Nanoencapsulation by Interfacial Polymerisation

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Innovations in Encapsulation 2017

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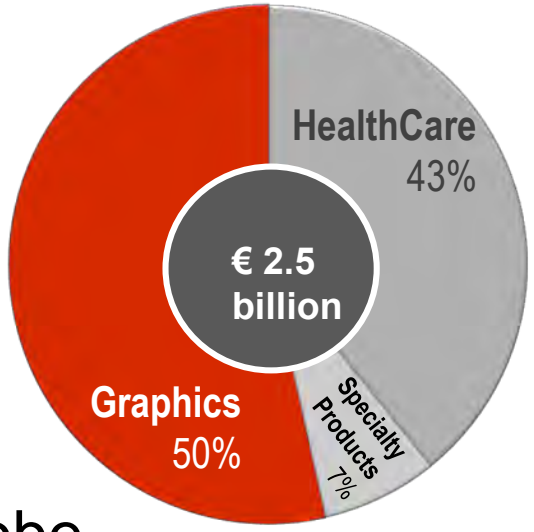
Agfa-Gevaert Group

- Founded in 1867, IPO in 1999 (Brussels)
- Headquartered in Antwerp, Belgium
- Sales of EUR 2.537 billion in 2016
- 10,360 employees (FTEs) worldwide
- Wholly owned sales organizations in more than 40 countries
- 25 R&D and production sites around the globe
- Global market leader in each of its divisions



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Encapsulation at Agfa-Gevaert

- An old history to build upon: First patents filed in 1962

1034437

PATENT SPECIFICATION

NO DRAWINGS

1034437

Date of Application and filing Complete Specification Feb. 20, 1963.

No. 6860/63.

Application made in Netherlands (No. 275045) on Feb. 20, 1962.

Complete Specification Published June 29, 1966.

Index at acceptance:—B8 CA

Int. Cl.:—A 61 j 5/00

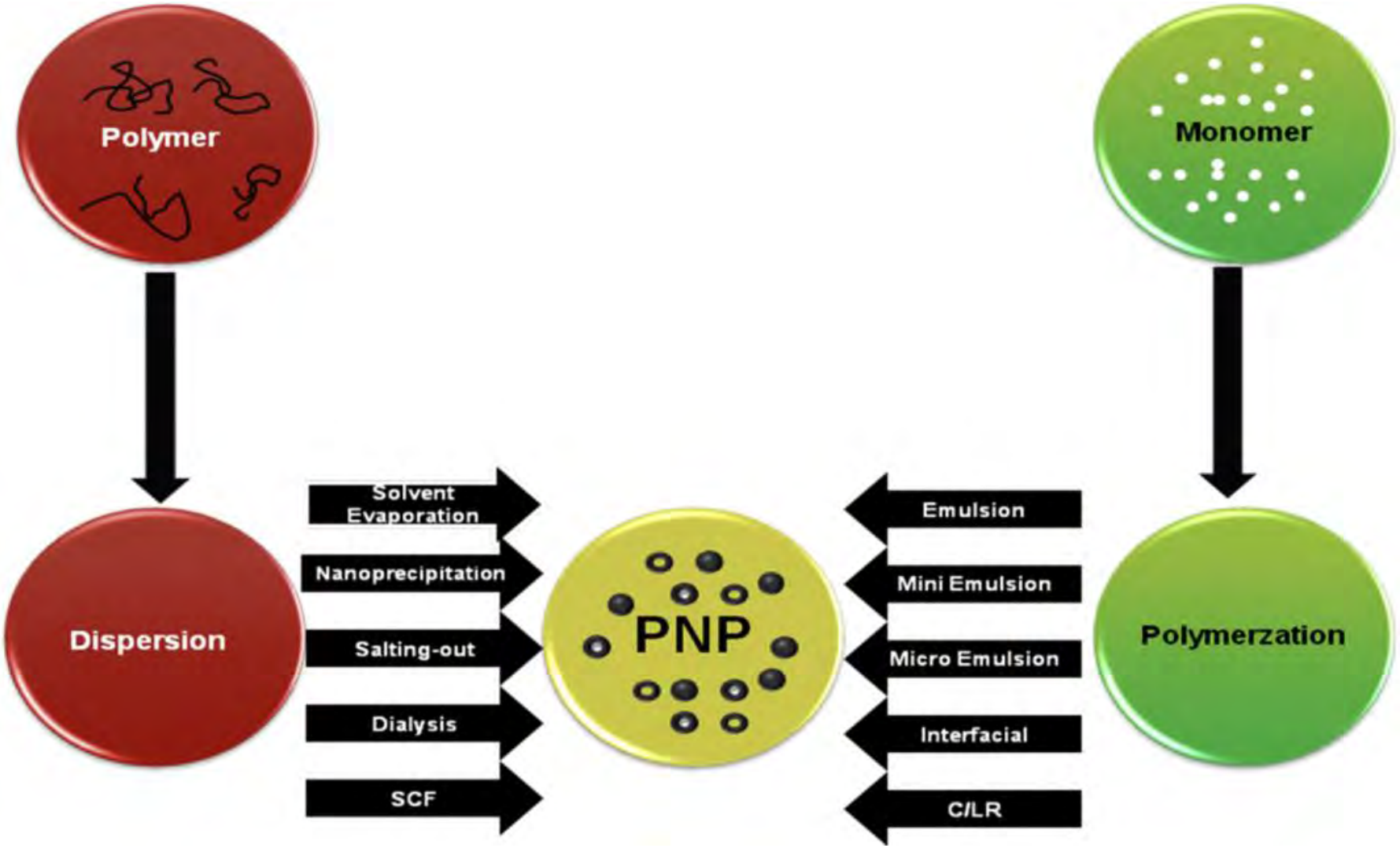
COMPLETE SPECIFICATION

Preparation of Microcapsules

We, GEVAERT PHOTO-PRODUCTEN N.V. a Belgian Company of Mortsel-Antwerp, Belgium, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

the oil with only one gellable hydrophilic colloidal sol whereupon coacervation occurs by adding a strongly concentrated salt solution to the emulsion. The colloid material is deposited around the oil droplets and the capsule-forming colloid material is gelled by cooling.

