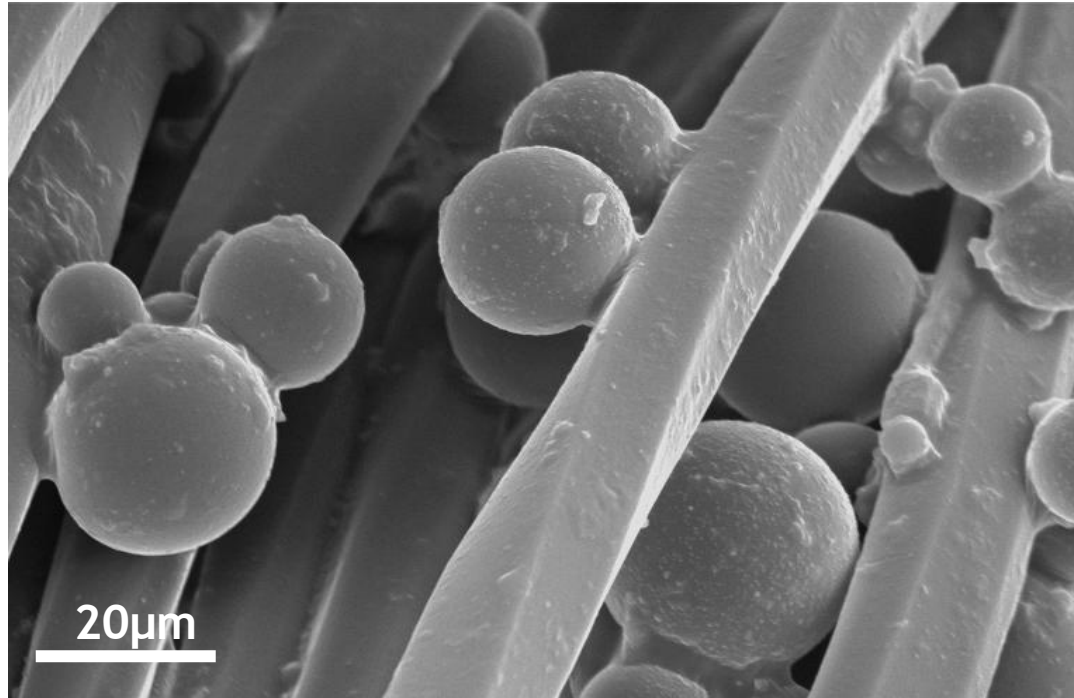




# Development of microcapsules as additives for advanced composites



**Royal Society of Chemistry Symposium 2015:  
Right on Target: Sector Specific Formulation**

**24 June 2015, Cologne, Germany**

**Isabel Martins, Roberto Teixeira & Maxime Durka**



# Outline

## □ Devan Chemicals

- Core business
- Reactive Microencapsulation Platform
- Reactive Microencapsulation Technology

## □ Ongoing Projects

- PUU Microcapsules
  - Functional Textiles using Microencapsulation
  - Microcapsules in Self-Healing
  - Encapsulation of Reactive Components in Coatings
- Other Types of Microcapsules



# Core business

Bringing textiles **to life** 

By creating innovative  
properties and functionality

Taking into consideration  
sustainability



# Inspiration for...



## Home textiles

- Bedding
- Upholstery
- Carpet
- Towels



## Apparel

- Underwear
- Socks
- Shirts
- Sportswear



## Technical textiles

- Medical
- Outdoor
- Filtration
- Workwear



# Reactive Microencapsulation Platform



Allergen Control by using reactive capsules containing probiotic endospores on textiles



Thermoregulation Control by using reactive capsules containing phase change materials on textiles



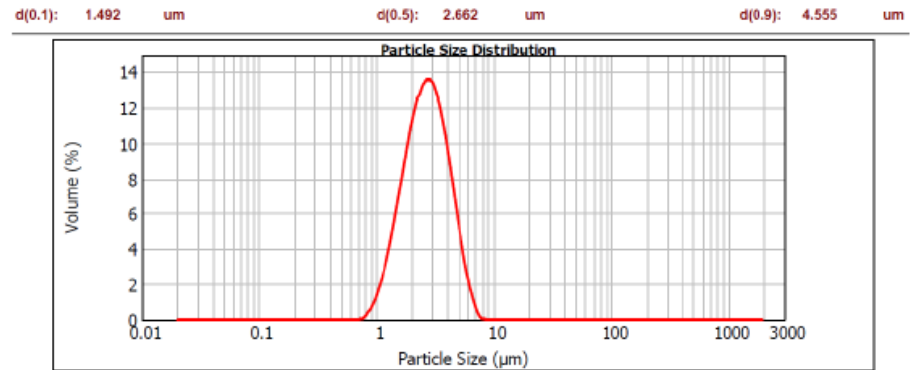
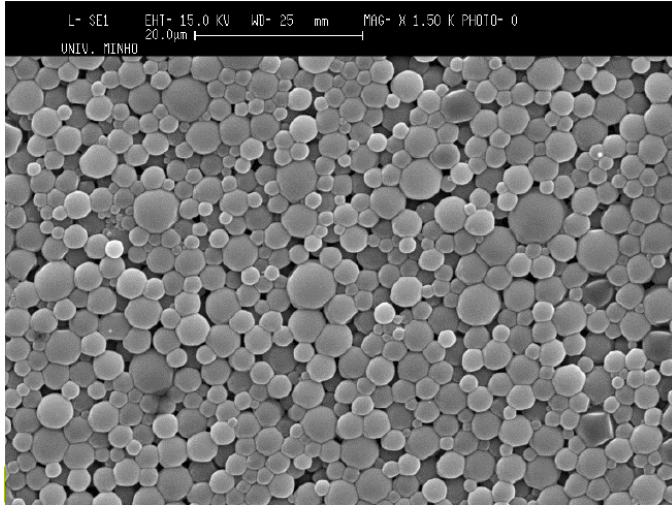
Insect Control by using reactive capsules containing natural & friendly repellents on textiles



