

High-throughput synthesis and characterization of micro-capsules



Speeding up
the development
of materials

J. Paul

InEn, 12 December 2014

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**

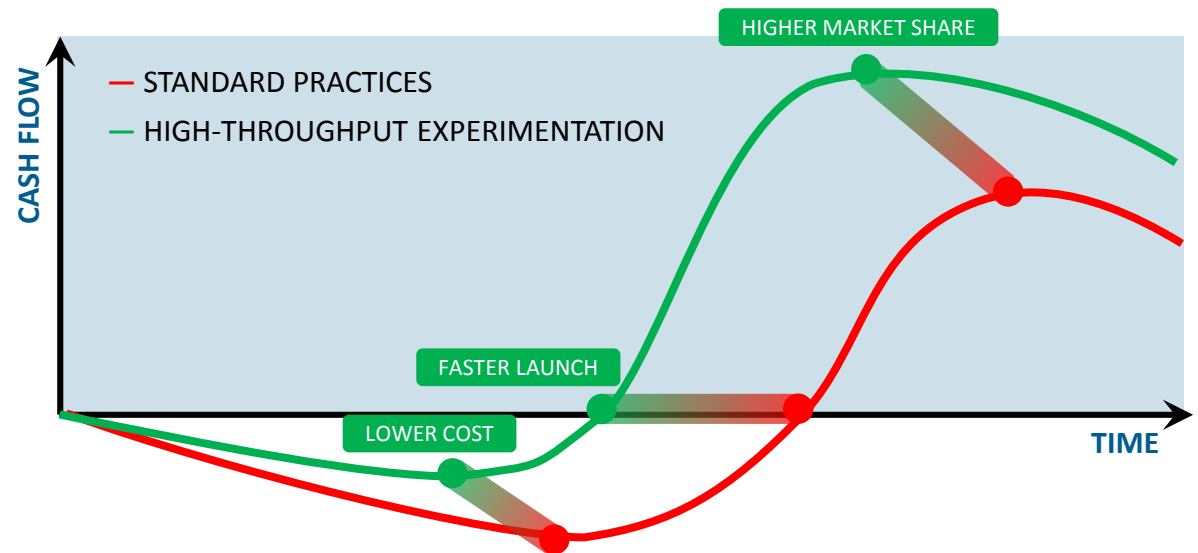
Development and application of unique high-throughput technologies for applied materials R&D

- **Activities**

Strategic long-term research and contract R&D

Introduction

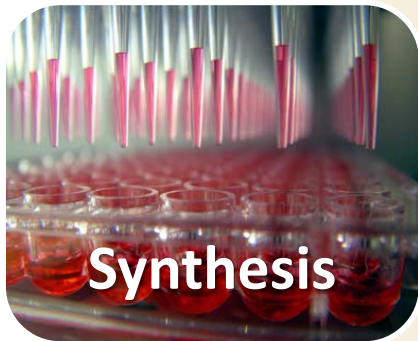
- **Main benefits of using high-throughput technologies**
 - Stay competitive
 - Increase productivity
 - Improve reproducibility
 - Serendipity