Encapsulation Methods



Rao, J. P.; Geckeler, K. E., *Progress in Polymer Science* **2011,** *36* (7), 887-913.

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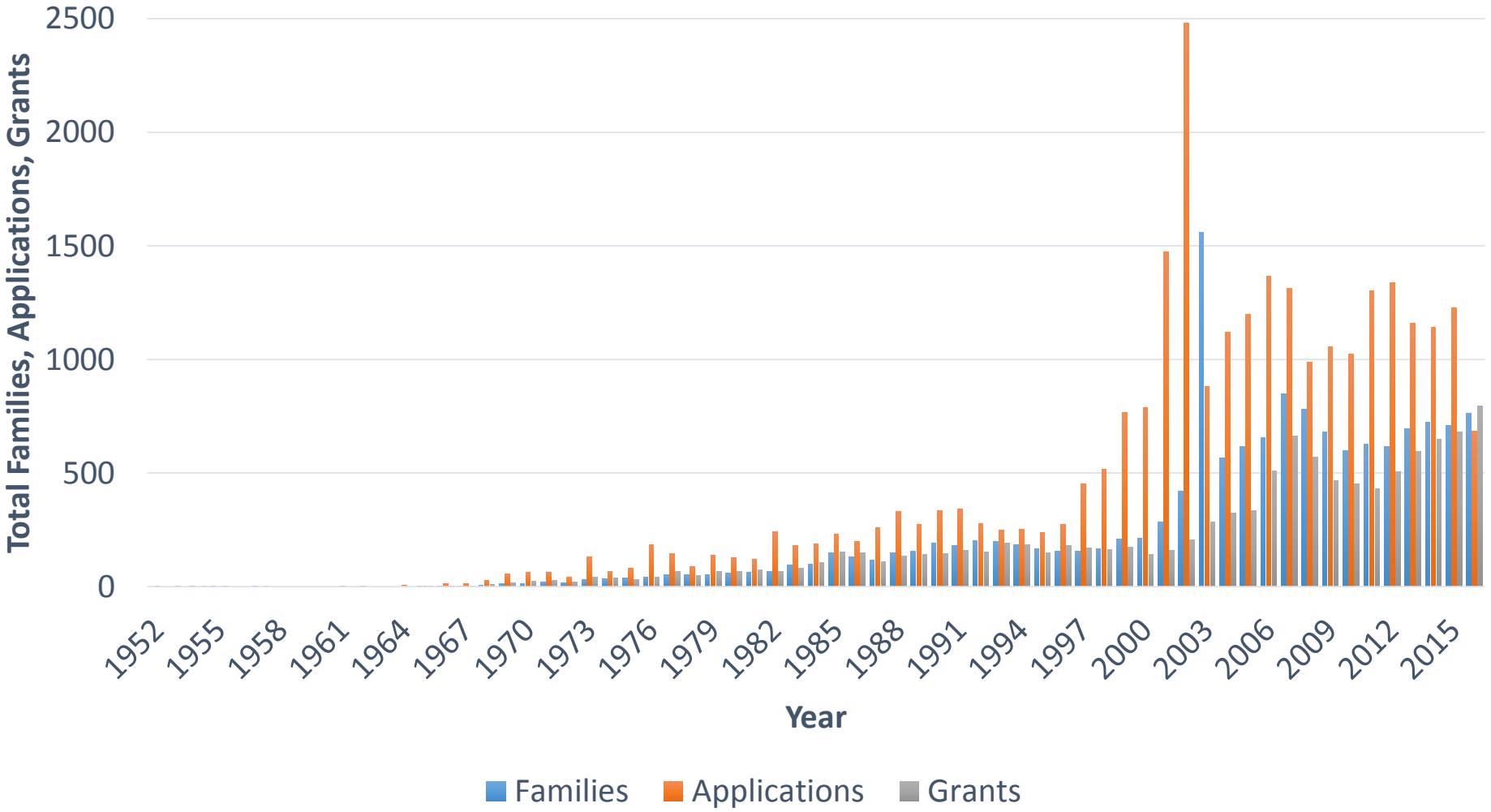
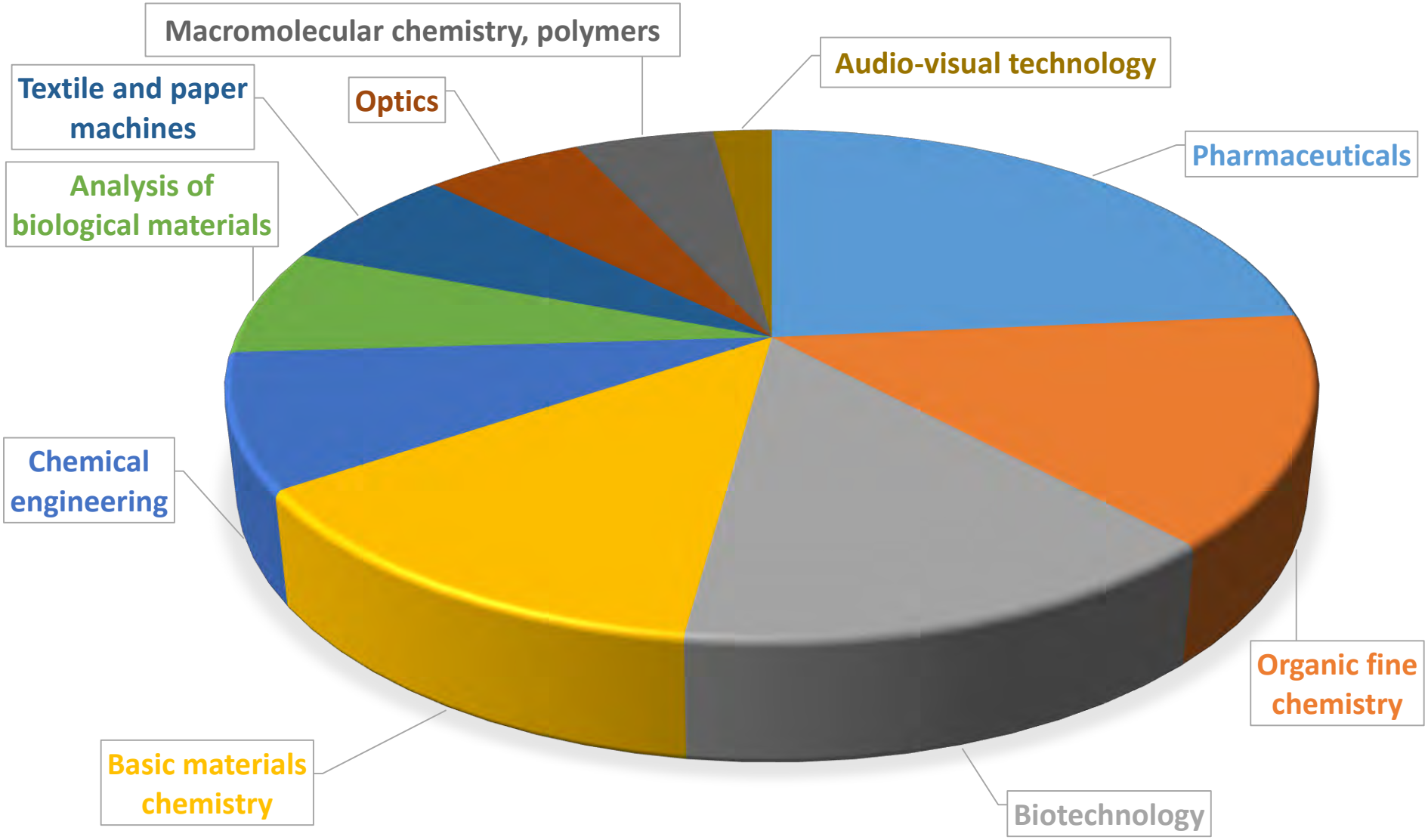
Patent Landscape

