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

European Commission

Techniques for encapsulation of food ingredients

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http://revalueprotein.com/

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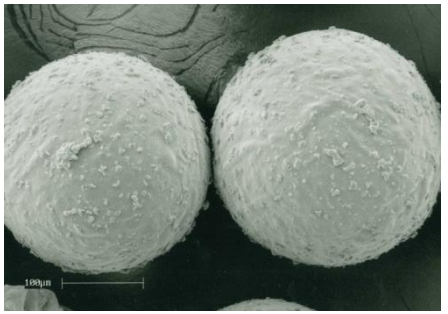
Encapsulation methods



- Physical (or mechanical) & Chemical
  - **Physical encapsulation**

Physical methods include encapsulation by gravity-flow, centrifugal extrusion, spray-drying, spray-chilling, spinning disk and others. In general, a coating is applied to the active material and then is dried to obtain coated microparticles.

Physical Encapsulation Characteristics
- Particle sizes of 1-10,000 microns
- Material versatility
- Narrow size distribution
- Scalability and high production capacity
- Continuous production

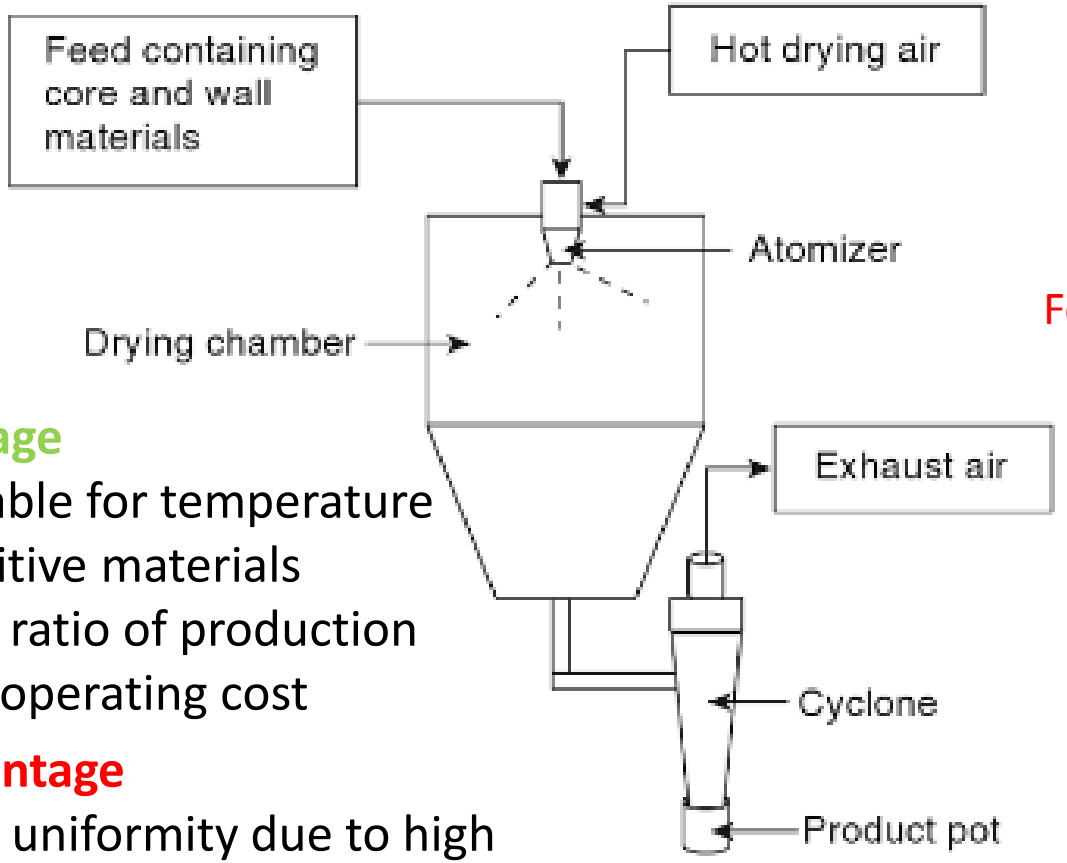
  - **Chemical encapsulation**

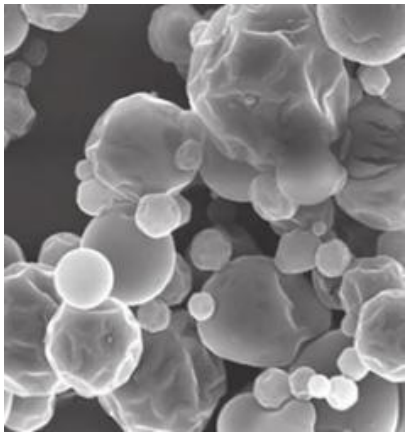
Chemical encapsulation methods include coacervation (simple and complex), *in situ* polymerization, interfacial polymerization, emulsion polymerization, layer-by-layer deposition, liposomes and others.

Chemical Encapsulation Characteristics
- Particle sizes of 0.1-500 microns
- High payload
- Uniform particle size distribution
- Scalability and high production capacity
- Batch production (mainly)

Physical encapsulation methods

- Spray-drying





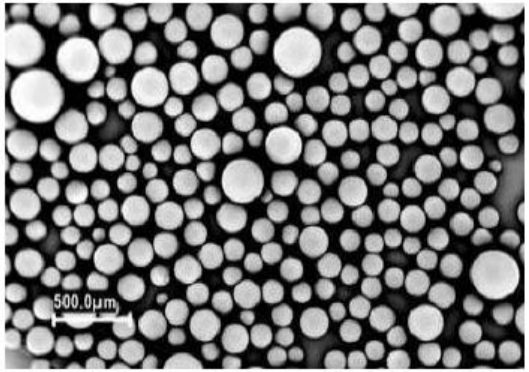
Food flavour encapsulated by spray-drying

Advantage

- Suitable for temperature sensitive materials
- High ratio of production
- Low operating cost

Disadvantage

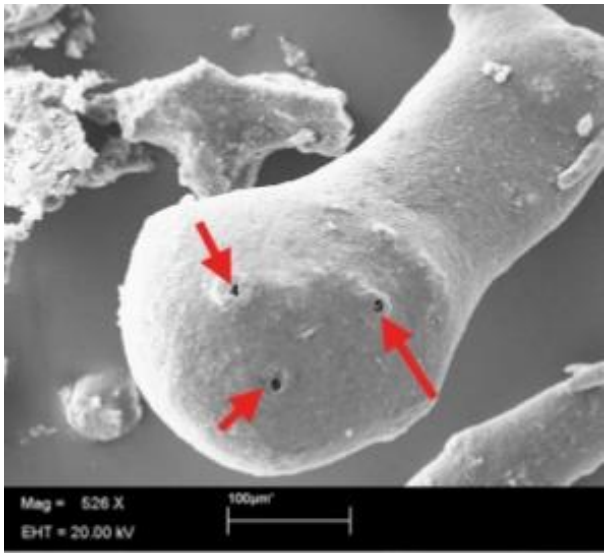
- Low uniformity due to high air velocities
- Aggregation



Microspheres of berries anthocyanin produced by spray-drying

Physical encapsulation methods

- Spray-cooling/chilling
  - Similar process to spray-drying, but using low temperatures;
  - Spray-cooling has been used mainly to encapsulate minerals and vitamins
  - Encapsulating material has low melting point. Cooling: 45-120C; Chilling: 32-42 C.



