

# Stabilization of luteolins from weld (*Reseda luteola* L.) and sawwort (*Serratula tinctoria* L.) by microencapsulation for natural dyeing

Carmo Serrano<sup>1,2(\*)</sup>, Margarida Sapata<sup>1</sup>, M. Conceição Oliveira<sup>3</sup>, Isabel Calha<sup>1</sup>, Luís Sá e Melo<sup>4</sup>

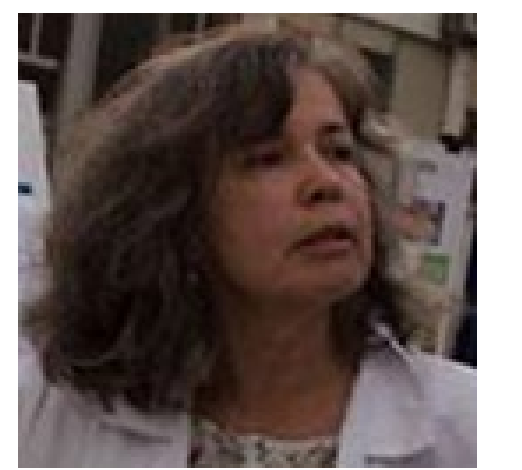
<sup>1</sup>Instituto Nacional de Investigação Agrária e Veterinária (INIAV, I.P.), Avenida da República 2780-157 Oeiras, Portugal

<sup>2</sup>LEAF – Linking Landscape, Environment, Agriculture and Food – Research Center, Instituto Superior de Agronomia, Universidade de Lisboa, Tapada da Ajuda, 1349-017 Lisboa, Portugal

<sup>3</sup>Centro de Química Estrutural, Institute of Molecular Sciences, Instituto Superior Técnico, Universidade de Lisboa, 1049-001 Lisboa, Portugal

<sup>4</sup>Aroma do Vale R. Luis Brandão de Melo,3, 3700-291 São João da Madeira

Contact: email carmo.serrano@iniav.pt



## Introduction

Weld and sawwort, are used for natural dyeing, on account to the presence of luteolins and derivatives. However, the use of luteolins has been limited due to instability, which is affected by several factors during processing, such as temperature, pH and light leading to the quinones formation, when compared with synthetic dyes. The aim of this work was to study the effect of luteolins microencapsulation by freeze-drying and spray-drying on color stability, using maltodextrin and Arabic gum as wall material.

## Mat & Methods

Flavonoid compounds were isolated by solvent extraction from dried and powdered plants. Freeze-drying and spray-drying were used to produce the water-soluble shell-coated matrix capsules with maltodextrin or Arabic gum as carrier agents. Microcapsules characterization were realized by physical methods, Scanning Electron Microscopy (SEM) and colorimetry assays. The extracts of non-encapsulated and encapsulated flavonoid compounds were elucidated by Liquid Chromatography and Tandem Mass Spectrometry. Stability tests of the release compounds were done at different pH and at 80°C. Overall, the results will show the dye plants with higher sources of flavonoid dye compounds, revealing the potential multipurpose use of the weld and sawwort plants.

## Results

### Solubility, encapsulation yield (EY) and encapsulation efficiency (EE)

The two encapsulation processes, for both encapsulating agents, increase the dispersion of the hydroalcoholic extracts of weld and sawwort.

Spray-drying process showed lower values for encapsulation yield and encapsulation efficiency than the freeze-drying process for both plants, regardless of the encapsulation agents.

### Morphology by Scanning Electron Microscopy (SEM)

The size distribution of maltodextrin microcapsules exhibited a range between 1.0-12.0 µm and those of Arabic gum a wider size range 0.8-20.8 µm (Figure 2 – A and C). The sawwort maltodextrin microcapsules exhibited a size range between 0.9-10 µm, and those of Arabic gum ranging between 0.7-18.8 µm (Figure 2 – B and D).

