FOOD stars — Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS

European Commission

Encapsulates in food applications (case studies)

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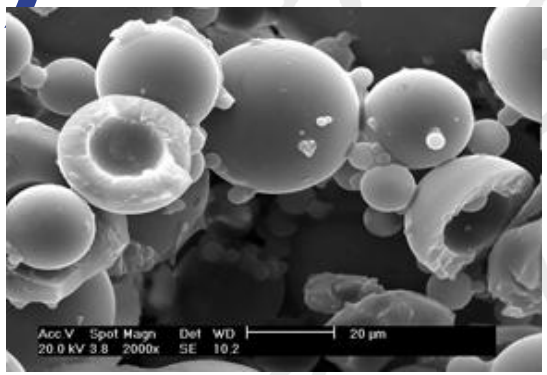

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Application 1 - Encapsulation of orange essential oil

Reference: "Encapsulation efficiency and thermal stability of orange essential oil microencapsulated by spray drying and by coacervation." Müller P.S., Perussello C.A., Zawadzki S.F., Scheer A.P. *Boletim do Centro de Pesquisa em Alimentos*, 33(2), 2016.

- Orange essential oil (OEL) is a formulation ingredient in foods, household cleaners and bath products. Can also be administered orally to help boost immunity, alleviate anxiety, anger and depression and relax muscular and nervous spams;
- Brazil is the third largest exporter of essential oil in the world, after only the USA and France. 91% of the whole production consists of essential oil of citrus, mainly oranges (80%), by-products of the juice industry.

In the food industry, OEL

- Enhances aroma and flavour of citric juices, soft drinks, candies, ice creams, cakes and biscuits
- However is **highly volatile , thermal sensitivity**

- **Objective of this research:** Assess quality of OEL obtained by two different methods, coacervation and SD

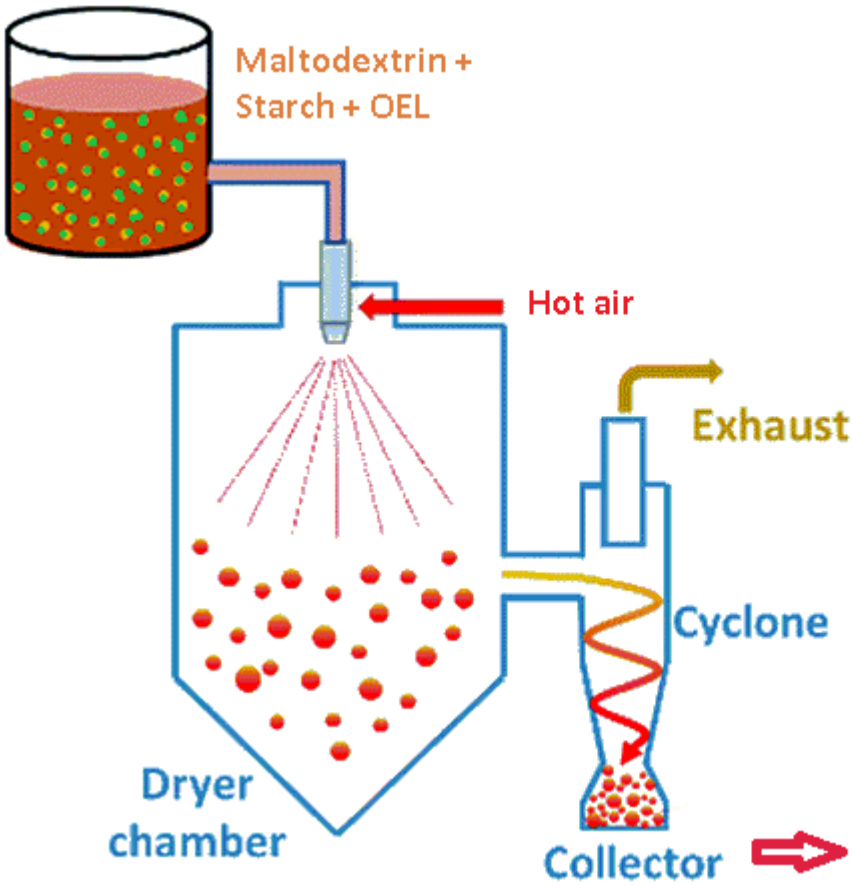
Methods for obtaining the microparticles

Microencapsulation method	Carrier	Crosslinking agent	Drying method
Spray drying	Starch and maltodextrin	None	Spray drying (150°C)
Coacervation	Sodium alginate	$CaCl_2$ 1%	Oven (30°C)

- Carrier materials are different according to the encapsulation method!
- Different encapsulation methods will result in different:
  - Morphology and size of the microparticles
  - Encapsulation efficiency
  - Release mechanisms and rates