Introduction

- Approach

High-throughput characterization



Design of Experiment

Automation

Data-mining

Introduction

- A wide range of applications



Consumer products



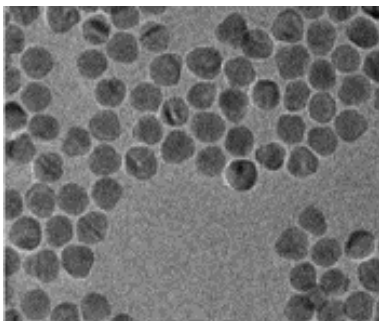
Flow efficiency coatings



Solar materials



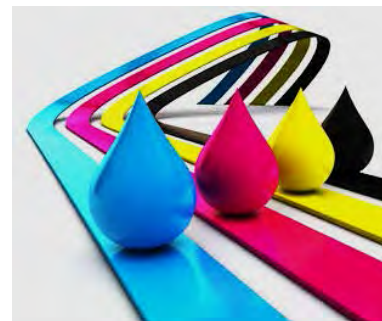
Corrosion protection coatings



Functional nano-particles



PU elastomers



Printing inks

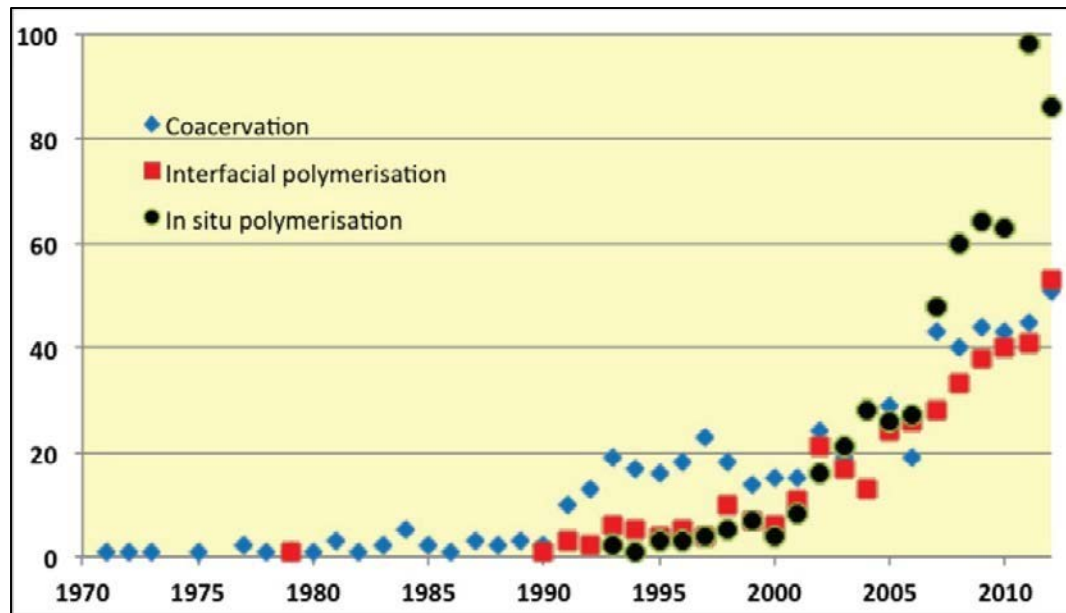


Encapsulation

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



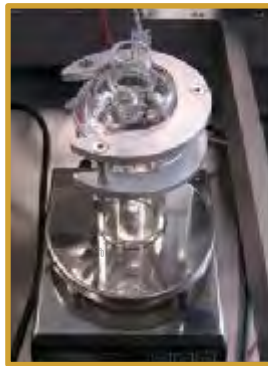
Powder dosing unit



Viscous liquid dosing unit



Custom-made synthesis reactor



High shear mixer



Automated synthesis of micro-capsules

- Different types of micro-capsules possible

Melamine-
formaldehyde shell

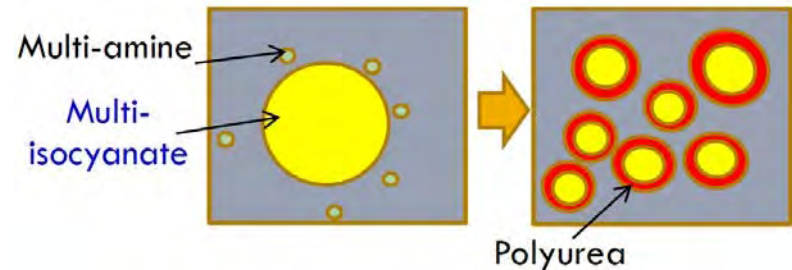
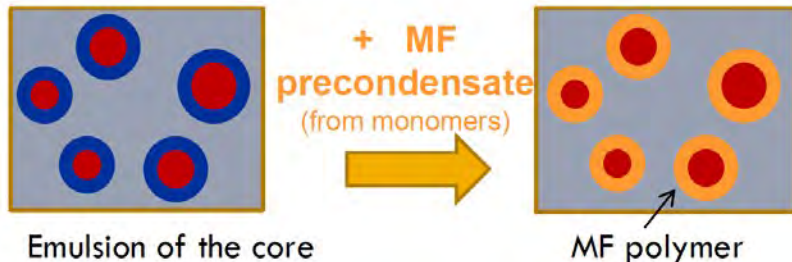