Advantage

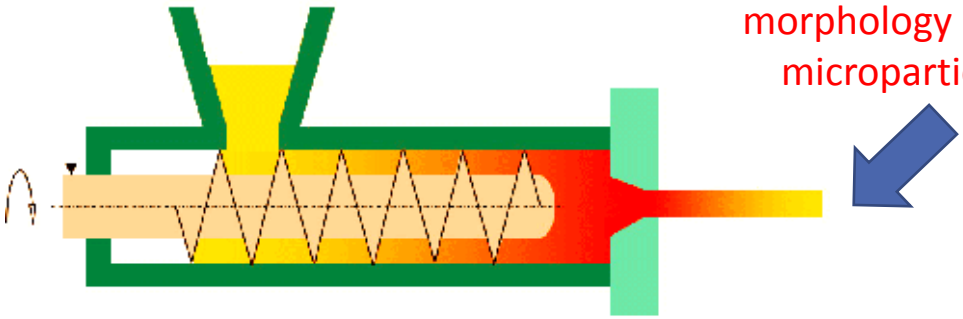
- Cheapest encapsulation method (low temperatures and high scale-up potential)

Disadvantage

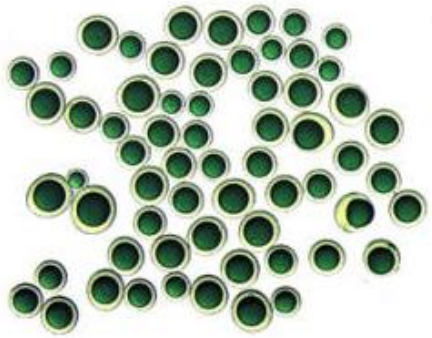
- Low encapsulation capacity (active material particles located at the surface)

Physical encapsulation metho

- Extrusion
  - An emulsion of active ingredient and carrier materials is extruded at a given temperature. The extruded emulsion is cut into pieces and dried or solidified



morphology of the microparticles

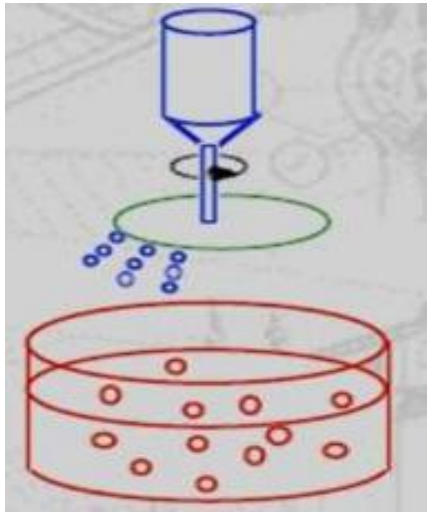


  - Widely used for volatile and unstable flavours in glassy carbohydrate matrices;
  - Provides very long shelf life to oxidation-prone flavour materials (atmosphere gases diffuse very slowly through the hydrophilic glassy matrix) = barrier almost impermeable against O_2.

Physical encapsulation methods

Video:
http://www.swri.org/4org/d01/microenc/microen/atomization.htm

- Extrusion Spinning disk
  - Suspensions of core particles in liquid shell material are poured into a rotating disc;
  - Due to the spinning action, the core particles become coated;
  - The coated particles are cast from the edge of the disc by centrifugal force;
  - The shell material is solidified by cooling.



Advantages

- Simple and low cost
- High production efficiency
- Suitable for high-solids and high-viscosity materials

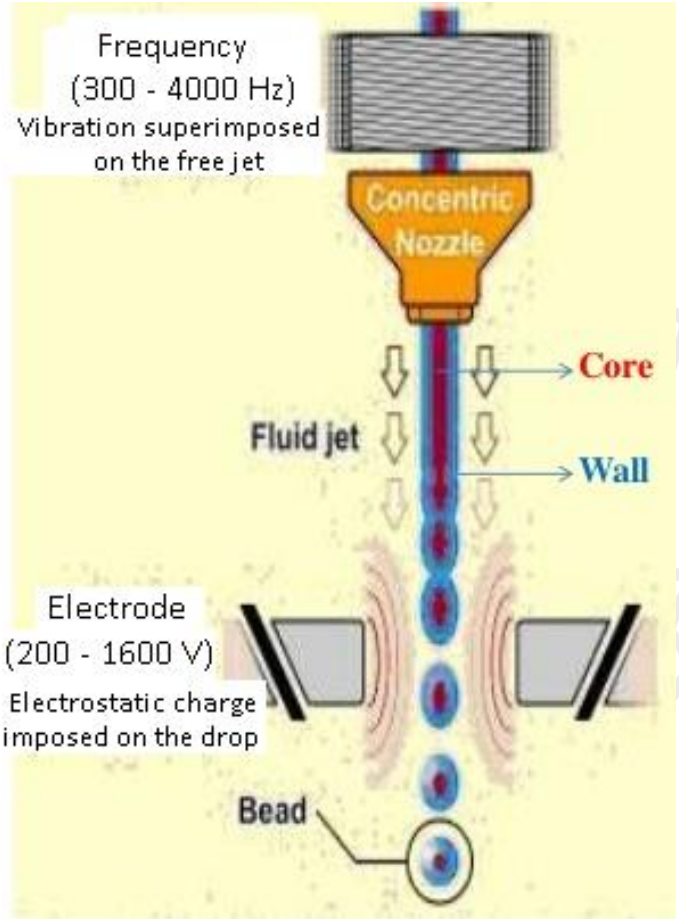
Disadvantages

- Large particle size (≥ 100 µm)
- Requires a large surface or volume to collect the beads

Physical encapsulation methods

- EHD (electrohydrodinamic) Coextrusion

  - involves the use of *high voltage* to generate an electrified coaxial jet



Advantage

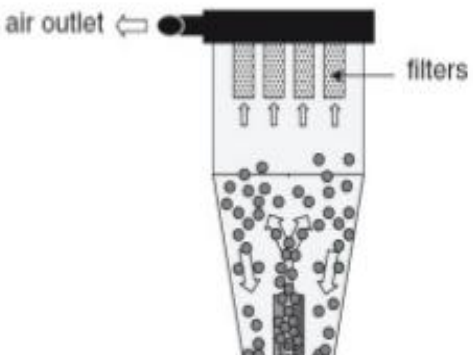
- High production efficiency (up to 22.5 kg of microcapsules/nozzle/h

