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# Encapsulation of an omega-3 concentrate by Particles from Gas Saturated Solutions (PGSS)-drying

## Particle characterization and oxidative stability


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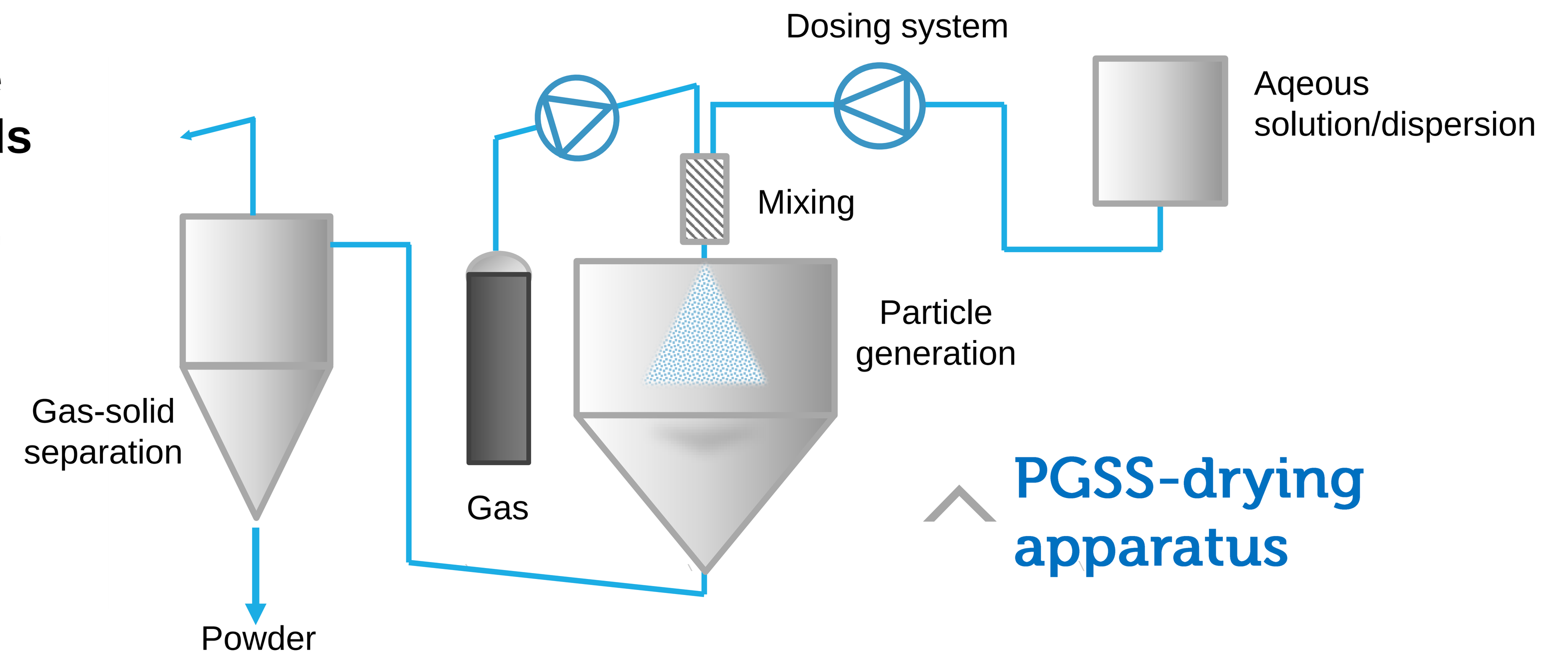
## INTRODUCTION

