High-throughput synthesis and characterization of micro-capsules

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InEn, 12 December 2014

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Outline

- Introduction
- Encapsulation technologies
- Automated synthesis of micro-capsules
- Automated characterisation of micro-capsules
- Case study
- Conclusions

Introduction

- **Flamac**

  Division of the Strategic Initiative Materials in Flanders

- **Started in 2005**

- **Mission**

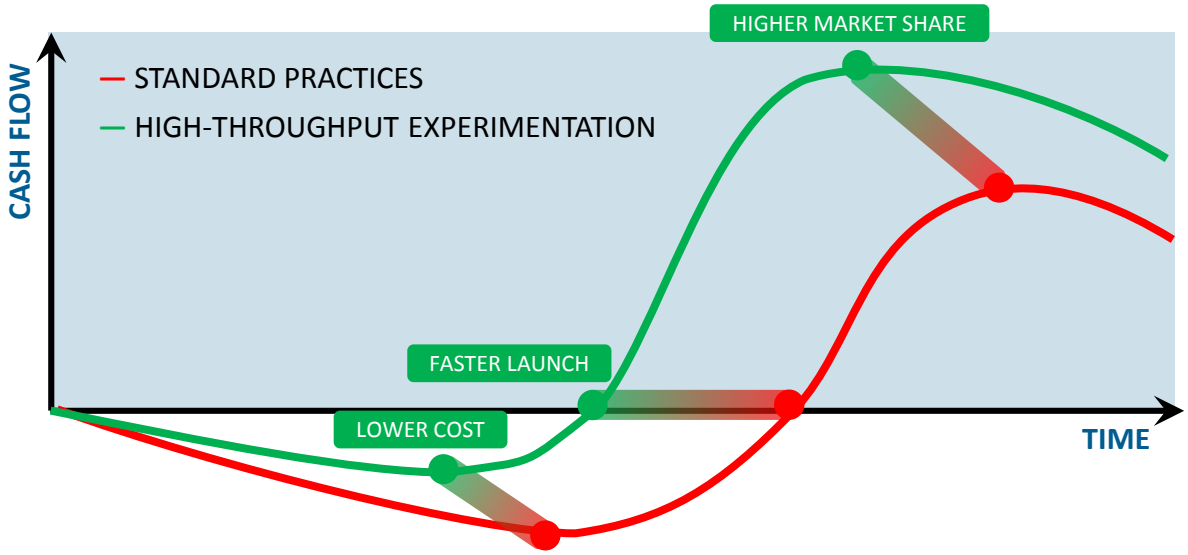
  Development and application of unique high-throughput technologies for applied materials R&D