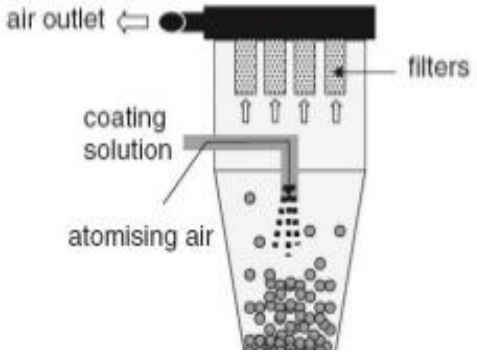
Disadvantage

- Since the drops are formed by the breakup of a liquid jet, the process is only suitable for liquid or slurries

Physical encapsulation methods

- Fluidized bed coating
  - Solid/powder particles are suspended in a temperature and humidity controlled chamber of high velocity air where the coating material is atomized;
  - Applicable for high-melt coatings (hydrogenated vegetable oil, stearines, fatty acids, emulsifiers and waxes) or solvent-based coatings (starches, gums, and maltodextrin).





Advantages

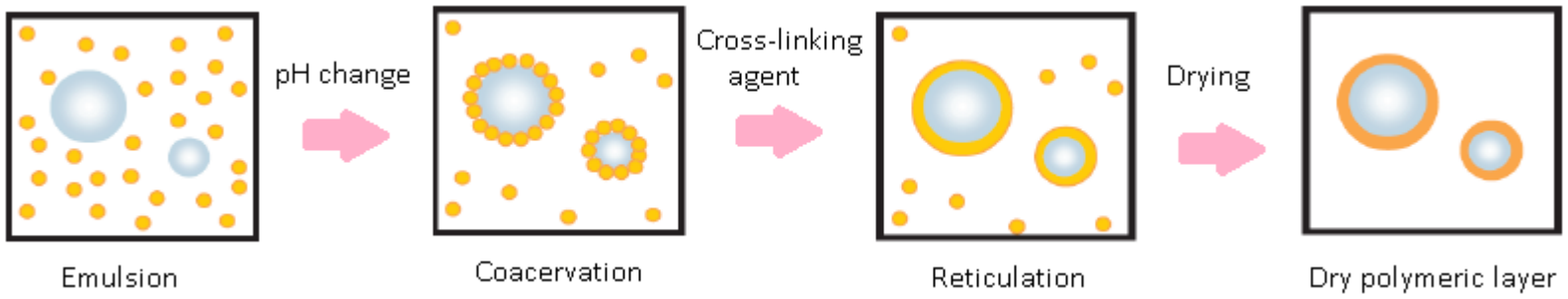
- Uniform layer of carrier onto solid particles
- Versatility of wall materials
- Multiple controlled release possibilities

Disadvantages

- Solidification of the lipid coating before it reaches the powder
- High particle aggregation

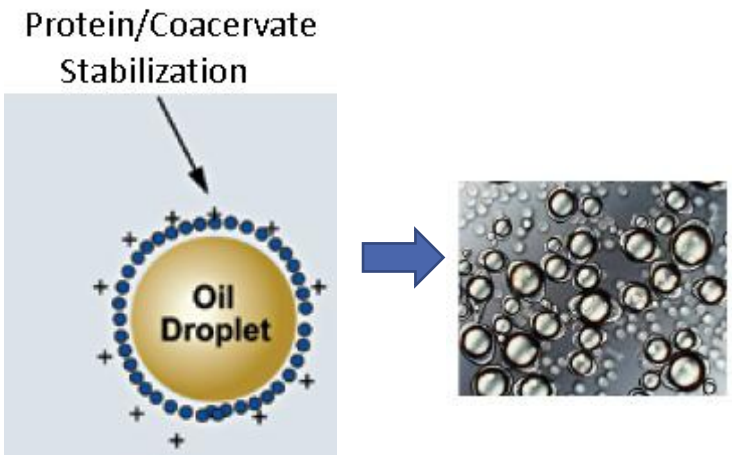
Chemical encapsulation methods

- Coacervation
  - O/W emulsions are prepared with lipophilic active in oil phase under stirring induces the formation of 3 immiscible phases;
  - A change of the pH value of the dispersion (e.g. by adding H_2SO_4, HCl or organic acids) reduces the solubility of the dispersed phase (shell material);
  - The shell material (coacervate) starts to precipitate from the solution and forms a continuous coating around the core droplets;
  - The shell layer is cooled down to harden or is dried and forms the final capsule. Hardening agents like formaldehyde can be added to the process.



Chemical encapsulation methods

- **Simple coacervation** (one type of polymer) versus **Complex coacervation** (two or more types of polymers of opposite ionic charges are present);
- Complex coacervation is based on the interaction among different polymers with opposite charges;
- This interaction forms insoluble complexes and produces phase separation. The deposition of such complexes around a hydrophobic core creates a barrier, allowing its encapsulation.



Advantages

- The core material is completely entrapped by the polymer(s)
- Mechanical and thermal resistance and release may be altered by cross-linking using chemical or enzimatic compounds (e.g. glutaraldehyde, transglutaminase, $CaCl_2$)