The best method will depend on the product **application**

➢ Encapsulation by spray-drying

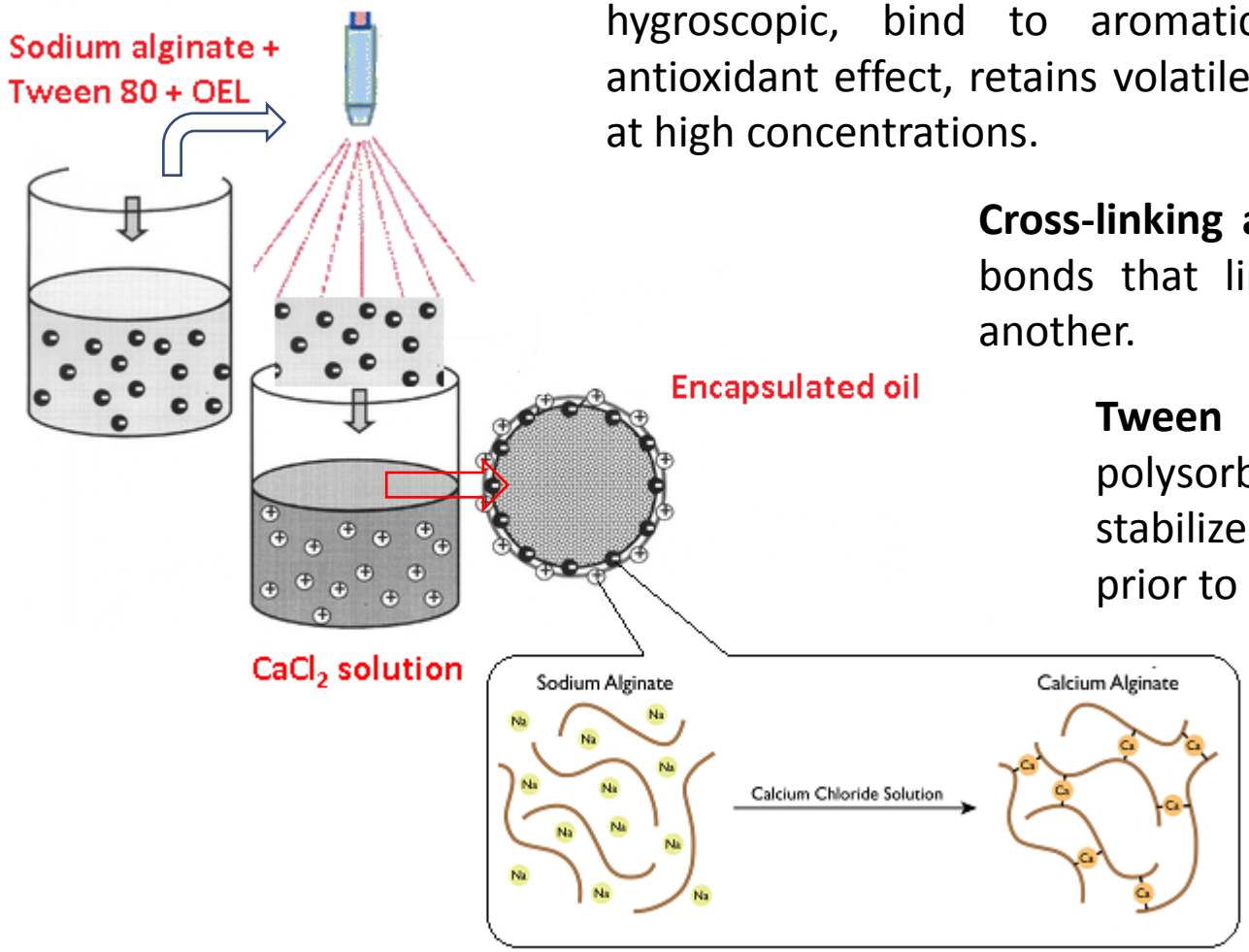


Maltodextrin: low cost, low hygroscopicity, bind to aromatic compounds, low viscosity at high concentrations, forms a film protecting the volatile material, antioxidant effect, retain volatiles (80 to 100%), **however does not act as an emulsifier.**

Modified starch: stabilizes emulsions (surfactant effects), low viscosity, good solubility in water, retention of volatiles over 93%, encapsulating and surfactant effects, **but exerts little protection against oxidation** during storage.

Maltodextrin + Starch

➢ Encapsulation by coacervation



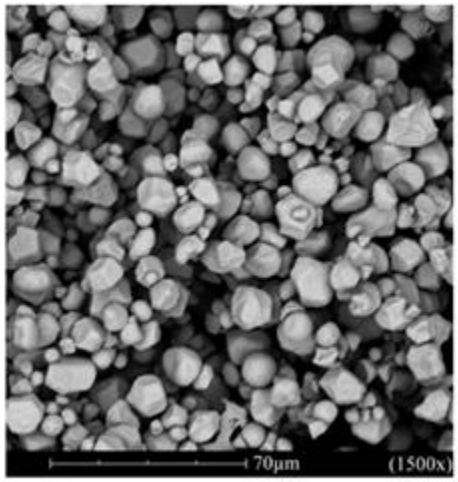
Sodium alginate: forms hydrogels, films, spheres, micro and nanoparticles in the presence of ions such as Ca^{++}, slightly hygroscopic, bind to aromatic compounds, low cost, antioxidant effect, retains volatiles (70-100 %), high viscosity at high concentrations.

Cross-linking agent: $CaCl_2$ helps forming bonds that link one polymer chain to another.

Tween 80: non-ionic surfactant polysorbate 80 was used to stabilize the oil-water emulsion prior to coacervation.

