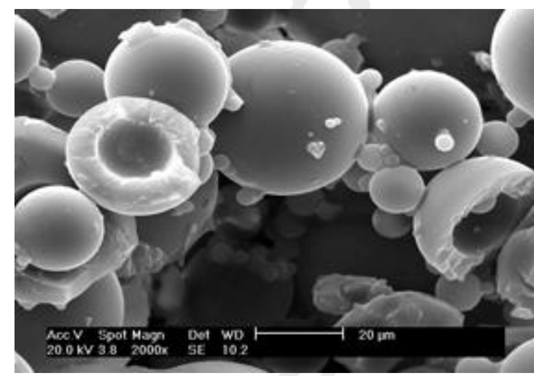
FOODstars *Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS*

European Commission

Factors affecting stability of encapsulated ingredients

Dr. Eng. Camila A. Perussello

camila.perussello@teagasc.ie



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Stability and quality of foods

- Food stability = **retention of quality** during processing and storage

Sensory aspects

Nutritional value

Functional properties

Oxidation

Microbial safety

Chemical contaminants

Composition

Rheological properties

etc…

FOOD stars

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

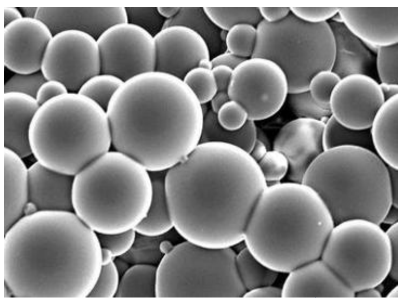
Factors affecting stability of encapsulated ingredients

- External factors

pH

temperature

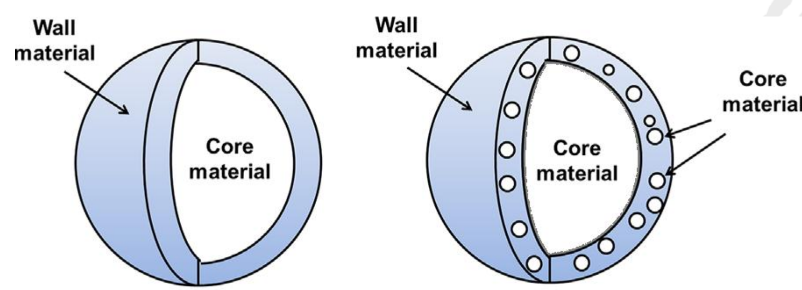
light



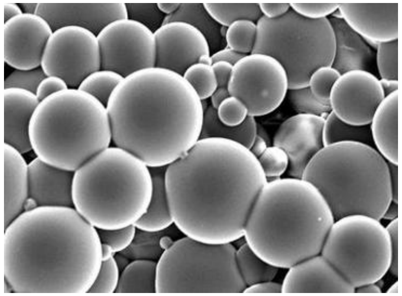
moisture/Aw

O_2

- Intrinsic factors
    - Carrier materials
    - Active materials
    - Payload
    - Particle size
    - Particle shape/morphology



- **External** factors affecting stability of encapsulated ingredients can lead to:
  - pH
    - Decreased probiotic viability in acidic media
    - Changes in chemical structure/ loss of functionality
  - O_2
    - Oxidation
    - Decreased nutritional value
  - Light
    - Carotenoids, vitamins, polyphenols and lipids may degrade, isomerize or generate free radicals
    - Discoloration
  - Moisture/Aw
    - Aggregation of particles
    - Decreased delivery of functional properties
  - Temperature
    - Protein denaturation
    - Decreased nutritional value
    - Color changes



- **Intrinsic** factors such as
    - Properties of carrier materials (μ, solubility, concentration, mass diffusivity)
    - Properties of shell materials (thickness, μ, solubility in water/oil)
    - Payload, morphology and size

  have influence on aspects such as:
    - release rates/mechanisms
    - sensory perception (texture/grittiness/roughness, odor, flavour, taste, appearance)

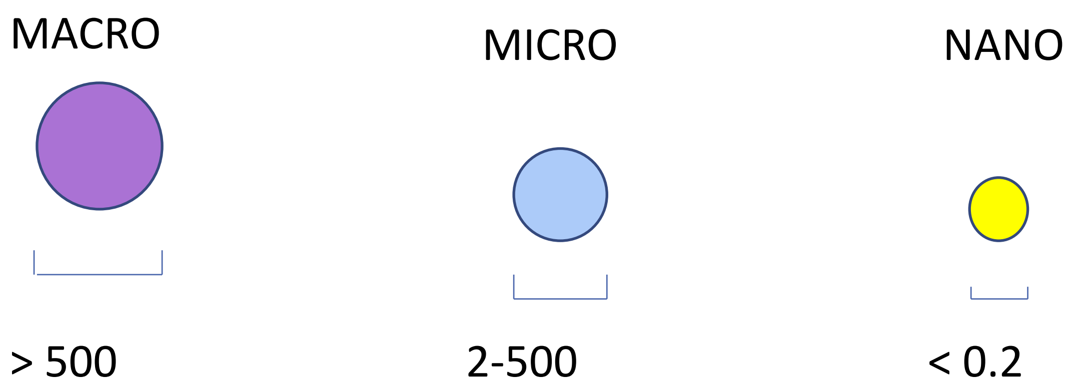
Consumer perception and acceptance!

Stability/quality of encapsulated ingredients

- Characterization of microparticles and encapsulated ingredients include determinations of:
  - Particle size, particle size distribution/uniformity;
  - Morphology of microparticles;
  - Properties of the encapsulated active ingredient (composition, thermal stability, volatility);
  - Properties of the microparticle (solubility);
  - Mass rate core/coating material (payload);
  - Thermal/oxidative stability of core and coating;
  - Sensory quality of the encapsulated material (color, flavor, odor).

