QCM-D: NEW POSSIBILITIES FOR OPTIMISED CLEANING AND DISINFECTION AT THE RISE TESTBED CLEANING INNOVATION

Mikael Kjellin

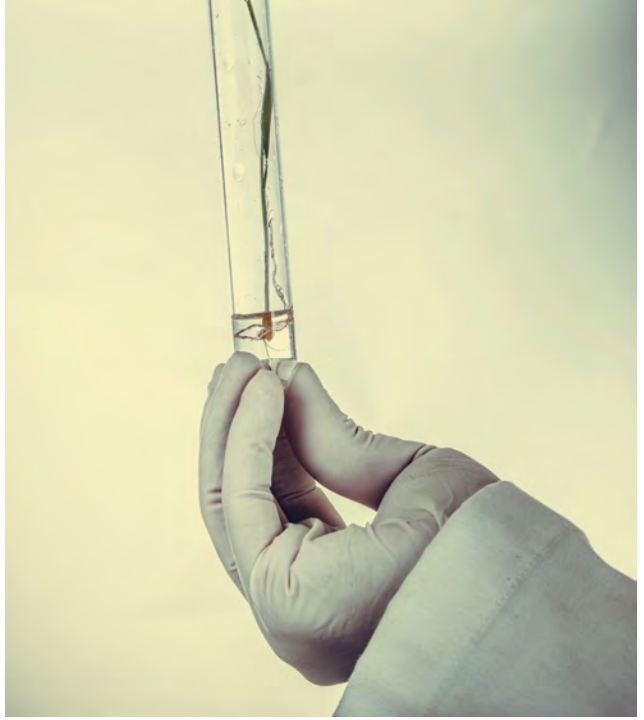
November 10, 2017

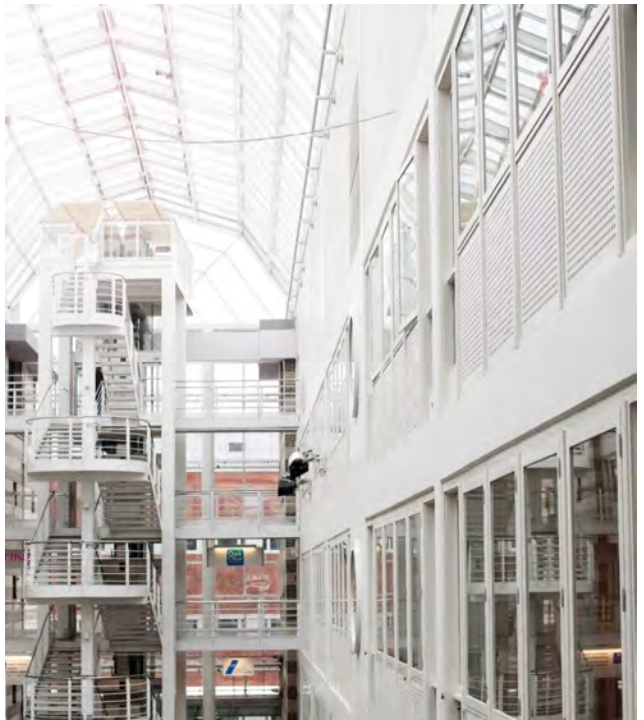
13:45-14:15

Research Institutes of Sweden

Division: Bioscience and Materials
Unit: Surface, Process and Formulation

Section: Formulation Development





Owned by the Swedish government

RISE=Research Institutes of Sweden

Merger between institutes Innventia, SP and Swedish ICT to become a stronger research and innovation partner for businesses and society.

One strong, unified institute for Sweden

- Compete on the international stage
- Build a stronger Swedish institute sector that will actively support Swedish industry, providing increased benefits for trade and industry, and society in general.

RISE in brief

- Present across the whole of Sweden. And beyond.
- 2,200 employees, 30 % with a PhD.
- Turnover approx. SEK 2.5 billion (2016).
- SME clients account for approx. 30 %.
- Runs 100s of test and demonstration facilities, open for industry, SMEs, universities and institutes (RISE is owner and partner in 60 % of all Sweden's T&D facilities).
- www.ri.se

With our broad range of competencies and unique expertise, we create benefits for many


