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France-Wallonie-Vlaanderen UNION EUROPÉENNE  
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**DURATEX**

Avec le soutien du Fonds européen  
de Développement Régional  
Met de steun van het Europees  
Fonds voor Regionale Ontwikkeling

# Development of a hydrophobic formulation

B. Kartheuser, Certech asbl

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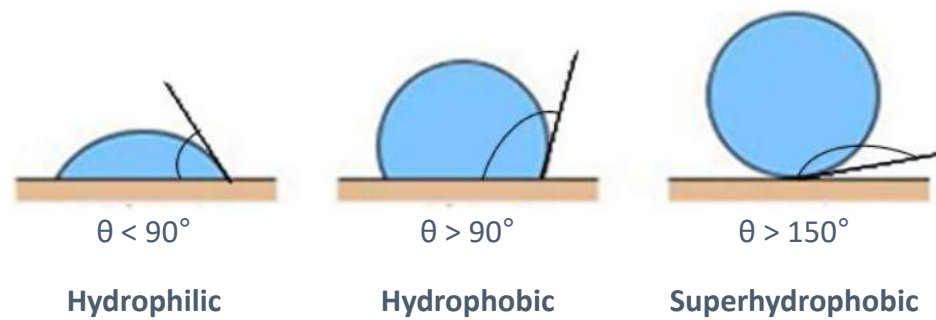


# Objective :

- » To develop a hydrophobic solution without fluorocarbon compounds that can be applied on fabric surfaces to obtain superhydrophobic properties.



# Hydrophilic - hydrophobic surface



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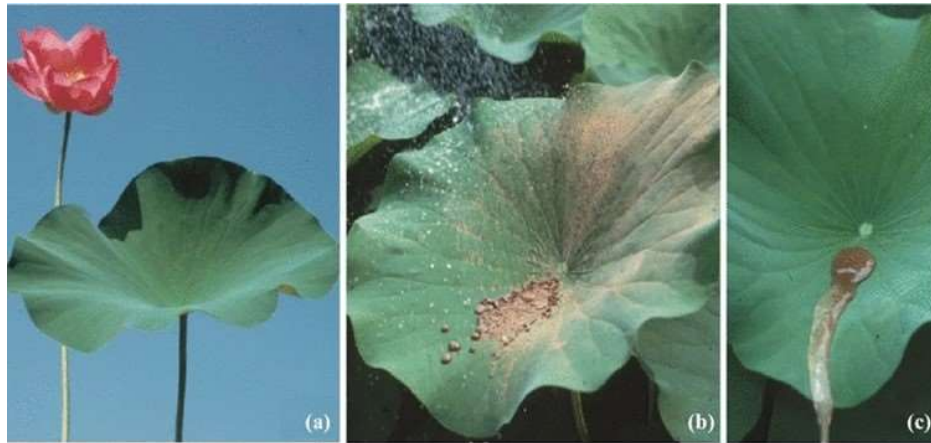
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UCLouvain

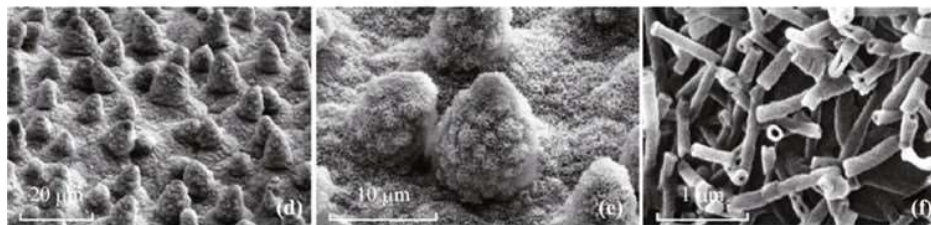


# How to obtain a hydrophobic surface ?

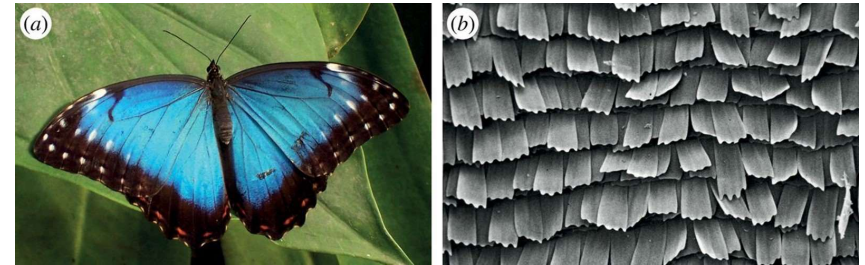
» Look to the nature :



The lotus plant (*Nelumbo nucifera*) (a): removal of dirt particles by water (b, c)



SEM micrographes of the lotus leaf (d) show the papillose cells (e) and wax tubules (f) on it



Barthlot et al, Phil.Trans.R.Soc.A 374:20160191

-Surface has a double structure :