Particle size

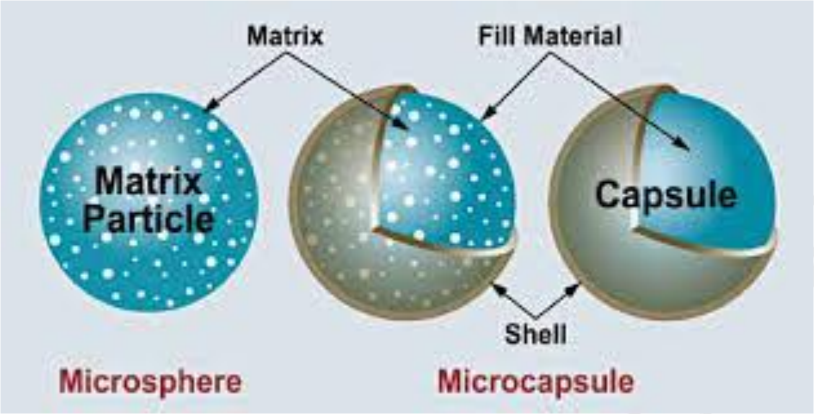
- Particle size varies according to the encapsulation method, active material, coating material and process parameters

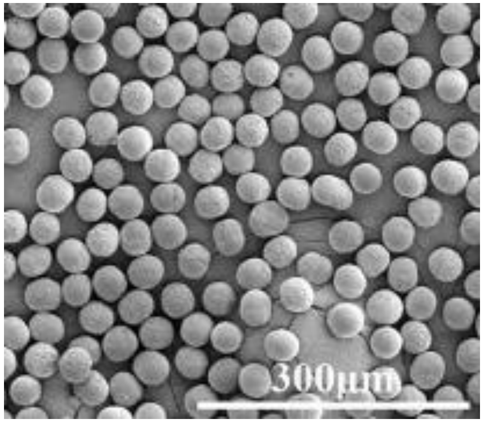


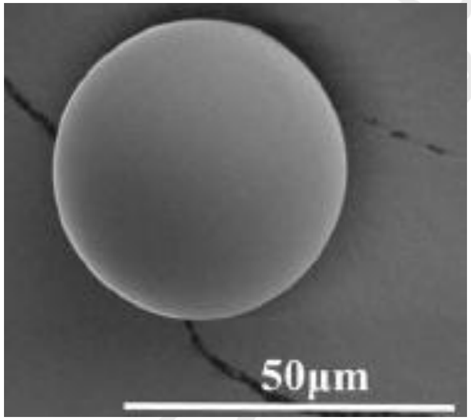
- Particle measurement techniques
  - Microscopy
  - Sieving
  - Sedimentation
  - Laser light scattering

Choosing a method for particle sizing

- Characteristics of the microparticles
  - Microsphere or microcapsule
  - Estimated size and size distribution
  - Toxicity
  - Level of detail/accuracy
- Application of the microparticles
- Cost
- Time



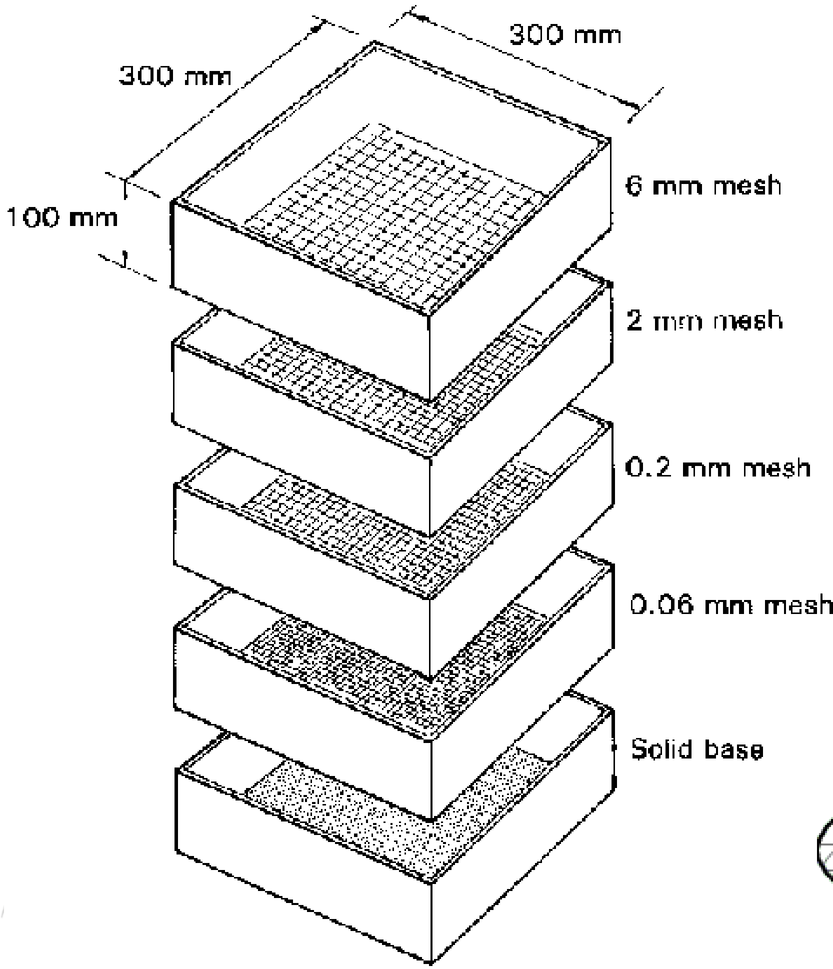


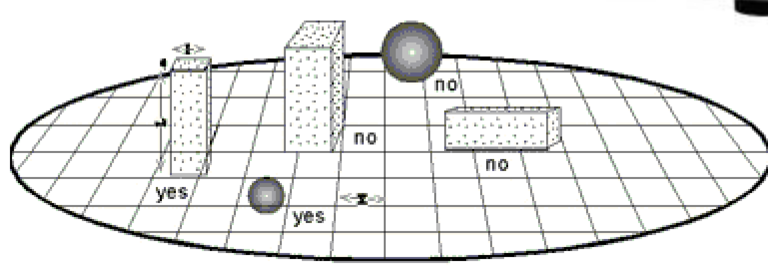


Particle sizing - Sieving

➢ Sieving is performed manually or by machines

using a stack of sieves with decreasing aperture size;





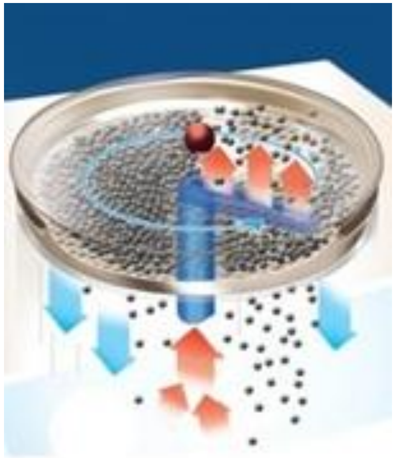
Particle sizing - Sieving

- Sieves have a specific aperture/mesh size (ex. 250 µm = No. 60; 125 µm = No. 120)
  - Sieve sizes are regulated by standards (ISO 565:1990 and ISO 3310-1:2000 (international), EN 933-1(European) and ASTM E11:01 (US);
  - Size range ≈ 5 µm – 3 mm (3000 µm).