PatBase Search (1/12/17): *encapsulation and (interfac* w1 polymeri?ation)



International Patent Classification (IPC): Application Fields

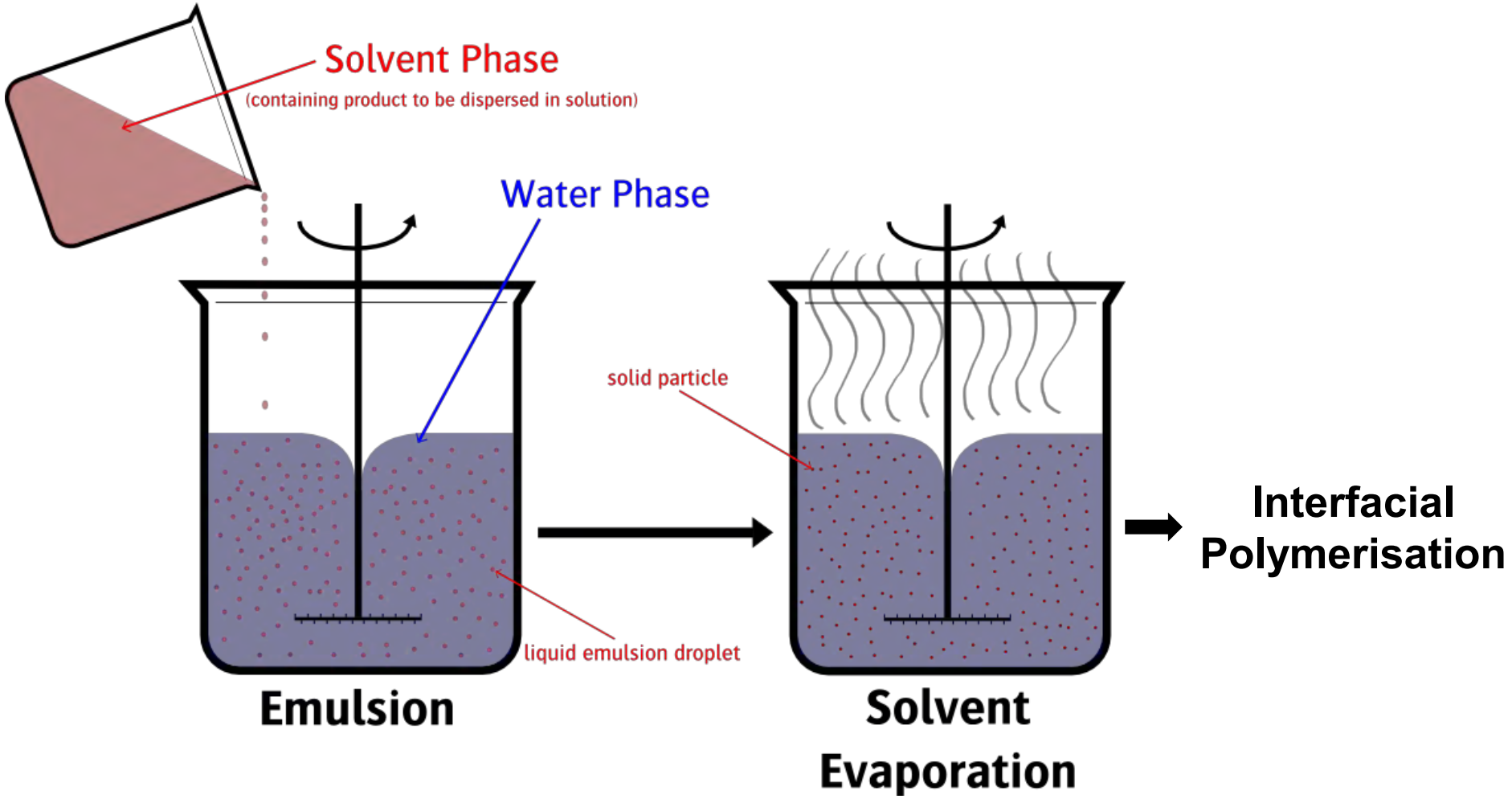


Encapsulation: Interfacial Polymerisation

- Advantages:
  - Good encapsulation degree
  - Can obtain a shell from a polymer not soluble in common solvents
  - Crosslinked capsules possible
  - Core-shell morphology

- Disadvantages:
  - Reactive chemistry
    - Residual monomer
    - Not always compatible with ingredients to be encapsulated

Encapsulation: Interfacial Polymerisation



Encapsulation: Interfacial Polymerisation

- Process:

