

Freeze drying versus Spray drying to obtain Grapefruit IntegroPectin powder

Giulia Di Prima¹, Antonino Scurria², Giuseppe Angellotti^{3,1}, Elena Belfiore^{3,1}, Mario Pagliaro²,
Francesco Meneguzzo⁴, Rosaria Ciriminna² and Viviana De Caro¹

¹Dipartimento STEBICEF, Università degli Studi di Palermo

³Dipartimento DICHIRONS, Università degli Studi di Palermo

²ISMN-CNR, Consiglio Nazionale delle Ricerche sede di Palermo

⁴IBE-CNR, Consiglio Nazionale delle Ricerche sede di Sesto Fiorentino

INTRODUCTION & SCOPE

IntegroPectin is a new family of citrus pectins of broad biological activity obtained via hydrodynamic cavitation of citrus biowaste carried out in water only. These pectins have already been isolated by freeze drying, a time-consuming and expensive procedure difficult to be scaled up. This work aims to develop a simple, fast, efficient and industrially advantageous procedure for pectins isolation. To this scope the spray drying technique is here proposed and evaluated as, nowadays, it is a greatly appreciated method for food and pharmaceutical applications also considering an industrial point of view.



Freeze drying vs. Spray drying



Freeze drying

- 30 mL of aqueous extract previously stored at -80°C overnight
- Freeze drying for 3 days
- 0.014 mPa
- ≈ -50°C
- in the dark

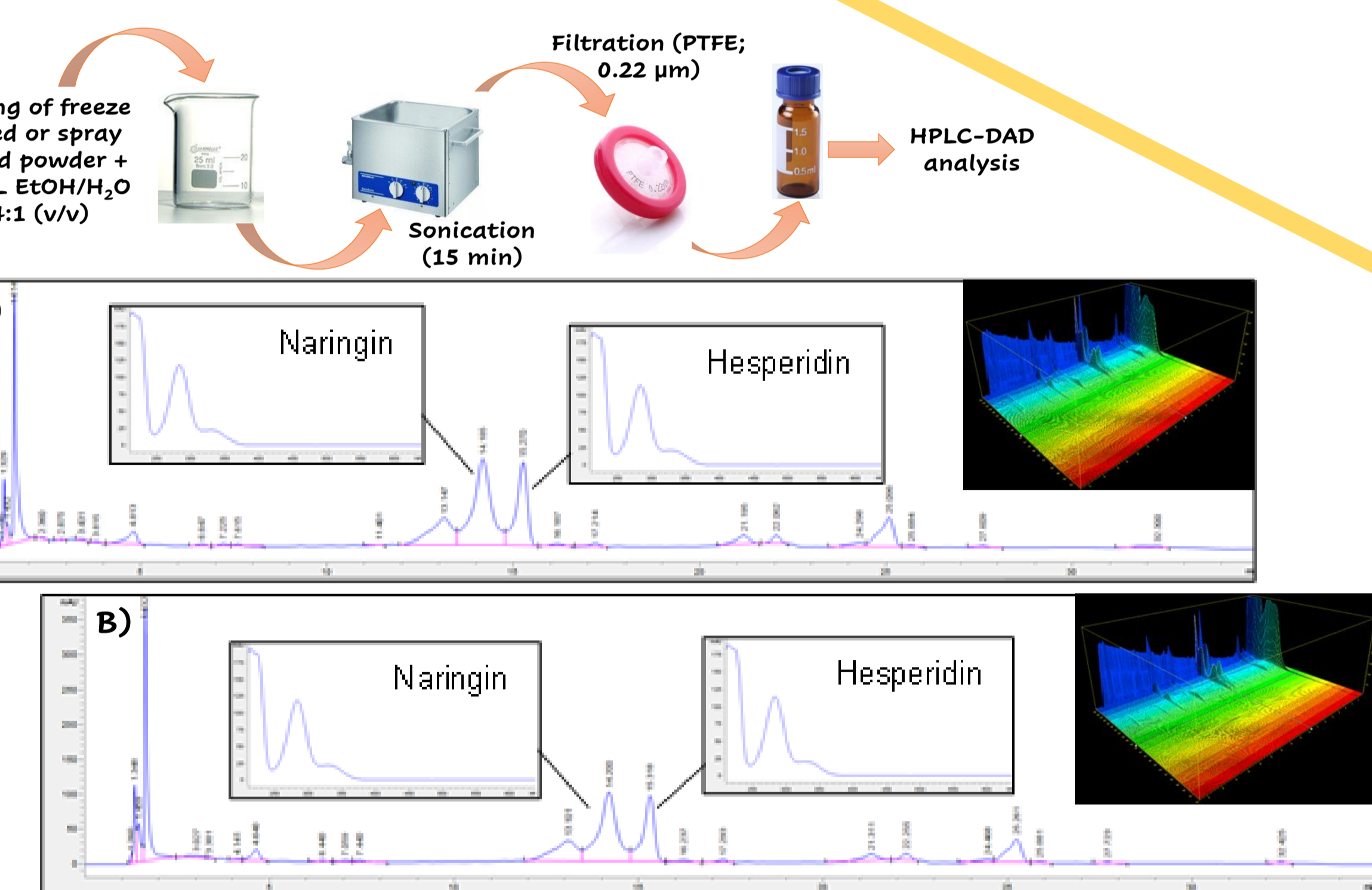
The yield of freeze drying process is assumed to 100% as this process does not lead to material loss
The yield of spray drying process is referred to the freeze drying one**