### Particles from Gas-Saturated Solutions (PGSS)-drying [1]

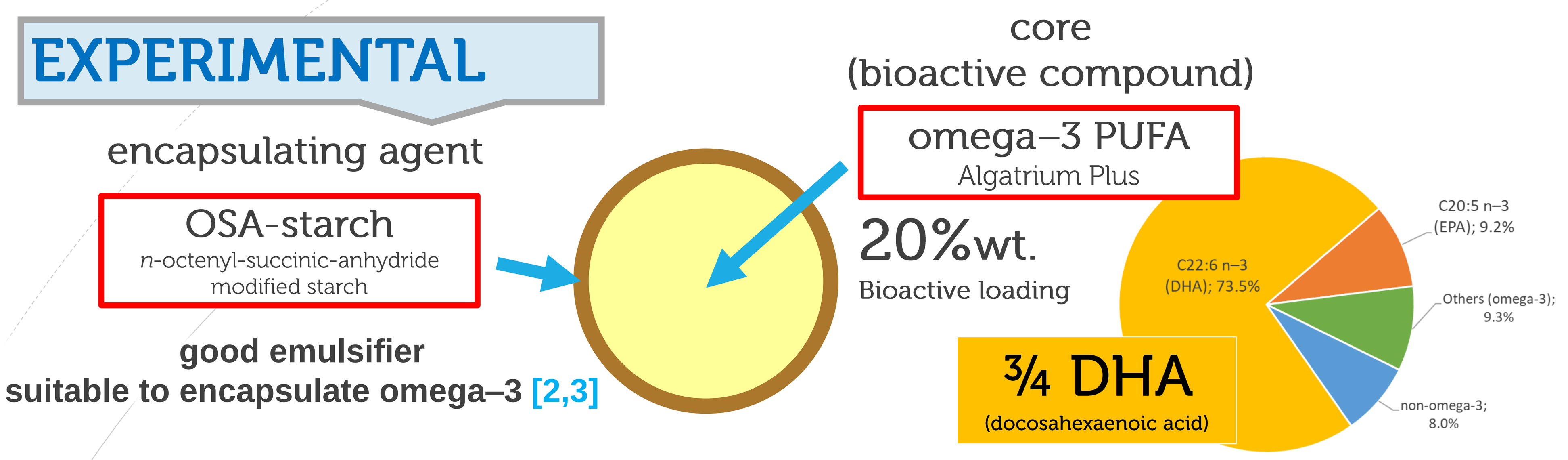
**Alternative encapsulation technique** based on the use of **Supercritical Carbon Dioxide ($SC\text{-}CO_2$)** to **micronize** and **dry aqueous solutions/dispersions of bioactive compounds**

- $SC\text{-}CO_2$ dissolves into the aqueous solution/dispersion at moderate p (10-15 MPa), **expanding the mixture** and **lowering its viscosity**
- The mixture is depressurized through a nozzle and dissolved $CO_2$ is rapidly released, **enhancing particle atomization**
- The **intense cooling** effect of the expansion and the **inert atmosphere** in the spraying tower **protect the bioactive compounds**

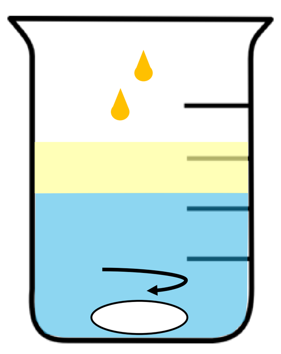



PGSS-drying apparatus

## EXPERIMENTAL

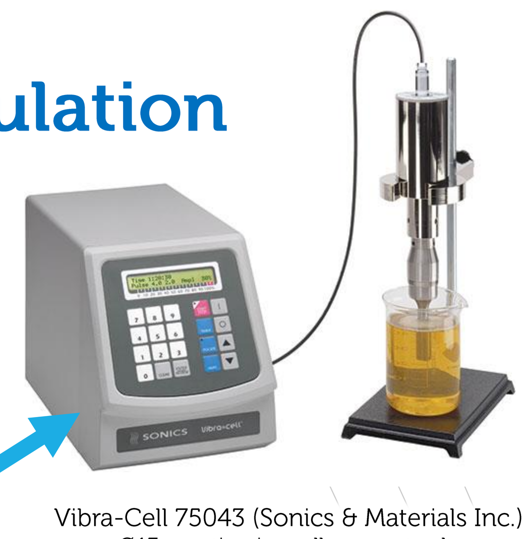



### Emulsion Formulation



6 % omega–3 concentrate
24 % OSA-starch
70 % distilled water

US-assisted emulsification



Vibra-Cell 75043 (Sonics & Materials Inc.)
Ø13 mm titanium alloy sonotrode
20 kHz 750 W