Polycondensation

Polyurea shell



Interfacial polymerization



Automated characterisation of microcapsules

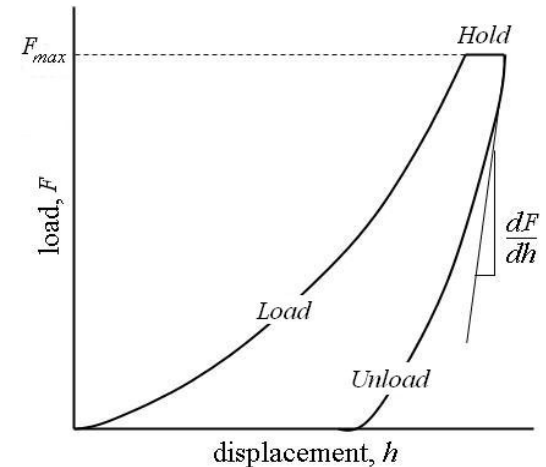
- **Overview**

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

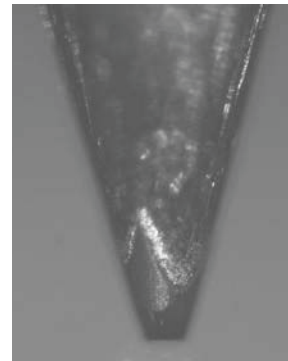
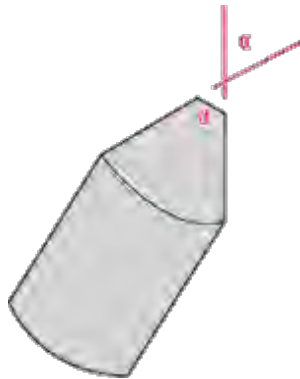
Automated characterisation of microcapsules