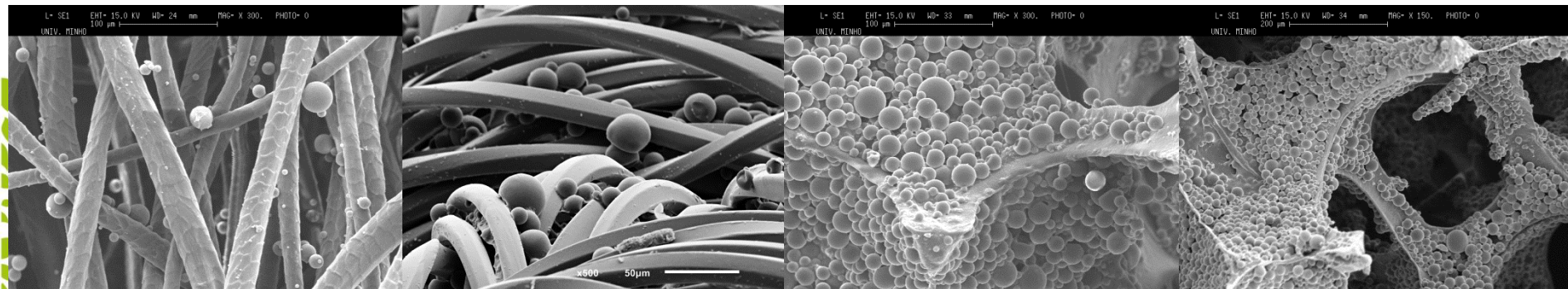
Sensorial Management Control by using reactive capsules containing fragrances and body/skin care extracts on textiles



# Microcapsules @ Devan



Small Size (d(0.9): 4.55μm) and monodisperse microcapsules



# Microcapsules @ Devan

## Reactive Microcapsule Technology

### Patented (WO/2006/117702)

- Controlled affinity and covalent reaction with fibres through available functional groups (shells to fibres).
- Well-established chemical bond that guarantees optimum adhesion and thus promoting higher resistance towards washing and abrasion.
- Efficient linkage compared with traditional binding routes.
- Enhanced microcapsules' release mechanism from treated substrates.

**Work on the adhesion and wash durability.**

5  $\mu$ m





# Ongoing Projects...

## ***Polyurethanes:***

- are one of the most versatile materials in the world today.
- represent an important class of **thermoplastics** and **thermoset** polymers.

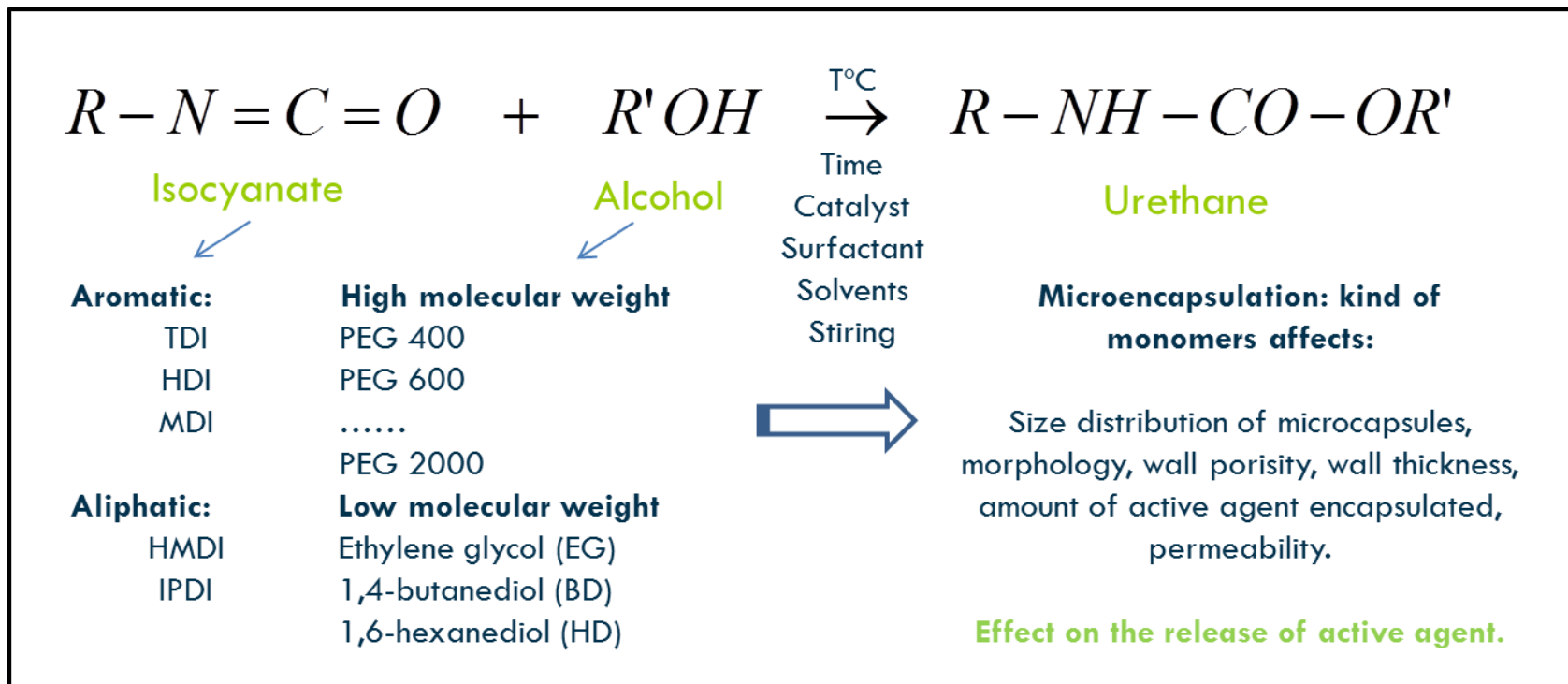
**Examples of current applications in which polyurethane dispersions are used.**



Source: <http://www.specialchem4adhesives.com/tc/polyurethane-dispersions/index.aspx?id=applications>

# Ongoing Projects...

## Polyurethanes



Variables involved in the production process of PU microcapsules.