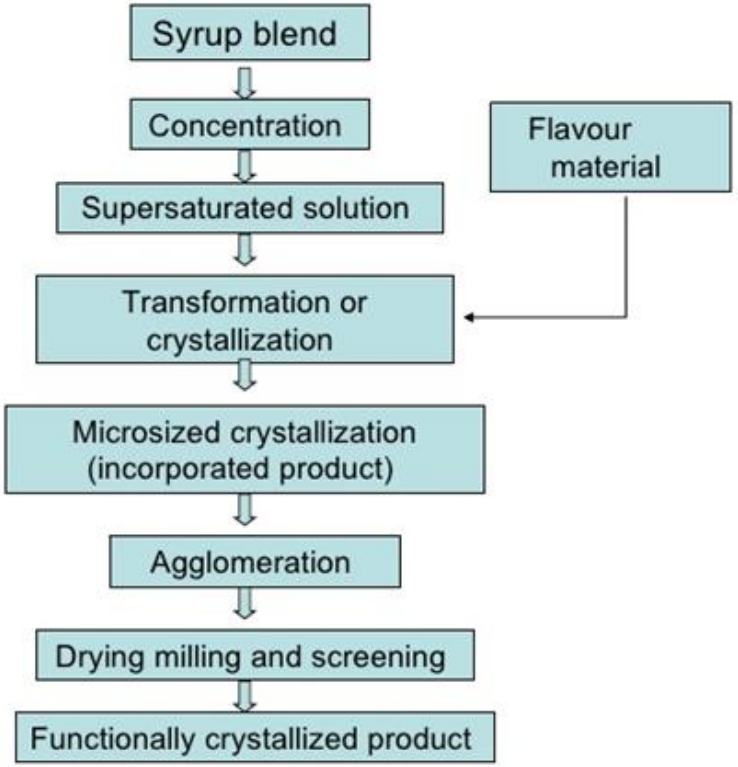
Disadvantages

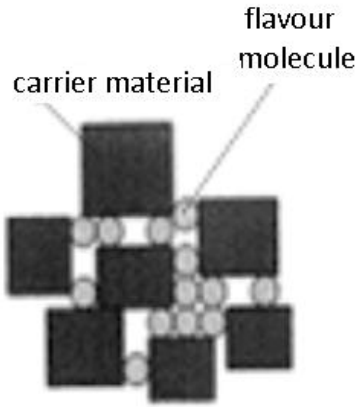
- Difficult to encapsulate water-soluble or polar components
- Occur at a narrow pH range (process operation and control)
- Expensive and complex

Chemical encapsulation methods

- Co-crystallization

  - Co-crystallization uses sucrose as a matrix for the incorporation of core materials;
  - The sucrose syrup is concentrated to the supersaturated state and the ingredients are incorporated (>120^{o}C and 95-97^{o}Brix);
  - Vigorous agitation provides spontaneous nucleation of the sucrose/active material mixture;
  - The encapsulated products are dried and screened to a uniform size;
  - Simple and economical method.





Emulsion technologies

- Emulsions can be generated by low or high energy methods.
- Low energy methods: emulsion is generated spontaneously or triggered by external factor (pH, temperature or salt concentration) when mixed oil, water and surfactant.
- High energy methods: emulsion generated by means of shear stress, cavitation, turbulent flow or high pressures.

Low energy

-High surfactant level narrows size distribution

-Thermal sensitive compounds

-High efficiency

-Limited choice of food grade ingredients

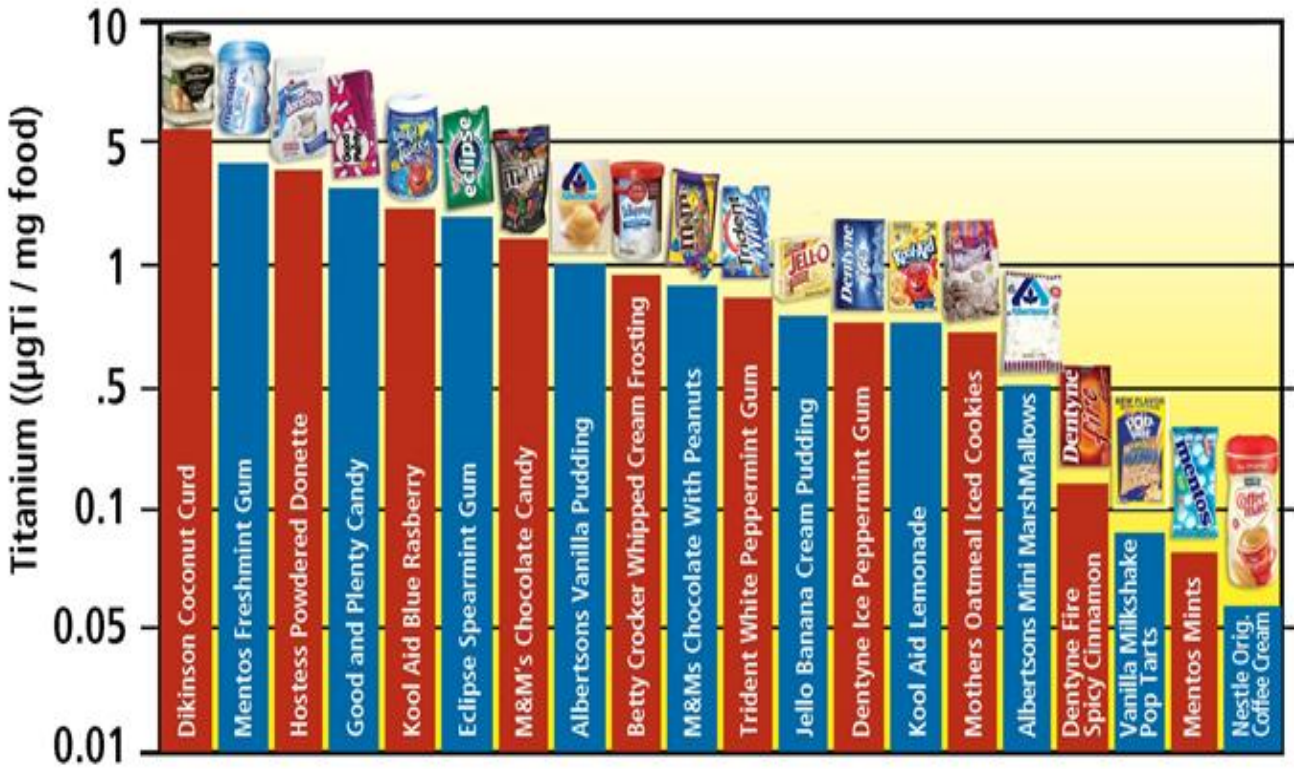
High energy

- Very narrow size distribution, smaller sizes

- **Higher variety of emulsifiers**
- **Expensive equipments. Low energy efficiencies**

Nanoencapsulation

- Nanoparticles (10-1000 nm) can help deliver nutrients, enhance flavour, and protect against UV light in packaging, and detect contamination;
- **Main differential: controlled release/delivery of small molecules**;
- Have low payloads and high surface area;



NE Techniques

-Micelles
-Liposomes
-Solvent evaporation
-Phase inversion/precipitation
-Layer-by-layer deposition
-Templating
-Molecular encapsulation
-Surfactant-free particle formation
-Controlled precipitation
-Polyelectrolyte complexes

Lipophilic vs. hydrophilic releasing

Lipophilic

-Release slower

-Incomplete release

-Release kinetic driven by erosion mechanism

-Highly permeable via active transport and facilitated diffusion in the intestine