Physical : micro and nano structure, trap air

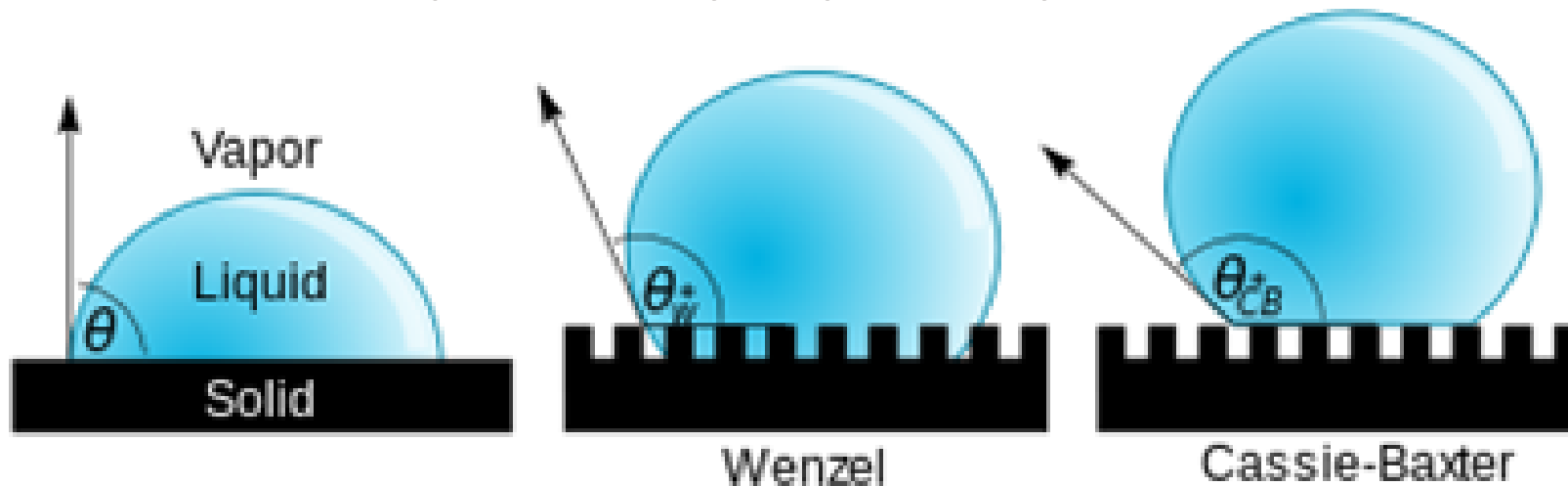
Chemical : wax , reduced the surface tension

Barthlott et al, Nano-Micro Lett. (2017) 9:23

DOI 10,1007/s40820-016-0125-1

# How to obtain hydrophobic surface ?

» Two models to explain the hydrophobicity.



Sticky surface : hydrophobic

slippery surface: superhydrophobic  
“fakir points”

slippery



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PRODUCT INSIGHT  
FOR YOUR TEXTILE SOLUTIONS



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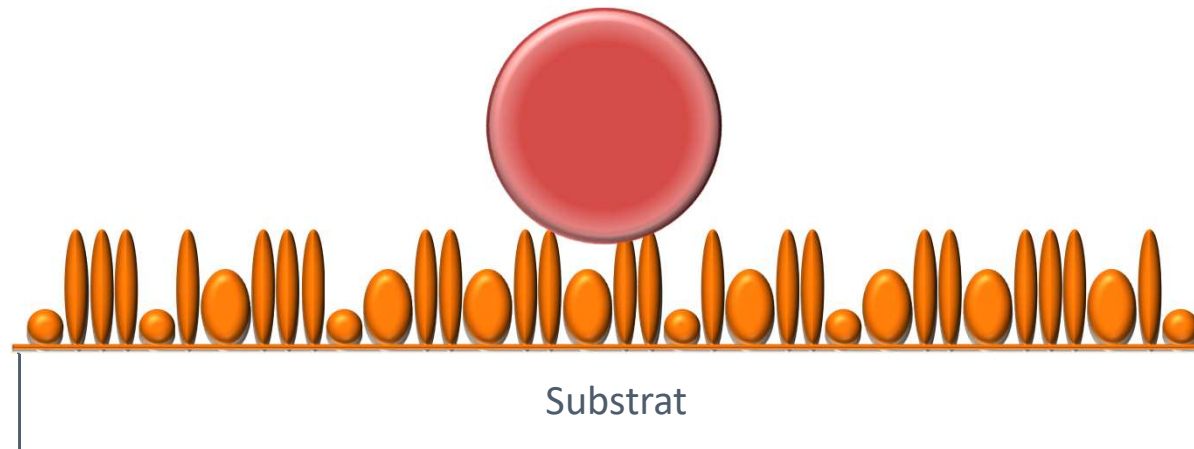
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# How to obtain hydrophobic surface ?