O/A emulsion formed by SA + $CaCl_2$ + Tween 80 + OEL

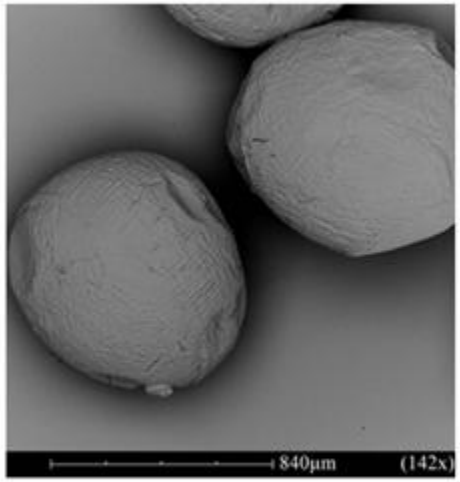
➢ Morphology (SEM) and sizing (OM)



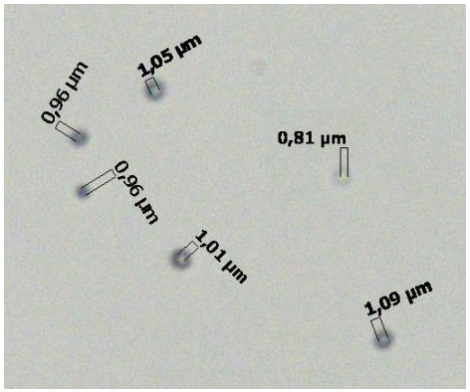
spray-drying

Spray-drying = **microspheres** of 1.02 µm average diameter (0.31 to 1.89 µm)

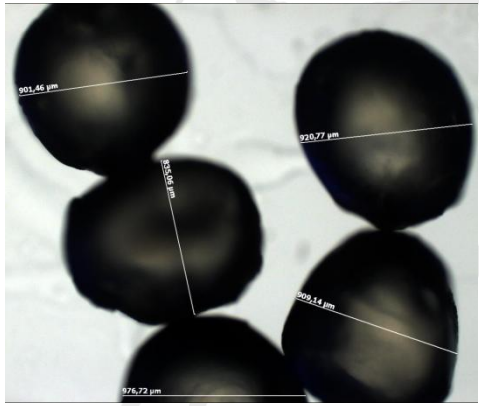
Coacervation = microcapsules 908.63 µm average diameter (346.37 to 1867.31 µm)



coacervation

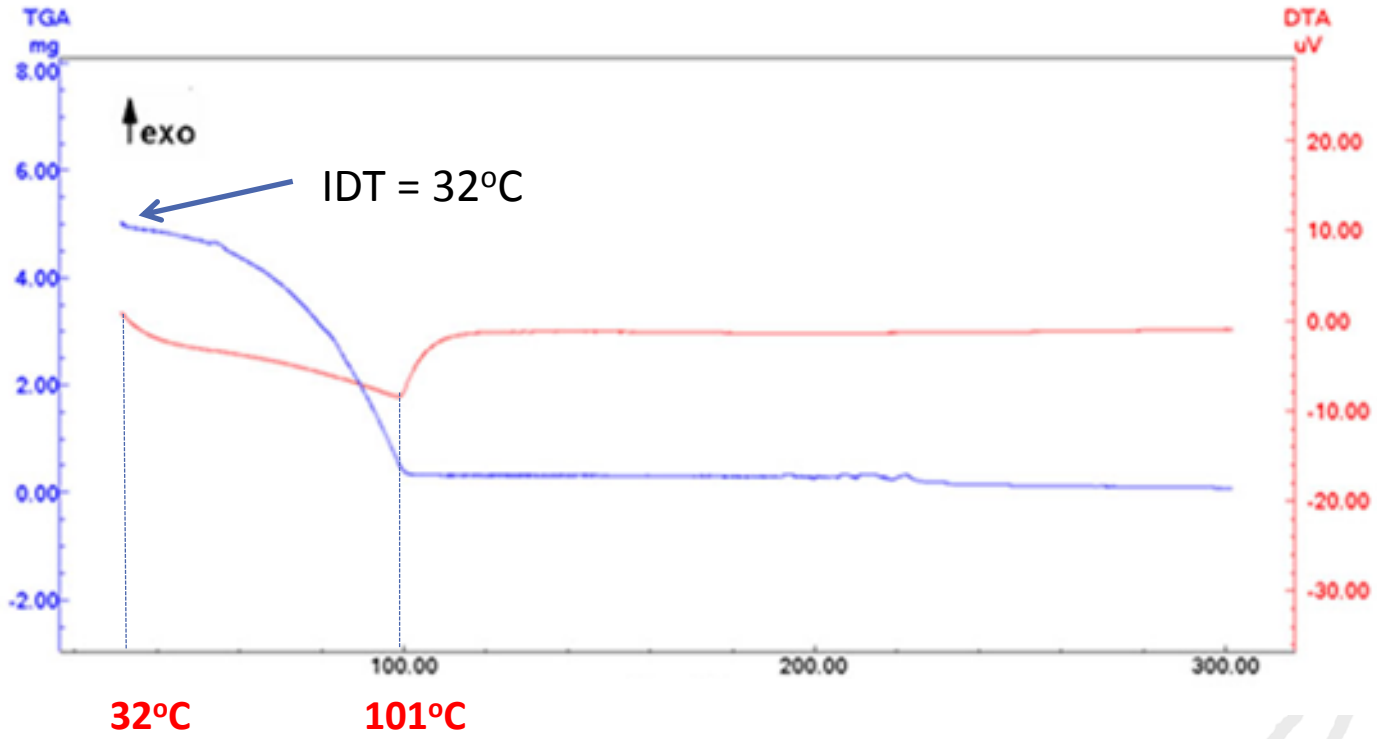


	d(0.1), µm	d(0.5), µm	d(0.9), µm	Uniformity
Spray-drying	0.31	0.85	1.89	0.89
Coacervation	346.37	504.21	1867.31	0.58