1. Oil phase:
   - Active product
   - Monomer (e.g. isocyanate)
   - Solvent (e.g. ethyl acetate)

2. Emulsification:
   - Oil in water (O/W)
   - Dispersing agent(s)

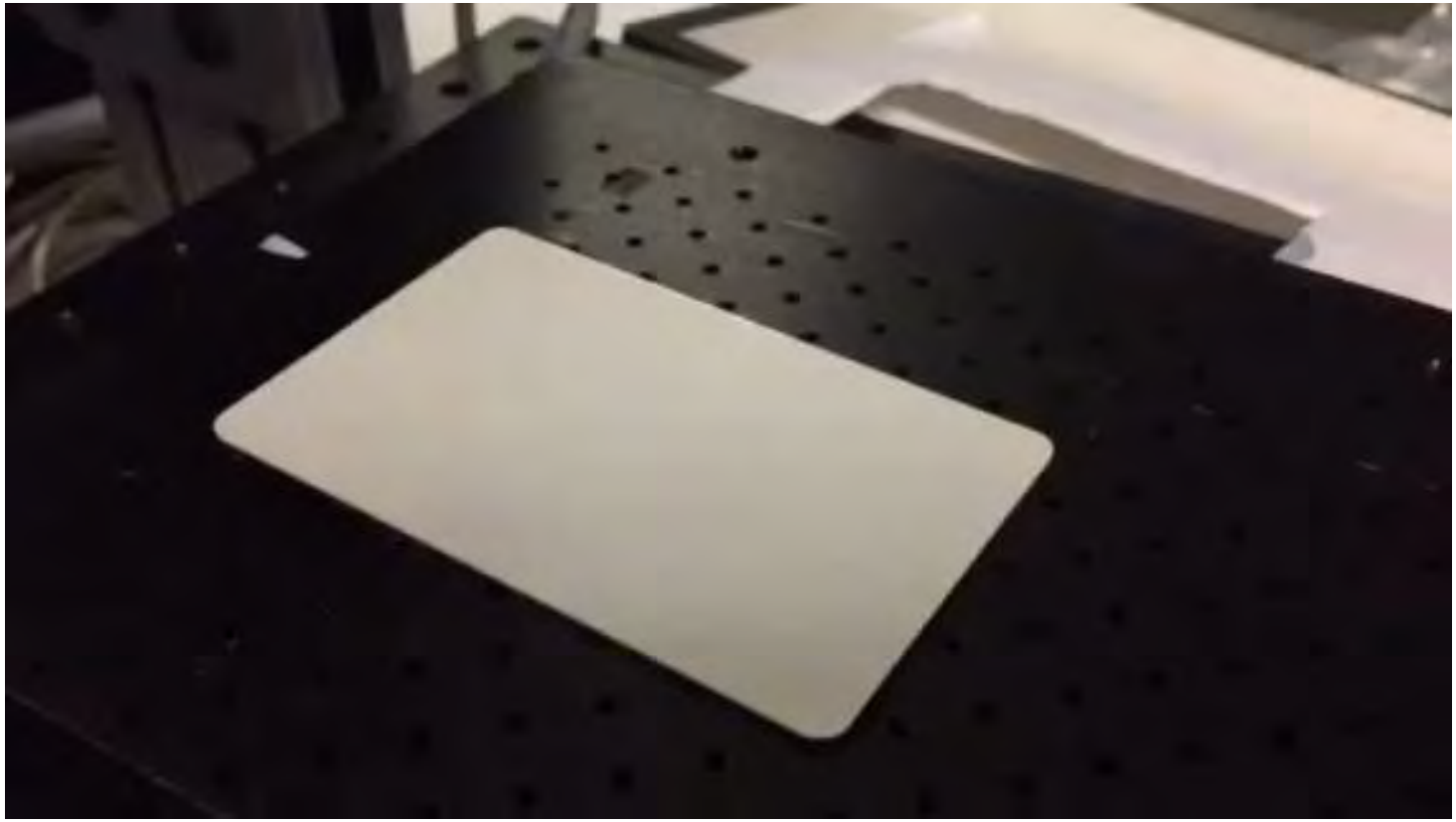
3. Solvent evaporation

4. Interfacial polymerisation

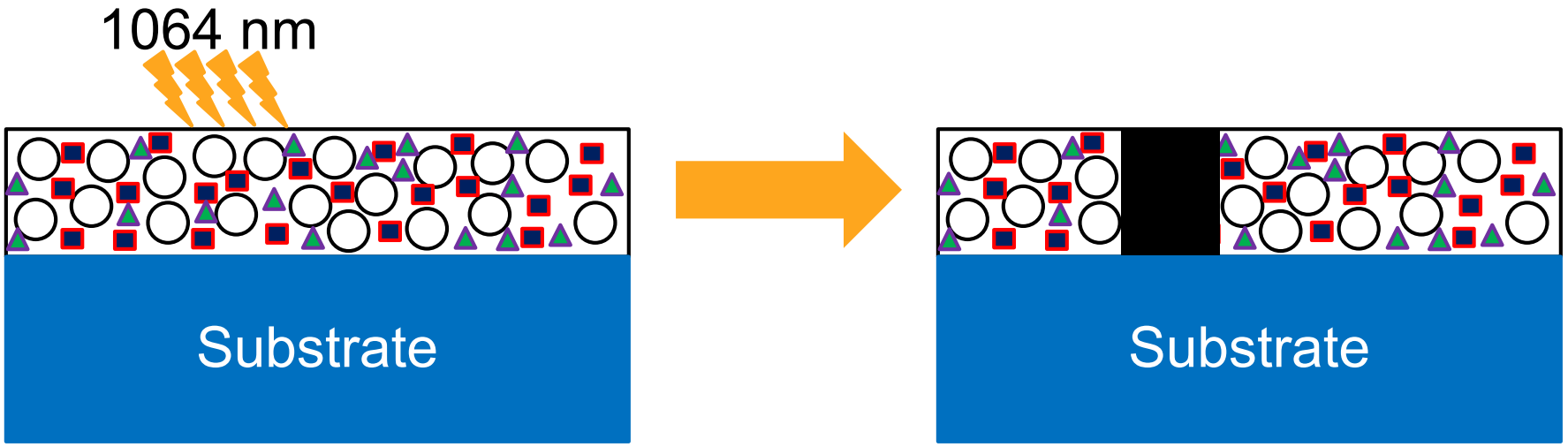
Nanoencapsulation

- Often in literature Microcapsule = Nanocapsule
  - 1 µm < Microcapsules < 100 µm