- » As seen in the nature, the surface needs to be structured :
  - > - Physically : use of particles
  - Chemically : use of hydrophobic compounds

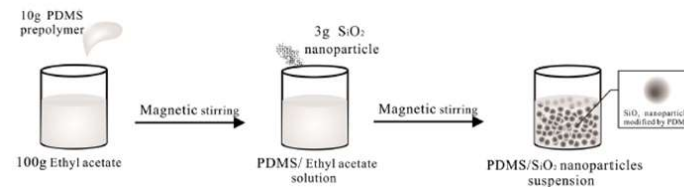


# Hydrophobic solution development

## » Different possibilities

- > Two independent solutions :
  - + 1° particle deposition
  - + 2° hydrophobisation of the particle by hydrophobic compounds like siloxanes such as polydimethylsiloxane (PDMS), wax, fatty acids...
- > One pot solution : particles + hydrophobic compounds + binder

+ Example

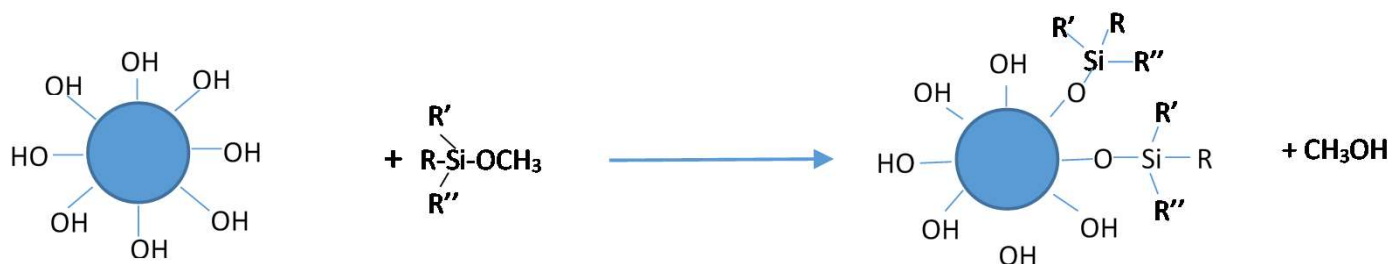
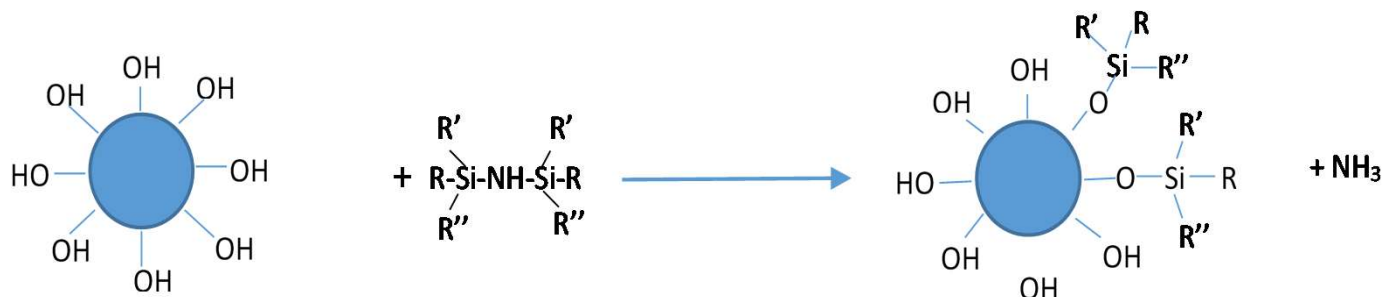


Ref: Wang et al. « Facile method to prepare a transparent superhydrophobic PET film », Appl. Phys. A 2016, 1–7.

- > One pot solution but with modification of the surface particles + binder

# Development of solutions for a super-hydrophobic coating

- Hydrophobisation of the solid with some alkyldisilazanes or alkylsilanes





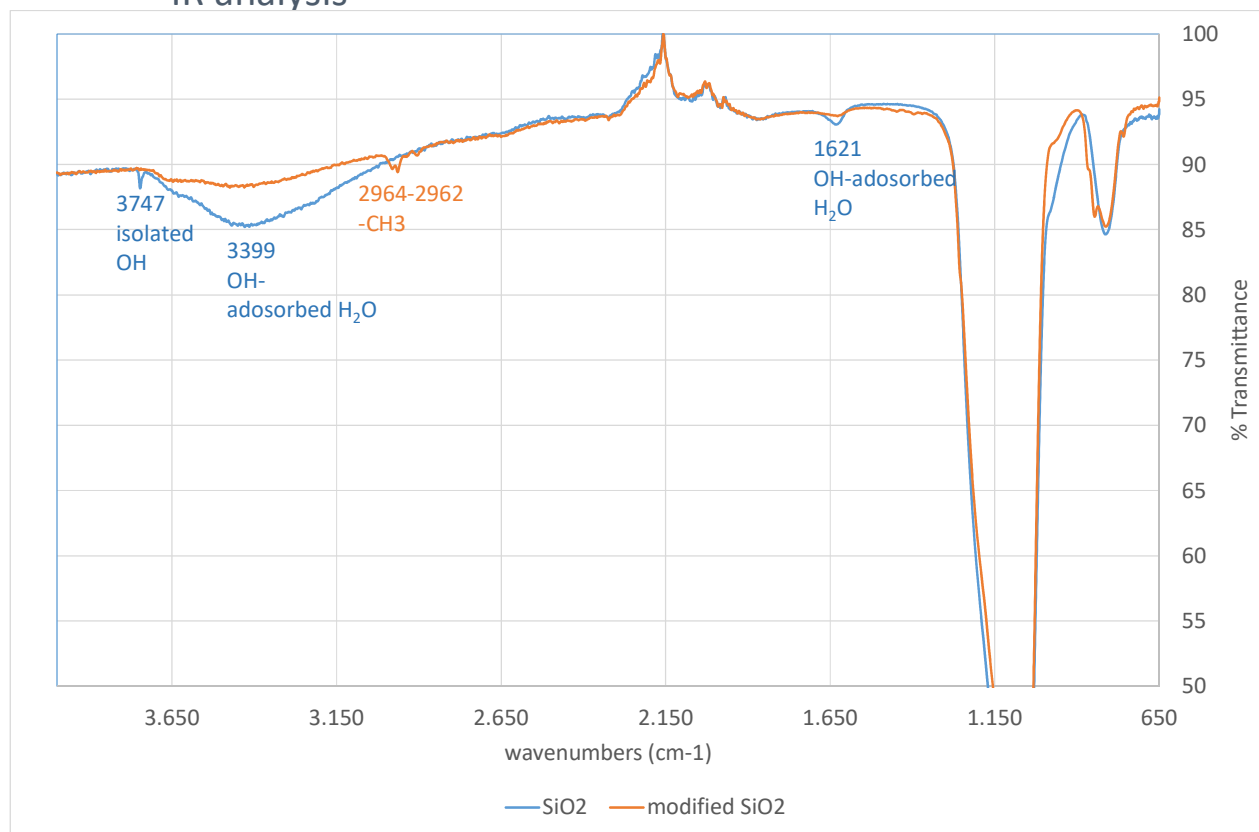
# Development of solutions for a super-hydrophobic coating