➢ Thermal analysis (TGA + DSC)

- For the **pure** essential oil there was **one single step of mass loss** of 90%, which corresponds to the volatilization occurred between 32°C and 101°C.

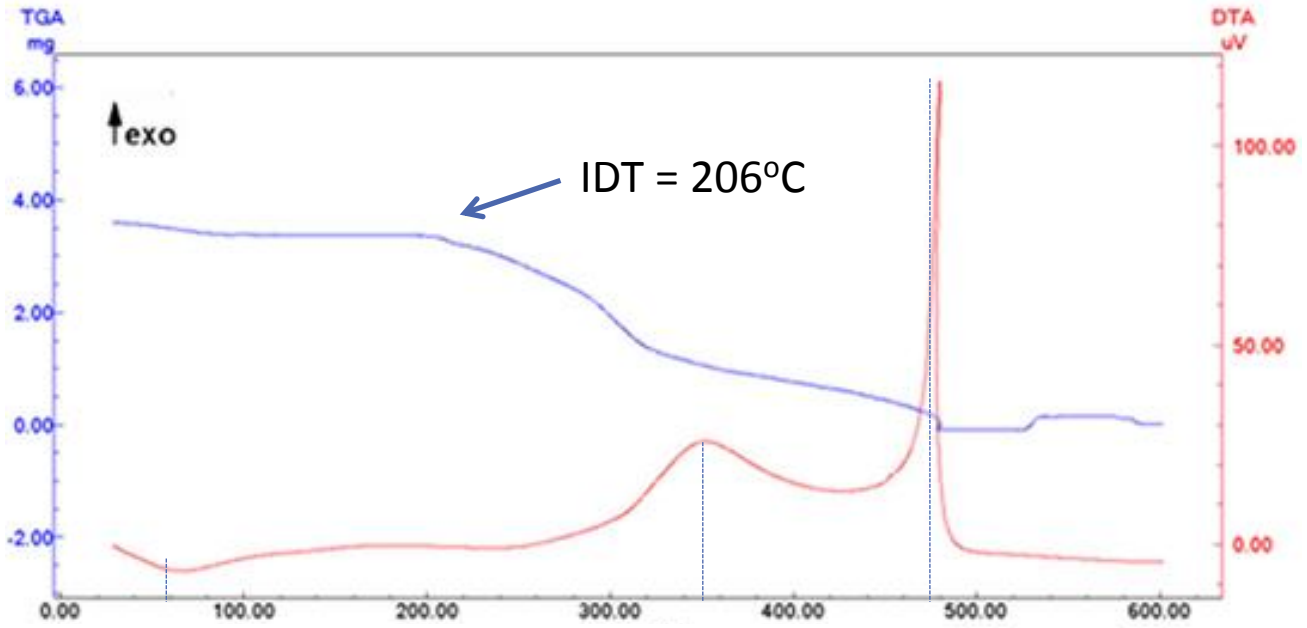


Thermogravimetric analysis for the pure orange essential oil
(10°C/min, synthetic air, 30-300°C, 100 mL/min)

- For the essential oil encapsulated by **spray-drying** there were **three thermal events**

  - **Mass loss** of 10% between 30 and 200°C;
  - **Mass loss** of 75% between 208 and 435°C;
  - **Mass loss** of 15% between 435 and 496°C.

Encapsulation **increased IDT** from 32 to 206°C; **total mass loss decreased** from 90 to 88%.