Sieve Mesh Chart			
APERTURE SIZE			
B.S.S(410/1969)	A.S.T.M. (11-70)	I.S. (469/1972)	MICRONS
4	5	4.00mm	4000
5	6	3.35mm	3353
6	7	2.80mm	2812
7	8	2.36mm	2411
8	10	2.00mm	2057
10	12	1.70mm	4000
12	14	1.40mm	1405
14	16	1.18mm	1204
16	18	1.00mm	1204
18	20	0.850mm	850

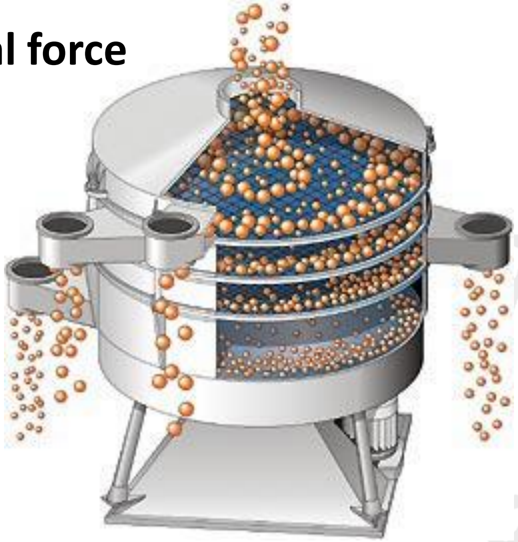
Particle sizing - Sieving

- Sieving may be performed manually or by machines aided by different mechanisms

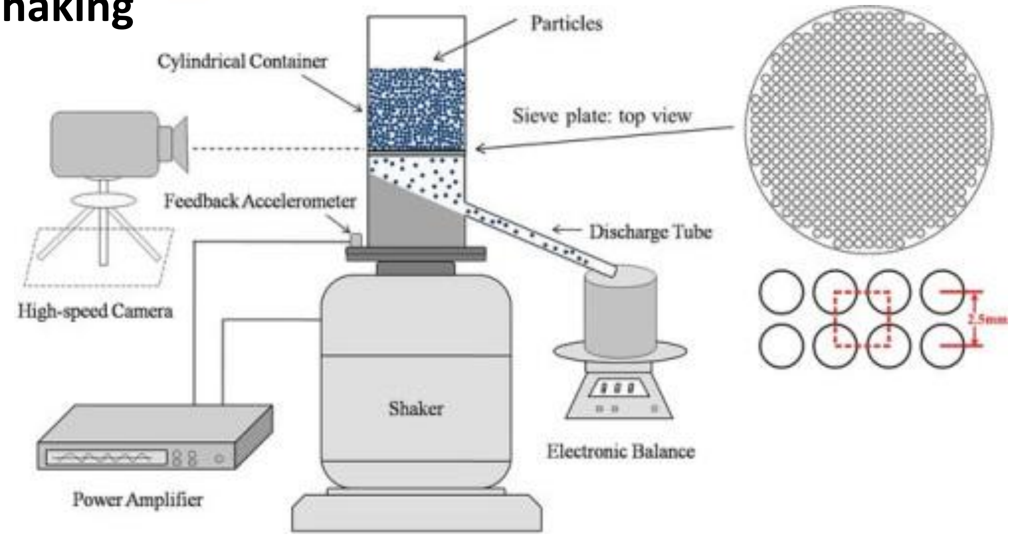


Air jet

Centrifugal force



Shaking

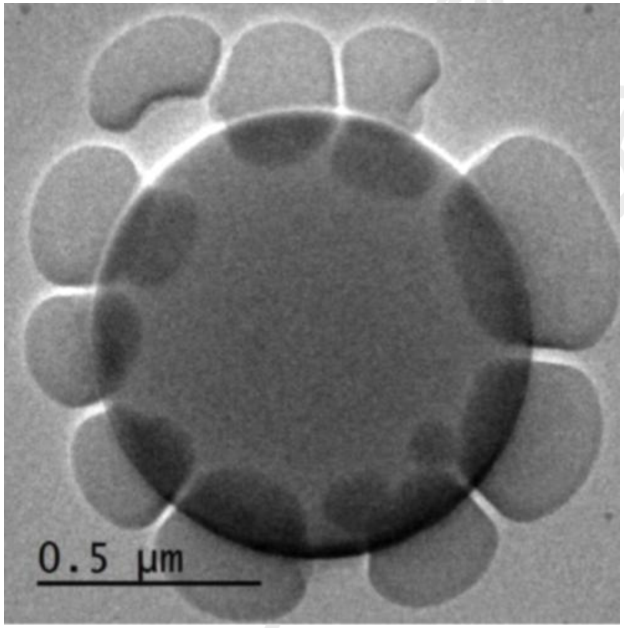


Ultrasonic

Particle sizing - Microscopy

- Optical microscopy (1 µm - mm) and Electron microscopy (≥ 0.001 µm)
  - Microscopy can distinguish aggregates from single particles;
  - Can be coupled to image analysis computers, each field can be examined, and a distribution obtained;
  - With small particles, diffraction effects increase causing blurring at the edges - determination of particles < 3 µm is less and less certain.

EM has **higher magnification and resolution** than OM: employs electron beams in place of light to illuminate specimens and uses electron lenses to magnify images