- Procedure : 1 particle modification example of  $\text{SiO}_2$ .



# Development of solutions for a super-hydrophobic coating

- Grafting verification :
  - IR analysis



- Reduction of the broad band at 3400 cm<sup>-1</sup>
- Disappearance of bands at 3474 and 1627 cm<sup>-1</sup>
- Appearance of bands at 2964 and 2962 cm<sup>-1</sup>

Replacement of the surface OH by Si(CH<sub>3</sub>)<sub>3</sub>

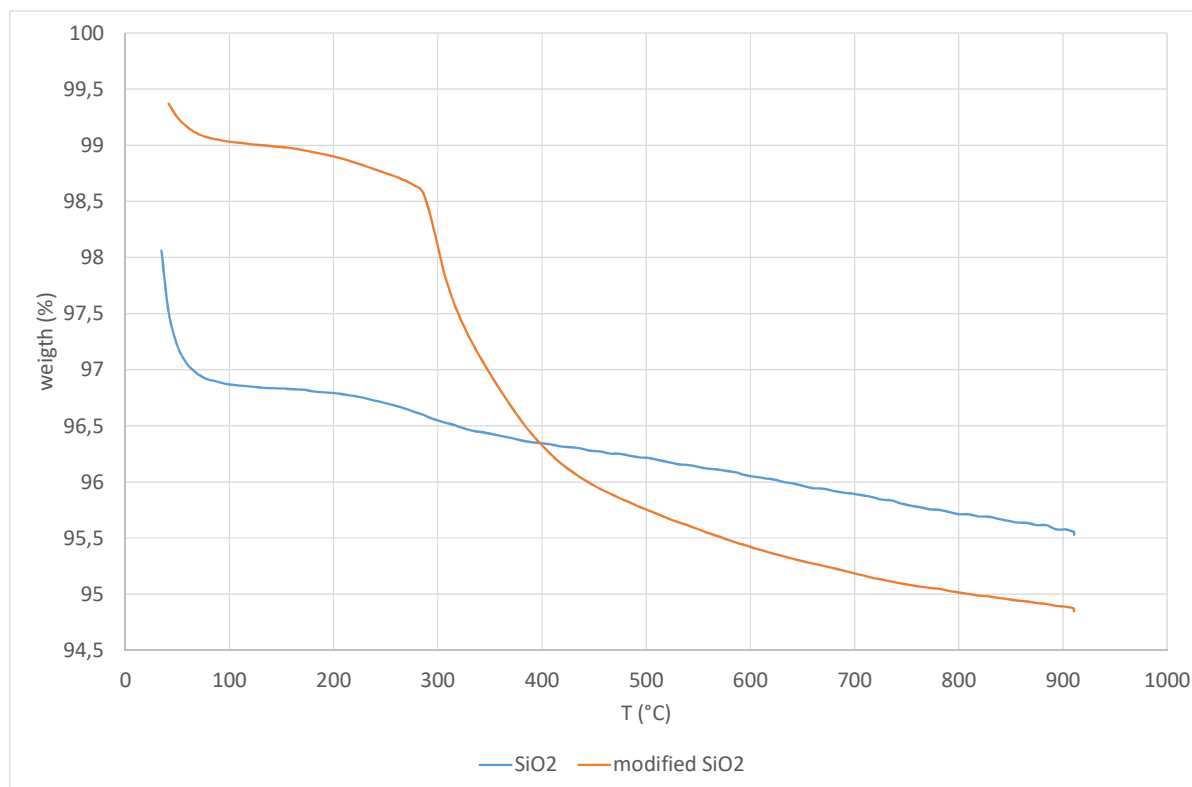


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# Development of solutions for a super-hydrophobic coating

- Grafting verification :
  - Thermogravimetric analysis under air flow



- Weight lost below 150°C is due to physisorbed water
- For SiO<sub>2</sub>, the increased temperature caused the dehydration of -OH  
$$\text{Si-OH} + \text{OH-Si} \longrightarrow \text{Si-O-Si} + \text{H}_2\text{O}$$
- The increased of the weight lost after 150 °C for the modified SiO<sub>2</sub> is due to the oxidation of the alkyl groups
- About 60 % of the available OH are replaced by the alkylsilazane