Hydrophilic

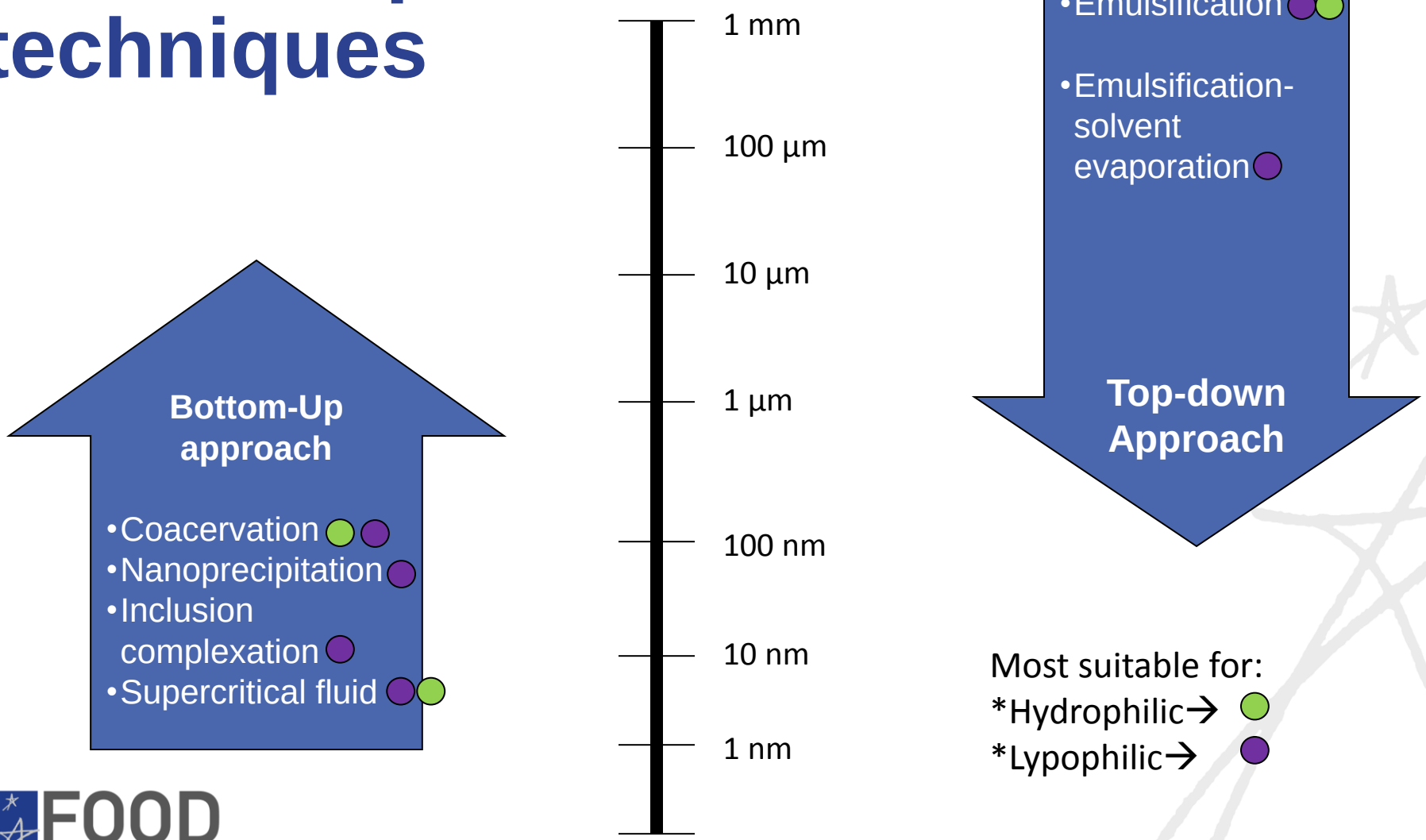
-Release faster

-Release kinetic driven by diffusion and erosion mechanism

-Low membrane permeability

-Only absorbed by active transport

Nanoencapsulation techniques



Most suitable for:

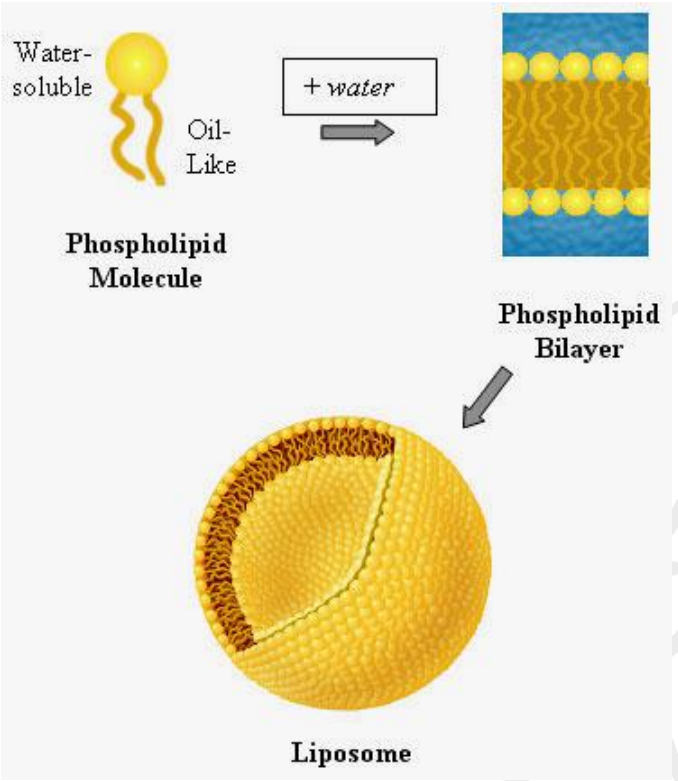
*Hydrophilic→

*Lypophilic→

Chemical encapsulation methods

- Nanoencapsulation – Liposomes

  - A liposome or lipid vesicle is a structure composed of bilayers of lipid molecules (phospholipids such as lecithin and cholesterol) that enclose liquid compartments;
  - They form when lipids are dispersed in aqueous media and exposed to high shear stress by using microfluidization or ultrasounds;
  - Particle size ranges from 25 nm to a few microns in diameter, are easy to make, and can be stored by freeze-drying.;
  - The underlying mechanism is basically hydrophilic-hydrophobic interactions between phospholipids and water molecules.