Volatility: HDI > TDI > MDI

# PUU Microcapsules



## Functional Textiles using Microencapsulation

**Goal:** scented men's suit

**Specifications:** 5 dry cleaning cycles and 9000 abrasion cycles.



**Chosen chemical system and process:**

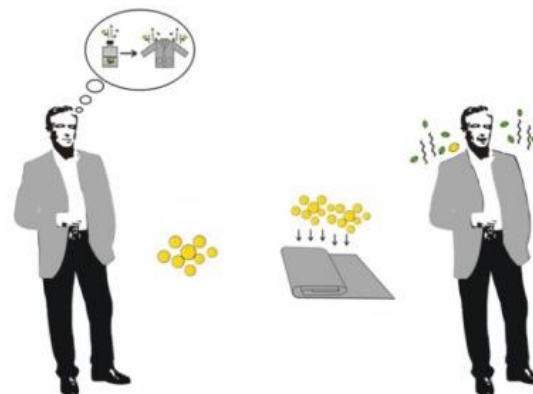
Polyurethane-urea (PUU)

Interfacial polymerization

**Active principle:**

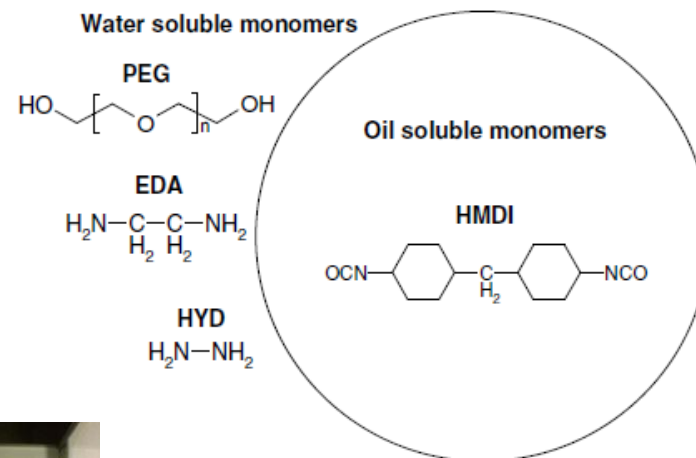
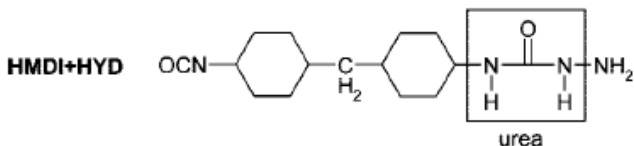
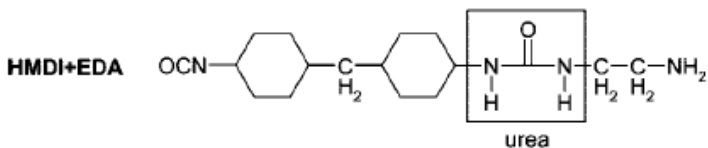
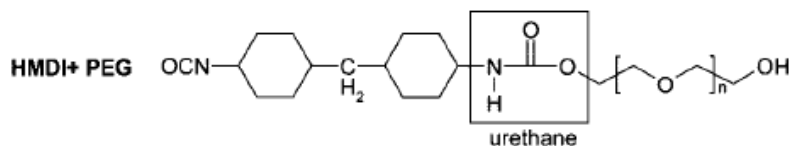
Model compounds (limonene)

Perfume (complex mixture)



Rodrigues, S.N., Fernandes, I., Martins, I.M., Mata, V.G., Barreiro, F., and Rodrigues, A.E., *Microencapsulation of limonene for textile application*. Industrial & Engineering Chemistry Research, 2008. 47(12): p. 4142-4147.

## Functional Textiles using Microencapsulation



**Reaction scheme for polyurethane-urea microcapsules production.**

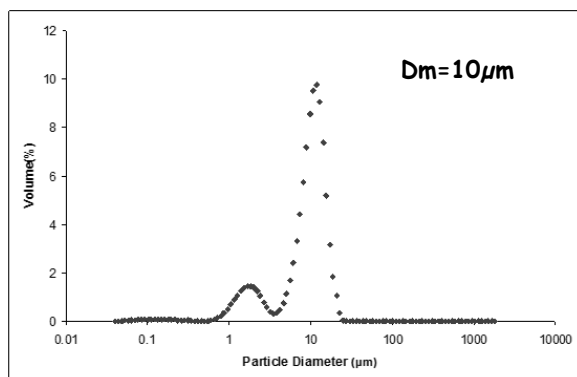


Rodrigues, S.N., Fernandes, I., Martins, I.M., Mata, V.G., Barreiro, F., and Rodrigues, A. E., *Microencapsulation of limonene for textile application*. Industrial & Engineering Chemistry Research, 2008. 47(12): p. 4142-4147.