### Drying Conditions

| PGSS-drying based on [3] | |
|---|---|
| Inlet T (static mixer) | 120 ºC |
| Outlet T (spraying tower) | 55 ºC |
| pressure | 10 MPa |
| GPR (gas-to-product ratio) | 30 g $CO_2$/g aq. feed |
| nozzle | Ø0.4 mm |

| Spray-drying | |
|---|---|
| Inlet T | 155 ºC |
| Outlet T | 100 ºC |
| Feed | 3.0 g/min |
| $N_2$ Flow | 360 L/h |
| nozzle | Ø1.5 mm |

Buchi B-290 mini Spray-dryer

| Freeze-Drying | |
|---|---|
| pressure | 1.5 · $10^{-4}$ mbar |
| time | 48 h |
| **Freezing methods** | |
| -20 ºC overnight | |
| Liquid $N_2$ (-196 ºC) | |

Labconco Freeze-Dry System

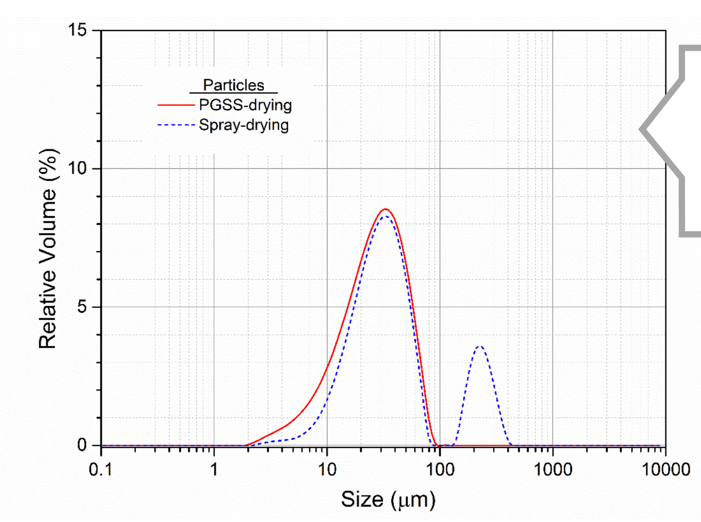
## RESULTS

| Emulsion /drying method | | EE (day 0) (%) | Yield (%) | Bioactive load (mg/g) | Moisture (%) |
|---|---|---|---|---|---|
| PGSS-drying | | 97.9[b] ± 0.3 | 61 ± 1 | 191 ± 8 | 3.3 ± 0.3[a] |
| Spray-drying | | 97.5[b] ± 0.1 | 30 ± 1 | 187 ± 3 | 5.6 ± 0.2[c] |
| Freeze-drying | -20 ºC overnight | 95.8[c] ± 0.2 | 99 ± 1 | 192 ± 2 | 4.66 ± 0.1[b] |
| | liq $N_2$ | 98.6[a] ± 0.1 | 99 ± 1 | 192 ± 2 | 4.7 ± 0.1[b] |

- High EE no matter the drying method
- Low yield in spray-drying
- High bioactive load
- Low moisture