| Freeze drying | Spray drying |
|---|---|
| *Yield %: 100% | **Yield %: 95.03 ± 6.79% |
| 30 mL → 3 days | 100 ml → 30-50 min |
| Difficult to scale up Expensive | Easy to scale up Low capital and operational costs |
| Appearance: light yellow, vaporous and needle-like powder | Appearance: light yellow, fine powder |
| pH after water dissolution: 4.41 ± 0.06 | pH after water dissolution: 4.38 ± 0.03 |

Spray drying

- 100 mL of aqueous extract □
- Inlet temperature: 110°C □
- Solution flow: 200 mL/h □
- Nitrogen aspiration: 100% □
- Cooling temperature (inert loop): 17°C □
- Starting water equilibration: 10 min □
- Final water cleaning: 10 min □

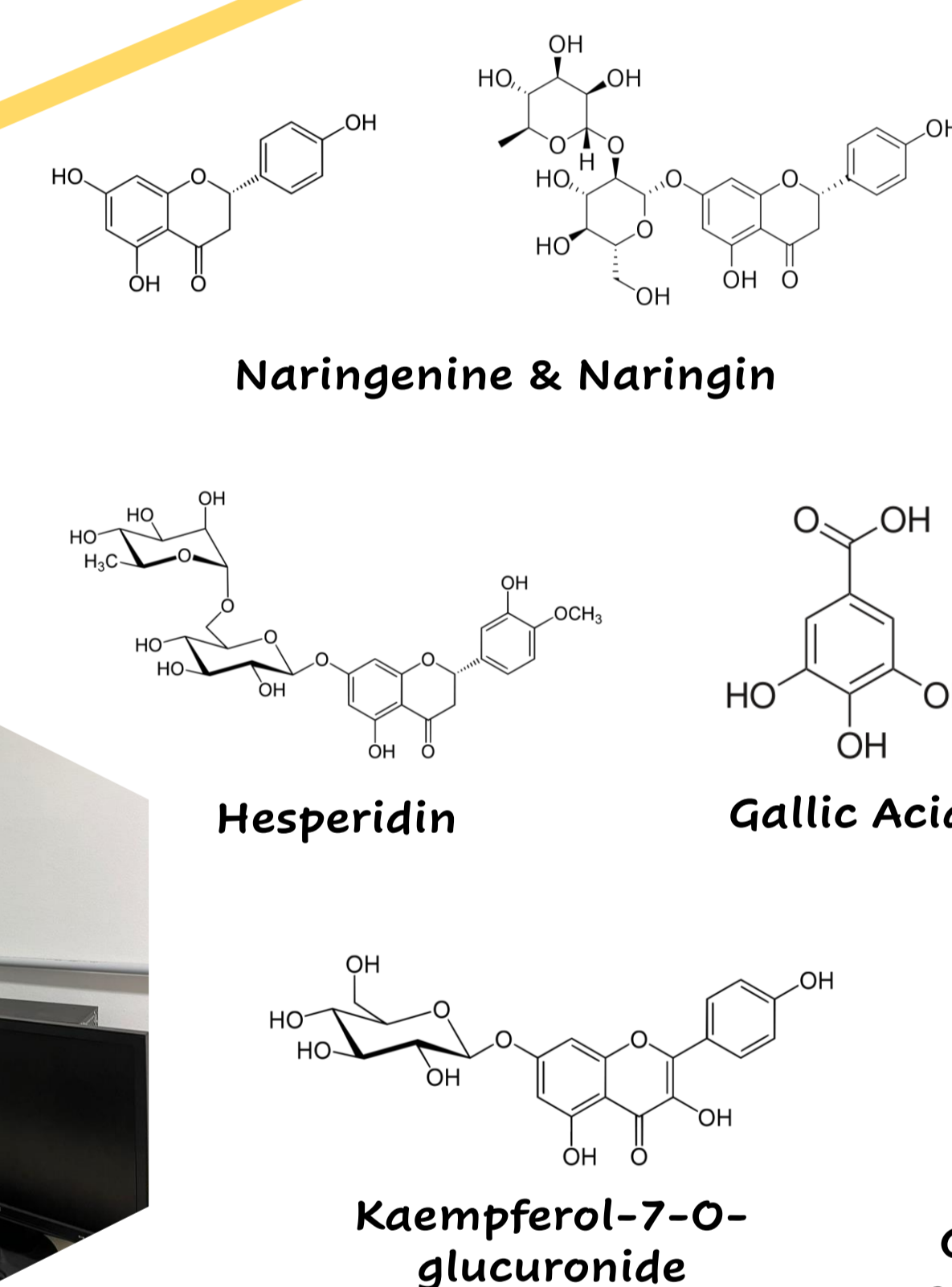
HPLC-DAD



| Sample | Naringin | Hesperidin |
|---------------------|-------------|-------------|
| Freeze dried powder | 3.08 ± 0.03 | 1.42 ± 0.03 |
| Spray dried powder | 2.87 ± 0.06 | 1.34 ± 0.03 |

Quantitative results of the HPLC-DAD analysis expressed as mg recovered in 100 mg of powder ± SE