- **Activities**

  Strategic long-term research and contract R&D

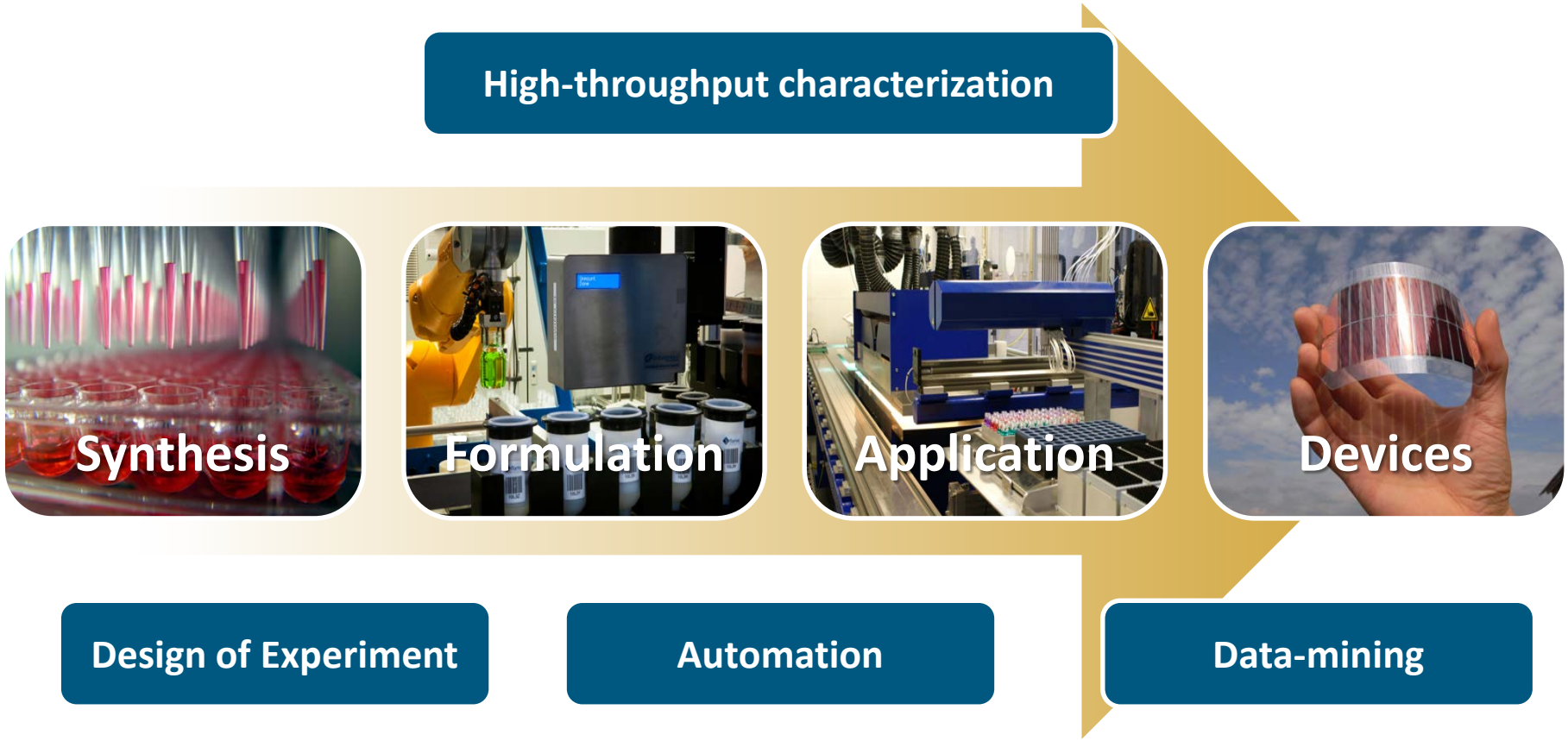
Introduction

- **Main benefits of using high-throughput technologies**
  - Stay competitive
  - Increase productivity
  - Improve reproducibility
  - Serendipity