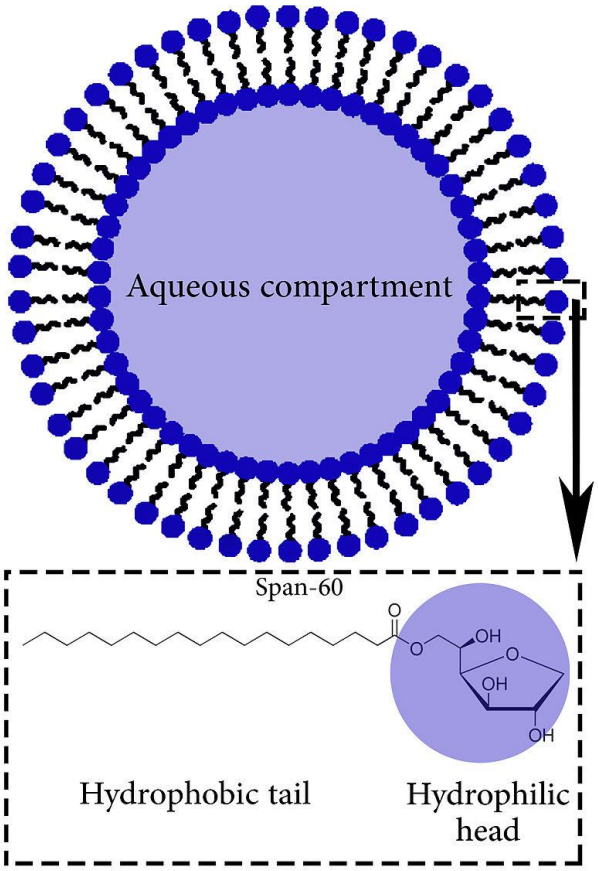
Video:
www.youtube.com/watch?v=B4Mz8pbBnSM

Permeability, stability, surface activity and affinity can be varied through size and lipid composition variations.

Chemical encapsulation methods

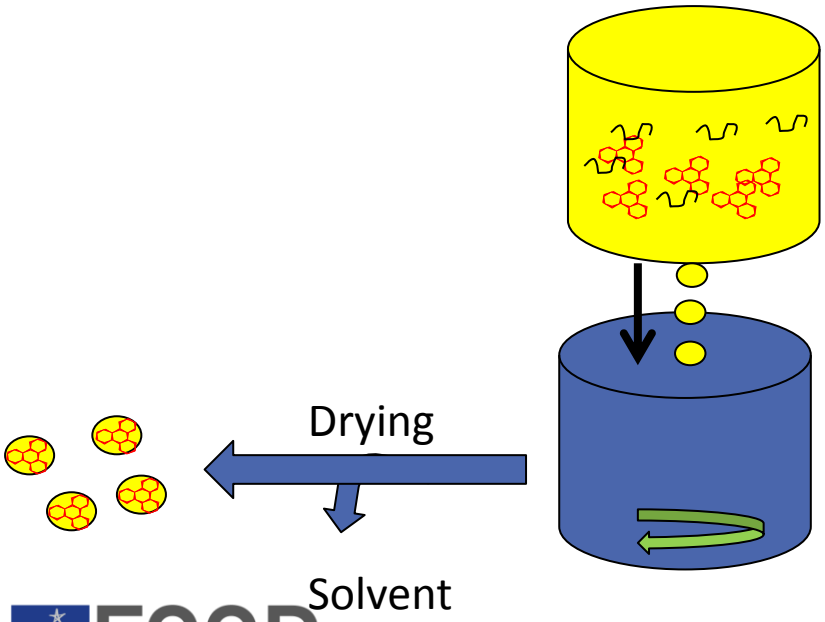
- Nanoencapsulation – Niosomes

  - A niosome is a vesicle which wall is composed by a non-ionic surfactant (Span-60, SDS) and cholesterol.;
  - Same techniques employed for liposomes are applied to generate niosomes;
  - Particle size ranges from 25 nm to a few microns in diameter;
  - They can entrap lipophilic drugs into vesicular bilayer membranes and hydrophilic drugs in aqueous compartments;
  - Niosomes are osmotically active, chemically stable and have long storage time compared to liposomes.



Nanoprecipitation or solvent displacement

- Polymer and lipophilic compound are dissolved in organic phase;
- Drop-wise added into stirred aqueous solution leading to spontaneous emulsification;
- Finally organic phase is removed.



Advantages

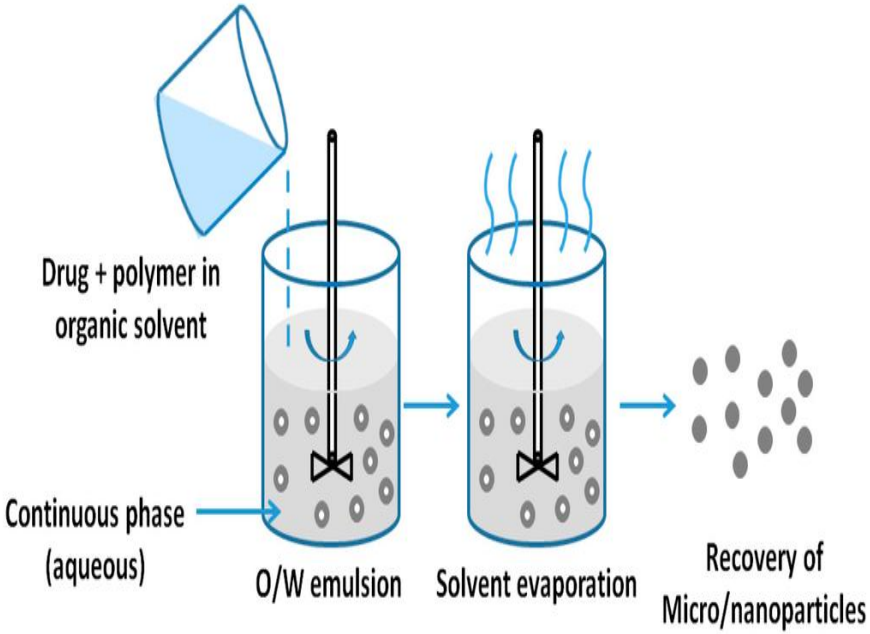
- Efficient when producing nanocapsules around 100 nm
- High stability, high encapsulation efficiency
- Sustained release of compounds
- Lipophilic drugs perform well

Disadvantages

- Only polymer based gels can be used (PEG or PLGA)
- Limited to water-miscible solvents with high diffusion rate

Emulsification-Solvent Evaporation

- A polymer is emulsified into an aqueous solution;
- Evaporation of polymer solvent leads to polymer precipitation as nanospheres;
- Active compound is dispersed in polymer matrix.