Folin-Ciocalteu



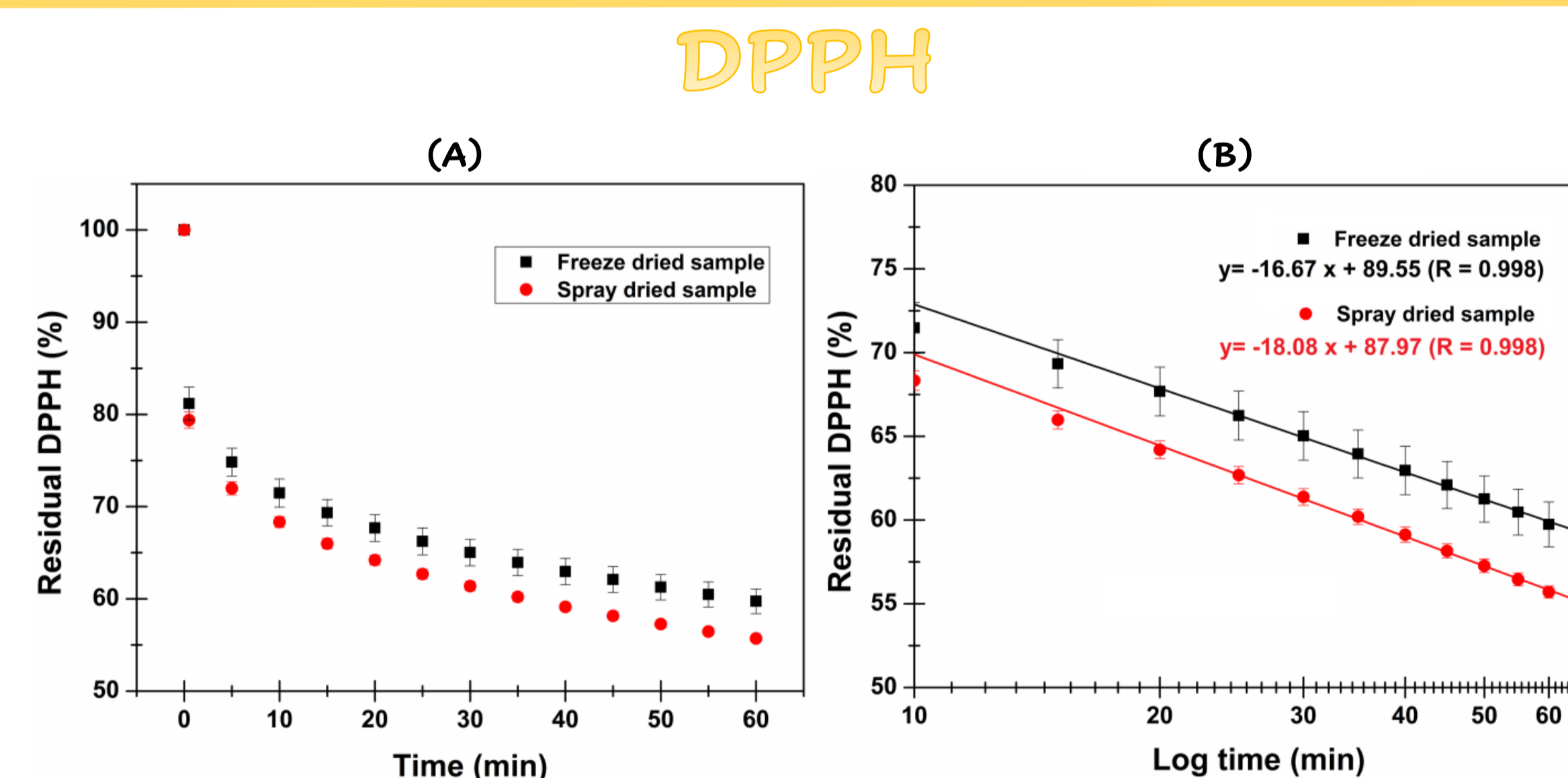
- 5 mg/mL of freeze dried or spray dried powder in ultrapure water
- Colorimetric response in presence of phenolic compounds
- 50 μL of sample into a plastic tube containing 2 mL of ultrapure water
 - + 130 μL of Folin-Ciocalteu reagent
 - + 370 μL of Na₂CO₃ water solution (1.89M)
- 2 h
 - Room temperature
 - In the dark

| Sample | Total phenolic content |
|---------------------|------------------------|
| Freeze dried powder | 3.85 ± 0.31 |
| Spray dried powder | 4.03 ± 0.07 |

Quantitative results of the Folin-Ciocalteu analysis expressed as equivalent to gallic acid (mg) recovered in 100 mg of powder ± SE