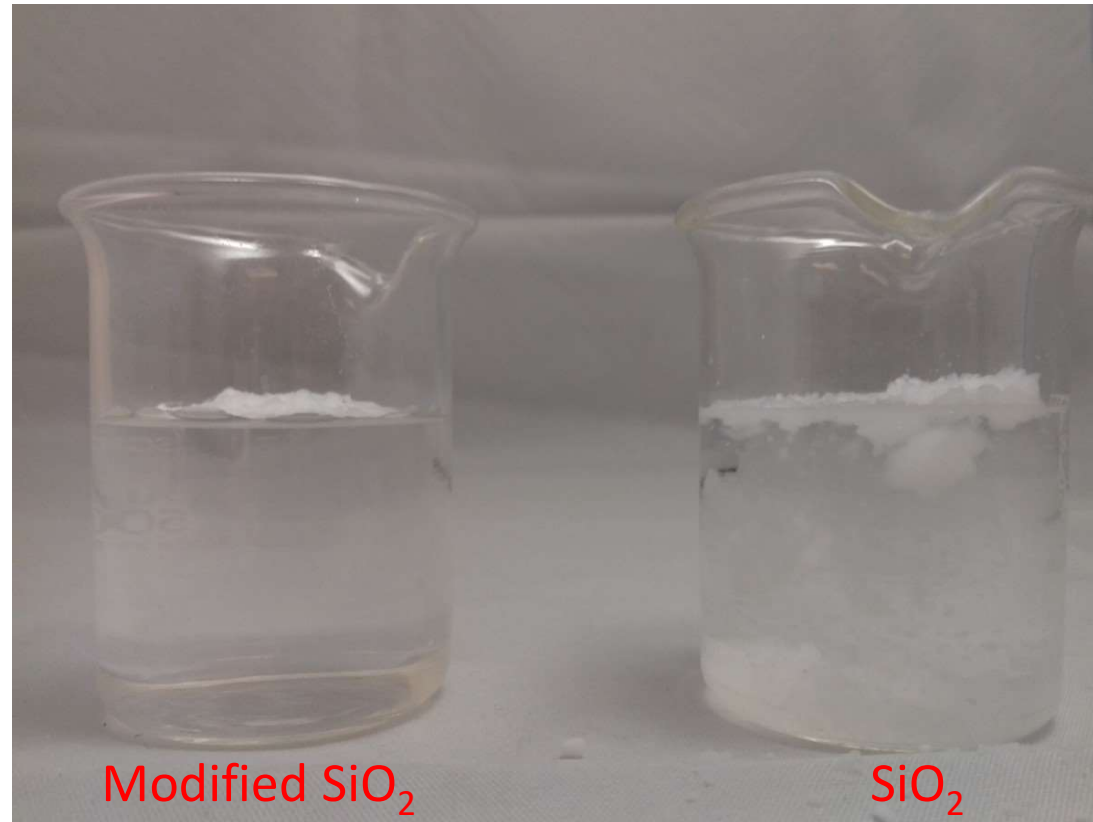


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# Development of solutions for a super-hydrophobic coating

- Hydrophobicity of the powder



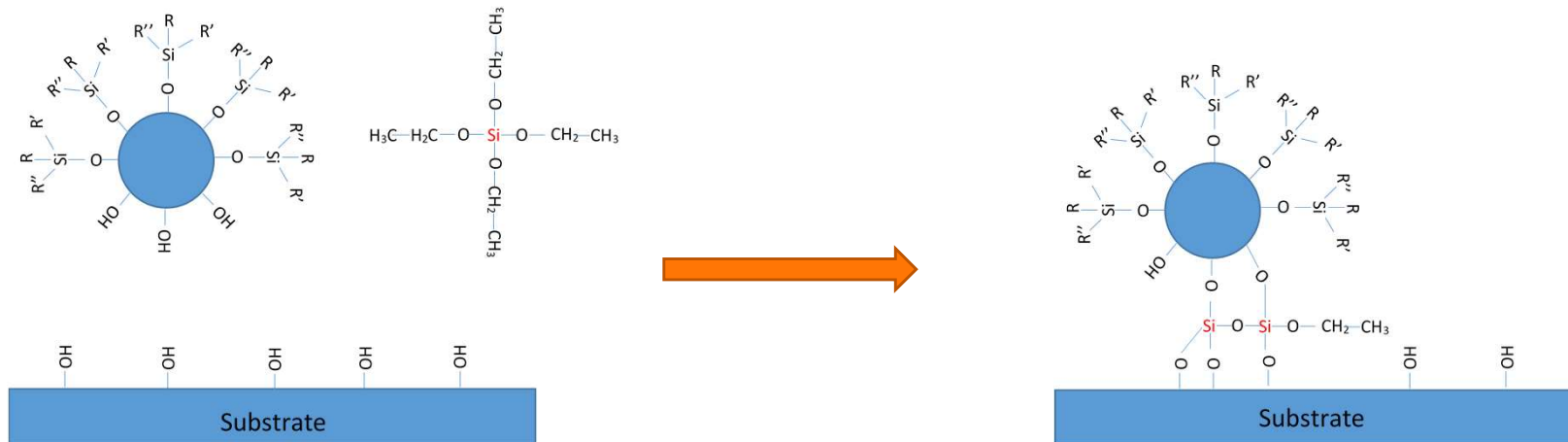
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# Development of solutions for a super-hydrophobic coating

How to graft the hydrophobic particles ?