  - 1 nm < Nanocapsules < 1 µm (EU definition: nano < 100 nm)
- Why nanoencapsulation?
  - System constraints
  - Optical properties
- How?
  - Intensive emulsification process
  - High dispersing agent concentration

Laser Marking



Laser Marking: Concept



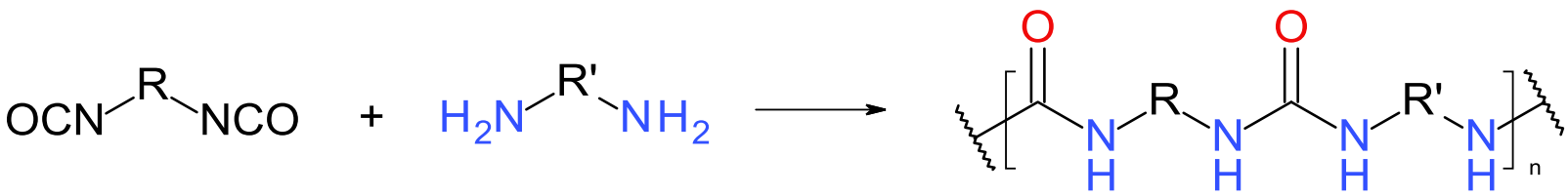
- Capsule containing a leucodye (furan dye):

- Developer: Acid (e.g. zinc 3,5-bis(α–methylbenzyl) salicylate).
- Infrared Absorber: e.g. carbon black or cyanine dye