Bioeconomy


Fire and safety


Cement, ballast and concrete


Certification


Circular economy


Design


Electronics


Energy and fuels


Glass


ICT and digitisation


Agriculture


Chemistry, materials and surfaces


Health and Life Science


Food


Mechanical engineering


Pulp and paper


Mechanics


Metrology


Mobility


Perception


Process development


Built environment


Systems analysis, service design


Applied measurement technology


Wood


Water

PERFORM

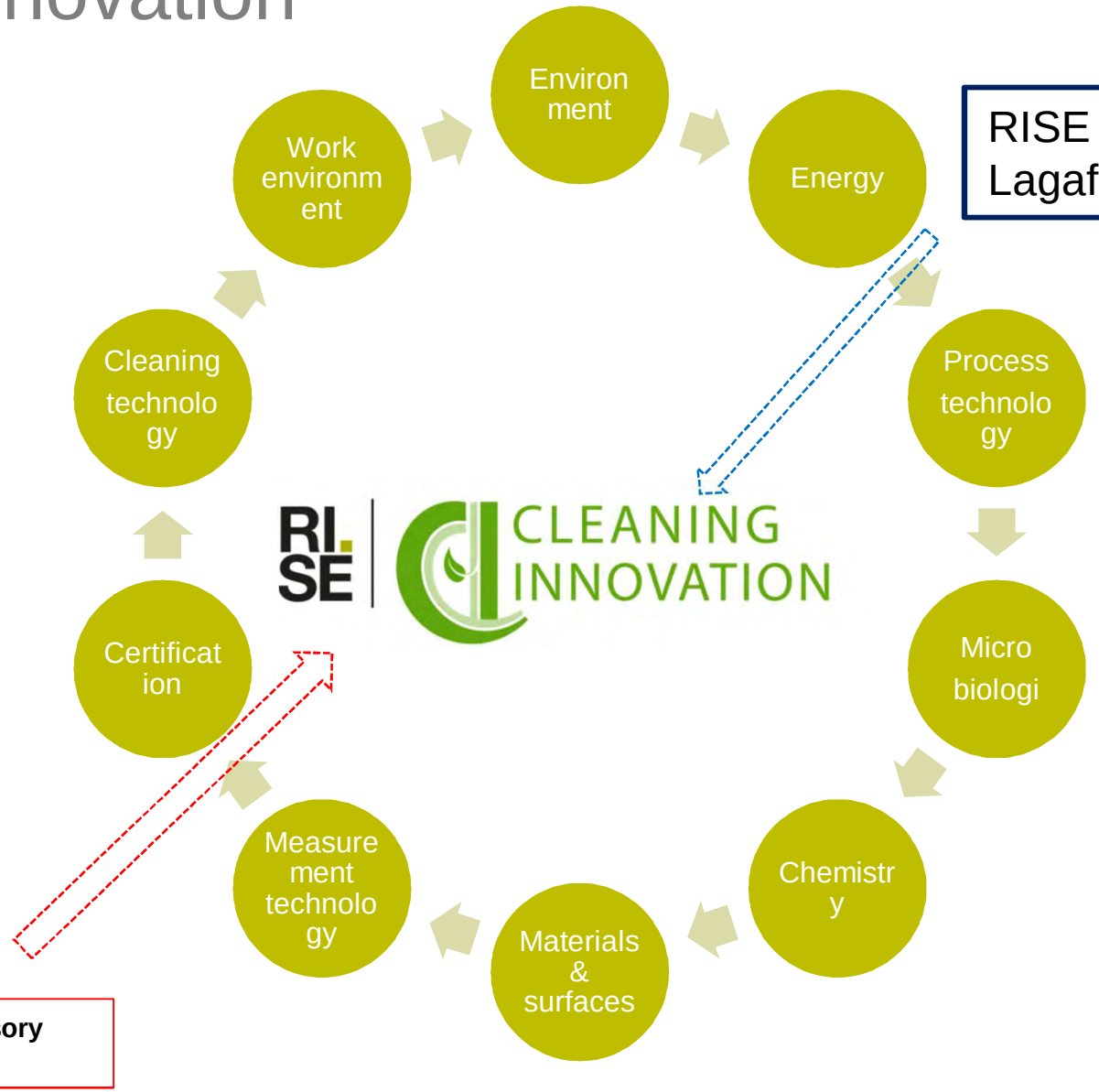
- Research and innovation in the formulation area
- Focus areas:
  1. Powder technology
  2. Formulation of biologics
  3. Controlled delivery and release
  4. Emulsions and disperse systems
- Collaborative projects, networking, courses
- www.rise-perform.com