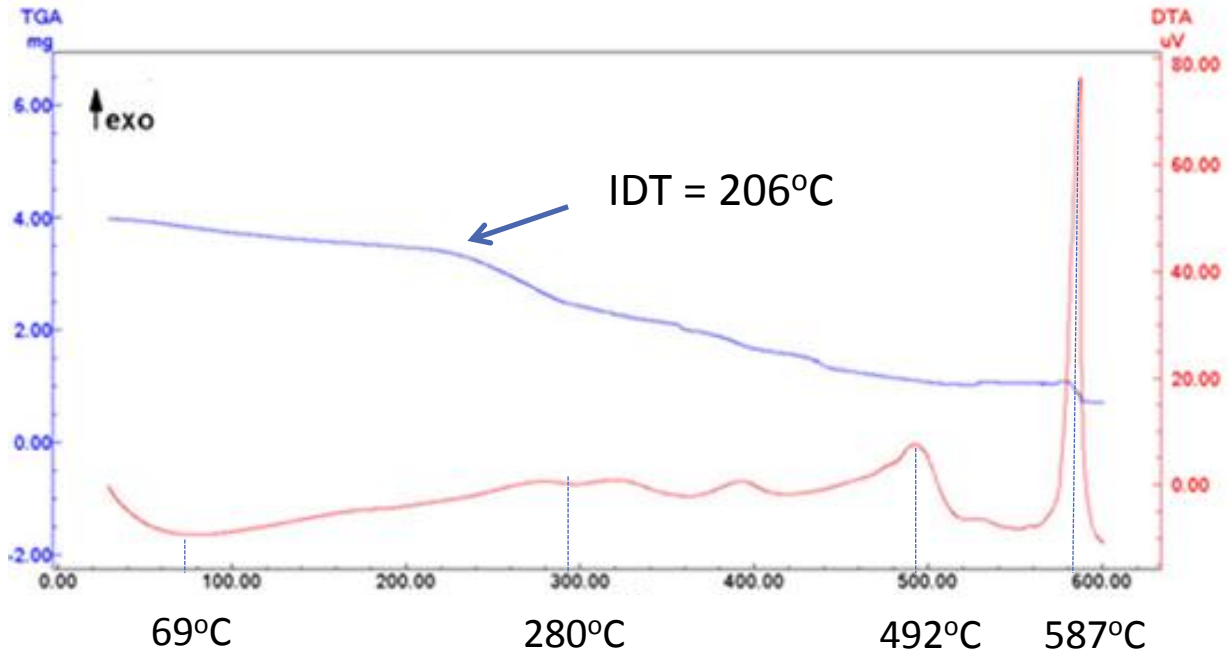
66°C 352°C 480°C

Thermogravimetric analysis for the orange essential oil encapsulated by SD
(10°C/min, synthetic air, 30-600°C, 100 mL/min)

- For the essential oil encapsulated by **coacervation** there were **four thermal events**

  - **Mass loss** of 6% between 30 and 100°C;
  - **Mass loss** of 32% between 100 and 290°C;
  - **Mass loss** of 34% between 290 and 500°C;
  - **Mass loss** of 9% between 500 and 600°C.

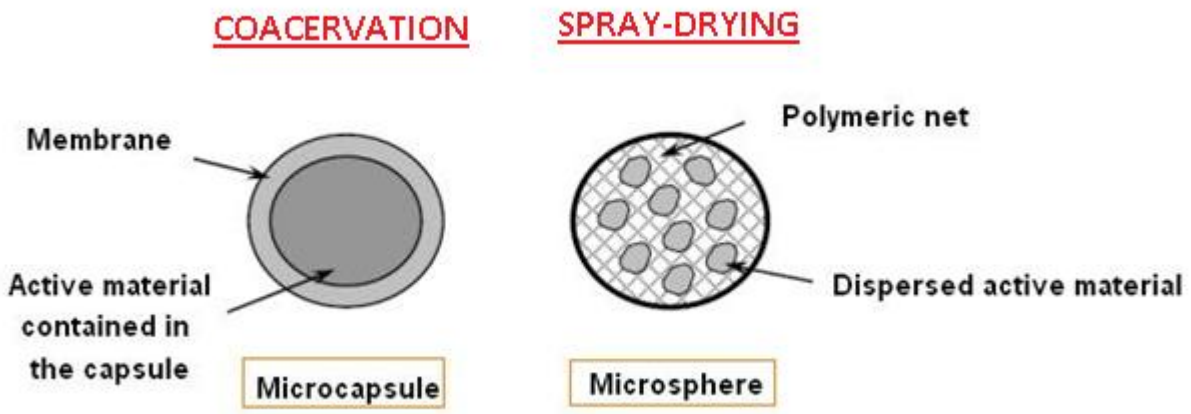
Encapsulation **increased IDT** from 32 to 206°C; **total mass loss decreased** from 90 to 45%.



Thermogravimetric analysis for the orange essential oil encapsulated by coacervation (10°C/min, synthetic air, 30-600°C, 100 mL/min)