Introduction

- **Approach**



Introduction

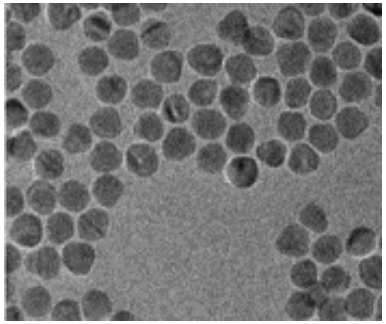
- **A wide range of applications**

Consumer products

Flow efficiency coatings

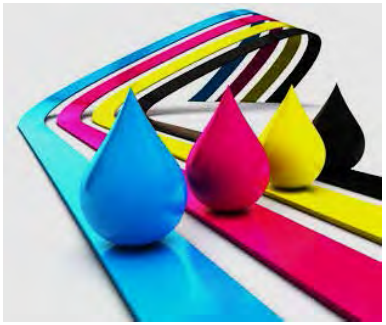
Solar materials

Corrosion protection coatings



Functional nano-particles

PU elastomers

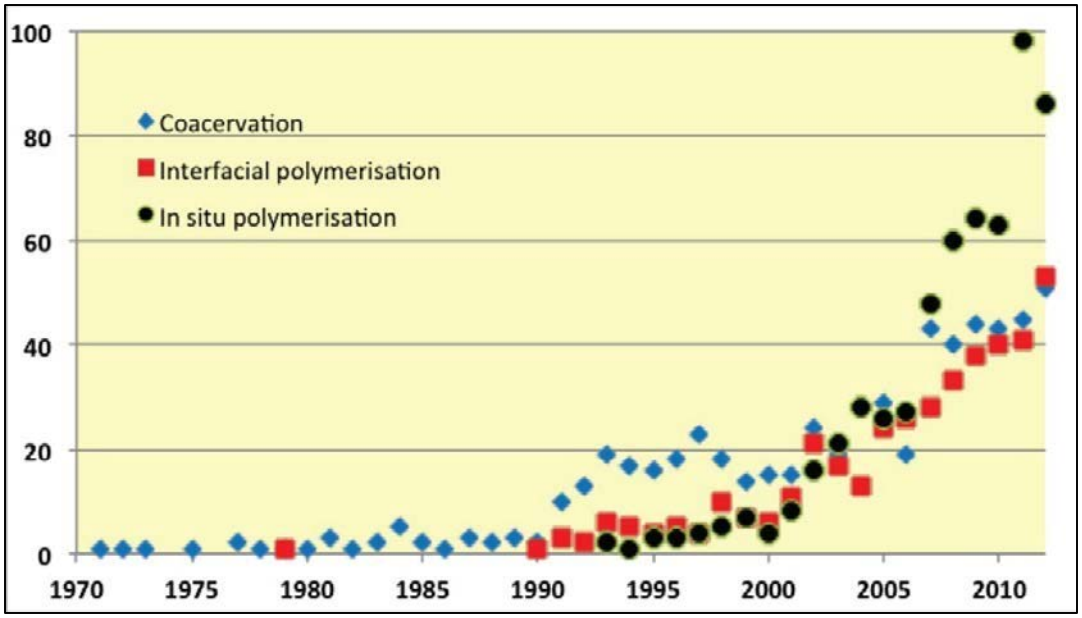


Printing inks

Encapsulation

Encapsulation technologies

- **Micro-encapsulation by chemical methods**
  - Based on polymerisation *or* polycondensation mechanisms
  - Interfacial and in situ polymerisation processes gained most scientific and industrial attention
  - Important alternatives to coacervation



Boh *et al.*, Bioencapsulation Innovations, March 2013

Encapsulation technologies

- **Requirements for micro-capsules**
  - Mechanical properties
    - Robust to survive their manufacture
    - Brittle to break when external trigger
  - Thermal stability
  - Resistance to solvents
  - Good shelf life to insert them into formulations or materials

Very often a large number of parameters need to be optimized !

Automated synthesis of micro-capsules

- **Automated synthesis platform**
  - From manual synthesis to an automated protocol

