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
Encapsulation at Agfa-Gevaert

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

**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.

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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:—

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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.

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Encapsulation Methods

Patent Landscape

PatBase Search (1/12/17): *encapsulation and (interfac* w1 polymeri?ation)
International Patent Classification (IPC): Application Fields

- Macromolecular chemistry, polymers
- Audio-visual technology
- Pharmaceuticals
- Organic fine chemistry
- Biotechnology
- Basic materials chemistry
- Chemical engineering
- Textile and paper machines
- Analysis of biological materials
- Optics

Graphic representation of the application fields with segments corresponding to each category.
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

1064 nm

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)

\[
\text{OCN}R\text{NCO} + \text{H}_2\text{N}R'\text{NH}_2 \rightarrow \overset{\text{polyurea shell}}{\begin{array}{c}
\overset{\text{linear or cross-linked}}{\text{structure}}
\end{array}}
\]
Laser Marking: Capsules

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

**Water phase:**
- Water
- Polyvinyl Alcohol

**Interfacial Polymerisation**

- Solvent Phase (containing product to be dispersed in solution)
- Water Phase
- Emulsion
- Solvent Evaporation
- Solid particle
- Liquid emulsion droplet
Laser Marking: Capsules

Leuco dye

[Chemical structure of Leuco dye]

[Microscope images of capsules]

[Text: Agfa]
Inkjet Inks

Medium

Binder

Pigment

Additives
Inkjet Inks

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

- Core: Blocked isocyanate

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\begin{align*}
\text{R} & \text{N} & \text{O} \\
\text{H} & \text{N} & \text{C} & \text{O} \\
\text{LV} & \text{LV} & \text{R} & \text{H-LV}
\end{align*}
\]

thermal activation

- 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

\[
\text{R'} \quad \text{N} \quad \text{R''} \quad \text{O} \quad \text{O}^- \quad \text{M}^+ \n\]

- High colloidal stability:
Encapsulation by Interfacial Polymerisation

A versatile tool
Thanks

- Fabienne Goethals
- Johan Loccufier
- Amandine Ligot
YEARS 150 JAAR
1867 - 2017