Laser Marking: Capsules

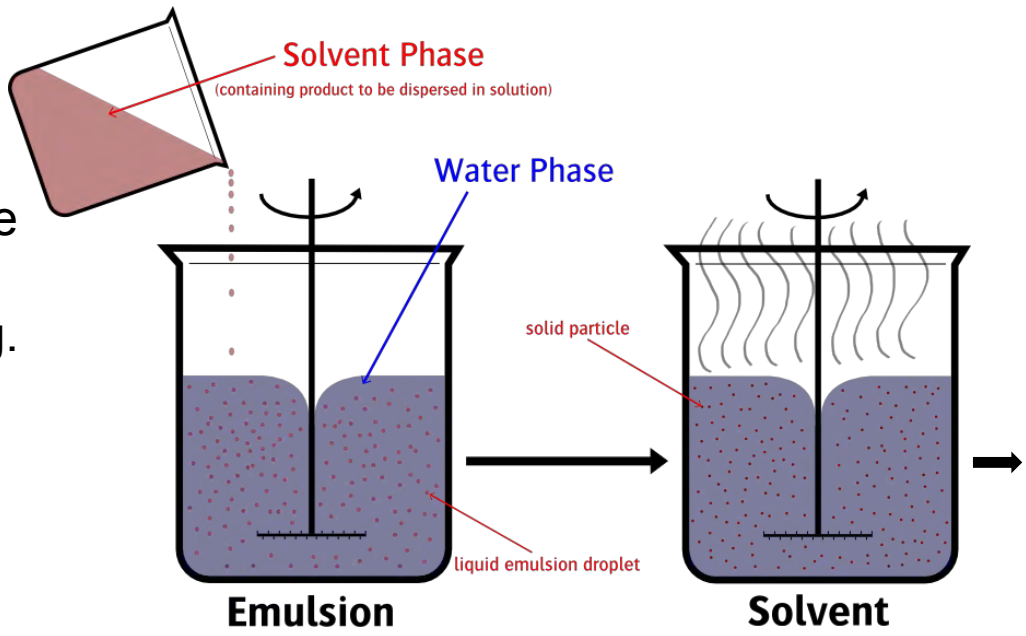
Capsule shell formed by interfacial polymerisation:

isocyanate + amine → polyurea shell (linear or cross-linked)



Laser Marking: Capsules

Organic phase:
- Leuco dye(s)
- Isocyanate(s) (e.g. Takenate D120N)
- Additives (e.g. UV absorber)
- Solvent (e.g. EtOAc)

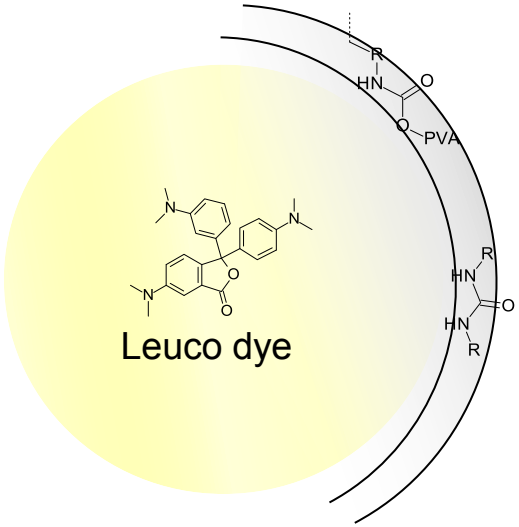


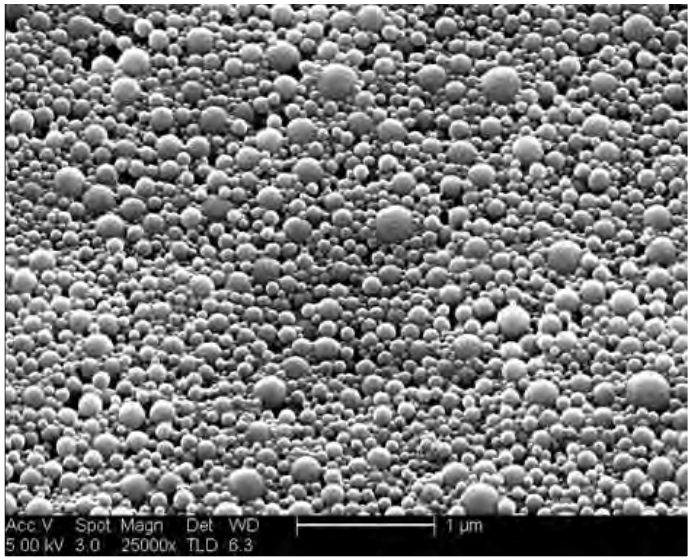
Interfacial Polymerisation

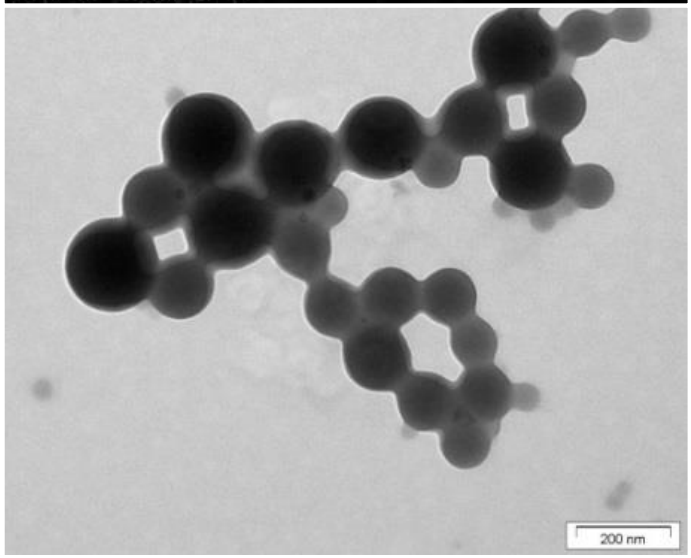
Water phase:
- Water
- Polyvinyl Alcohol