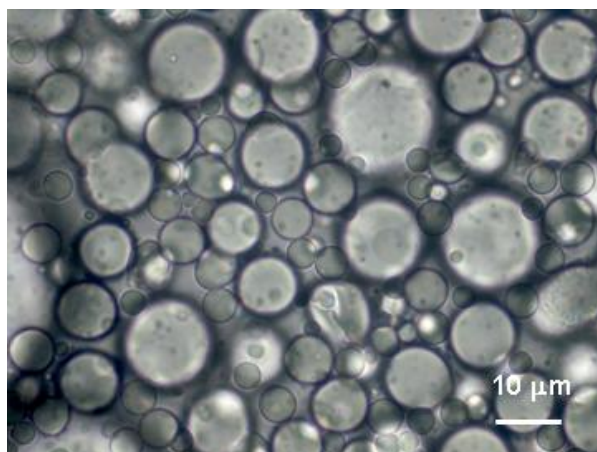
# PUU Microcapsules

## Functional Textiles using Microencapsulation

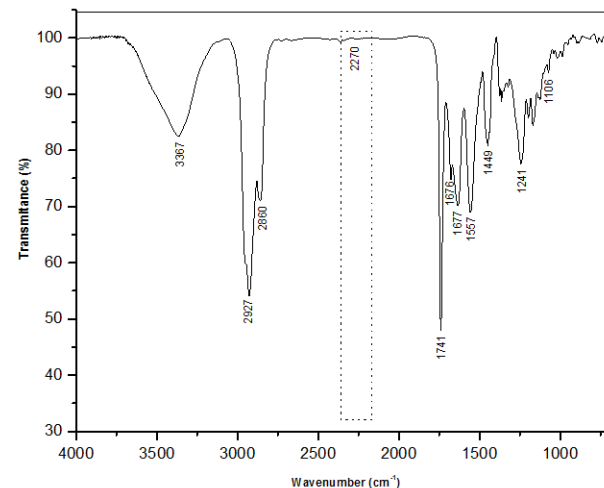
### Particle size distribution



### Morphology

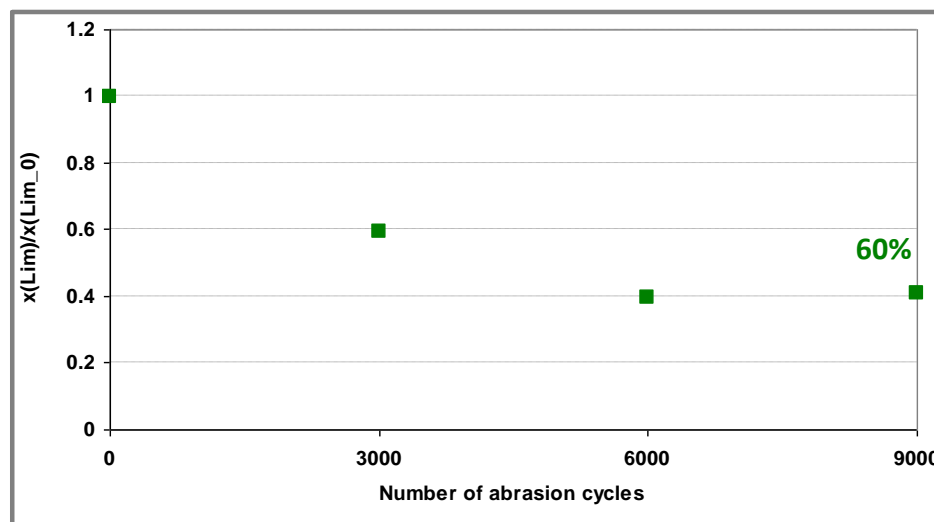
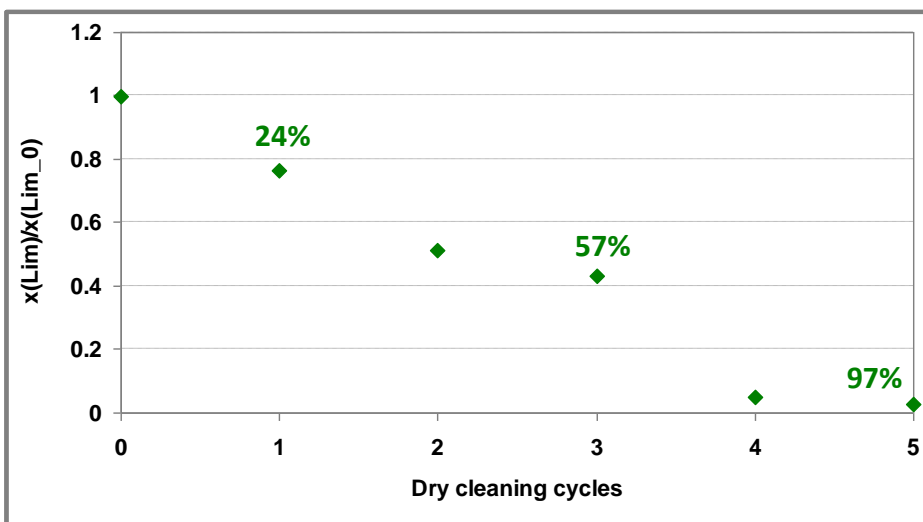


### Isocyanate group: $2270\text{ cm}^{-1}$



Rodrigues, S.N., Fernandes, I., Martins, I.M., Mata, V.G., Barreiro, F., and Rodrigues, A.E., *Microencapsulation of limonene for textile application*. Industrial & Engineering Chemistry Research, 2008. 47(12): p. 4142-4147.

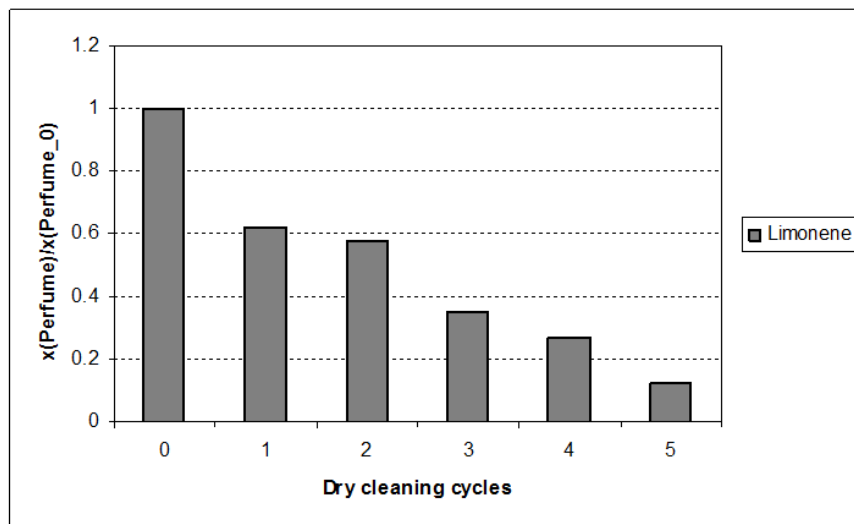
## Functional Textiles using Microencapsulation