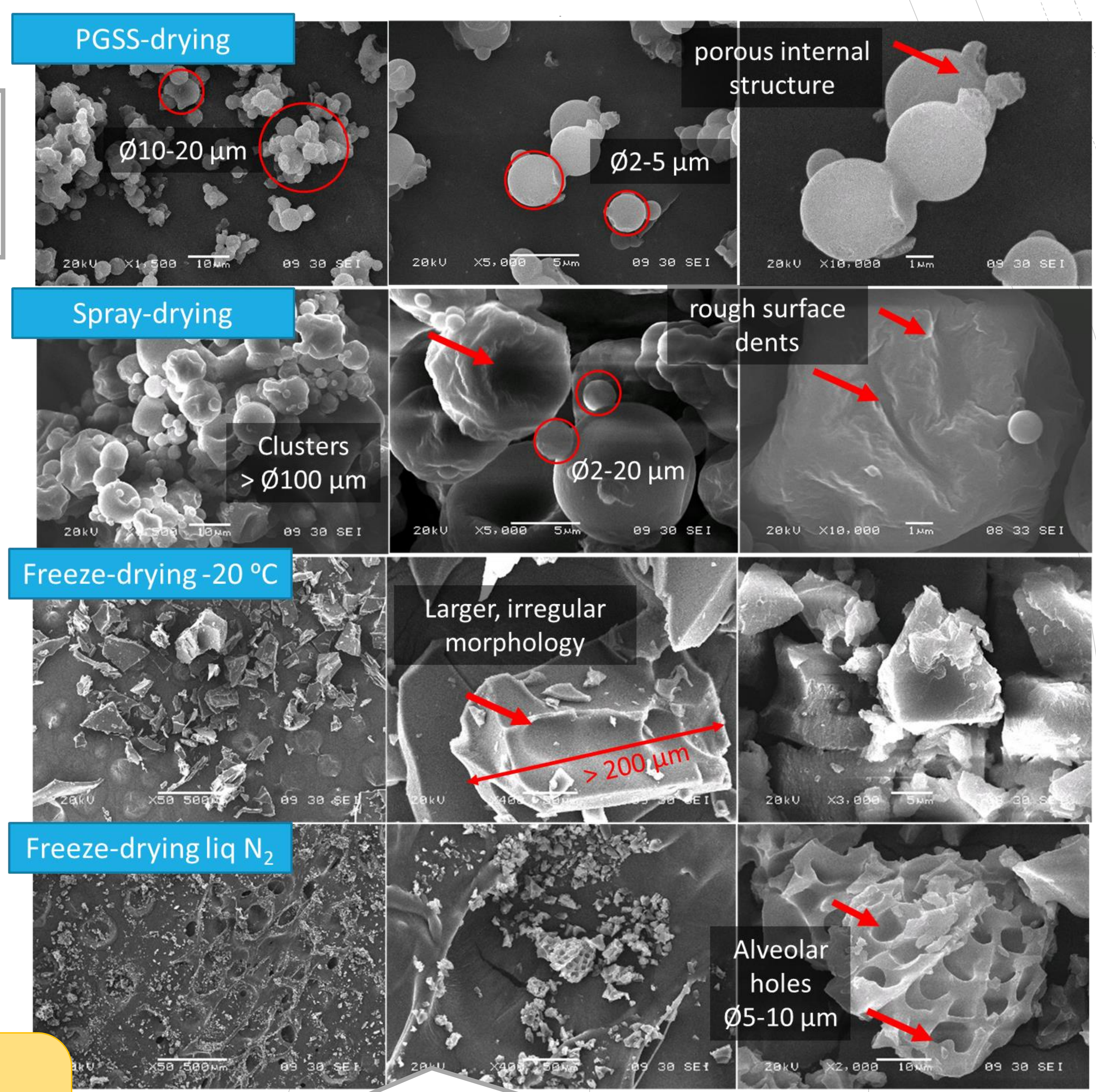
$$\text{Encapsulation Efficiency, EE }(\%) = \frac{\text{total oil} - \text{non-encapsulated oil}}{\text{total oil}} \times 100$$

### Particle Size Distribution




PGSS-drying – monomodal $d_{0.5}$ = 28.6 µm
Spray-drying – bimodal $d_{0.5}$ = 35.4 µm