### Degradation kinetics

The stability of weld flavone aglycones, at pH 3 and temperature of 80°C, showed a less detrimental effect on colour and in the content of the dye flavone aglycones obtained from encapsulation by freeze-drying with maltodextrin, since the Kd values were the lowest (Figure 3). The results show that the major degradation occurs in the spray-drying process and Arabic gum is the carrier agent that least protects flavones.

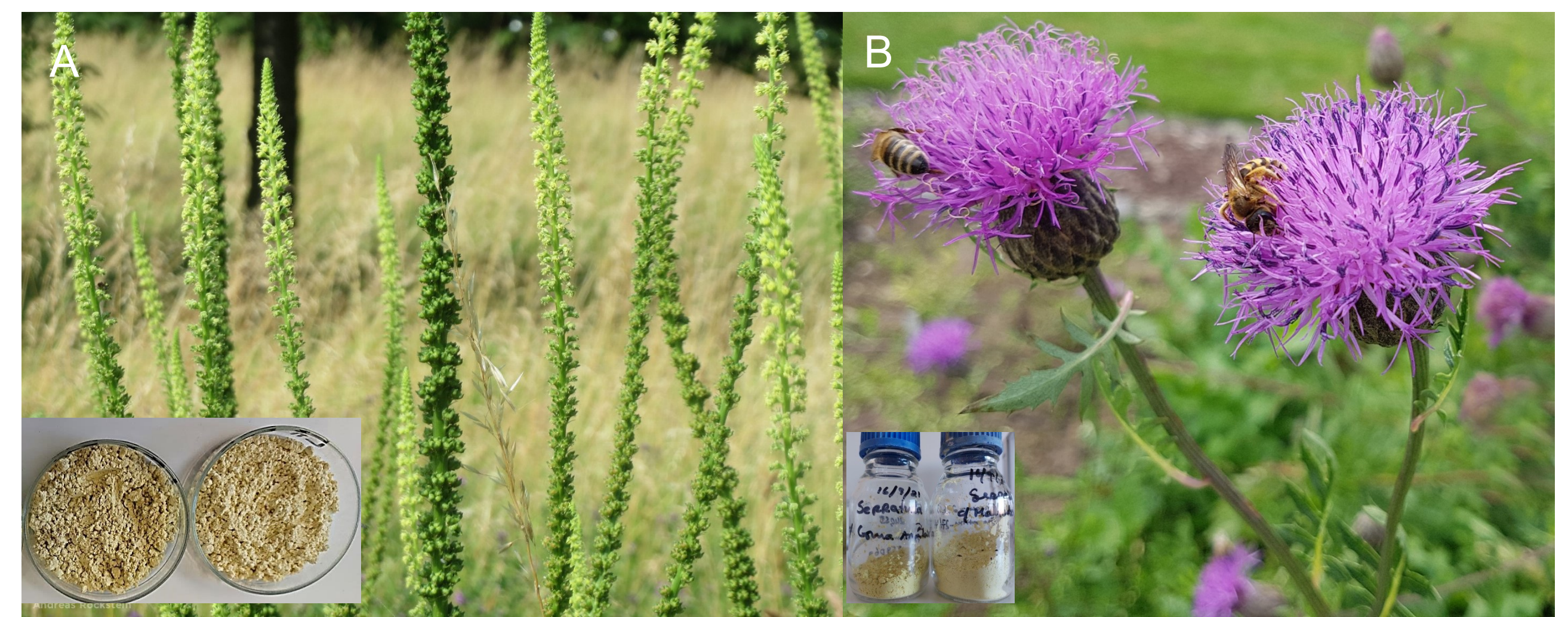


Fig. 1 Weld (A) and sawwort (B) with dyes in powder form.