Contact: Lovisa Ringstad, lovisa.ringstad@ri.se

RI. SE

The RISE testbed Cleaning Innovation

- Cleaning Innovation is a platform to develop new technologies, new cleaning formulations, perform testing, education etc in the area of cleaning in the food- and nearby-industry.
- Within Cleaning Innovation RISE and the industry have gathered experts, courses, venues and experience to enable new green cleaning technologies based on knowledge.
- www.cleaninginnovation.se



Cleaning Innovation Services

1. Cleaning Innovation - Smart for the environment and clean!
2. Training sessions on cleaning
3. Cleanability of process equipment
4. Development & Evaluation of cleaning formulations
5. Cleaning and disinfection - Comparison of techniques
6. Numerical Cleanness Analysis
7. Clean - and with a small footprint
8. Avoid Process Downtime & Problems – Analyze Your Cleaning Processes
9. Safety and Exposure - Cleaning in Production Units
10. Swat team - investigation of cleaning problems

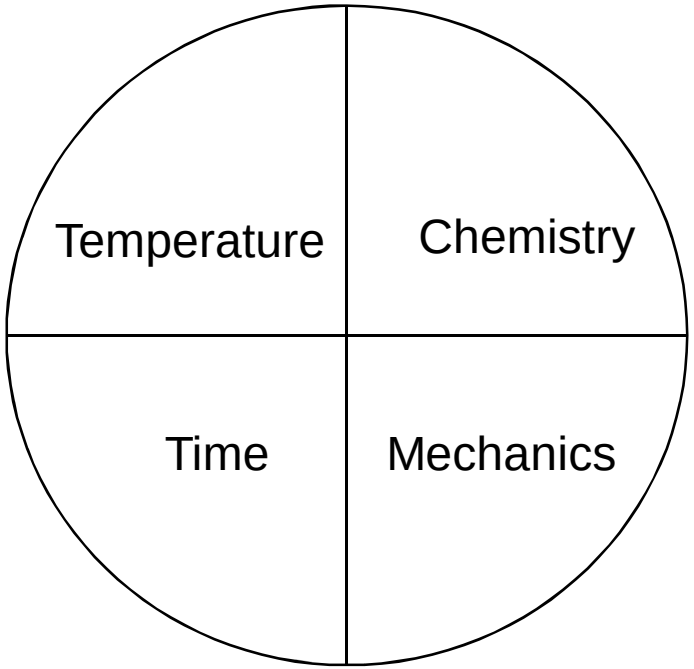
Seminar 2017-11-29, Gothenburg, RISE Jordbruk och livsmedel

www.cleaninginnovation.se

Cleaning-Important control parameters

- Temperature
- Time
- Mechanical energy
- Chemistry. Concentration of active substances

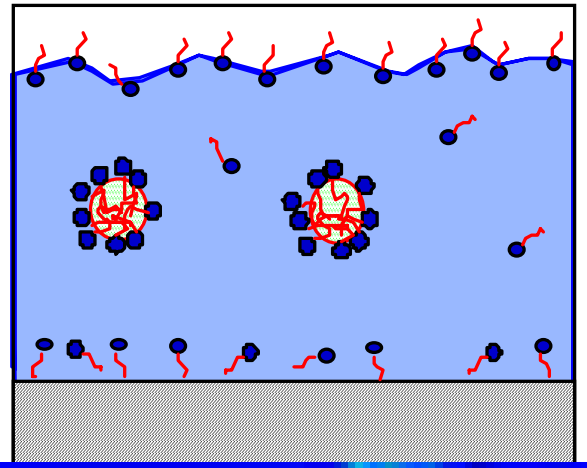
- Also important: What is the demand of cleanliness?



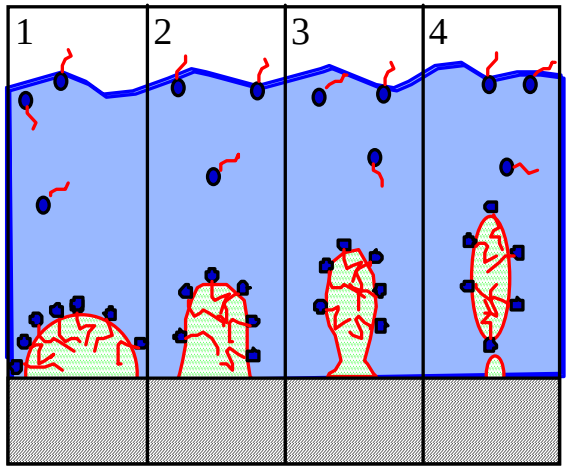
Sinner's circle
4 factors that influence the cleaning result.

Cleaning mechanisms for oily soil. Three main types.