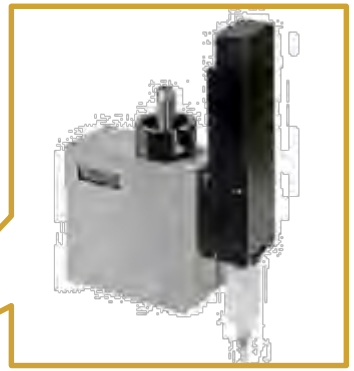
Automated synthesis of micro-capsules

- **Automated synthesis platform**

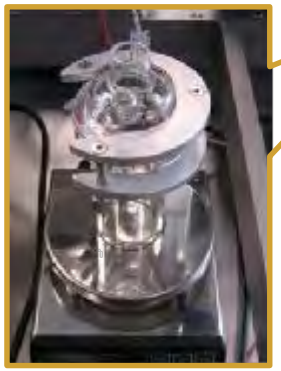
Liquid handling & pH trimming unit

Powder dosing unit

Viscous liquid dosing unit



Custom-made synthesis reactor



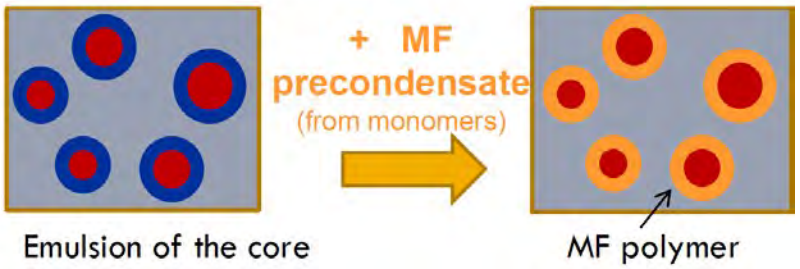
High shear mixer

Automated synthesis of micro-capsules

- **Different types of micro-capsules possible**

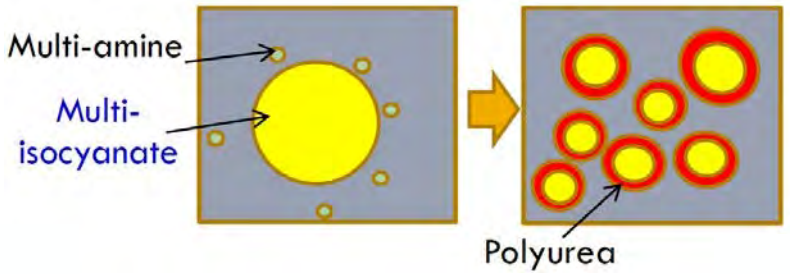
Melamine-formaldehyde shell

Polycondensation



Polyurea shell

Interfacial polymerization

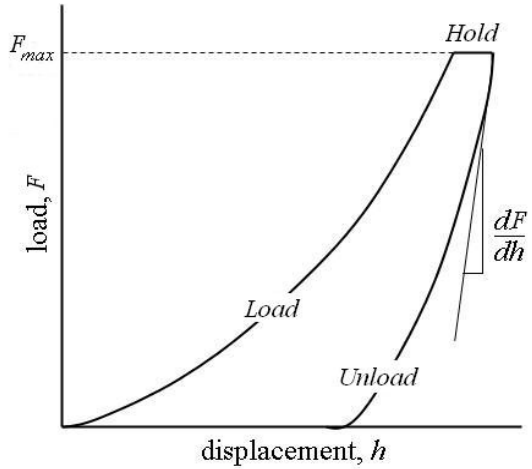


Automated characterisation of microcapsules

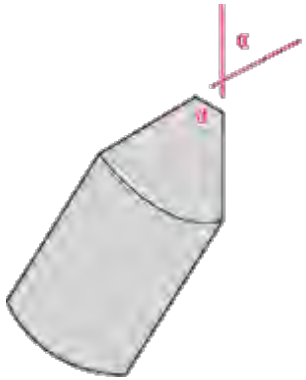
- **Overview**
  - Mechanical properties
  - Morphology
  - Particle size distribution
  - Leaching of active ingredients
  - …

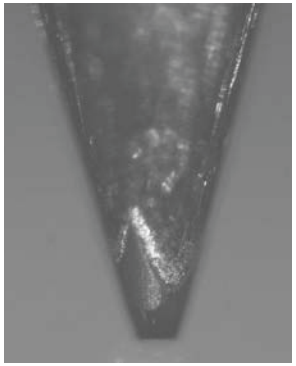
Automated characterisation of microcapsules

- **Mechanical properties**
  - Using nano-indentation technique
  - Visco-elastic properties
    - Stiffness
    - Elasticity
    - Hardness
  - Flat punch tip (100 µm width)