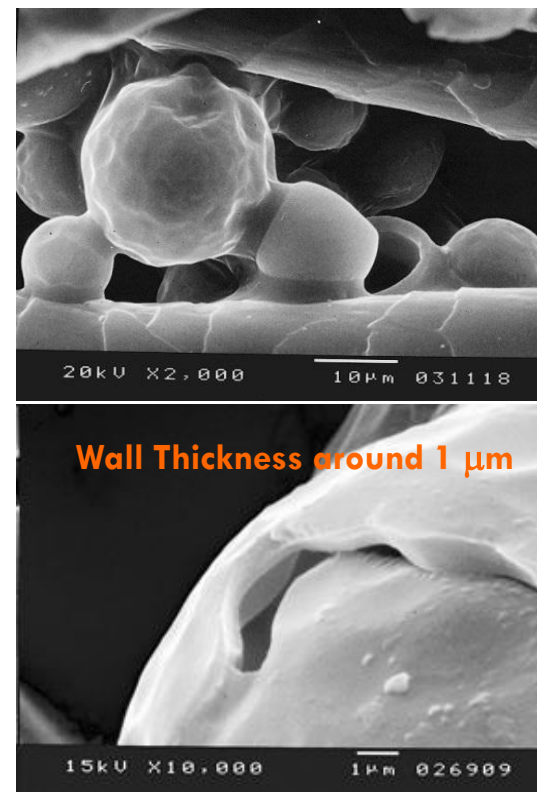
**Quantity of limonene present in the headspace according to the number of dry cleaning cycles and abrasion cycles.**

Rodrigues, S.N., Fernandes, I., Martins, I.M., Mata, V.G., Barreiro, F., and Rodrigues, A.E., *Microencapsulation of limonene for textile application*. Industrial & Engineering Chemistry Research, 2008. 47(12): p. 4142-4147.

## Functional Textiles using Microencapsulation



Quantity of limonene present in the headspace according to the number of dry cleaning cycles.



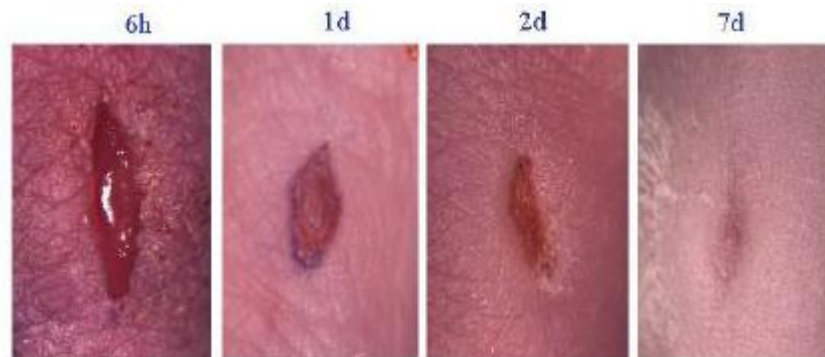
Rodrigues, S.N., Martins, I.M., Fernandes, I.P., Gomes, P.B., Mata, V.G., Barreiro, M.F., and Rodrigues, A.E., Scentfashion®: Microencapsulated perfumes for textile application. Chemical Engineering Journal, 2009. 149(1-3): p. 463-472.

# PU Microcapsules – Self Healing

## Self-Healing Concept

*‘Self-healing can be defined as the ability of a material to heal (recover/repair) damages automatically and autonomously, that is, without any external intervention.’\**

**Common goal: mimicking biological systems**



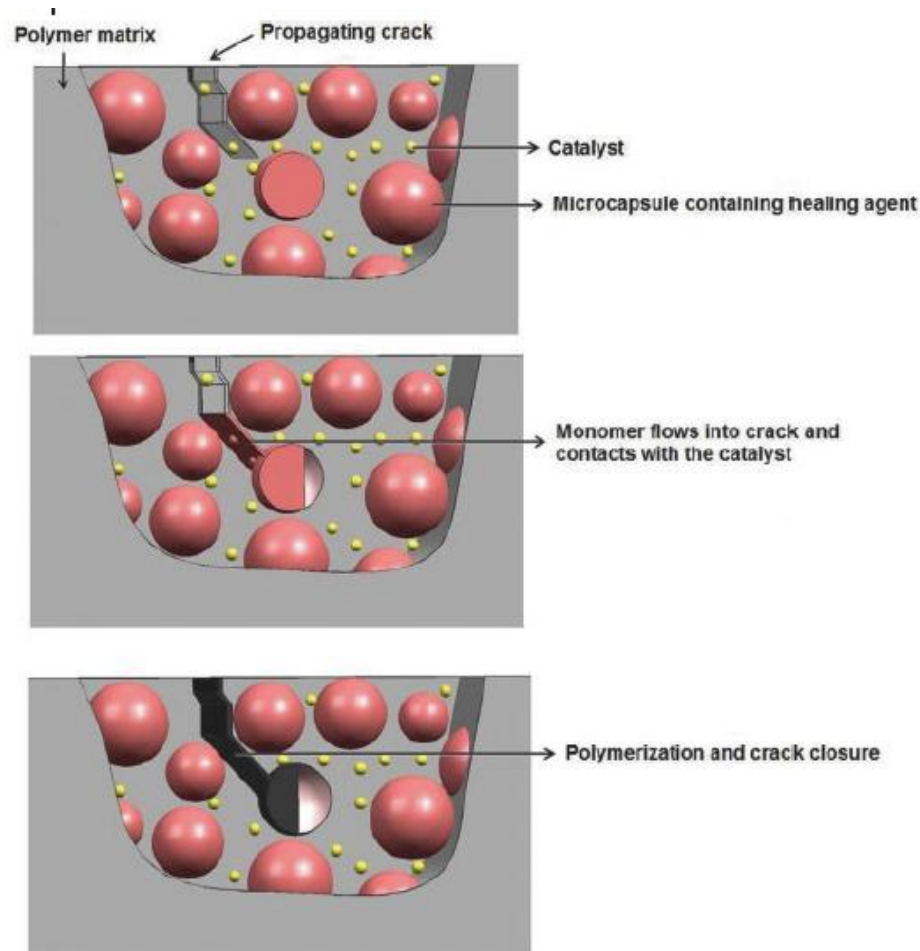
\***Source:** Gosh, S.K.(2008); Self-healing Materials: Fundamentals, Design Strategies and Applications; Wiley-VCH.





# PU Microcapsules

## Microcapsules in Self-Healing



### Self-healing concept:

Autonomous: no external stimulus needed

Thermoset materials with microcapsules containing healing agents

Different matrix materials: epoxy, polyurethanes, unsaturated polyesters

Schematic representation of self-healing concept using embedded microcapsules\*

\*Source: Gosh, S.K.(2008); Self-healing Materials: Fundamentals, Design Strategies and Applications; Wiley-VCH.