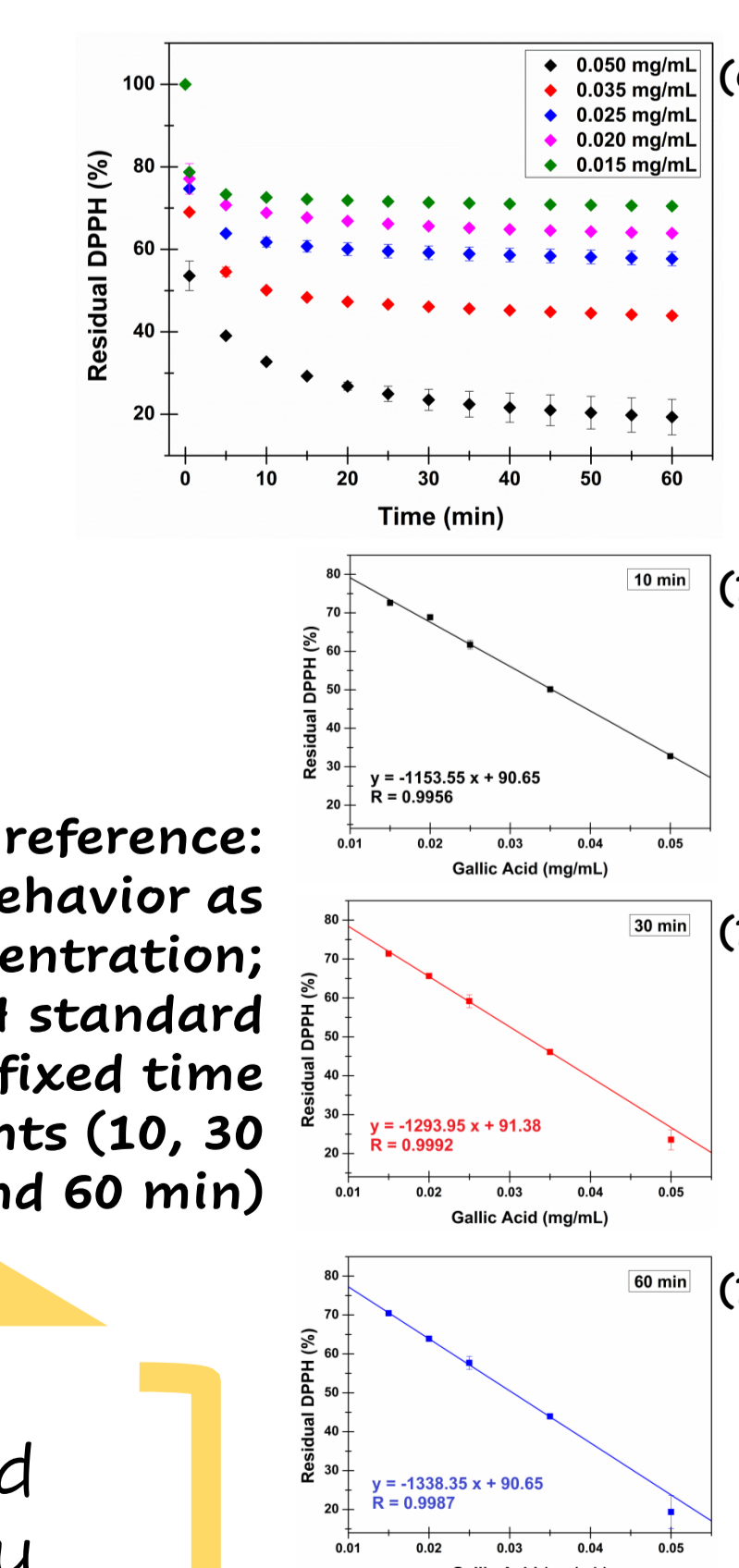
HPLC-DAD

DPPH

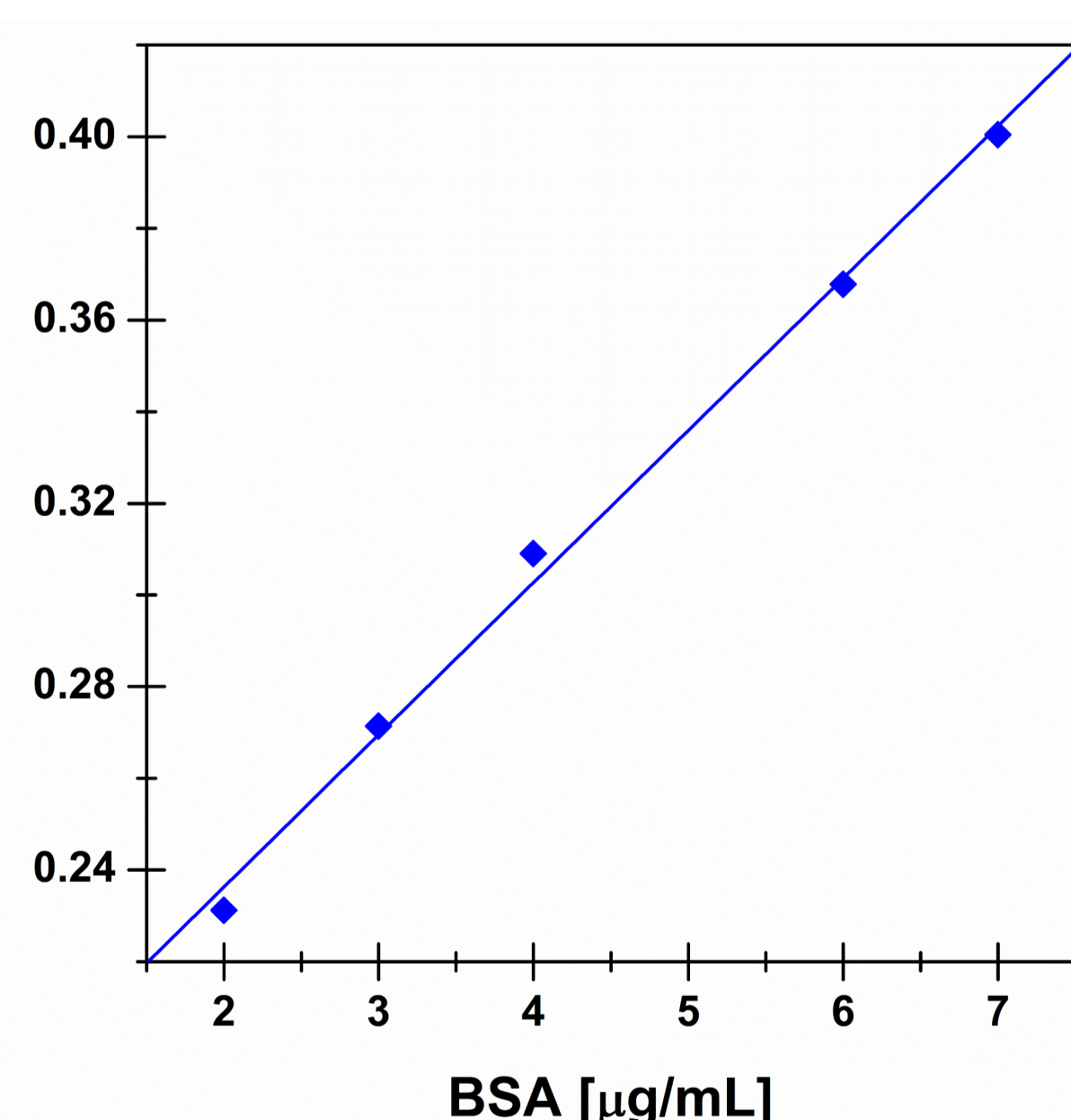


| | Freeze dried powder | Spray dried powder |
|----------------------------|---------------------|--------------------|
| Calibration curve (10 min) | 0.0166 ± 0.0013 | 0.0193 ± 0.0005 |
| Calibration curve (30 min) | 0.0204 ± 0.0011 | 0.0232 ± 0.0004 |
| Calibration curve (60 min) | 0.0231 ± 0.0009 | 0.0261 ± 0.0003 |

Antioxidant power expressed as equivalent to gallic acid (mg/mL) ± SE



Bradford Assay



| Sample | Total protein content |
|---------------------|-----------------------|
| Freeze dried powder | 0.246 ± 0.004 |