Water phase:
- Amine (e.g. pentamine)

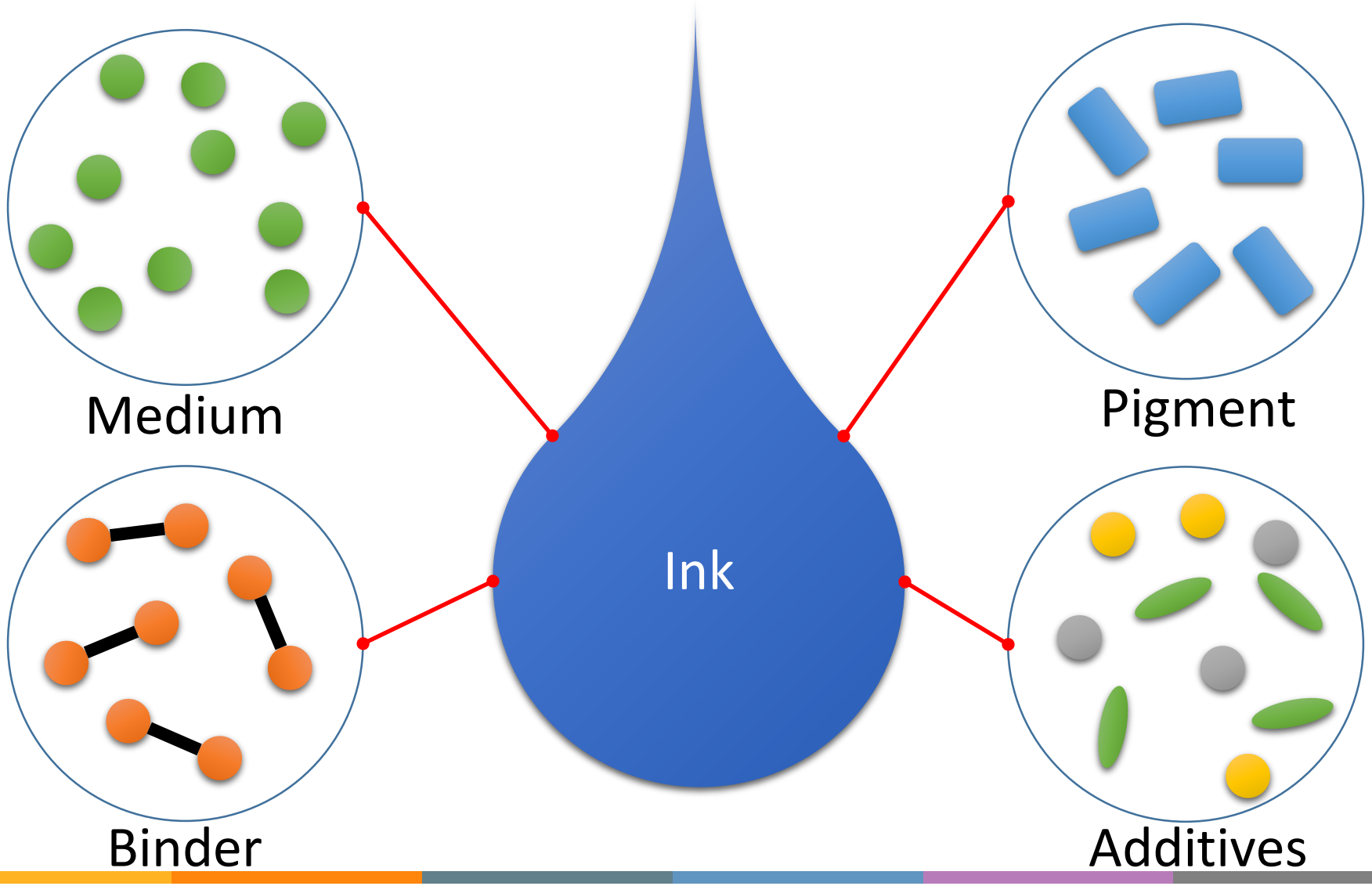
Laser Marking: Capsules





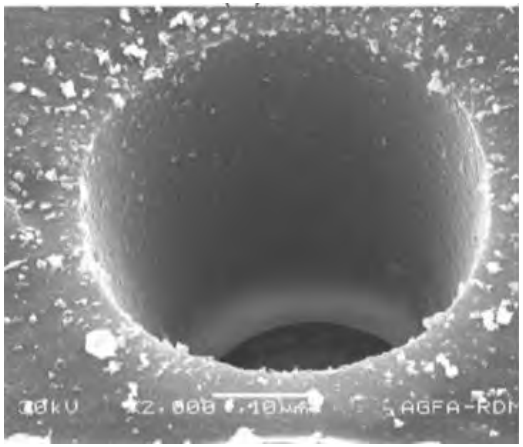


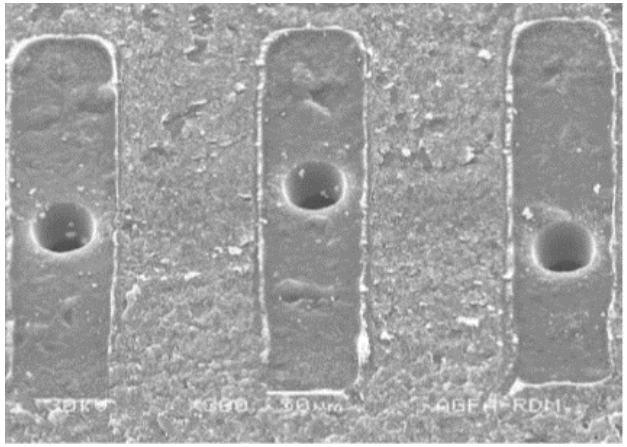
Inkjet Inks



Inkjet Inks

- Print head nozzle → 20 – 50 µm diameter
- Low viscosity → 1 – 15 mPa.s at jetting temperature
- Colloidal stability and rheology critical



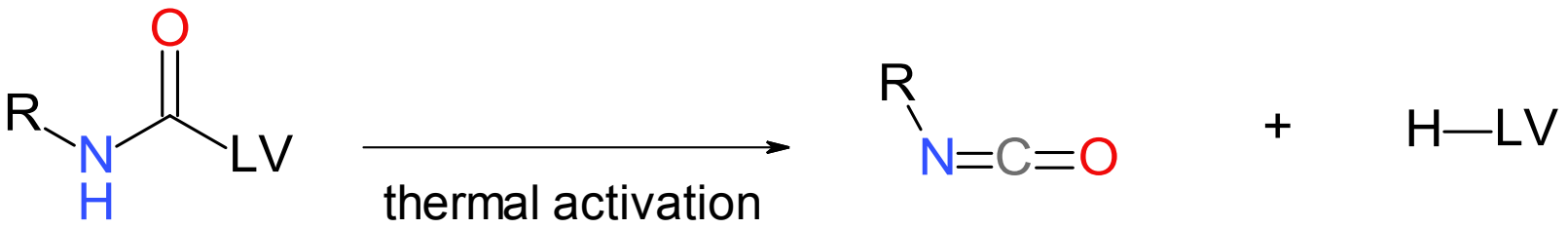


Print head nozzle

Ink Jetting

Inkjet Inks: Self-Dispersing Capsules

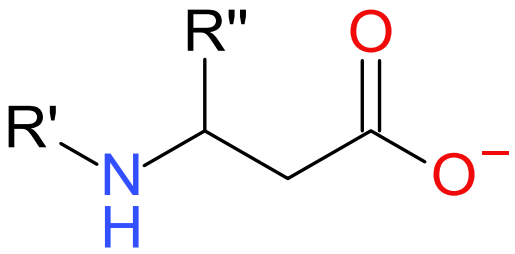
- Core: Blocked isocyanate