Advantages

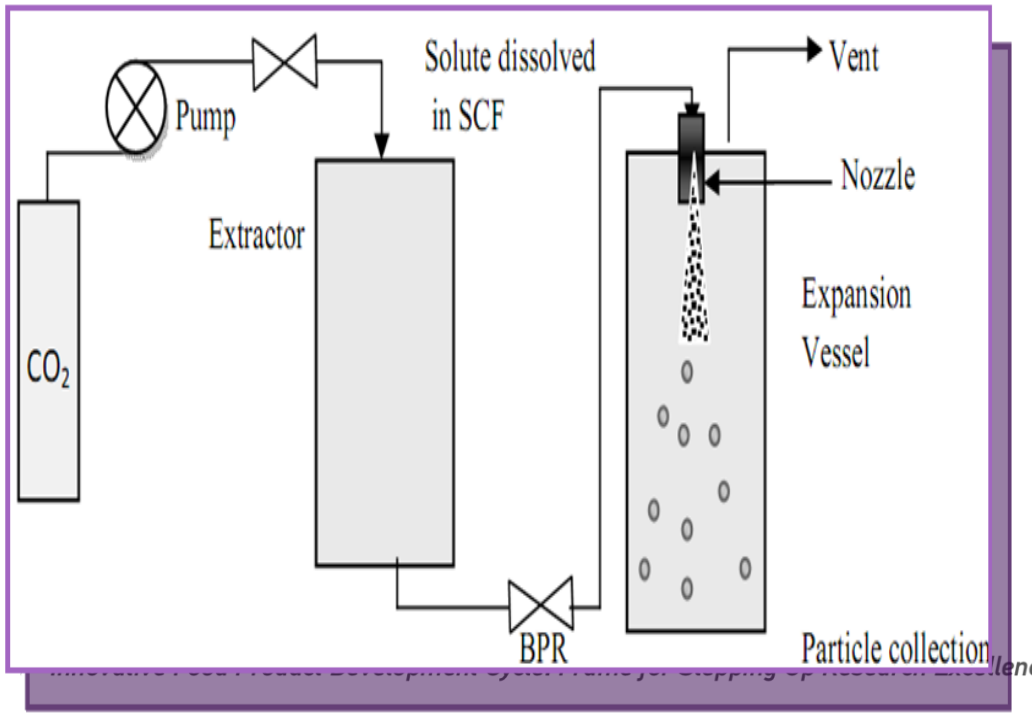
- Efficient when producing nanocapsules below 100 nm
- Size can be tailored depending on emulsion technique
- Spherical shape, high loading content
- Sustained release of compounds

Disadvantages

- Depends on suitable emulsification technique.
- Scaling-up is limited due to high energy requirements
- Solvent employed must be food grade
- Only lipophilic material can be encapsulated

Supercritical fluid technique

- The polymer and the ingredient is solved in a supercritical fluid (CO2, water, propane, nitrogen);
- Then the solution is expanded through a nozzle and the supercritical fluid is evaporated;
- Polymer precipitates encapsulating the active compound;



Advantages

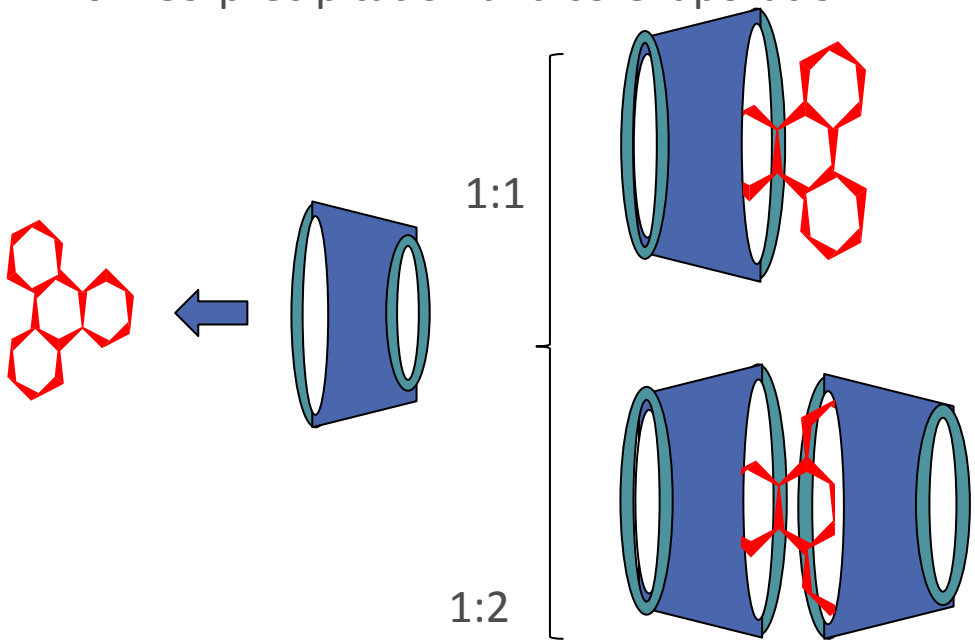
- CO_2 provides inert medium →suitable easily oxidable compounds.
- Minimum use of organic solvents
- Particle size narrow and controllable morphollogy

Disadvantages

- High capital investment for high pressure equipment

Inclusion Complexation

- Generally refers to the encapsulation of a ligand (bioactive ingredient) into a cavity-bearing substrate (shell material), mainly α and β-cyclodextrin;
- Driving forces are hydrogen bonding, van der Waals or hydrophobic effect.
- Co-precipitation and co-evaporation



Advantages

- Mainly used for volatile organic molecules (essential oil and vitamins)
- Mask odours
- Preserve aromas
- High stability and efficiency