| Spray dried powder | 0.062 ± 0.004 |

Quantitative results of the Bradford assay investigation expressed as equivalent to bovine serum albumin (mg) recovered in 100 mg of powder ± SE

Bradford assay: bovine serum albumin calibration curve

Bradford Assay

Conclusions

Freeze dried and Spray dried IntegroPectin powders are superimposable in terms of total phenolic content, amount of some representative polyphenols and chromatographic profile. The Spray dried IntegroPectin demonstrates a slightly higher antioxidant power. The Spray Drying method offers the following further advantages:

- Remarkable Yield (>95%)
- Quick procedure
- Easy to scale up
- Possible inactivation of oxidative and hydrolytic enzymes leading to bioactive polyphenols protection from subsequent degradation

Funding

This research was funded by the Ministero dell'Università e della Ricerca, PON FSE REACT-EU Research and Innovation 2014-2020 Action IV.5 "Dottorati su tematiche green" and Action IV.6 "Contratti di ricerca su tematiche Green", PON FSE-FESR R&I 2014-2020, Action I.1, "Dottorati innovativi a caratterizzazione industriale", and by the Regional ERDF Operational Program 2014-2020 of Sicily, Action 1.1.5, "CoSMetici della filiera vitivinicola biologica (SMILING)" no. 087219090480