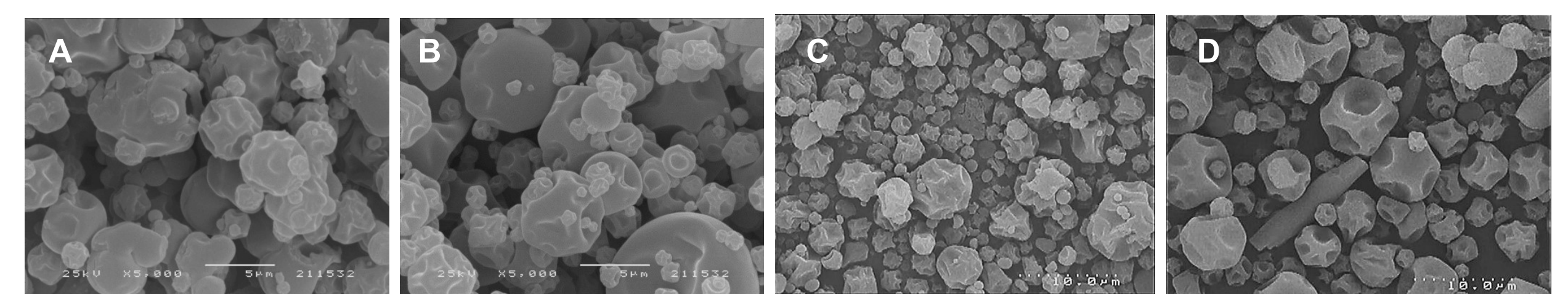


Fig. 2 SEM images of weld and sawwort microcapsules obtained by spray-drying by maltodextrin (A and C) and Arabic gum (B and D).