# PUREPAIR - Polyurethane foams and elastomers with autonomic repair functions



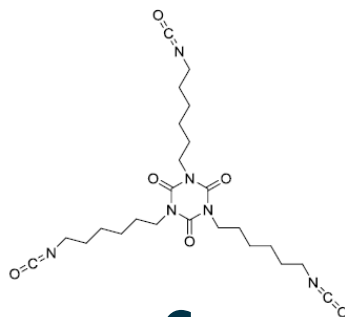
## Microcapsules in Self-Healing

### Polyurea shell

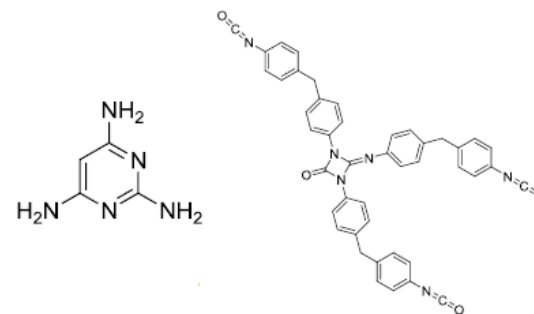
Isocyanate component

Amine component

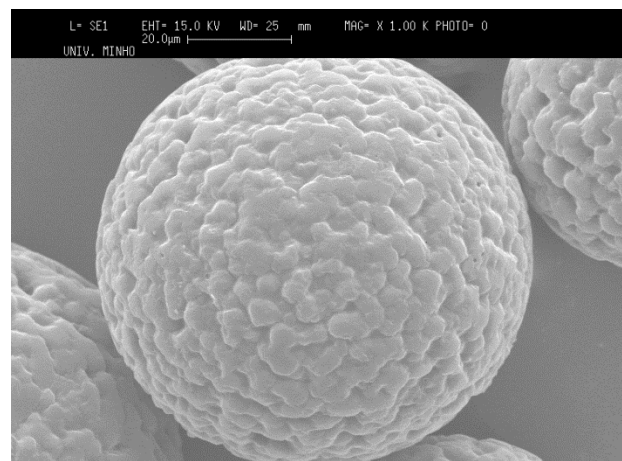
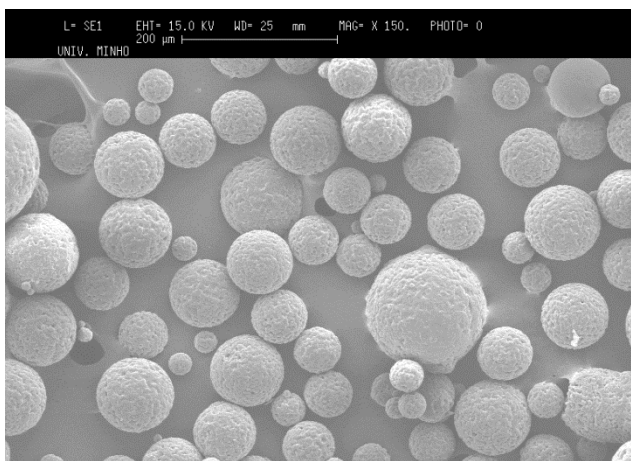
#### ■ SEM images



Core



Shell



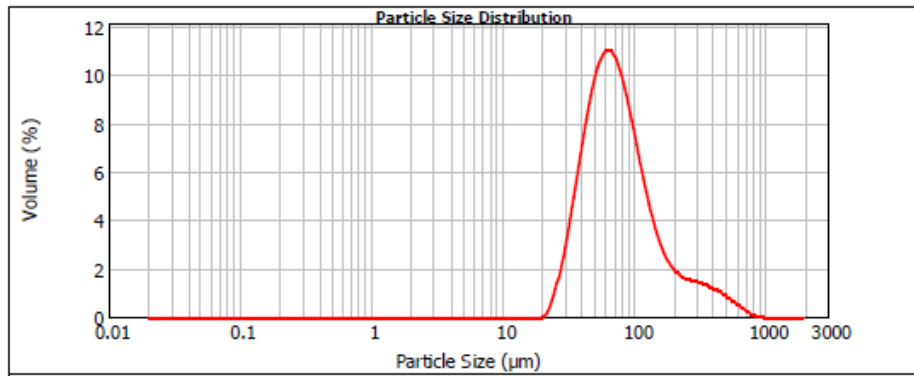
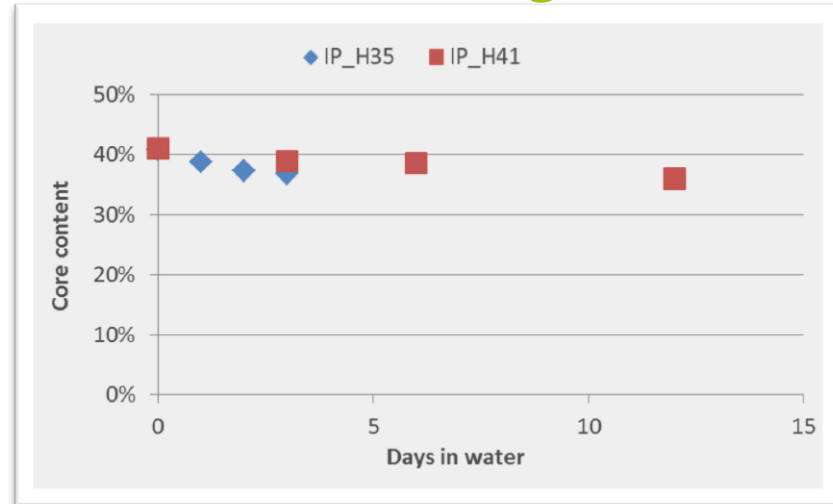
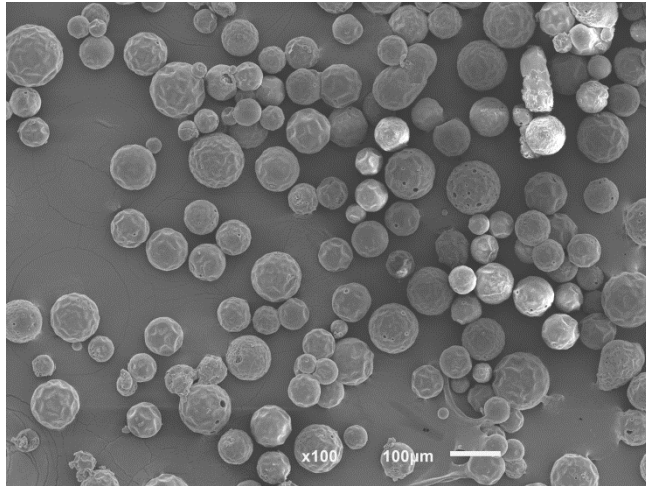
L. T. T. Nguyen, X. K. D. Hillewaere, R. F. A. Teixeira, O. Berg, F. E. Du Prez, Polymer Chemistry, Article ASAP