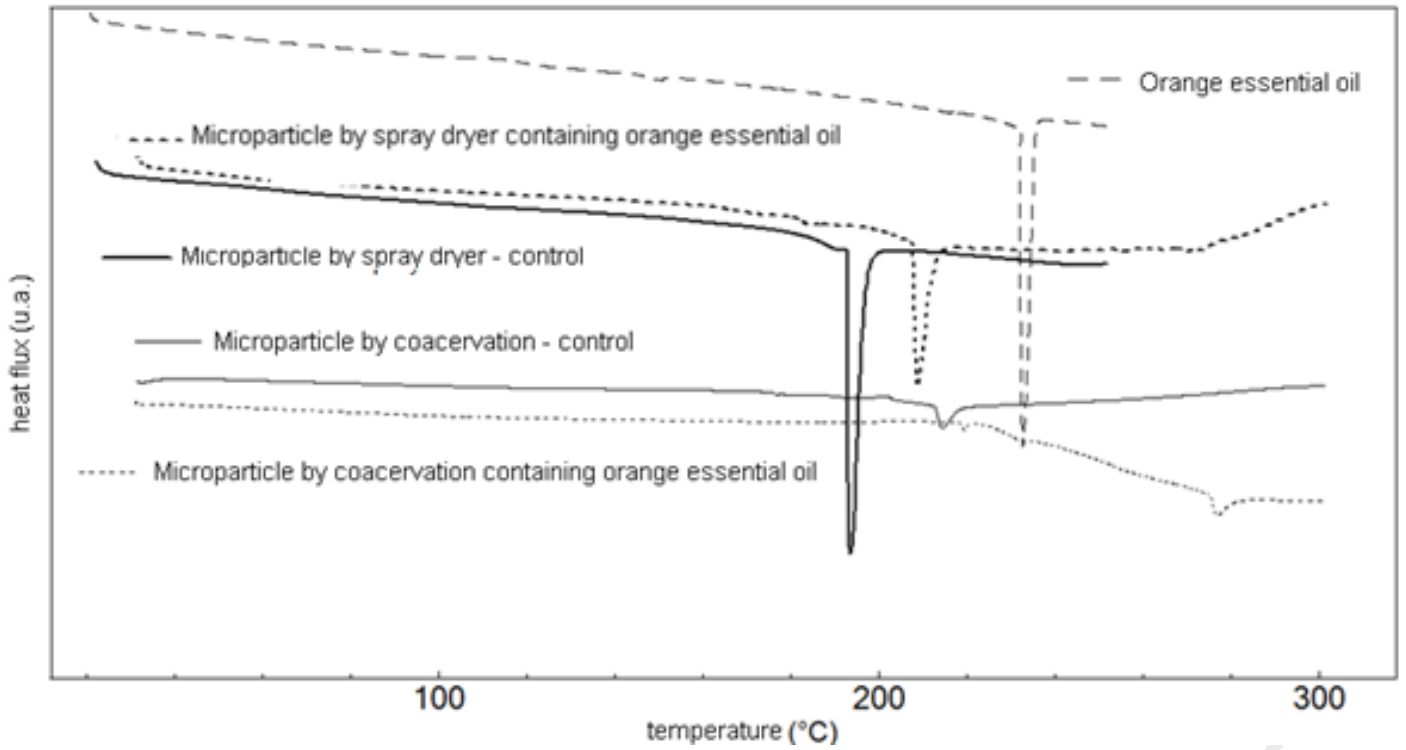
➢ Results from TGA analysis

- **Total mass loss decreased after encapsulation:** 90% pure essential oil X 88% SD X 48% coacervation;
- **IDT increased after encapsulation:** 32ºC pure essential oil X 206ºC encapsulated oil;
- Due to crosslinking, the coacervated microparticles protected more efficiently the OEL (presence of an outer layer), resulting in a higher thermal stability for the product respect to the spray dried particles.



➢ Results from the DSC analysis

- The **endothermic peaks are shifted to the left** when comparing the microparticles with essential oil to the control samples, therefore the process promoted an interaction between the carrier polymers and the essential oil;
- The essential oil encapsulated by coacervation presented **higher boiling point and IDT** respect to the pure essential oil and the OEL encapsulated by SD.



Differential Scanning calorimetric analysis (10°C/min, synthetic air, 30-600°C, 100 mL/min)

Composition of the OEL – GC/MS

- A gas chromatograph provided the mass spectra of the compounds;
- Retention times of standard orange essential oil and samples were compared using a mass spectra data bank (NIST).

Component	Mass percentage %			
	Reported by Bertolini (2001)	Before encapsulation ± SD	After Coacervation ± SD	After spray-drying ± SD
Ethanol	0.100%	NQ	NQ	NQ
Ethyl Acetate	0.005%	NQ	NQ	NQ
Acetal	0.002%	NQ	NQ	NQ
Hexanal	0.020%	NQ	NQ	NQ
Acetate Butyrate	0.100%	NQ	NQ	NQ
Trans-2-hexenal	0..005%	NQ	NQ	NQ
Alpha-pinene	0.400%	0.447±0.003%[a]	0.339±0.002% [b]	0.331±0.003% [b]
Beta-pinene	NQ	0.461±0.007% [a]	0.360±0.008% [b]	0.356±0.005% [b]
Trans-isolimonene	NQ	0.046±0.001% [a]	0.046±0.003% [a]	0.032±0.001% [b]
Sabinene	0.400%	NQ	NQ	NQ
Mircene	1.800%	1.767±0.011% [a]	1.651±0.007% [b]	1.672±0.006% [b]
Octanal	0.500%	0.291±0.003% [a]	0.289±0.005% [a]	0.288±0.002% [a]
Pseudo- limonene	NQ	0.087±0.001% [a]	0.087±0.001% [a]	0.078±0.002% [b]
D-limonene	93.600%	95.930±0.013% [a]	95.781±0.009% [b]	95.622±0.003% [c]
Linalol	0.500%	0.325±0.012% [a]	0.324±0.013% [a]	0.308±0.009% [b]
Alpha-terpineol	NQ	0.048±0.001% [a]	0.039±0.001% [b]	0.041±0.002% [b]
Decanal	0.600%	NQ	NQ	NQ
1-dodecene	NQ	0.265±0.006% [a]	0.265±0.004% [a]	0.258±0.003% [a]
Neral	0.200%	0.229±0.004% [a]	0.228±0.003% [a]	0.236±0.005% [b]
Geranial	0.100%	0.103±0.003% [a]	0.101±0.001% [a]	0.098±0.002% [a]
Valencene	1.700%	NQ	NQ	NQ
TOTAL	100.000%	99.999±0.005% [a]	99.510±0.005% [b]	99.320±0.004% [c]

Oil composition may vary according to harvesting time, soil, climate, extraction methods and other factors

Analysis conditions: injection temperature 250°C, injection volume 0.5 µL, CP-SIL8CB fused silica column (0.25 mm of internal diameter, 30 m of length and 0.25 µm of liquid film), helium as a carrier gas at a flow of 1.0 mL/min, column temperature 40°C to 300°C (20°C/min) remaining for 45 min at 300°C.