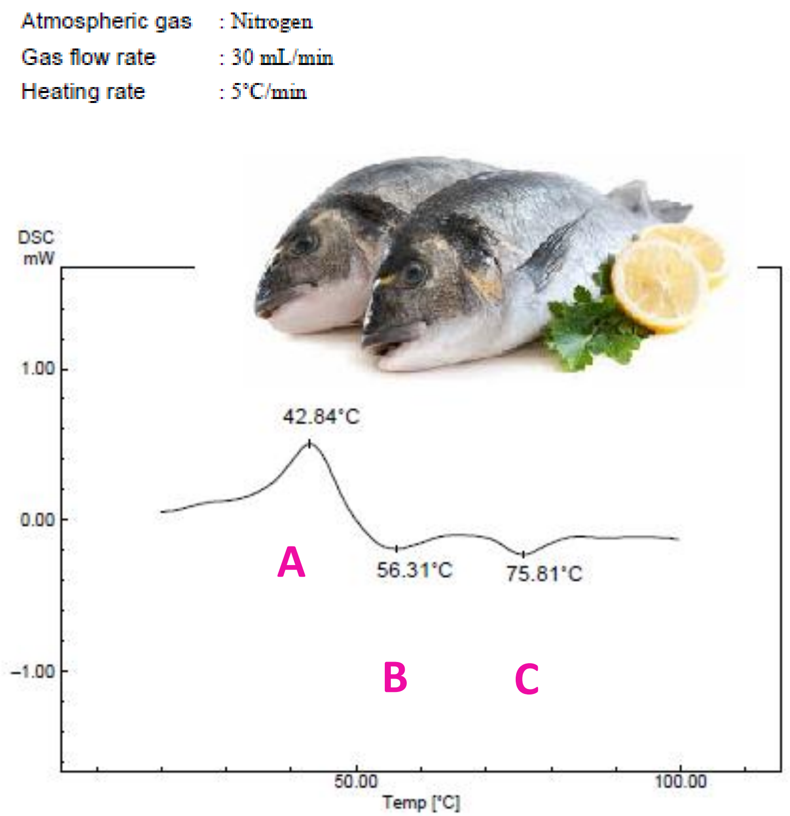
Thermal analysis

- Thermal analyses can be used to determine the thermal behavior of substances subjected to temperature variations;

- The main thermal analysis techniques are:
  - DSC = Differential scanning calorimetry
  - TG = Thermal gravimetry
  - TMA = Thermo-mechanical analysis
  - DMA = Dynamic mechanical analysis

- The results may bring conclusions such as decomposition behaviour of materials, melting point, Tg, drying, evaporation, adsorption of substances, oxidation and purity of substances.

DSC for the decomposition of carp meat

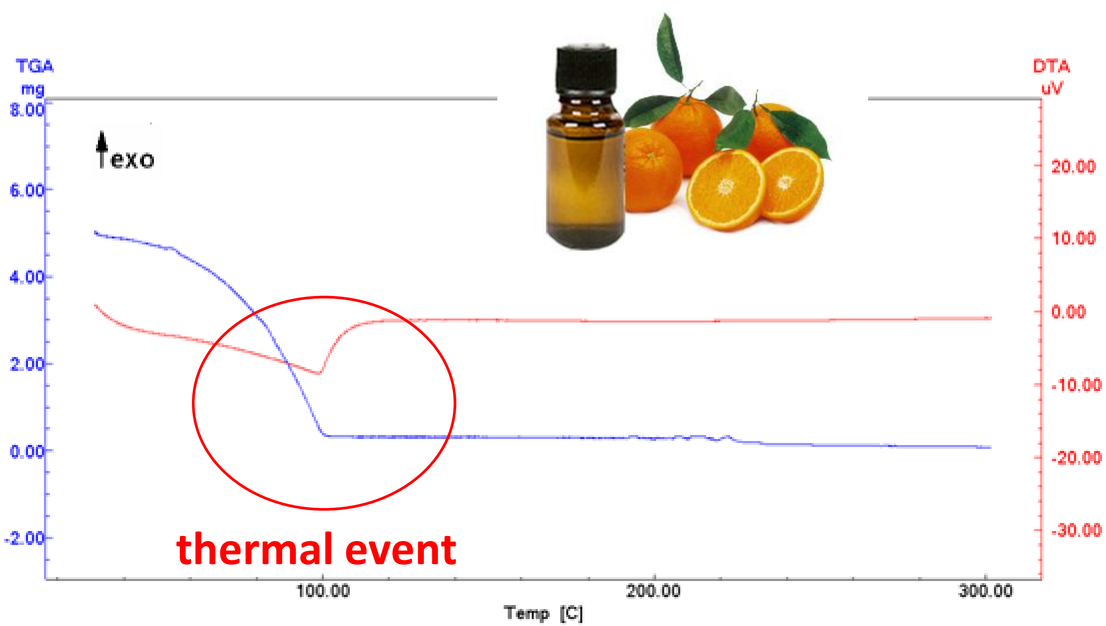


A - Shrinkage of myosin and actin caused by ATP remaining in the fish meat.

B and C - Denaturation of myosin and actin, respectively.

Thermal analysis – TGA and DTA

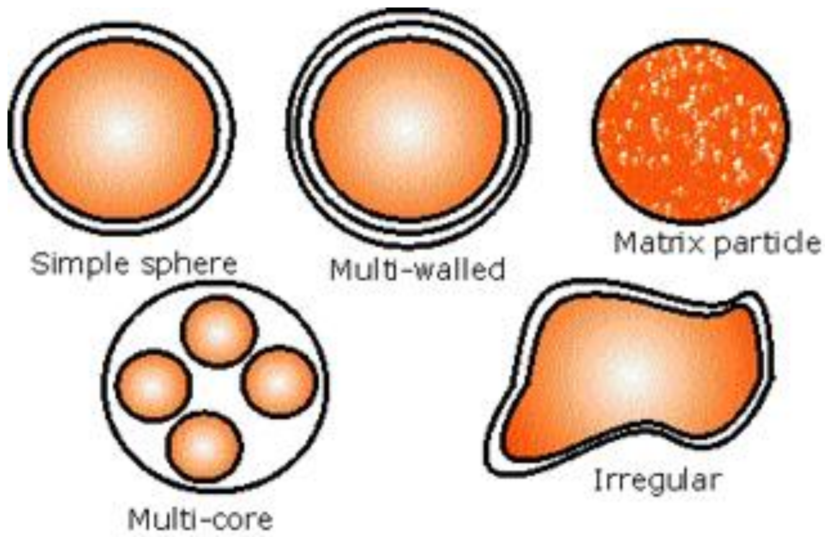
- Thermal Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) are used to determine the thermal behavior of substances subjected to **temperature variations**:
  - TGA curves provide results of mass loss (or mass gain) of a substance during a temperature programme;
  - DTA curves, usually plotted together with TGA curves, provides a registration of thermal fluxes during a temperature programme.



TG and DTA curves for orange essential oil (air synthetic atmosphere 10°C/ min, 100 mL/min, 30 - 300°C, mass versus temperature)

Payload

- Payload is the amount of core material encapsulated within a shell or matrix;
- Range from 1 to 99% depending on the **particle morphology**, encapsulation materials, and process;
- Microspheres accommodate payloads of 10-30%;
- Microcapsules accommodate more active material than microspheres (≥ 90%).