- **Mechanical properties**

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



Load displacement curve

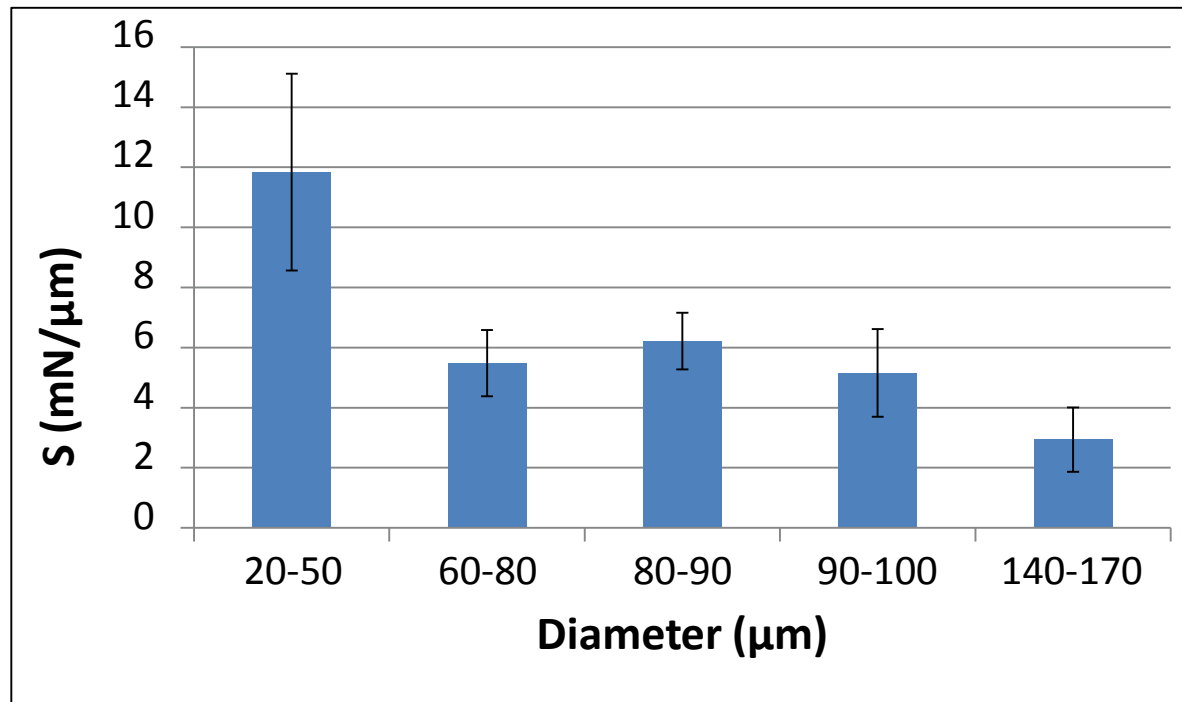


Automated characterisation of microcapsules

- **Mechanical properties**

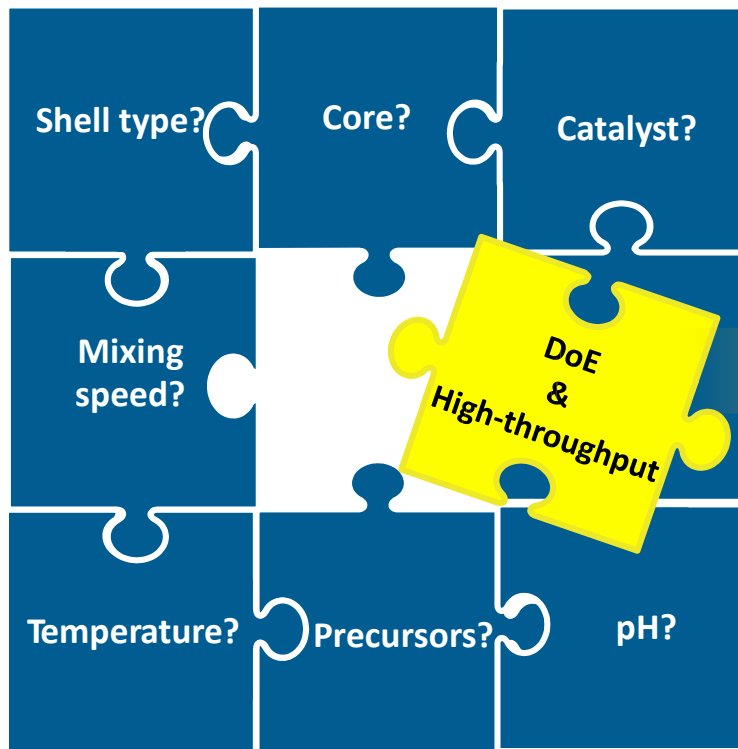
- Melamine formaldehyde micro-capsules

- ⇒ effect of size on capsule stiffness



Case study

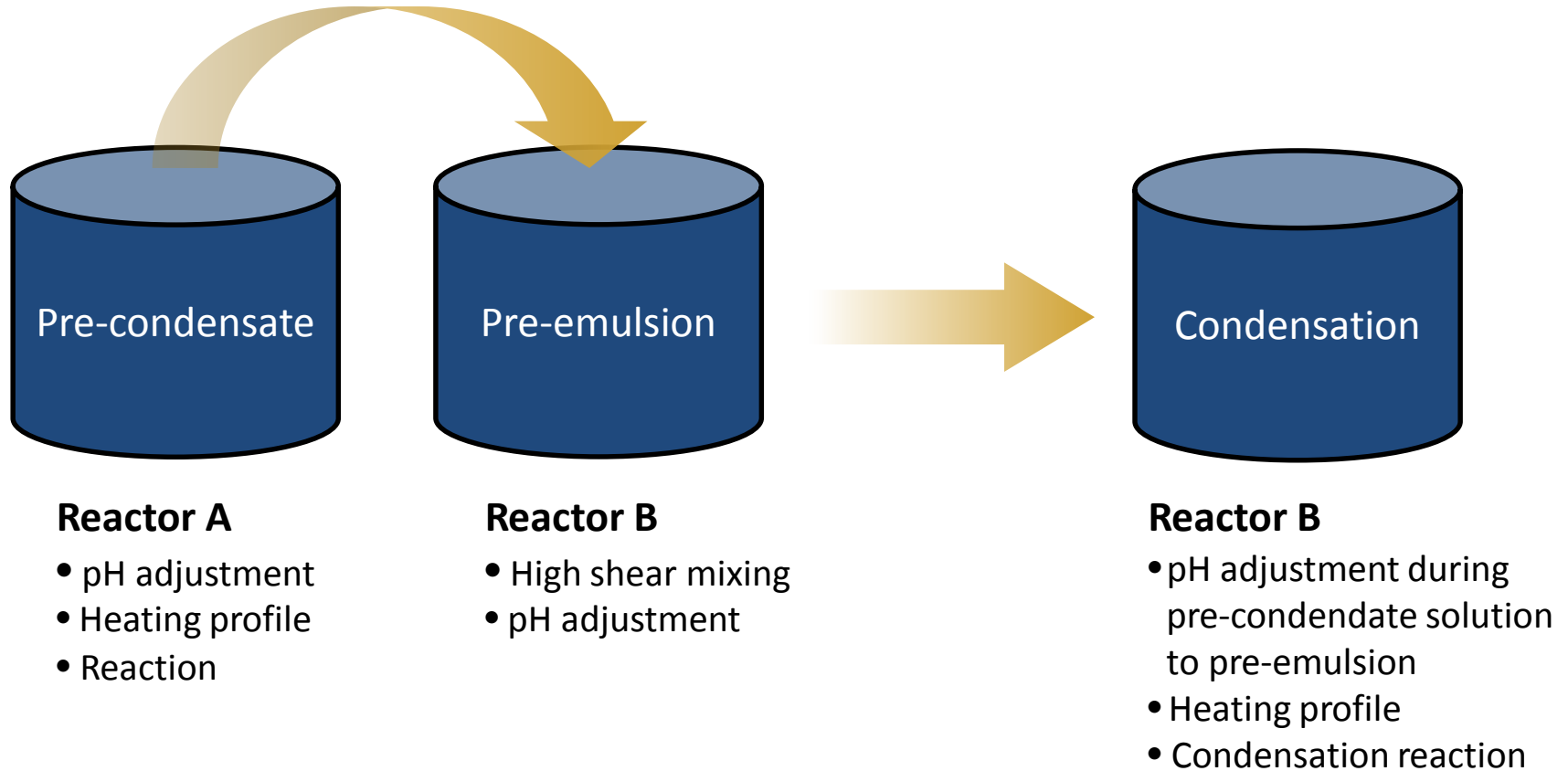
- **Melamine formaldehyde micro-capsules**
 - Encapsulation of cyclohexane
 - Multi-parameter space to be optimized