Load displacement curve





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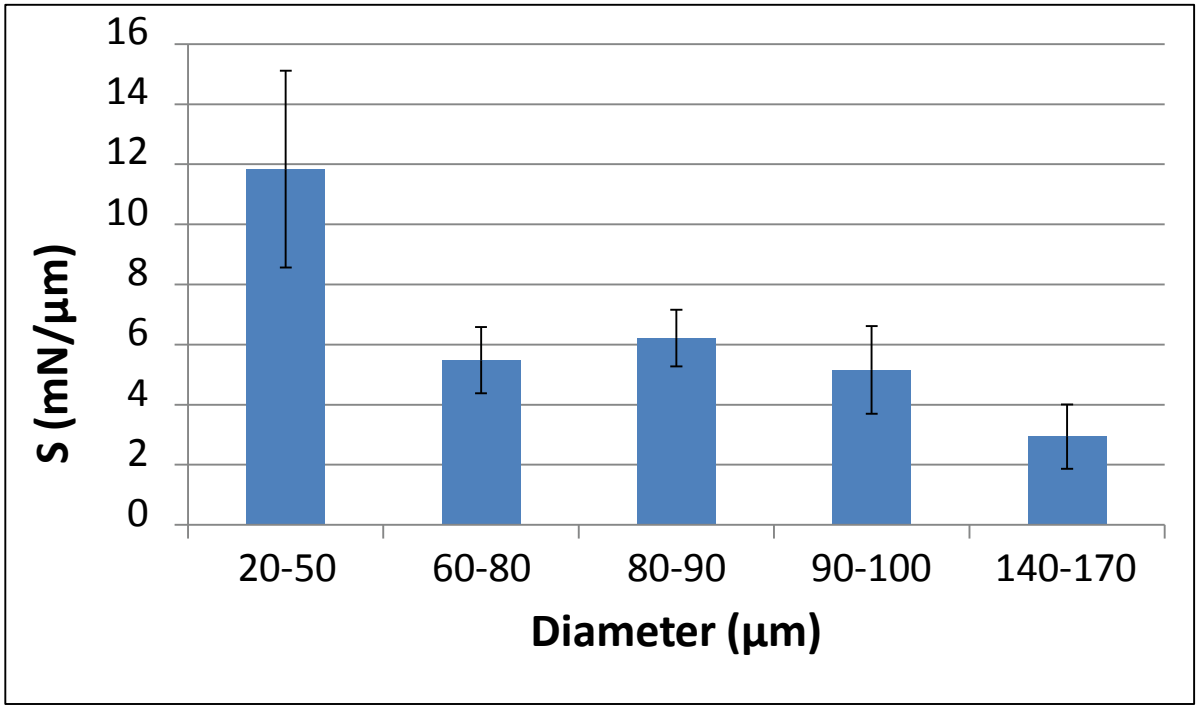
Automated characterisation of microcapsules