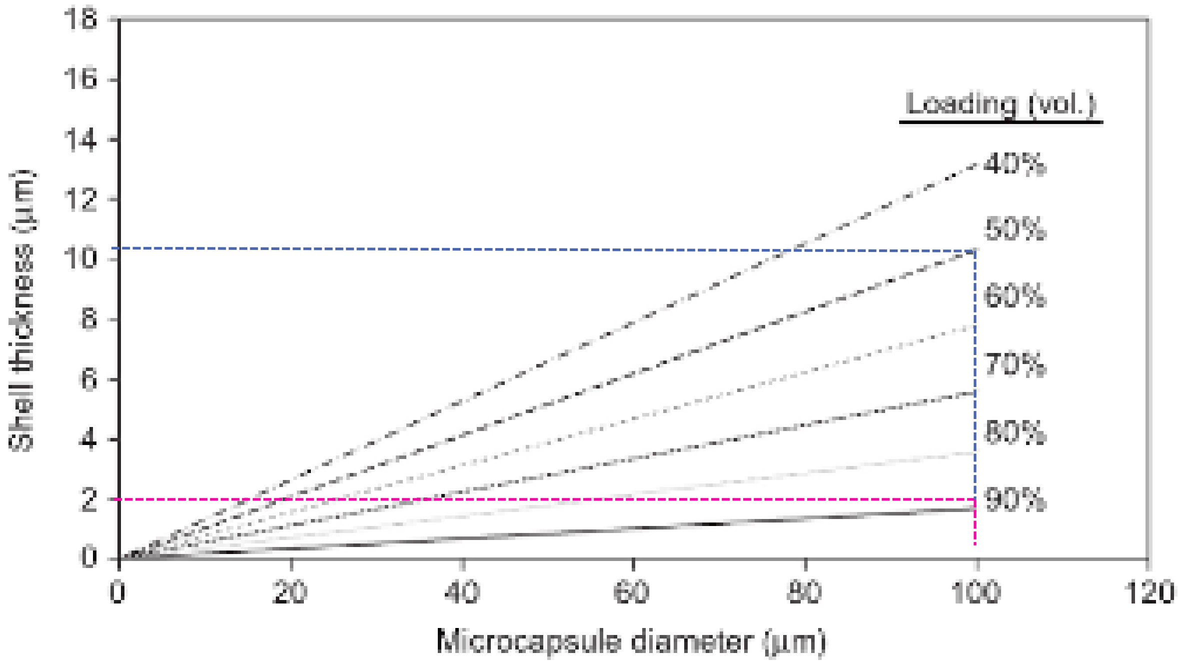
Microcapsule shapes

➢ Higher payloads mean

- Lower protection against environmental conditions (thinner shell)
- Higher costs with active ingredient
- Higher amount of core material near or at the surface (faster degradation and/or faster release – sometimes desirable)
- Inverted morphology in microspheres with payload >50%
- Thinner shells = less mechanical strength to be broken

➢ Lower payloads mean

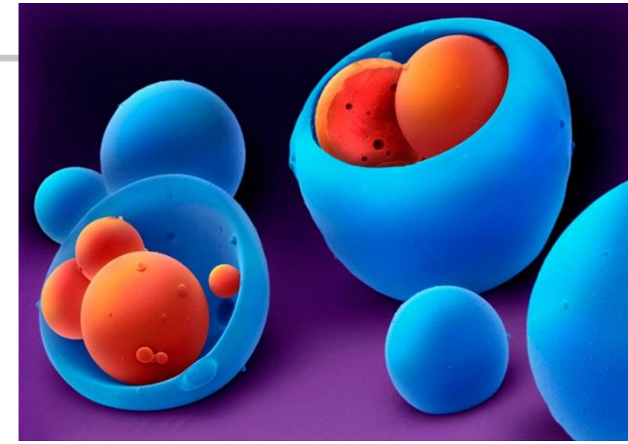
- Thicker shell (more protection)
- Higher costs with coating materials



Loading versus shell thickness for microcapsules

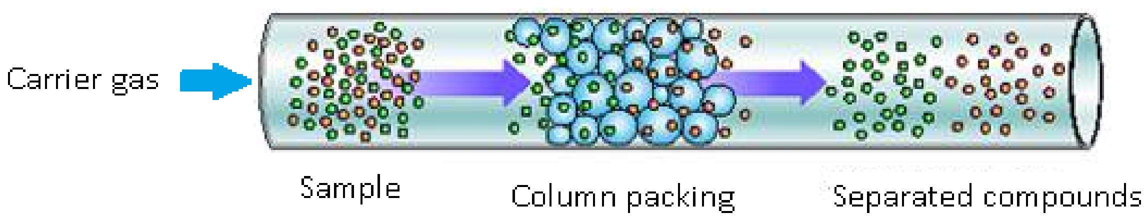
- A 100 µm capsule with 90% payload will have a 2 µm-thick shell
- Increasing shell thickness to from 2 to 10 µm reduces payload to 50%