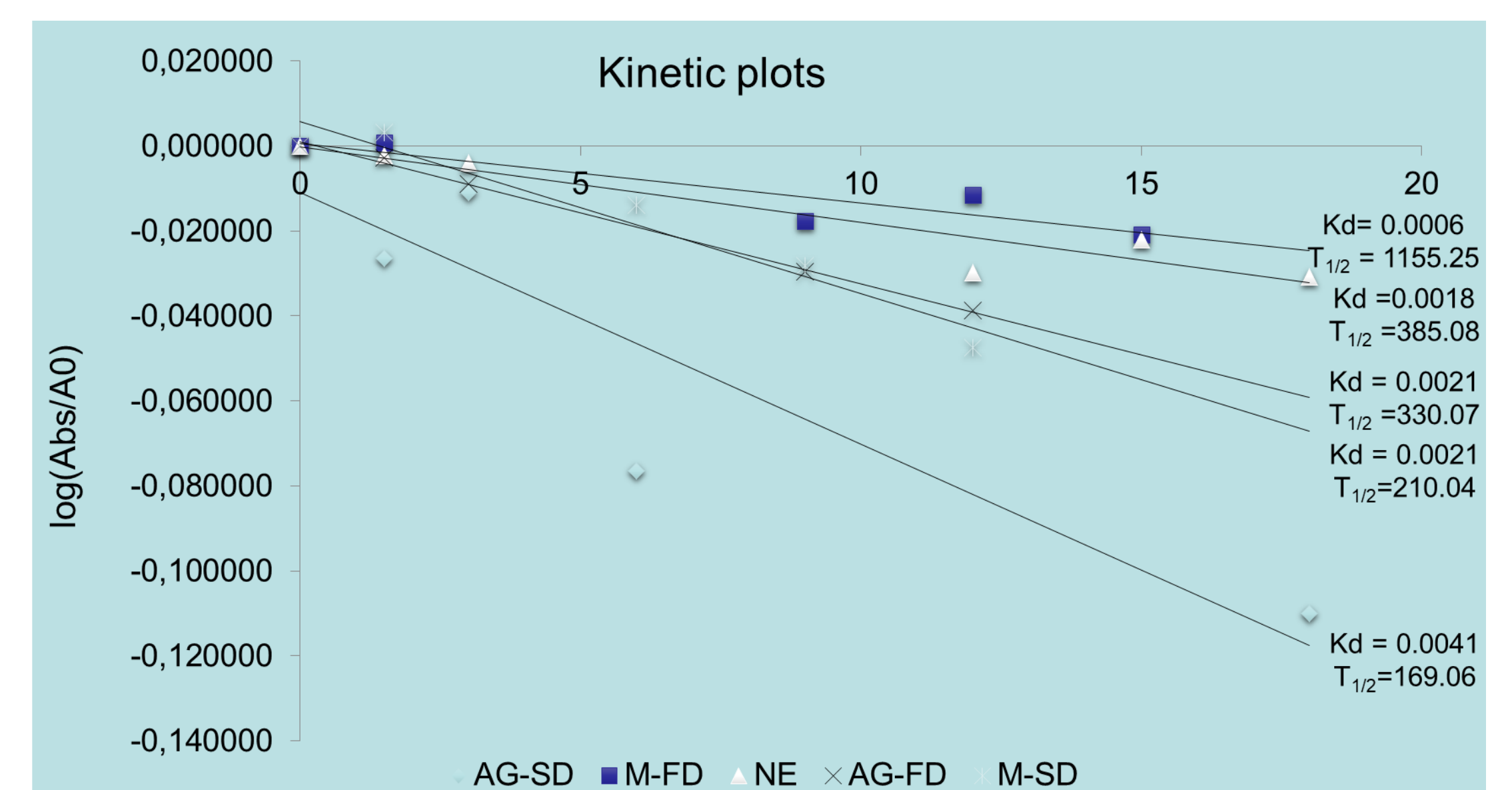


Fig. 3 Degradation kinetics of non-encapsulated and encapsulated flavonoids in weld extract as temperature (T=80°C) function.