<http://www.sim-flanders.be/project/purepair>

# PUrePAIR - Polyurethane foams and elastomers with autonomic repair functions



## Microcapsules in Self-Healing



Core Content: ~50 %

$d(0.5) = 71.6 \mu\text{m}$

L. T. T. Nguyen, X. K. D. Hillewaere, R. F. A. Teixeira, O. Berg, F. E. Du Prez, Polymer Chemistry, Article ASAP

<http://www.sim-flanders.be/project/purepair>

# EnReCom - PUU Microcapsules

## EnReCom - Encapsulation of Reactive Components in Coatings

### Partners



- Coating Resins
- High Throughput Experimentation

### PUU shell

Isocyanate component

Amine component

### Core

Plasticizers

Allnex technical options

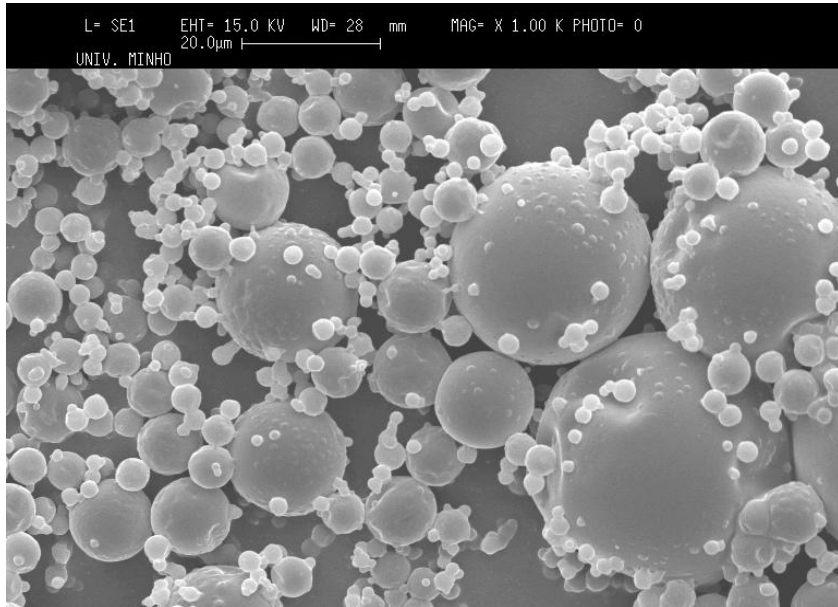
### Main Objective:

- Develop a two component coating system, masked as a one component system.



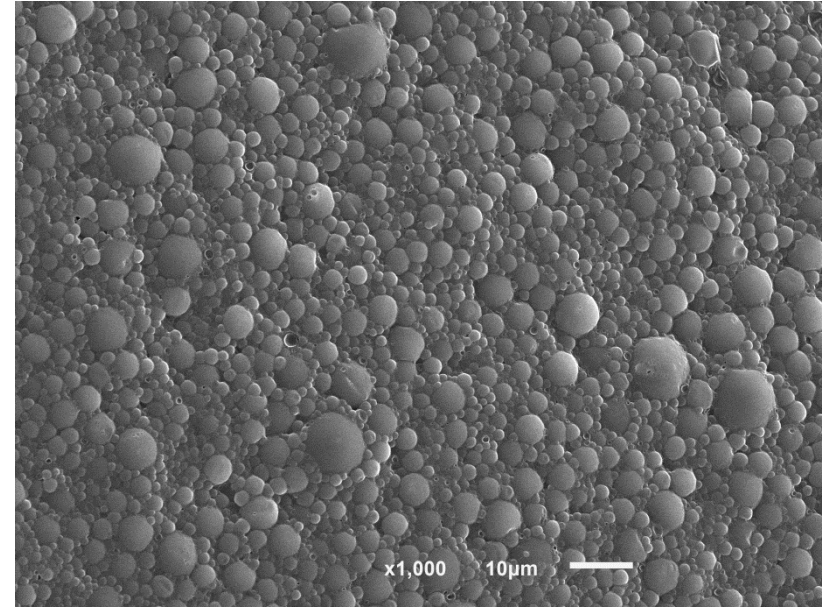
# EnReCom - Encapsulation of Reactive Components in Coatings

## ▪ Encapsulation of plasticizers



### ▪TOTM

Solid Content: 24.7%  
 $d(0.9) = 88.4 \mu\text{m}$



### ▪ADIPATE

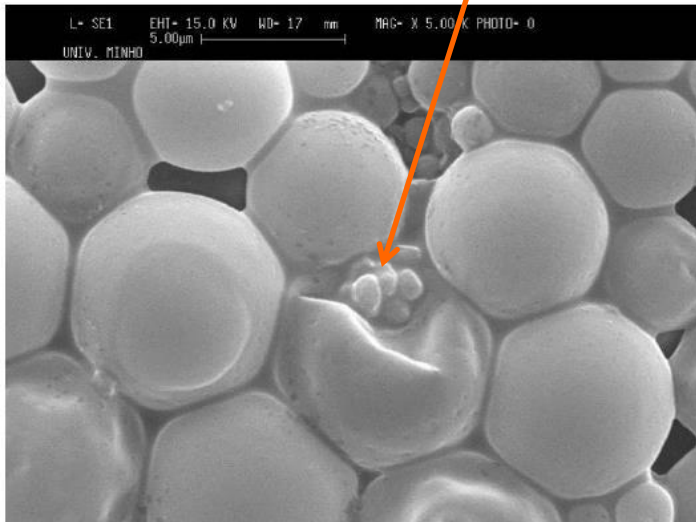
Solid Content: 40 %  
 $d(0.9) = 17.4 \mu\text{m}$