Need a binder to link the particles to the substrate's surface, such as for example tetraethylorthosilicate (TEOS).

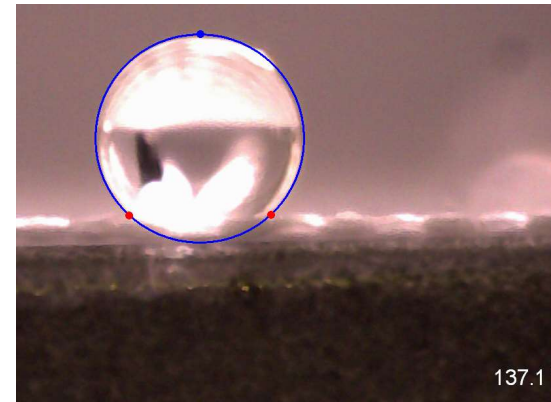


# Development of solutions for a super-hydrophobic coating

How to graft the hydrophobic particles ?

Make a solution :

1° dispersion of the particles in an organic solution containing the binder

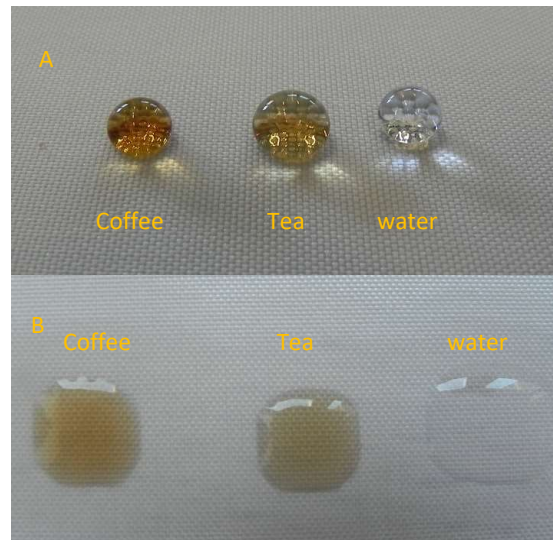


# Development of solutions for a super-hydrophobic coating

How to graft the hydrophobic particles ?

2° : Make an aqueous solution of the hydrophobic particles :

water + organic solvent + particles + binder (used ultra turrax for dispersion)



Droplets of coffee, tea and water on (A) treated PET tissue, and (B) untreated PET tissue

able to obtain a water based solution, stable for more than 4 months



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# Development of solutions for a super-hydrophobic coating



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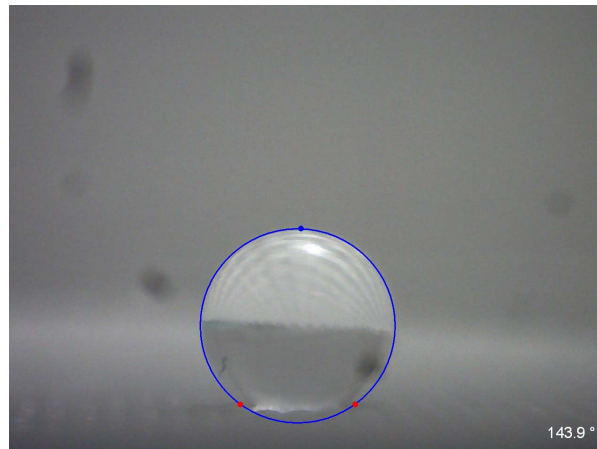
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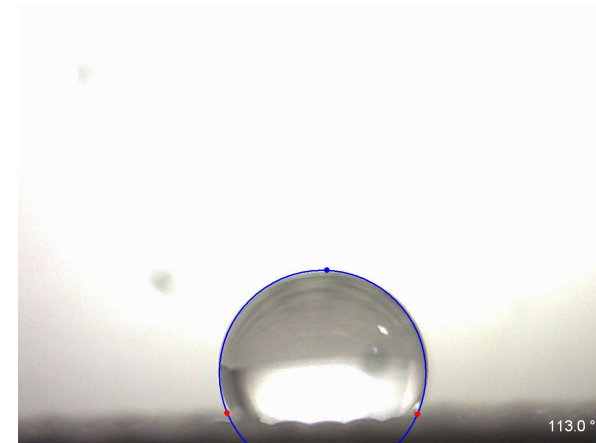