- **Mechanical properties**
  - Melamine formaldehyde micro-capsules

    ⇒ effect of size on capsule stiffness



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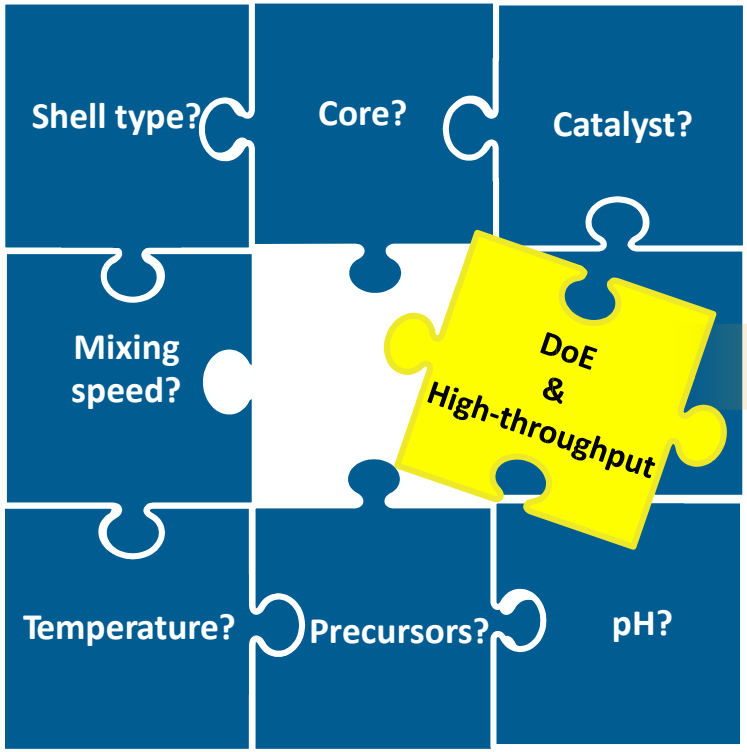
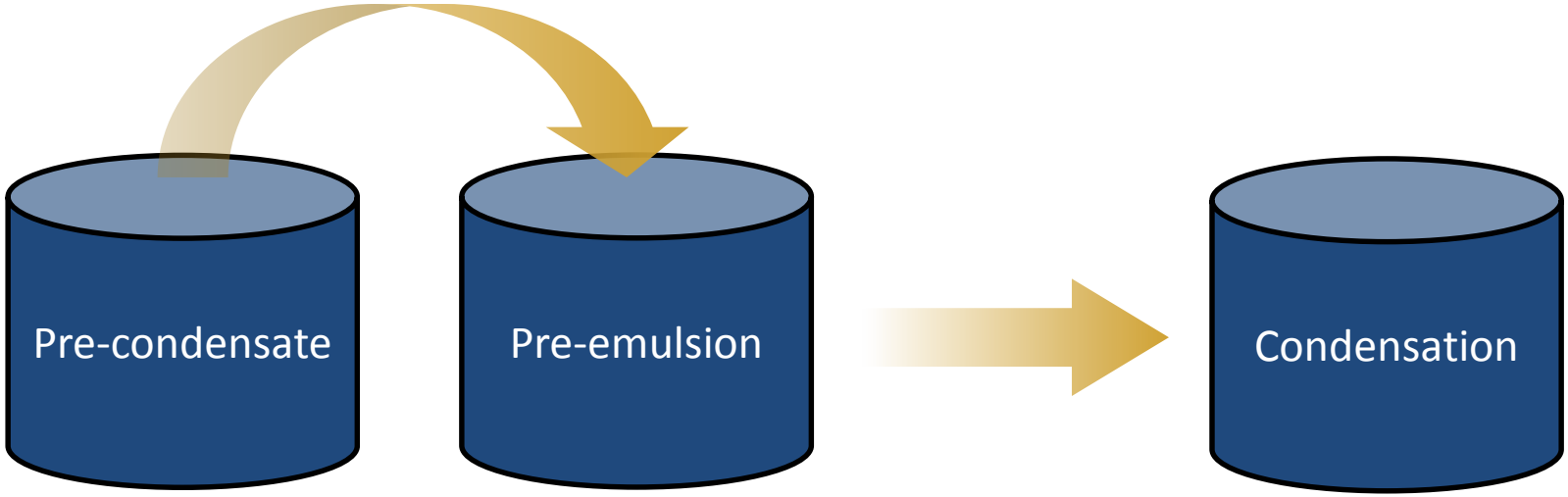
Case study

- **Melamine formaldehyde micro-capsules**
  - Encapsulation of cyclohexane
  - Multi-parameter space to be optimized



