High-throughput synthesis and characterization of micro-capsules
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

High-throughput characterization

Design of Experiment
Formulation
Application
Devices

Automation

Data-mining
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
- High shear mixer
- Custom-made synthesis reactor

InEn, 12 December 2014
Automated synthesis of micro-capsules

- Different types of micro-capsules possible

**Melamine-formaldehyde shell**

**Polyurea shell**

Polycondensation

Interfacial polymerization
Automated characterisation of microcapsules

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

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Automated characterisation of microcapsules

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

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

⇒ effect of size on capsule stiffness

![Bar chart showing the effect of diameter on capsule stiffness](chart.png)
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

**Condensation**
- pH adjustment during pre-condensate solution to pre-emulsion
- Heating profile
- Condensation reaction
Case study

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

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

PSMA + PVA 5%
Ratio shell/core = 1

PSMA 5%
Ratio shell/core = 1

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
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See also poster of Seda Cakir
“Microcapsules for Self-Healing”!
Thank you for your attention