Disadvantages

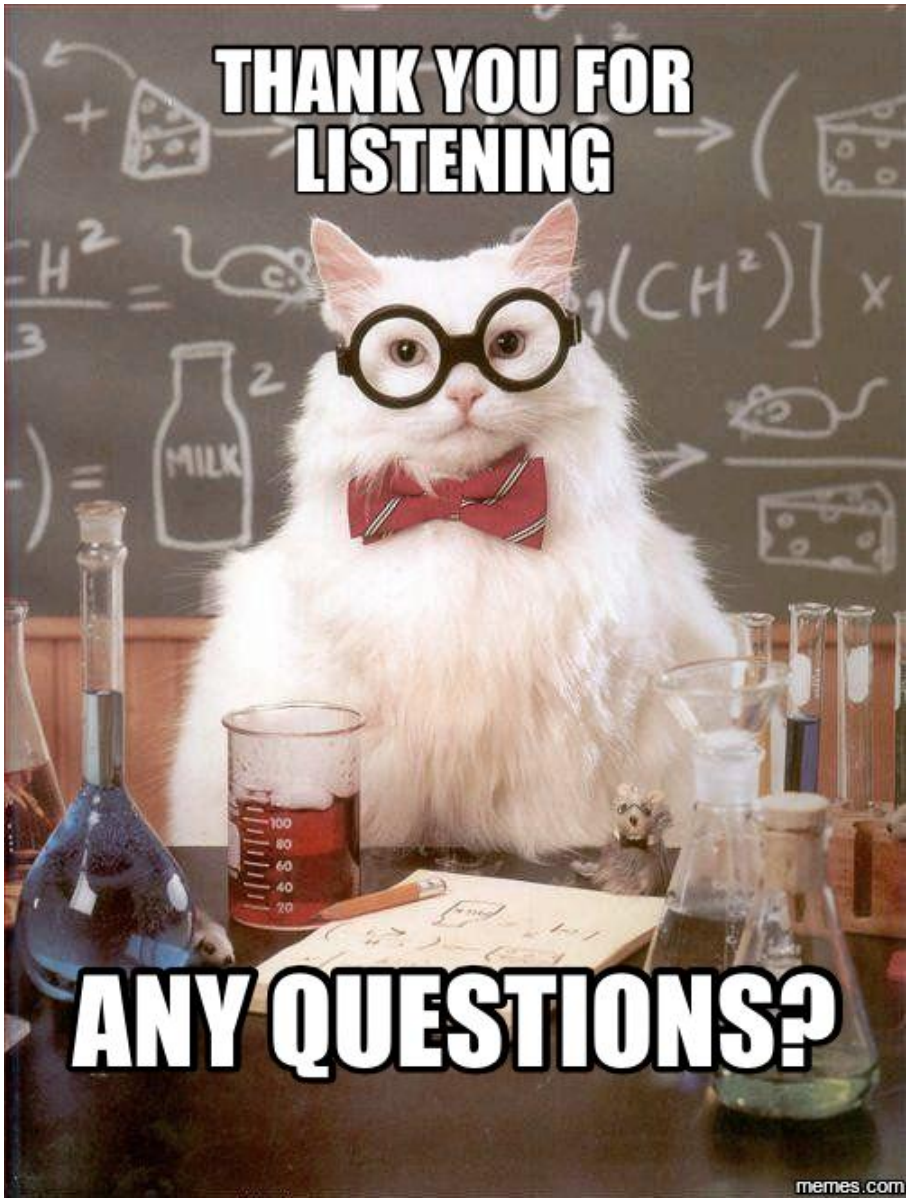
- Suitable shell materials are rarely available in food industry

Methods	Commonly encapsulated actives	Properties of methods	Particle size	Morphology (structure)	Properties of encapsulation/ delivery systems
Spraying technologies					
Spray drying	Hydrophobic compounds Lyophilized proteins	Easy handling Low operating costs High production rates Reproducibility Wide choice of encapsulating materials Encapsulation efficiency 10–90 % Difficult to control particle size Not suitable for high temperature-sensitive actives Moderate yields for small batches	Powders (10–300 μm) or Large-scale particles (2–3 mm)	Matrix	Spherical particles (powders) Good stability of encapsulates Non-uniform particles Tendency of particles to aggregate
Spray cooling/ chilling	Hydrophobic compounds	Lower operating costs compared to spray drying Suitable for heat-sensitive actives Encapsulation efficiency 10–100 % Particle size is not easily controllable Moderate yields for small batches	20–200 μm	Matrix	Microcapsules are insoluble in water Rapid release of the actives Special storage conditions can be required
Fluidized bed coating	Solid-core materials	Low-cost process Particle size distribution is controllable Can be used to add layer on preformed encapsulates Encapsulation efficiency 5–50 % Temperature-sensitive actives can be degraded	5–5,000 μm	Core/shell Multiwall Multicore	Slow release of actives
Microgel encapsulation					
Extrusion process	Cells Plant compounds Enzymes Proteins	Cost-effective Gentle/no organic solvents and extreme temperature and/or pH conditions are required Can be performed under both aerobic and anaerobic conditions Multiple-nozzle systems, spinning disk atomizer, jet-cutter techniques can have higher production capacity Encapsulation efficiency 20–50 % Difficult to scale up Difficult to process high viscous polymer solutions Microgel particles must be separated from the liquid bath	50–2,000 μm	Matrix Core/shell Multiwall Multicore	Spherical particles Burst release of actives from matrix type

Đorđević, Verica, et al. "Trends in encapsulation technologies for delivery of food bioactive compounds." *Food Engineering Reviews* 7.4 (2015): 452-490.

Methods	Commonly encapsulated actives	Properties of methods	Particle size	Morphology (structure)	Properties of encapsulation/delivery systems
Emulsification process	Hydrophilic and hydrophobic/lipophilic compounds	Easier to scale up than extrusion Microgel particles must be separated from the liquid bath More expensive compared to extrusion More difficult to control the process	50–2,000 μm	Matrix Core/shell Multiwall Multicore	Spherical particles Burst release of actives from matrix type
Complex coacervation	Hydrophobic compounds Flavors Essential oils	Expensive Complex mechanisms Sensitive to environmental conditions Can be applied for heat-sensitive actives Encapsulation efficiency 40–90 % The use of organic solvents	1–500 μm	Core/shell Multiwall	Spherical, multinucleate particles Heat-resistant Controlled release of actives Good storage stability
Emulsion technologies	Hydrophilic and hydrophobic/lipophilic compounds	*Low-energy procedures*: Low-cost method Suitable for thermolabile actives *High-energy procedures*: Can be scaled up Small droplet size (0.3–1 μm) Wide range of particle size and shape Encapsulation efficiency up to 100 % Higher operational costs/low-energy efficiency Polymerization during capsule formation Restricted number of food-grade emulsifiers	(Macro) Emulsions > 200 nm 5 nm < Micro emulsions < 100 nm 20 nm < Nanoemulsions < 200 nm	Core/shell Multicore Multiwall	Spherical particles Can be polydisperse Conventional emulsions are unstable when exposed to food processing Multilayer emulsions are stable Controlled release of actives from multilayer emulsions
Encapsulation in liposomes	Hydrophilic and hydrophobic/lipophilic compounds Enzymes	Encapsulation efficiency 5–50 % High costs of scale-up	10–1,000 μm	Phospholipid bilayers	Controller release of actives
Encapsulation in cyclodextrins	Apolar molecules Flavors	Inclusion efficiency, from 30 to 100 % High price of cyclodextrins Restricted to low-molecular-weight compounds	From nano- (~1 nm) to microsize (~1,000 nm)	Molecular inclusion	Increase solubility of hydrophobic molecules Controlled release of actives Particles (β-cyclodextrins) tends to agglomerate
Supercritical fluid-based encapsulation	Hydrophilic and hydrophobic/lipophilic compounds	Avoid the use of organic solvents, ware, high temperatures, and mechanical stress Encapsulation efficiency 20–100 % Difficult to control process	10–400 μm	Matrix Core shell	Particles tend to agglomerate
Encapsulation in yeast	Aromas and antioxidants	Simple process Cost-effective process	~5 μm	Core shell	Thermostable up to 250 °C Gradual release of actives Specific in-mouth delivery of actives

Đorđević, Verica, et al. "Trends in encapsulation technologies for delivery of food bioactive compounds." *Food Engineering Reviews* 7.4 (2015): 452-490.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ANY QUESTIONS?
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Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS

European Commission

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