Case study

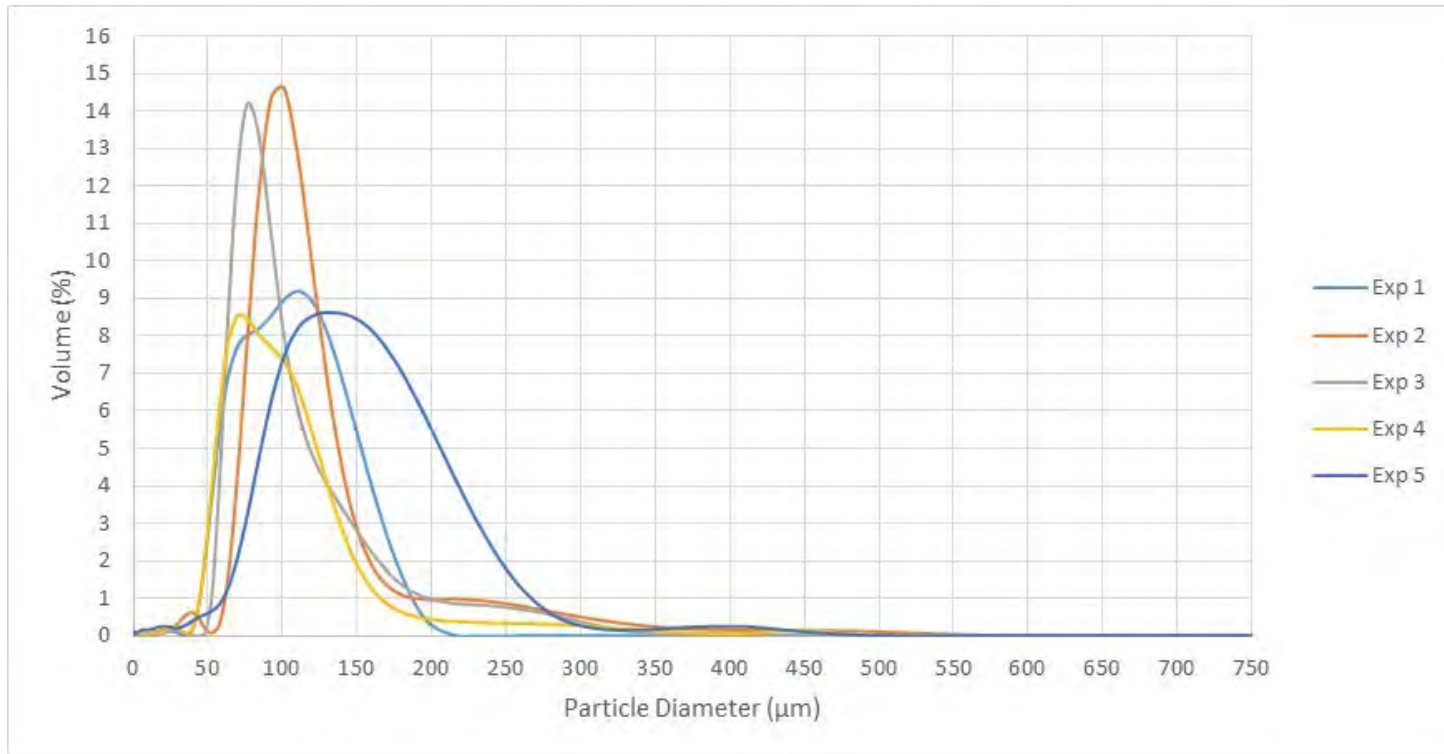
- **Melamine formaldehyde micro-capsules**