➢ Encapsulation efficiency – GC/MS detector

- Extraction conditions:

  - samples of 1 g extracted with 2.4 g of ethanol;
  - sample and syringe temperature 35°C;
  - incubation period 10 min, stirring 500 rpm each 20 sec;
  - extracted samples were quantified by GC/MS;

$$\%EE = \frac{m^{OEL}_{final}}{m^{OEL}_{sample}} \times 100$$

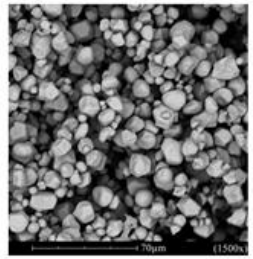
99.32% for spray drying versus **99.51%** for coacervation

➢ Payload – GC/MS detector

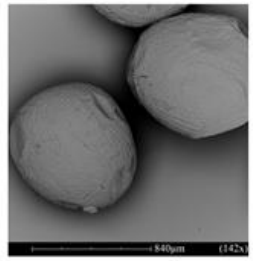
- SD microspheres = **16.4%**
- Coacervated microcapsules = **80.0%**

➢Conclusions

- Polymeric microparticles containing orange essential oil were successfully obtained either by spray drying or coacervation:



spray-drying

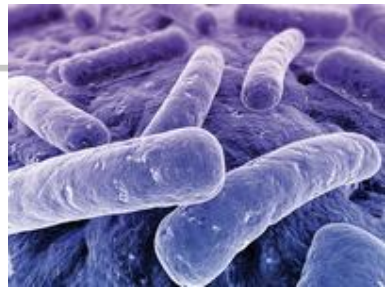


coacervation

- ✓ **Higher thermal stability** (oxidation) than the pure OEL for both methods: IDT increased from 32 to 206ºC;
- ✓ **Different sizes and morphologies**: spray-dried microspheres of 1.02 µm versus coacervated microcapsules of 908.63 µm;
- ✓ **Different encapsulation efficiencies**: 99.32% for spray drying versus 99.51% for coacervation;
- ✓ **Different payloads**: 16.4% for spray drying versus 80.0% for coacervation;

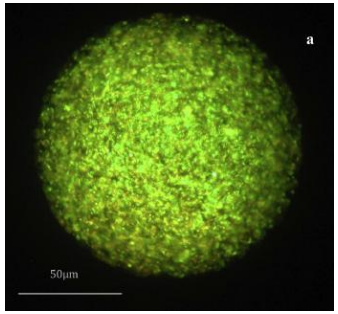
- D-limonene was identified as the OEL's main compound (95.7%), followed by mircene (1.7%);
- The higher thermal stability of the **coacervated** microparticles is attributed to crosslinking;
- The encapsulation method must be chosen basing on the final use of the product, production rate and system cost.

Practical applications - 2

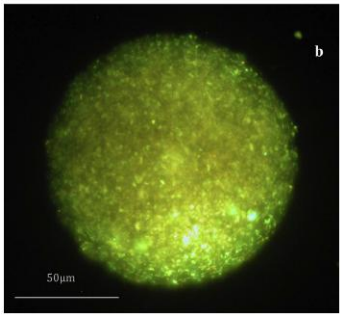


- Microencapsulation of the probiotic strain *Lactobacillus reuteri*
  - Wall materials: alginate (MC) and chitosan-alginate (CMC);
  - Method: vibration nozzle (a laminar flowing jet liquid breaks up into equal size droplets by a superimposed vibration; the selectable vibration frequency determines the quantity of droplets produced; e.g. 700 Hz= 700 droplets/s;
  - Goal: enhance survival in foods and in gastrointestinal environment;
  - Viability was tested for 28 days storage in different solutions simulating gastrointestinal conditions;
  - **Results:** 100% survival rate, encapsulation efficiency of 99% for MC and 92% for CMC.

Alginate



Chitosan



Reference: De Prisco et al. (2015). Microencapsulation by vibrating technology of the probiotic strain *Lactobacillus reuteri* DSM 17938 to enhance its survival in foods and in gastrointestinal environment. LWT - Food Science and Technology, 61 (2015), 452-462.

Practical applications - 3

- Microencapsulation of antioxidant mushroom extracts
  - Wall material: maltodextrin;
  - Core material: hydroalcoholic extracts of two mushrooms species, *Suillus luteus* and *Coprinopsis atramentaria;*
  - Method: spray-drying;
  - Goal: study the antioxidant effect in cottage cheese;
  - **Result 1:** the spray-drying of the extracts using an extract/maltodextrin ratio of 1/20 and an inlet temperature of 170ºC resulted in good encapsulation efficiency (43.5-62.6%.);
  - **Result 2:**

Table 2
Antioxidant activity, expressed as EC_{50} values (mg/ml), of microencapsulated (Mic) individual and combined extracts prepared from *S. luteus* (Sl) and *C. atramentaria* (Ca).