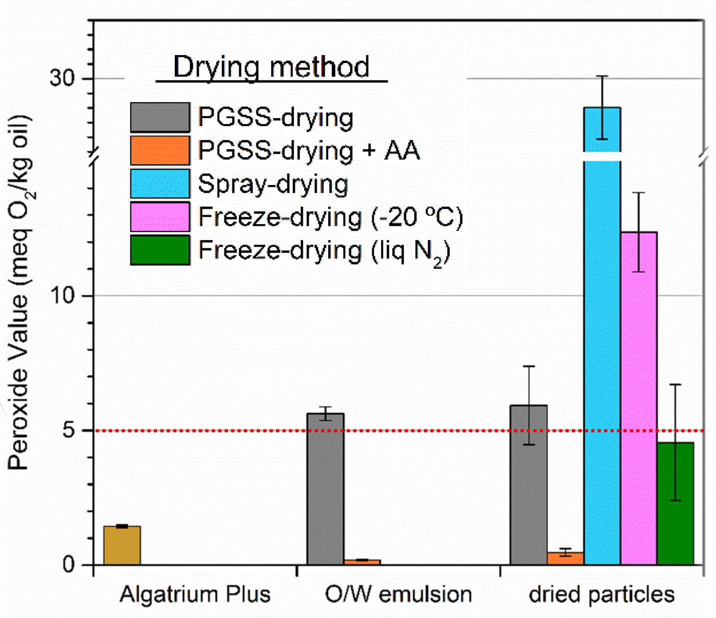
PGSS-drying promotes efficient atomization [4]

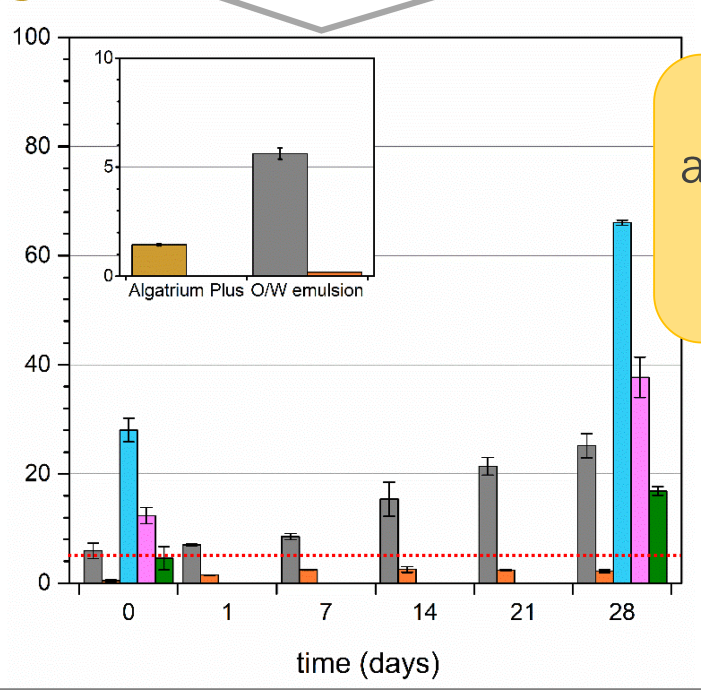



- PGSS-drying – spherical // Ø1.5-5 µm up to 10 µm internal porous structure
- Spray-drying – spherical // Ø1-10 µm up to 20 µm (larger) rough surface and dents collapse of hollow core
- Freeze-drying – irregular // some dimensions > 200 µm internal porous structure

### Oxidation - Peroxide Value

- Oxidation increases during processing
- Ascorbic acid prevents oxidation of omega–3
- Highest oxidation in spray-drying
- Conventional freezing at ambient [oxygen]
- PGSS-drying and FD with liq $N_2$ are comparable




PGSS-drying + ascorbic acid protect omega–3 from oxidation [5]


### REFERENCES

[1] Türk, M. (Ed.) (2014). Particle Formation with Supercritical Fluids Challenges and Limitations, Supercritical Fluid Science and Technology (Vol. 6). Elsevier.
[2] de Paz, E., Martín, T., Bartolomé, A., Largo, M., Cocero, M. J. (2014). Food Hydrocolloids, 37, 14–24.
[3] Varona, S., Martín, A., Cocero, M. J. (2009). Chem. Eng. Process.: Process Intensification, 48(6), 1121–1128.
[4] Martín, Á., Weidner, E. (2010). J. Supercrit. Fluids, 55, 271–281.
[5] de Paz, E., Martín, Á., Cocero, M. J. (2012). J. Supercrit. Fluids, 72, 125–133.



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