Case study

- **Melamine formaldehyde micro-capsules**
  - Recipe



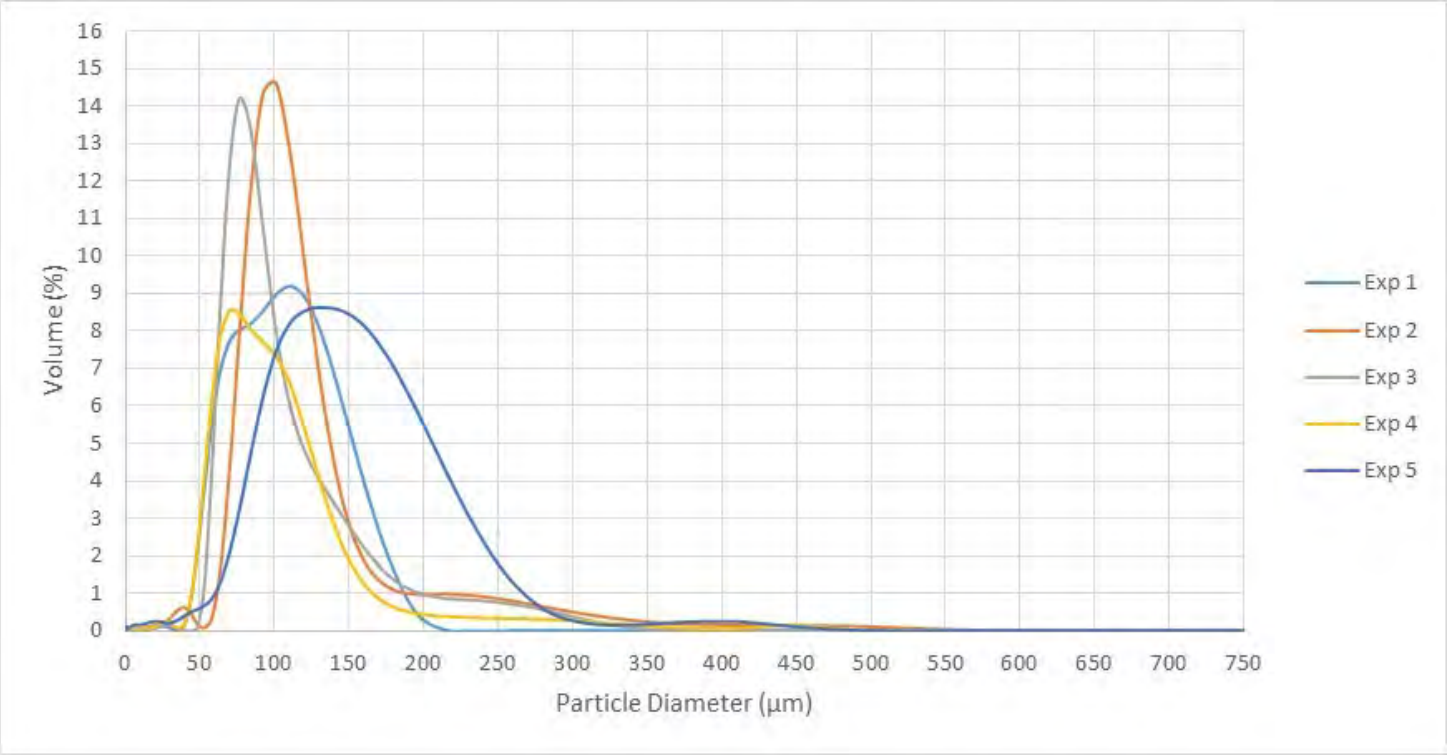
Reactor A
- pH adjustment
- Heating profile
- Reaction

Reactor B
- High shear mixing
- pH adjustment

Reactor B
- pH adjustment during pre-condendate solution to pre-emulsion
- Heating profile
- Condensation reaction

Case study

- **Melamine formaldehyde micro-capsules**
  - Impact of process parameters on size distribution



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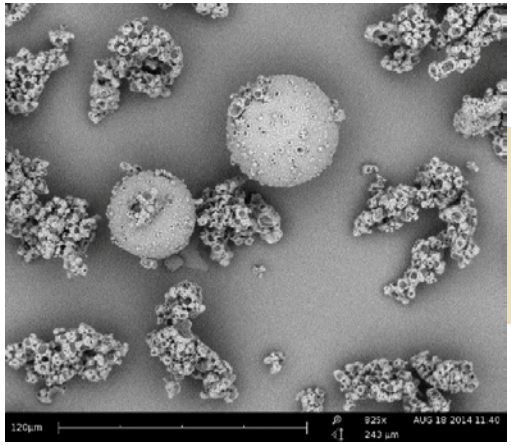
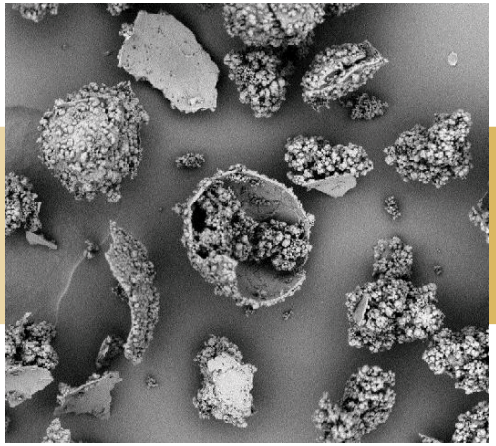
Case study