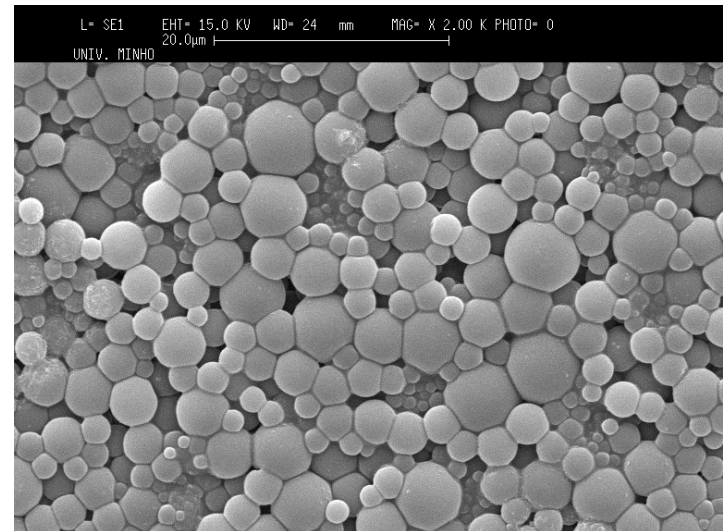
# Encapsulation of Biogenic Agents and Yeast extracts using MF Shell

1. Suspension in inert oil
2. Emulsion is formed with addition of water & shell precursors
3. Formation of the shell under temperature treatment

**Spores**

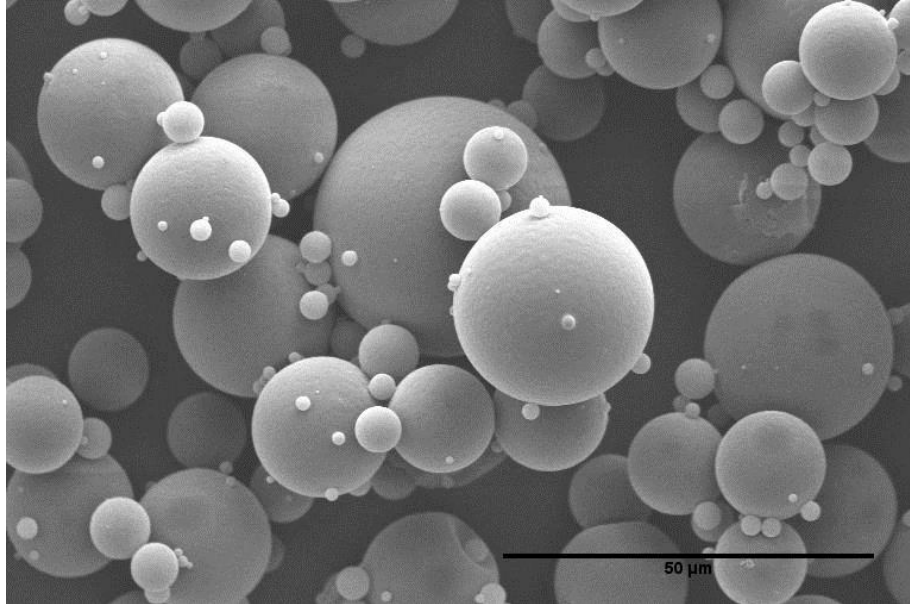


**FOOD SOURCE** in separate container  
**Microcapsules containing Yeast extracts**

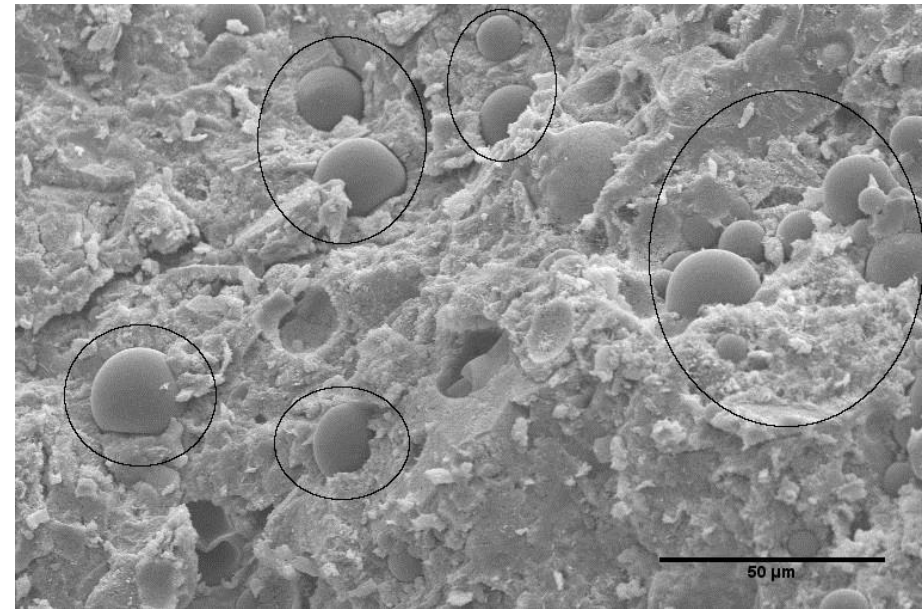


# MF Microcapsules dispersed in concrete

healCON  
concrete which repairs itself



**MF microcapsules before mixing with Cement**



**MF microcapsules x 150**

Pictures from  
“The Magnellaboratory”, UGent



# Summary

## □ Reactive Microencapsulation Platform @Devan

- Reactive Microencapsulation Technology

## □ PUU Microcapsules

- Functional Textiles using Microencapsulation
- Microcapsules in Self-Healing
- Encapsulation of Reactive Components in Coatings





# Acknowledgments



*Thank you for your attention*

**Isabel Martins**

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**24 June 2015, Cologne, Germany**