# Development of solutions for a superhydrophobic coating

- Test of padding at Centexbel at two different pressures: 0,5 and 4 bar



Spray deposition

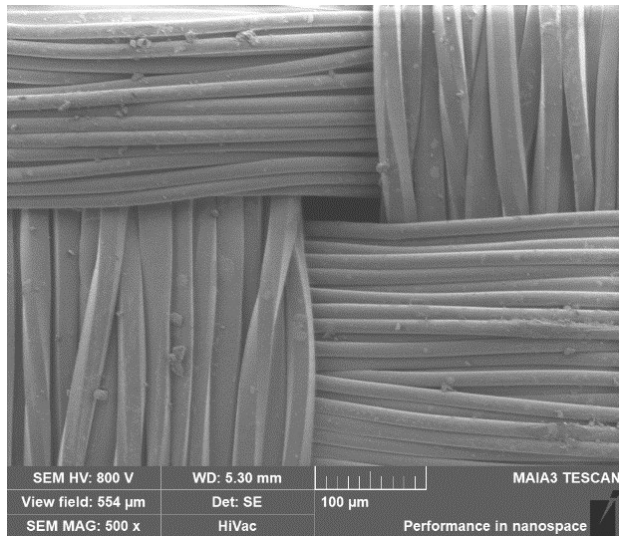


padding deposition

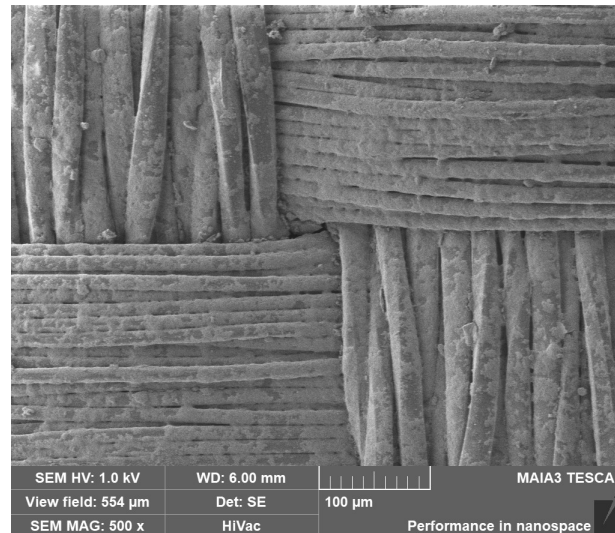
➔ Loss of the superhydrophobicity after padding deposition

# Development of solutions for a super-hydrophobic coating

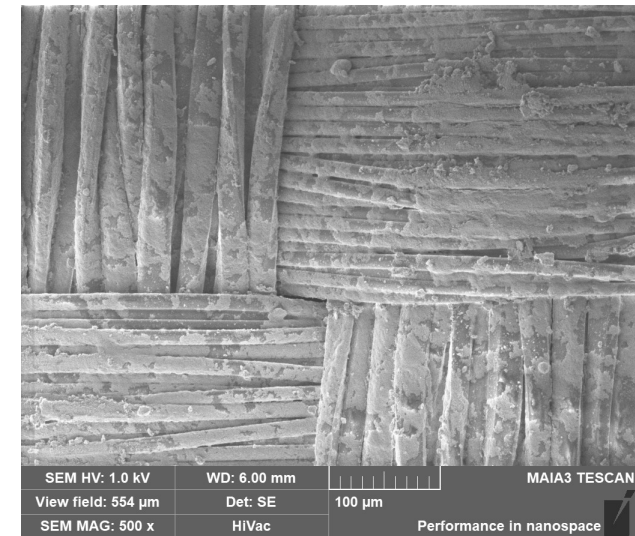
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Spray