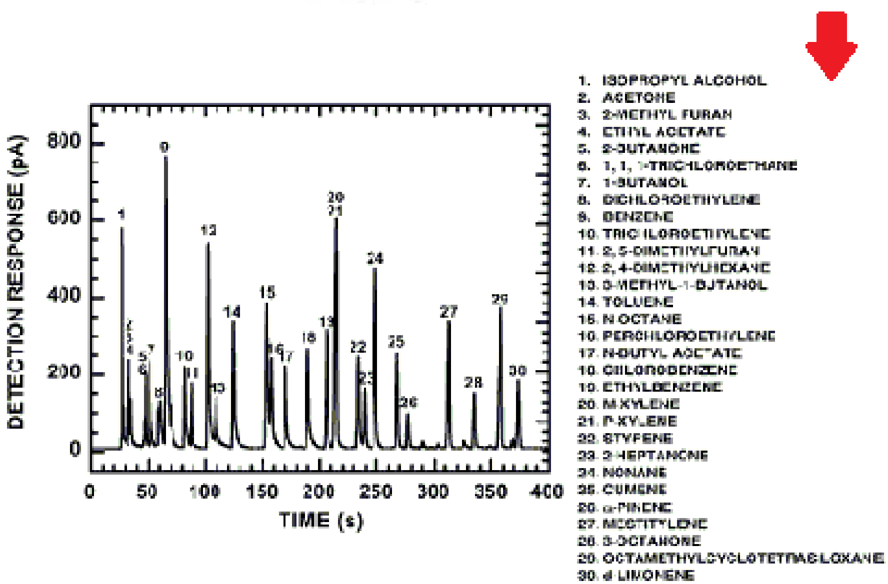
➢ How to determine payload



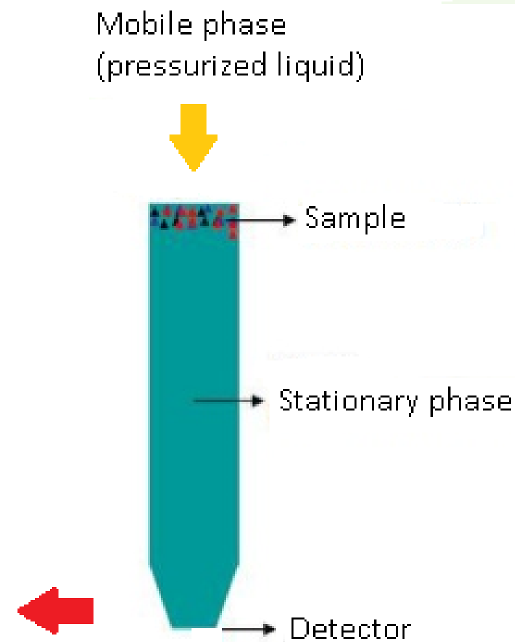
- ➢ Main techniques
  - GC/MS
  - HPLC

GAS CHROMATOGRAPHY





HIGH PERFORMANCE LIQUID CHROMATOGRAPHY



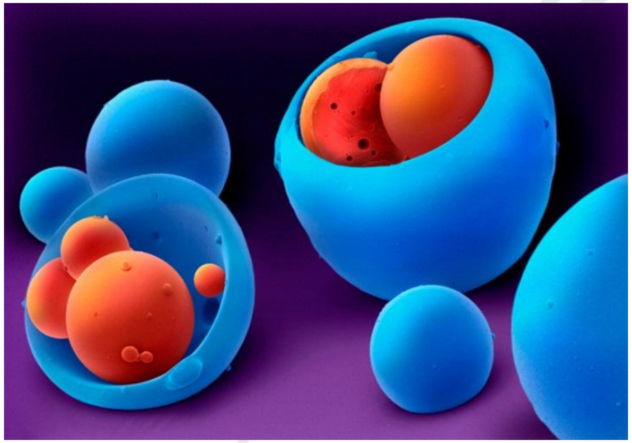
➢ Typical payloads for different encapsulation methods

- Atomization techniques: 10-30%

  - Spray-drying can accommodate higher loading through adjustment of the core/shell ratio in the SD solution and residence time;
  - Spray-chilling/freezing is limited to lower loadings because the feed slurry for atomization must remain t a low viscosity to form an aerosol;

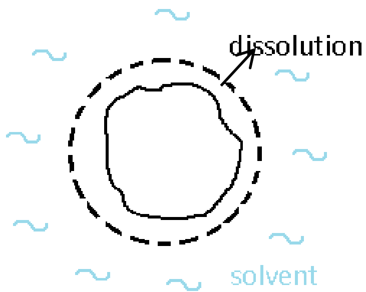
- Extrusion: can be tuned to generate capsules with a range of payloads (nozzle geometry and size, shell materials);

- Emulsion-based processes (layer-by-layer) offer the highest payloads (up to 99%).

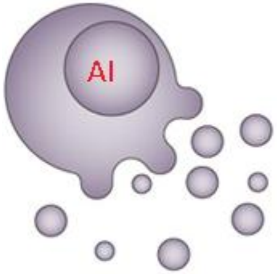


Release mechanisms

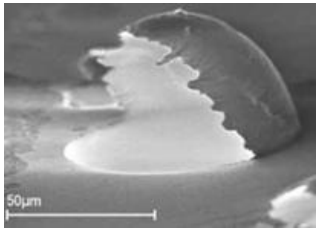
A. Dissolution (pH, solvent, moisture)



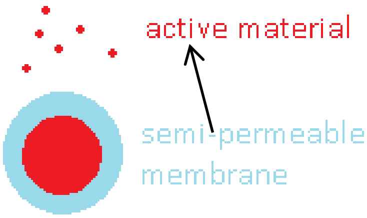
B. Thermal (fusion)



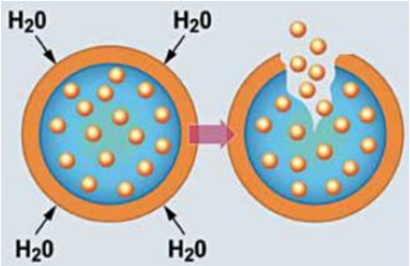
C. Mechanical (pressure, cut, friction)



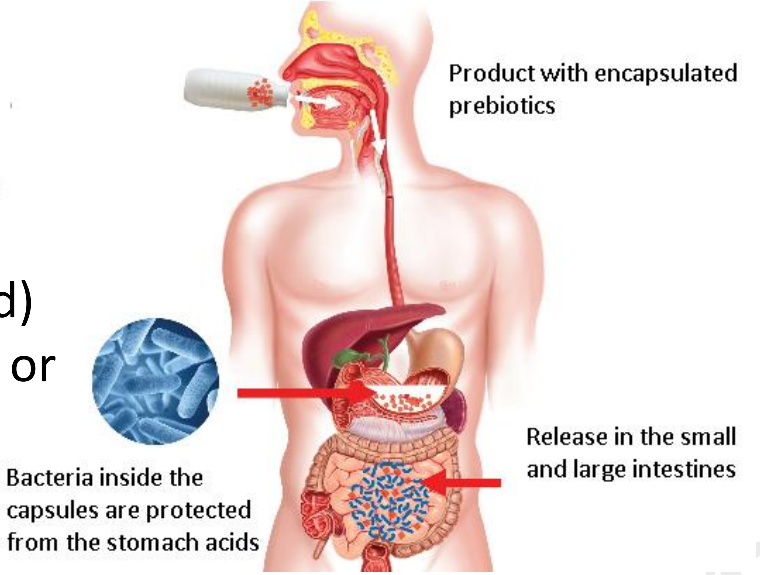
D. Diffusion (selective permeability)



