

P50: ENCAPSULATION AND CONTROLLED RELEASE OF BIOACTIVE PHENOLIC COMPOUNDS: PHENOLIC ACIDS, FLAVONOIDS AND PHENYLPROPANOIDS

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Abstract – In this work the effectiveness of spray drying encapsulation as a new strategy to increase bioavailability of bioactive compounds has been evaluated. Thus, an experimental design based on a central composite 2² model was carried out to encapsulate different phenolic compounds from vegetable sources. Inlet air temperature and ratio sample/encapsulation agent (inuline) were selected as independent variable. The results pointed out a great yield was obtained under all experimental conditions. Encapsulation efficiency presented variability depending on the chemical group studied.

Keywords: Microencapsulation, phenolic compounds, inuline.

1. INTRODUCTION

Currently, phenolic compounds bioactivity has increased their pharmacological interest due to these compounds may be used as an alternative to treat different diseases such as obesity or cancer. However, the limited bioavailability of these compounds hinders the ability to exert these beneficial effects. For this reason, many strategies focus on control release are being developed. On this sense, spray drying encapsulation is a rapid, inexpensive and reproducible method for bioactive compounds encapsulation. In this work, this technique has been applied using an experimental design based on response surface methodology to microencapsulate phenolic compounds belonging to phenolic acids, flavonoids and phenylpropanoids and to know the effect of the main technological factors on the effectiveness of microencapsulation process.

2. EXPERIMENTAL

2.1. Material and methods

- Phenolic compounds from *Lippia citriodora* and *Hibiscus sabdariffa* (Monteloeder®, Spain).
- Inuline as encapsulating agent (Fagron®, Netherlands).
- Commercial standards: 4-hydroxycoumarin, chlorogenic acid, gallic acid, quercetin and delphinidin-3-O-sambubioside.

2.2. Spray drying microencapsulation

Phenolic compounds were encapsulated by spray drying process using inuline as encapsulating agent (EA). The inuline belongs to a class of dietary fibers known as fructans, being available to control the release of bioactive compounds on gastrointestinal tract. Response surface methodology based on a central composite 2² design with central and star points (12 experiments) was applied. Inlet air temperature and ratio sample/inuline were selected as independent variable. Process factors and levels of experimental design used are shown in table 1. Response variables were yield and encapsulation efficiency (EE). These variables were calculated according to Equation 1 and Equation 2. Spray drying microencapsulation was carried out using a spray dryer model 4M8-TriX (Procept®, Zelzate, Belgium).

$$EE (\%) = \frac{(Total\ polyphenols - Superficial\ polyphenols)}{Total\ Polyphenols} \times 100 \quad (1)$$

$$Yield (\%) = \frac{Experimental\ powder\ (g)}{Theoretical\ powder\ (g)} \times 100 \quad (2)$$

Table 1. Experimental central composite 2² design

Process factors	Levels				
	- α	-1	0	1	α
Temperature (°C)	134.75	140	165	190	195.25
Sample/EA ratio	1:0.34	1:0.5	1:1.25	1:2	1:2.15

2.3. Analytical characterization of phenolic compounds

Characterization of phenolic compounds present in both, samples and microparticles, was carried out by using High Performance Liquid Chromatography coupled to time of flight mass spectrometry (HPLC-DAD-ESI-TOF/MS). Concentrations of each compound were determined using the area of each individual compound (three replicates) and by interpolation in the corresponding calibration curve. Phenolic compounds which had no commercial standards, were tentatively quantified on the basis of other compounds having similar structures. Calibration curves shown a good linearity over the study range ($r^2=0.9943$).

Concentrations of each compound were used to determine EE (equation 1) where extraction of non-encapsulated and encapsulated compounds in microparticles was carried out according to Idham, Z., Muhamad, I. I., & Sarmidi, M. R. (2012) [1].

3. RESULTS AND DISCUSSION

Figure 1 displays the Extracted Ion Chromatograms (EIC) of the main phenolic compounds obtained by HPLC-DAD-ESI-TOF/MS. Four compounds were characterized as phenolic acids, twelve compounds were flavonoids and six of them phenylpropanoids.

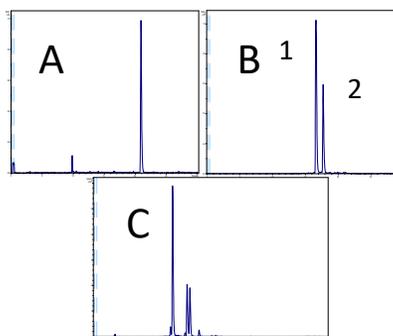


Figure 1. EIC of chrysoeriol-7-diglucorinide (A), verbascoside (1) and isoverbascoside (2) (B) and chlorogenic acid isomers (C) obtained by HPLC-DAD-ESI-TOF/MS.

Table 2 shows that a higher yield than 74% was achieved for all experimental conditions. Conditions with higher yield were conditions A (165°C; 1:2.15), E (165°C; 1:1.25) and I (140°C; 1:2).

Concerning EE, the obtained values were in the range from 34.7 to 66.2. Flavonoid showed the highest EE, above 60%, whereas phenolic acids had an EE of 52.5% and phenylpropanoids showed the smaller EE, 36%.

Table 2. EE and yield obtained by Spray-drying encapsulation

Condition (Temperature; Sample/EA ratio)	Yield (%)	EE (%)
A (165°C; 1 :2.15)	83.91	66.22
B (140°C; 1 :0.5)	81.28	34.69
C (190°C; 1 :2)	74.71	52.93
D (165°C; 1 :0.34)	79.60	36.67
E (165°C; 1 :1.25)	84.47	52.17
F (190°C; 1 :0.5)	75.98	38.06
G (165°C; 1 :1.25)	78.13	50.26
H (165°C; 1 :1.25)	79.93	49.64
I (140°C; 1 :2)	85.35	62.53
J (195.25°C; 1 :1.25)	80.63	52.13
K (165°C; 1 :1.25)	82.12	56.55

L (134.75°C; 1 :1.25)	83.61	43.82
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Individual phenolic compounds displayed a variable trend of encapsulation behaviour. With regard to sample/inuline ratio, the higher amount of encapsulation agent was, the greater EE was achieved.

Unexpectedly, stability of phenolic compound was not affected by temperature, being this independent variable not a determinant factor for EE.

Figure 2 shows the estimated response surface for a representative phenolic compound evaluated.

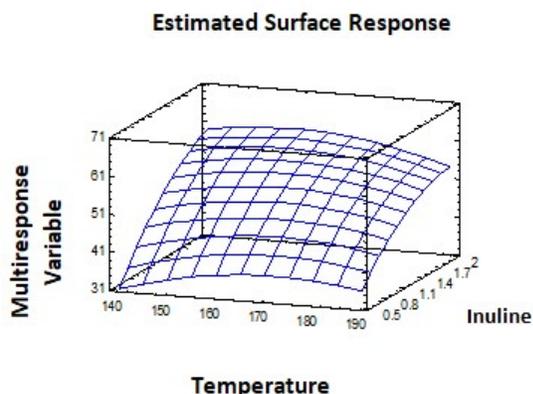


Figure 2. Estimated Surface Response of β -Hydroxyverbascoside

4. CONCLUSIONS

Phenolic compounds belonging to phenolic acids, flavonoids and phenylpropanoids were encapsulated with a high yield up to 85% determining that the temperature did not affect the stability of bioactive compounds. However, the independent variable sample/EA ratio was the most determinant to achieve higher EE. Future trends are guaranteed to evaluate the controlled release by *in vitro* digestion essays.

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