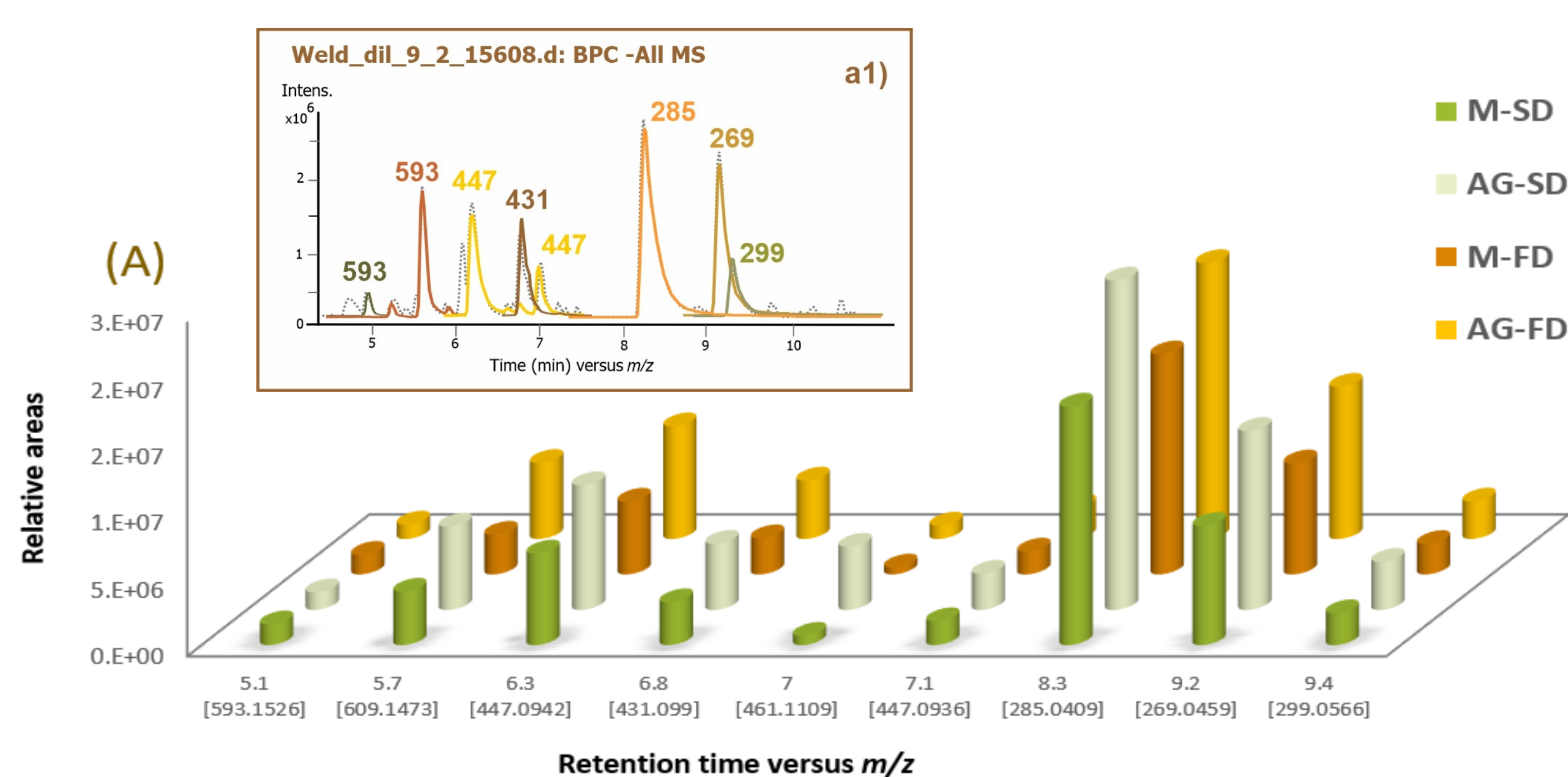


Fig. 4 (A) Comparative diagram of the relative abundances of flavonoid compounds present in freeze-drying weld extracts encapsulated with maltodextrin (M-FD) and Arabic gum (AG-FD), and spray-drying extracts encapsulated with maltodextrin (M-SD) and Arabic gum (AG-SD), respectively; LC-ESI(-)HRMS profile of the weld extract: (a1) extracted ion chromatograms for the main yellow chromophores.

### Yellow dye profile of weld (W) and sawwort (S) extracts

The results allowed the identification of the same number of flavonoid compounds with high relative abundance in the two drying processes, obtained in release tests from the microcapsules.

The microcapsules prepared with Arabic gum show higher relative abundances than those obtained with maltodextrin, indicating that this encapsulant retains a greater amount of the yellow flavonoid chromophores, for the two drying processes.