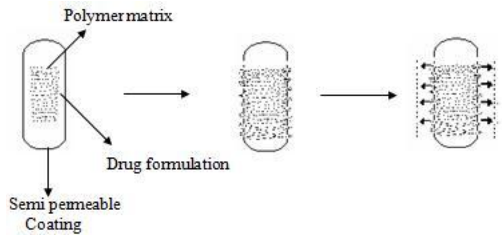
E. Osmotic



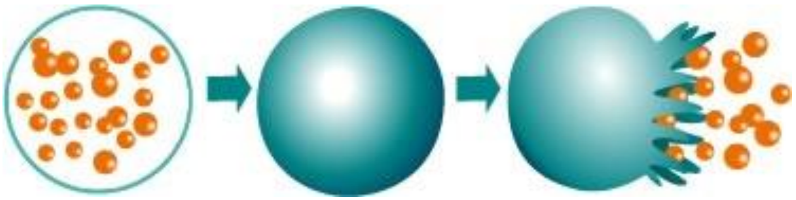
Release profiles



- Triggered release
  - Triggers: pH, T, moisture, P, electromag. field)
  - Allows to achieve immediate, delayed or pulsatile release profiles
- Sustained release
  - Release occurs for an extended period of time
  - Allows to achieve constant active ingredient to exposure for a fixed period

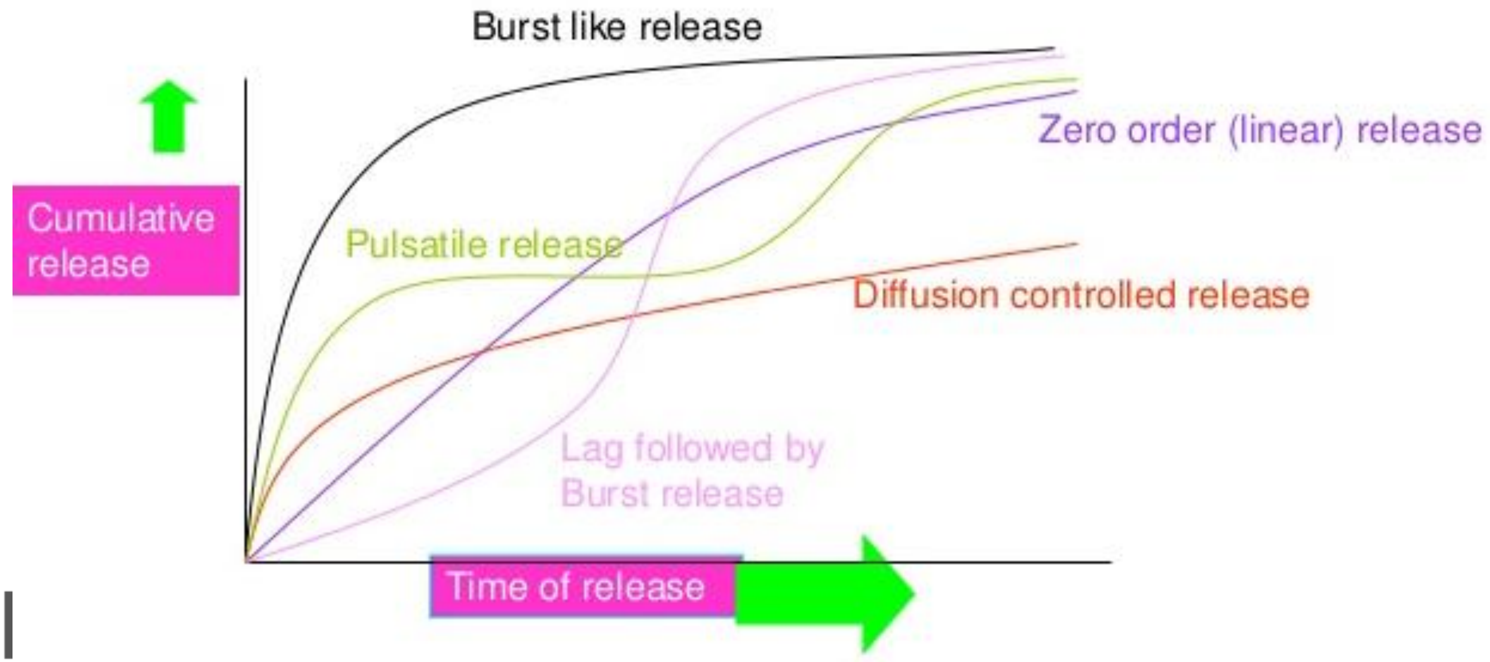


- Burst release
  - Release due to mechanical forces



Release profiles/rates

- Release profiles and rates depend on factors such as:
  - Characteristics/properties of coating polymers and active ingredients
  - Payload
  - Morphology and size of the encapsulated particles



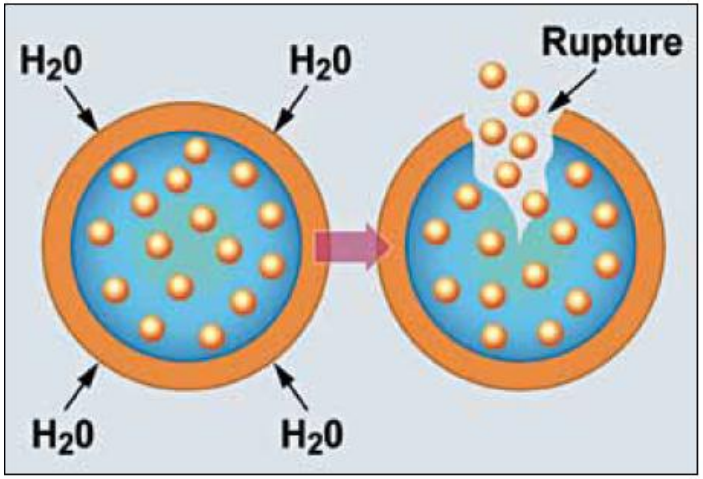
Mathematical modelling of release profiles

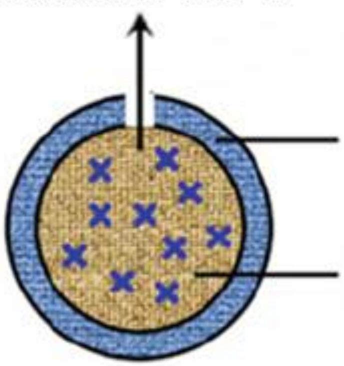
Release rate model	Equation	Application
Zero order kinetics	$C_t = C_0 - kt$	Microspheres, fast release
First order kinetics	$ln[C_t] = ln[C_0] - kt$	Slow release
Higuchi's equation	$\frac{C_t}{C_0} = k_H\sqrt{n} + b$	Microcapsules, diffusion, low-soluble matrices
Korsmeyer-Peppas	$\frac{C_t}{C_0} = kt^n + b$	Diffusion + erosion
Peppas-Sahlin	$\frac{C_t}{C_0} = k_1t^m + k_2t^{2m}$	Diffusion + erosion

