5M, was provided by SIGMA ALDRICH.

### 2.2 Microcapsules obtention

This encapsulation process is when a fluid flows in laminar state and breaks into droplets of a same size through an overlap vibration. Then these drops gel on an ionic solution, resulting, encapsulation of the active ingredient (Core) coated with a polymer (Shell).

Microcapsules were obtained by BUCHI B-390, the internal nozzle diameter (core) was 0,2mm and the external nozzle diameter (shell) was 0,4mm.

Potencial and frequency values are shown in table 1.

*Table 1: Potencial and frequency values used*

Potencial (V)	2500	2000	1500	1000	500	250
Frequency (Hz)	6000	3000	1500	750	300	150

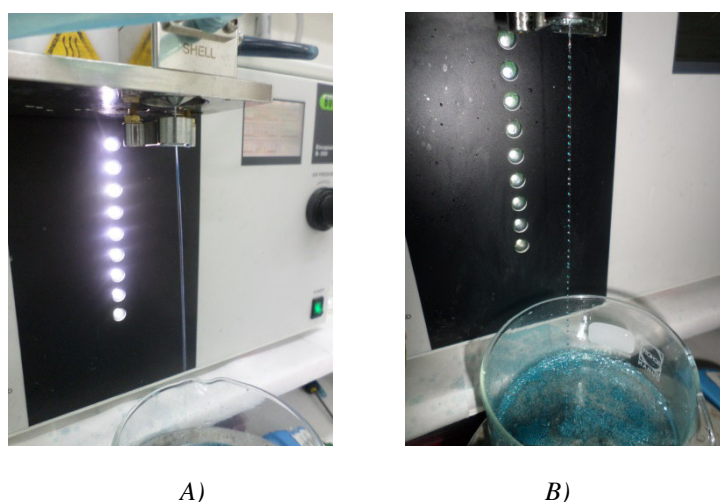
### 2.3 Instrumental techniques

A stereomicroscope Olympus SZX7 was used in order to study microcapsules morphology.

## 3. RESULTS AND DISCUSSION

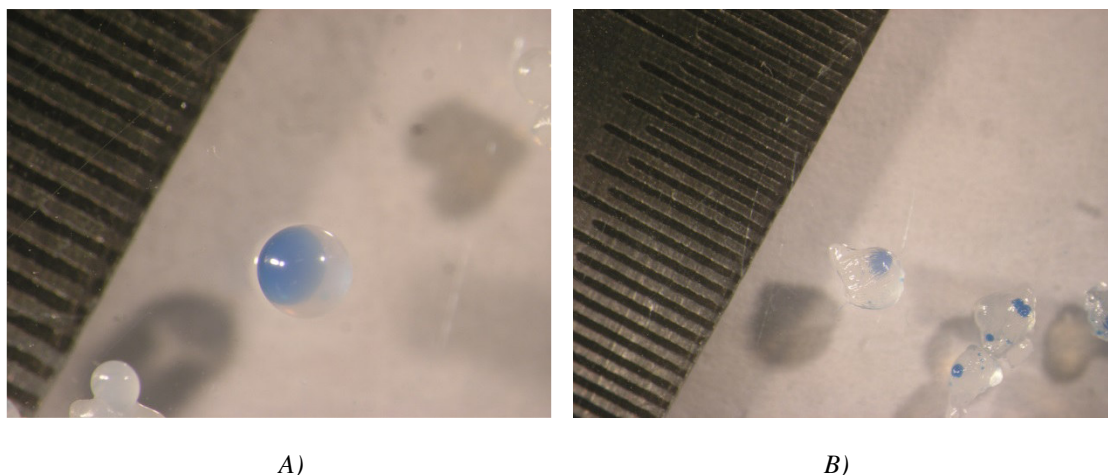
The values of frequency and potencial was optimized depends on the morphology of the chain, due to the strobe light.

Frequency range where the chain wasn't appropriated to produce spherical microcapsules, using the optimal materials concentration was 750-6000Hz. Microcapsules with the correct shapes were obtained with frequency values between 750-150Hz. We can observe in the next figures these differences.



*Fig. 1: Chain morphology. A) Inadequate shape; B) Adequate shape*

The chain that we can observe in picture A doesn't allow the formation spherical microcapsules this is because the frequency is too high, whereas the picture b allows obtain microcapsules with the correct morphology like in the following figures .



**Fig. 2:** *Microcapsules shape. A) Spherical shape; B) Non-spherical shape*

If potential is too high, the microcapsules agglomerate upon the electrode as we can see the next figure. For this work, this effect could be shown in the potential ranges between 2500-500 V.



**Fig. 3:** *Agglomerate microcapsules upon the electrode*

## 5. CONCLUSIONS

The present work optimizes the frequency and potential in a microencapsulation process, coextrusion and gelling. Certain differences can be observed. Depending on the frequency values the chain changes and as a result, the microcapsules morphology changes too. Highly potentials produce in the microencapsulation process particles agglomerates on the electrode.

Specially at this work, after several assays, we obtain the optimal parameters. They are 300Hz of frequency and 250V of potential.

## ACKNOWLEDGEMENTS

The authors want to thank the IVACE (Valencian Institute of Business Competitiveness) and the European Regional Fund for the financial supported.

## REFERENCES

- [1] N. Garti and D.J. McClements, “*Encapsulation technologies and delivery systems for food ingredients and nutraceuticals*”, Ed. WoodHead Publishing, 2012.
- [2] N. Henry, L. Buchala., F. Gueraud and D. Poncelet, “*Optimization of microspheres formulation by a Co-extrusion Method*,” presented at the XXI International Conference on Bioencapsulation, Berlin, Germany, 2013.
- [3] B. Sarmiento and J.D. Neves “*Chitosan-Based systems for biopharmaceuticals, delivery, targeting and polymer therapeutics*” Ed. Wile, 2012.
- [4] L. Capablanca., M. Bonet., E. Bou, M. Ferrandiz, E. Franco and C. Dolcà, “*Aplicación de técnicas biotecnológicas y de microencapsulación para la funcionalización de agrotexiles*”, presented at the II Congreso de I+D+i, Campus de Alcoy, “Creando Sinergias”, Alcoy, España, 2014.
- [5] L. Piazza, T. Roversi, “*Preliminary study on microbeads production by co-extrusion technology*” *Procedia Food Science*, vol. 1, 2011, pp. 1374 – 1380.
- [6] S. J. Risch, “*Encapsulation of flavors by extrusion, in Flavor Encapsulation*”, American Chemical Society, 1988, pp.103-109.