- **Melamine formaldehyde micro-capsules**
  - Impact of recipe on morphology



PSMA + PVA 5%
Ratio shell/core = 1



PSMA 5%
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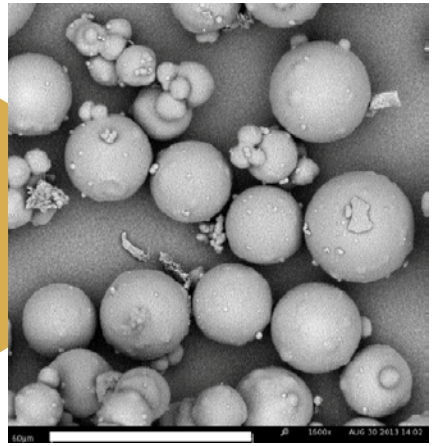
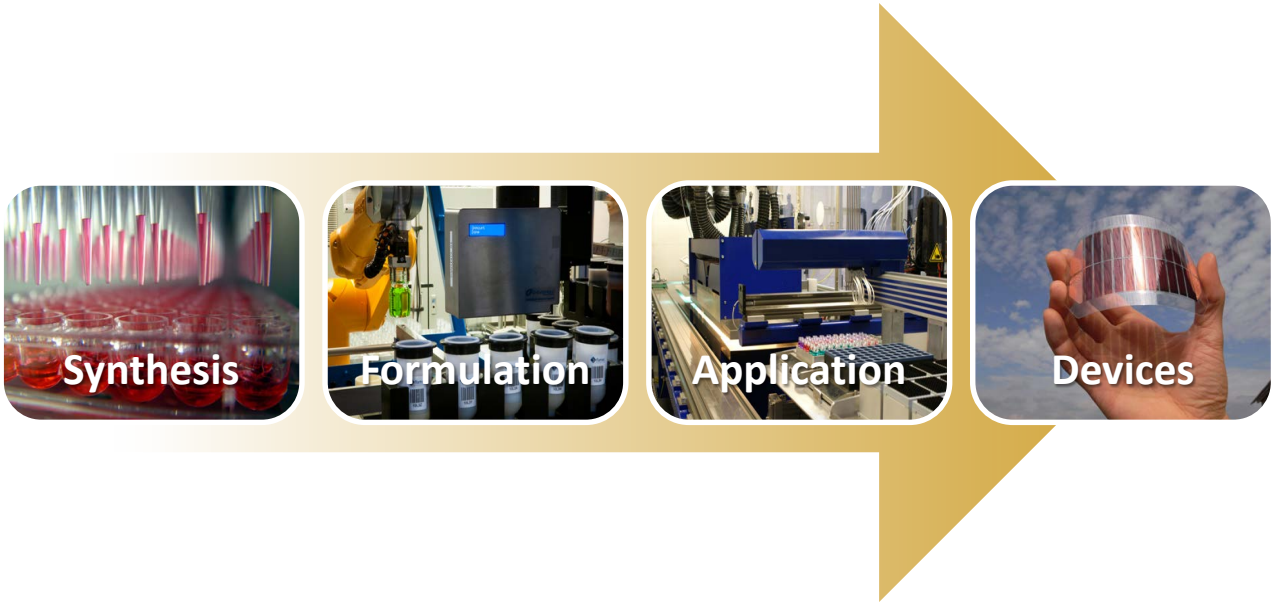


PSMA 2,5%
Ratio shell/core = 1

(*) PSMA: poly(styrene-maleic anhydride)
PVA: polyvinyl alcohol

Conclusions

- **A unique platform for automated capsule synthesis and characterization, allowing**
  - Composition screening
  - Process conditions screening
- **Speeding-up the complete material development chain**



Acknowledgements

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  - Erwin Bauters
  - Pieter Castelein
  - Geoffrey Coppens
  - Tom Parasote
- **UGent**
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  - Dr. Seda Cakir

See also poster of Seda Cakir
“Microcapsules for Self-Healing” !

Thank you for your attention