Release methods

Common Controlled Release Profiles

- Triggered release – Release occurs due to a change in environment, such as pH, temperature, moisture, pressure, electromagnetic. This is used to achieve immediate, delayed or pulsatile release profiles.
- Sustained release – Release occurs for an extended period of time. This can be used to achieve constant active ingredient exposure for a fixed period.
- Burst release
- Combination release profiles



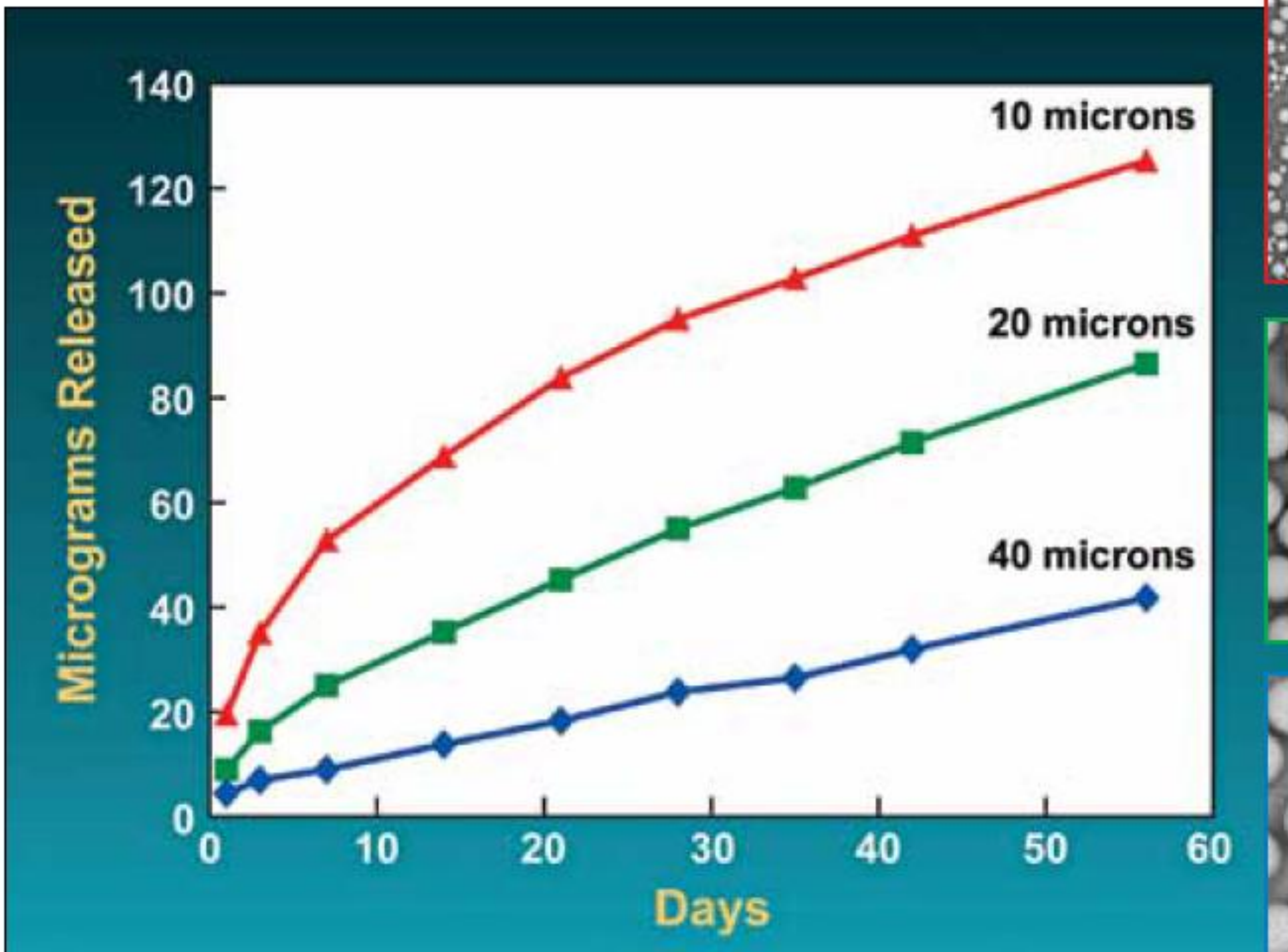


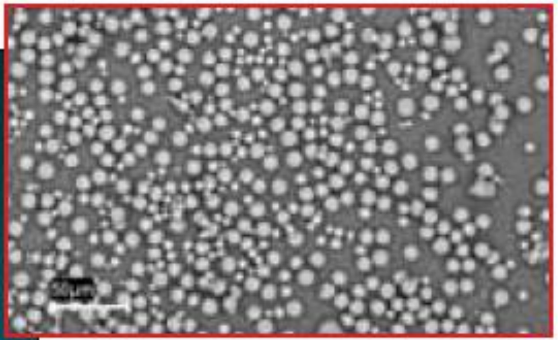
Release Mechanisms

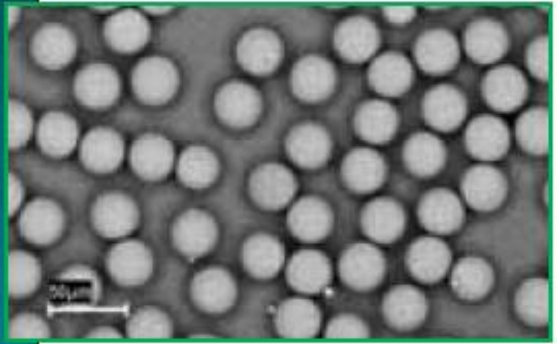
- Diffusion
- Dissolution
- Molecular trigger (such as pH)
- Biodegradation
- Thermal
- Mechanical
- Osmotic

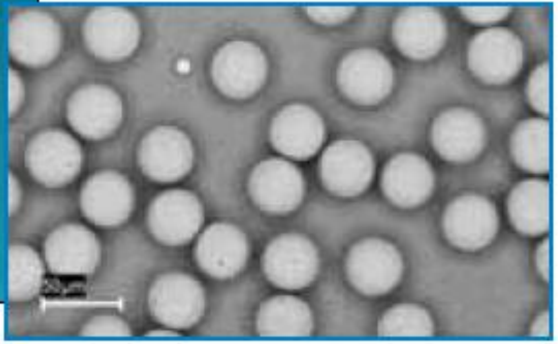
Release rates

➢ Particle size is one of many parameters that may be adjusted to control release rates of encapsulated ingredients.









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