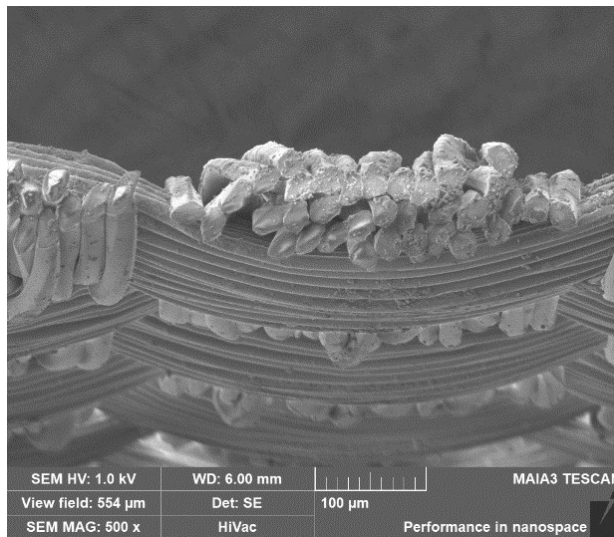
Padding 0,5 bar



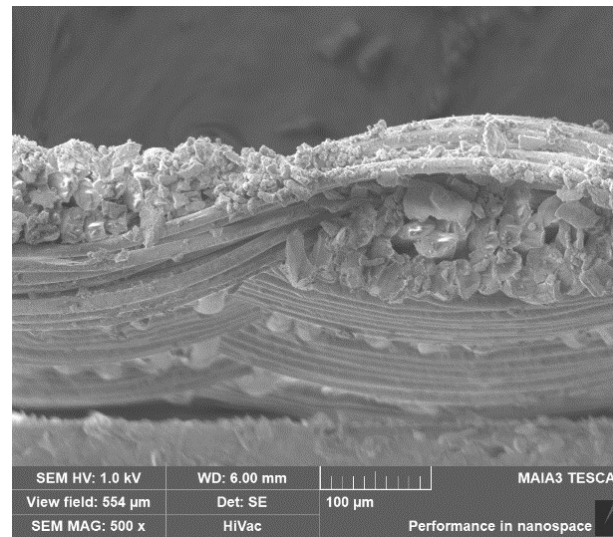
Padding 4 bar

# Development of solutions for a super-hydrophobic coating

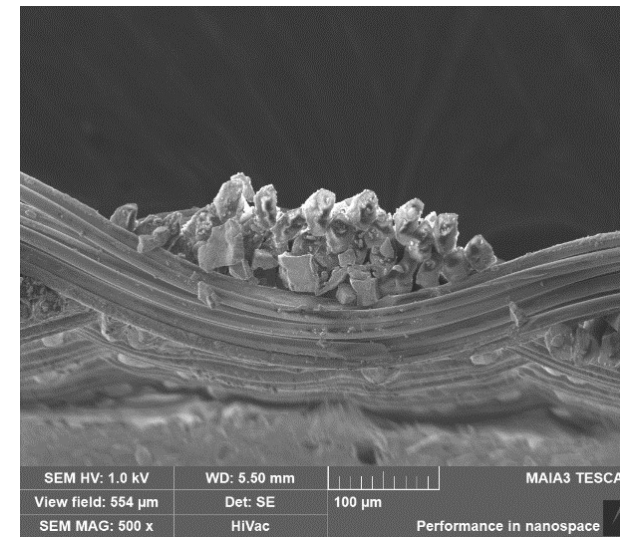
- Test of padding at Centexbel at two different pressures: 0,5 and 4 bar



Spray



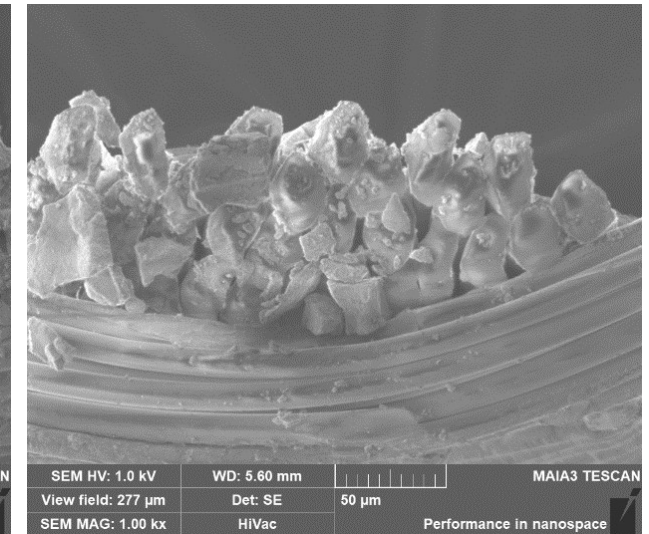
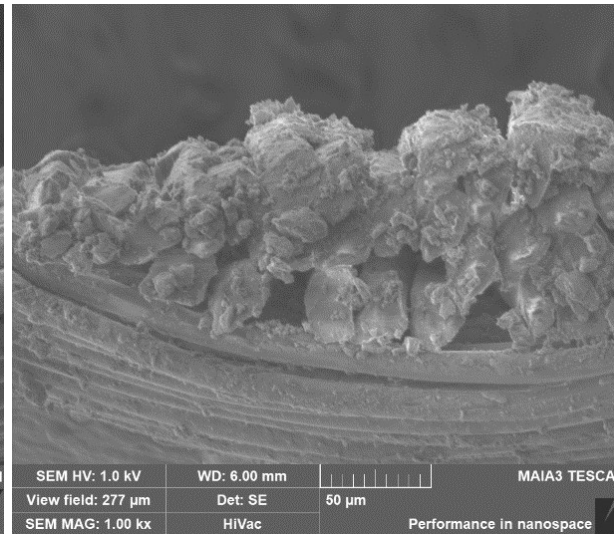
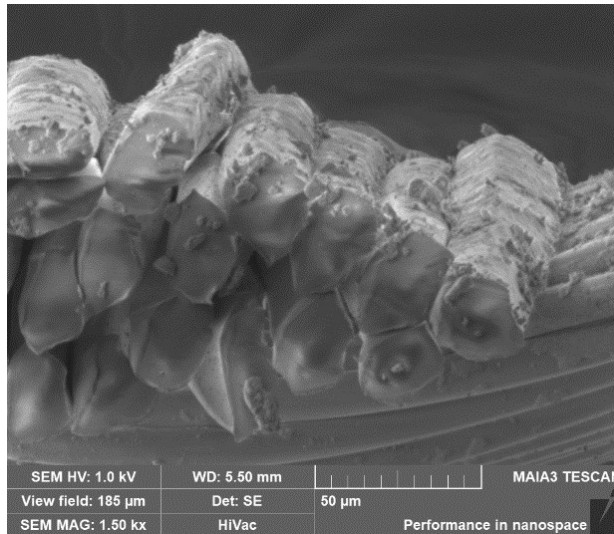
Padding 0,5 bar



Padding 4 bar

# Development of solutions for a super-hydrophobic coating

- Test of padding at Centexbel at two different pressures: 0,5 and 4 bar



# Conclusions

Possibility to make a water based hydrophobic solution.  
Superhydrophobic surface is obtained by spray coating but not with padding.  
Still needs some improvements of the adhesion of the hydrophobic particles.

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# Acknowledgment



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Projet soutenu par  
Project ondersteund door

**Interreg**   
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