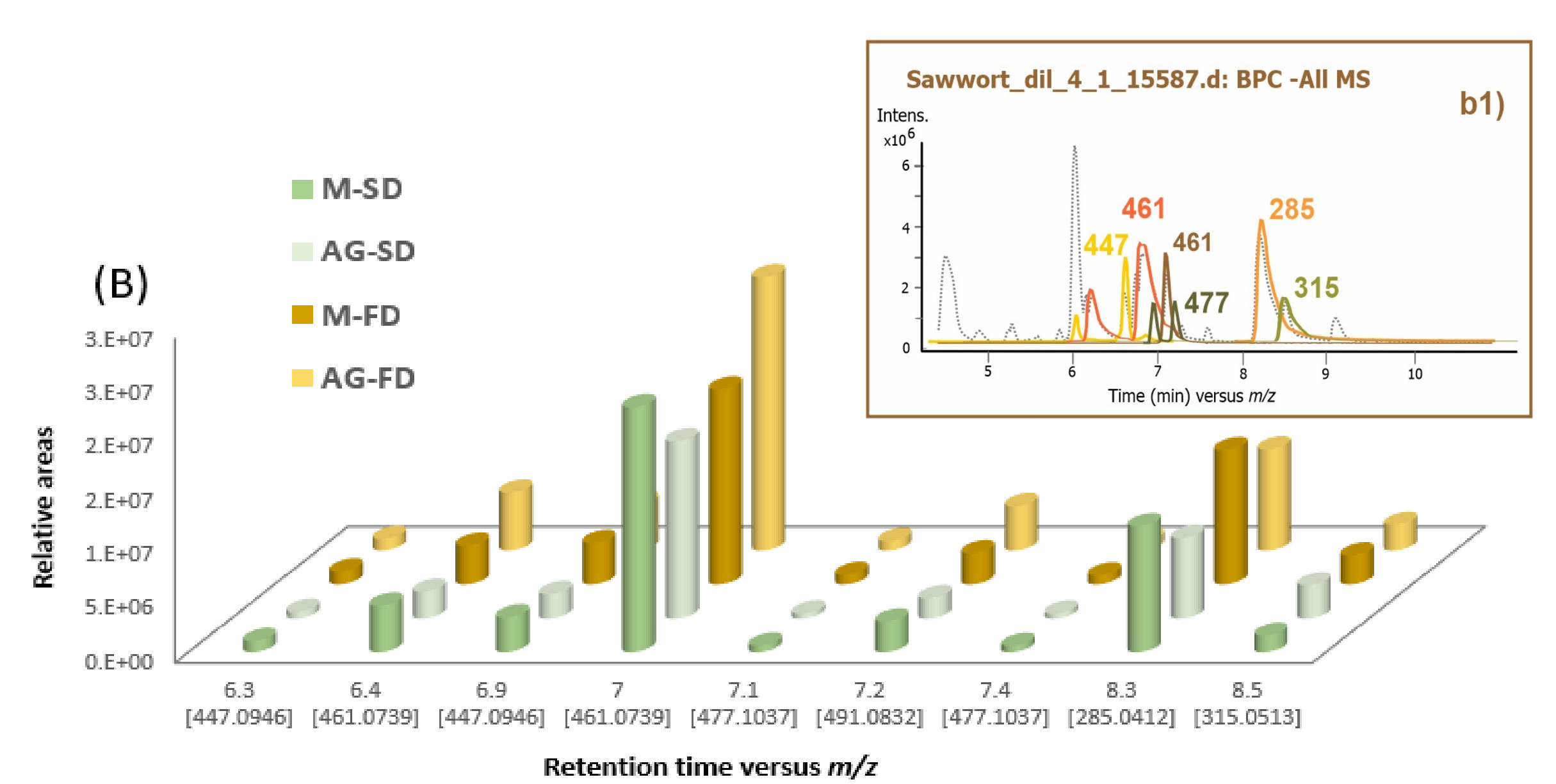


Fig. 5 (B) Comparative diagram of the relative abundances for the main yellow chromophores present in freeze-drying sawwort extracts encapsulated with maltodextrin (M-FD) and Arabic gum (AG-FD), and spray-drying extracts encapsulated with maltodextrin (M-SD) and Arabic gum (AG-SD), respectively; LC-ESI(-)HRMS profile of the sawwort extract: (b1) extracted ion chromatograms for the main yellow chromophores.

## Conclusions

Microcapsules of maltodextrin and Arabic gum showed better dispersion, high yields and good results for the encapsulation of weld and sawwort flavones in freeze-drying process.

The stability of weld flavone aglycones, at pH 3 and temperature 80°C, were higher in maltodextrin microcapsules obtained by freeze-drying process.

The microencapsulation of weld dye extracts in freeze-dried maltodextrin constitutes a promising application for the textile industry.



References: Serrano, C., et al. (2007-2008). Bulletin du Cietá, 84-85, 77-91. Serrano, C., et al. (2020). Acta Scientiarum Polonorum Technologia Alimentaria 19(1):57–71. Carota et al. (2017) Acta Hort. 760(1)99-104. Thuillot et al. (2022) J. Exp. Bot. 89:105-112.

Acknowledgements: Work developed within the project GO - Natural Dyeing - Use of natural dyes in natural fibres, financed by the Rural Development Programme - PDR2020, action 1.1 Operational groups. PDR2020-101-001. COMPETE 2020 - FCT - Foundation for Science and Technology, and UIDB/00100/2020 and UIDP/00100/2020 FCT funding.