- Recipe



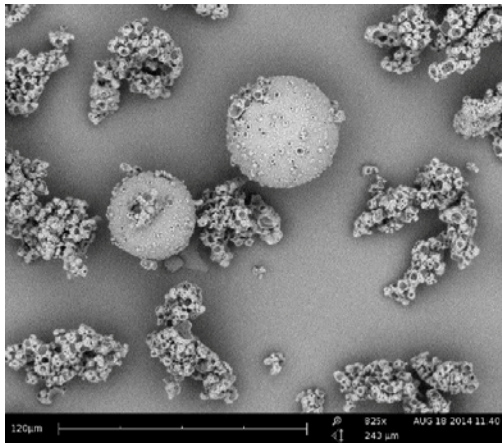
Case study

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

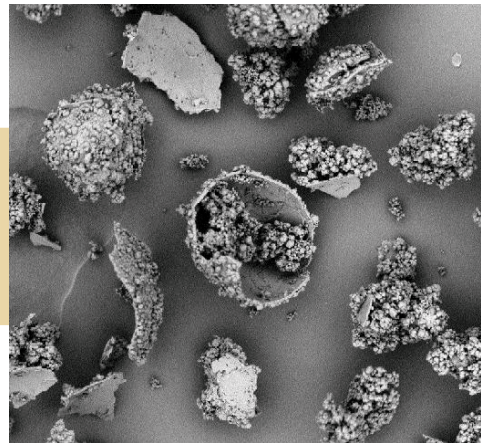


Case study

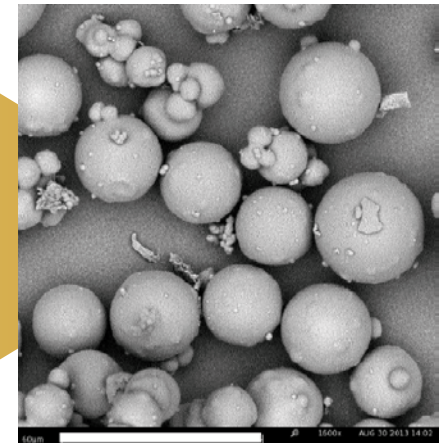
- **Melamine formaldehyde micro-capsules**
 - Impact of recipe on morphology



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



Acknowledgements

- **Flamac**

- Erwin Bauters
- Pieter Castelein
- Geoffrey Coppens
- Tom Parasote



- **UGent**

- Prof. Filip Du Prez
- Dr. Guadalupe Rivero
- Dr. Seda Cakir


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



Thank you for your attention



International conference - Ghent, 2-4 June, 2015



High-throughput Materials Development: Bridging the Gap between Lab and Market

Home	Home
Scientific & Organising Committees	Dear colleagues,
Call for Abstracts	Flamac and DECHEMA have great pleasure in inviting you to participate in the sixth International Conference on Combinatorial Materials Research , to be held in Ghent on 2, 3 and 4 June 2015. The conference will focus on several topics, including materials for energy applications, recycling, novel formulated products, computational materials design and advanced characterisation methodologies.
Programme	High-throughput and combinatorial methods offer an efficient tool to vary chemical composition and material processing. Consequently, there is a great deal of interest within the industry in using high-throughput experimentation (HTE) in materials R&D. In addition to oral presentations , there will be poster presentations and one-to-one meetings . Sponsors will get the opportunity to demonstrate their products and services with attendees, hold informal meetings or just give more exposure to their brand via an exhibition stand.
Venue	
Hotels	
Registration	
Sponsoring	
One-to-one networking	
Social event	
Past conferences	

Key dates

Call for abstracts opens:
30 June 2014

Deadline for abstracts:
27 February 2015

Early bird registrations
until 31 March 2015

Deadline for registration:
25 May 2015

This year's conference will be a very special edition as Flamac will celebrate its **10th anniversary**. I hope that we will have the opportunity to meet you in **Ghent**, a city that is rich in architecture and culture - and much more besides.
I look forward to welcoming you personally in **June 2015**.

Dr Johan Paul