- Improved ink adhesion after thermal treatment (100 °C – 160 °C):
  - Capsule breaks-up
  - Reactive isocyanate generated in-situ

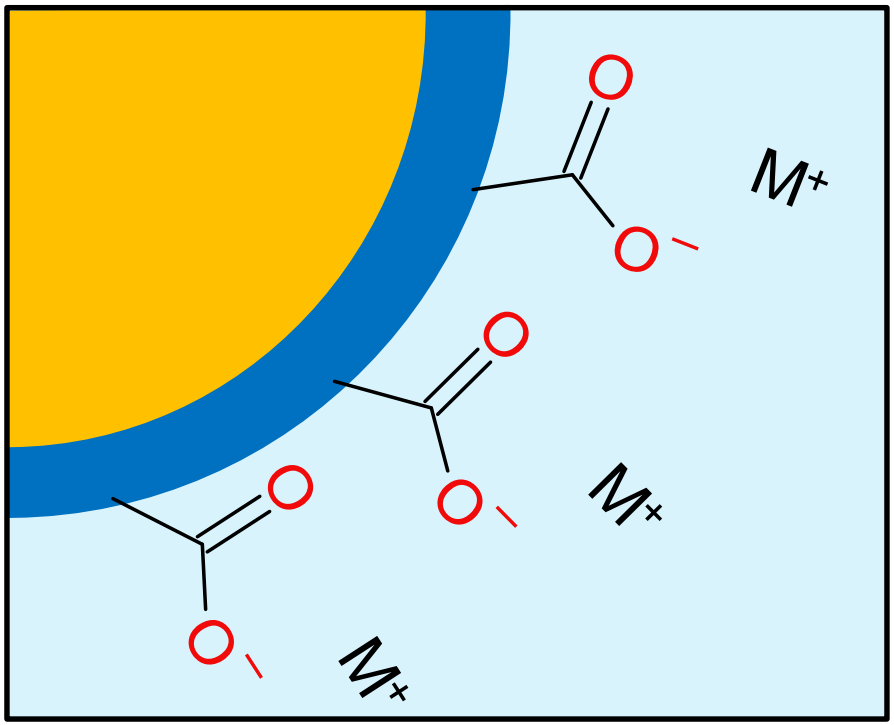
Inkjet Inks: Self-Dispersing Capsules

- Shell
  - Self-dispersing: reactive surfactant copolymerised with isocyanate



M+

  - High colloidal stability:



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Encapsulation by Interfacial Polymerisation

A versatile tool

Thanks

- Fabienne Goethals
- Johan Loccufier
- Amandine Ligot


YEARS 150 JAAR
Agfa
1867 - 2017