Assay	Mic Sl	Mic Ca	Mic Sl + Mic Ca (1:1)	
			Experimental	Effect
DPPH radical scavenging activity	2.13 ± 0.06	2.36 ± 0.16	2.04 ± 0.04	SE
Reducing power	0.85 ± 0.01	0.78 ± 0.01	0.83 ± 0.02	AE

Reference: Ribeiro et al. (2015). Spray-drying microencapsulation of synergistic antioxidant mushroom extracts and their use as functional food ingredients. Food Chemistry, 188, 612-618.

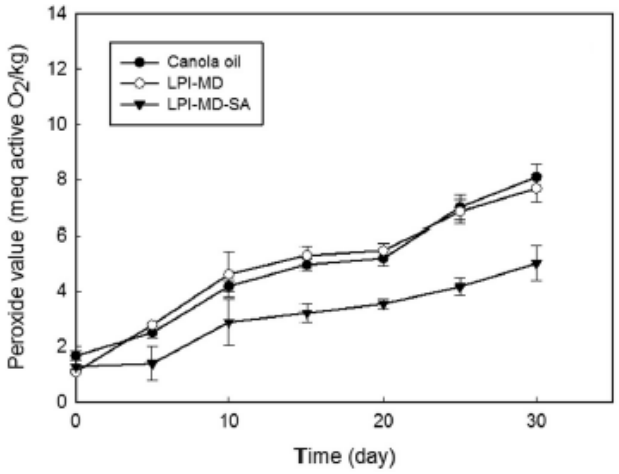
Practical applications - 4

- Microencapsulation of bioactive compounds from blackberry
  - Core material: blackberry (*rubus fruticosus*) aqueous extract + 2% citric acid;
  - Wall material: gum arabic (GA) and polydextrose (PD) at concentrations 10 and 15 %;
  - Method: spray-drying at 140 to 160°C;
  - **Result 1**: anthocyanins retention in the microcapsules was 878.32-1300.83 mg/100 g, and phenolics retention was 2106.56-2429.22 mg (GAE)/100 g;
  - **Result 2:** antioxidant activity DDPH from 31.28 to 40.26 % and ABTS from 27 to 45.15 %;
  - **Result 3:** the best encapsulation condition was atomization at 140 °C and 15 % gum arabic.

Reference: Rigon and Noreña (2015). Microencapsulation by spray-drying of bioactive compounds extracted from blackberry (*rubus fruticosus*). Journal of Food Science and Technology, 53(3), 1515-1524.

Practical applications - 5

- Microencapsulation of canola oil by spray-drying
  - Encapsulation material: lentil protein isolate and maltodextrin with/without lecithin and/or sodium alginate;
  - Method: spray-drying at 180°C;
  - **Result 1:** lentil protein + maltodextrin + sodium alginate resulted in the highest entrapment efficiency (88%);
  - **Result 2:** lentil protein + maltodextrin + alginate microcapsules showed better oxidative stability and had a stronger wall structure than the lentil protein + maltodextrin microcapsules;



Reference: Chang et al. (2016). Microencapsulation of canola oil by lentil protein isolate-based wall materials. Food Chemistry, 212, 264-275.

Practical applications - 6

- Microencapsulation of lactic acid bacteria in liposomes
  - Core material: concentrated form of cell free extract (CFE) derived from attenuated *Lactococcus lactis* supsb. *lactis* 303 CFE;
  - Wall material: two different proliposome preparations (Prolipo S = 30% (w/w) unsaturated soybean phospholipids and 70% (w/w) aqueous media; Prolipo Duo = 50% (w/w) unsaturated soybean phospholipids and 50% (w/w) aqueous media);
  - Method: liposomes/microfluidization;
  - **Result 1:** the encapsulated LAB partitioned within the curd during Cheddar cheese production and prevented excessive losses of CFE into the whey;
  - **Result 2:** better flavour development (sensory and volatile attributes) in the liposomal encapsulated CFE cheeses versus Control cheeses;
  - **Result 3:** Intact liposomes were found in the cheese up to 28 days of ripening.

Reference: Nongonierma et al. (2013). Encapsulation of a lactic acid bacteria cell-free extract in liposomes and use in cheddar cheese ripening . Foods, 2, 100-119.

Practical applications - 7

- Microencapsulation of rosemary essential oil
  - Encapsulation materials: gum arabic, inulin, maltodextrin and modified starch;
  - Method: spray-drying;
  - **Result 1:** the oil composition did not change due to encapsulation;
  - **Result 2:** inulin decreased hygroscopicity and increase wettability, however decreased the encapsulation efficiency;
  - **Result 3:** larger particles were produced using gum arabic or modified starch;
  - **Result 4:** replacement of gum arabic with starch + maltodextrin (1:1 m/m) did not affect encapsulation efficiency;
  - **Result 5**: inulin, a fibre with proven functional activity, is an alternative encapsulating material for the production of foods with functional claims.

Reference: Fernandes et al. (2014). Gum arabic/starch/maltodextrin/inulin as wall materials on the microencapsulation of rosemary essential oil. Carbohydrate polymers, 101, 524-32.

DISCLAIMER:

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This presentation reflects only
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