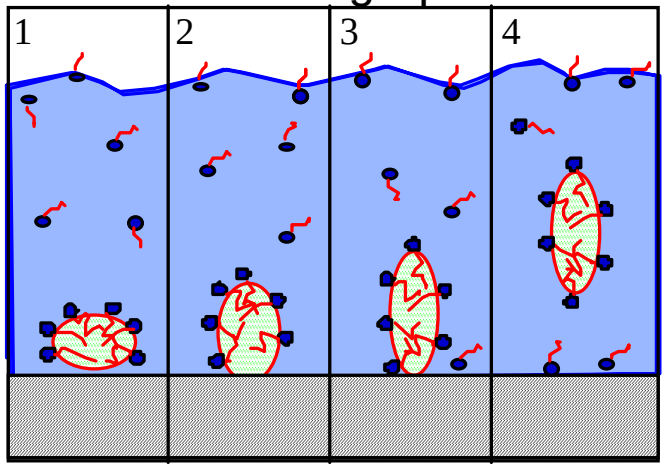
Solubilisation

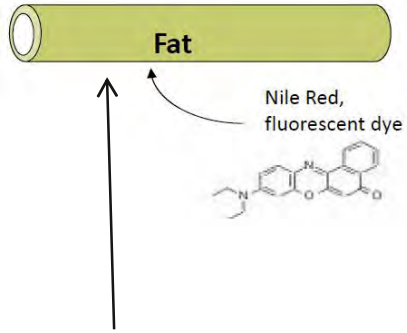


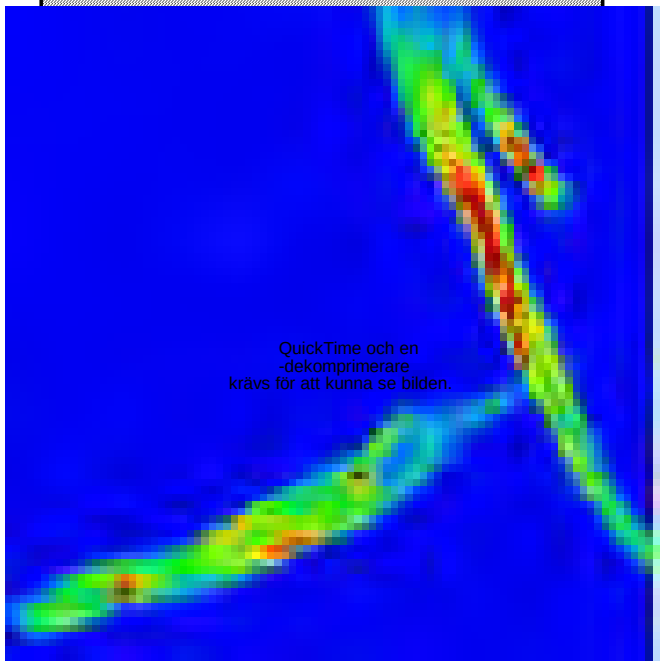
Emulsification

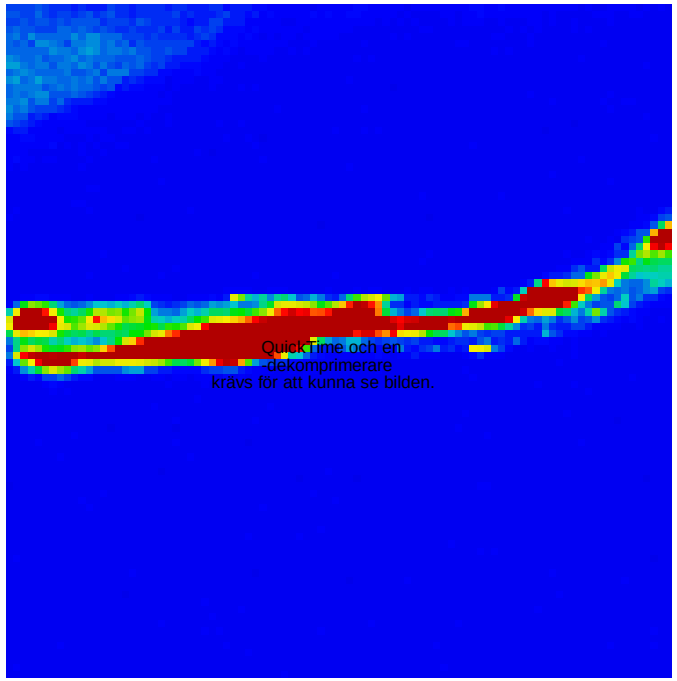


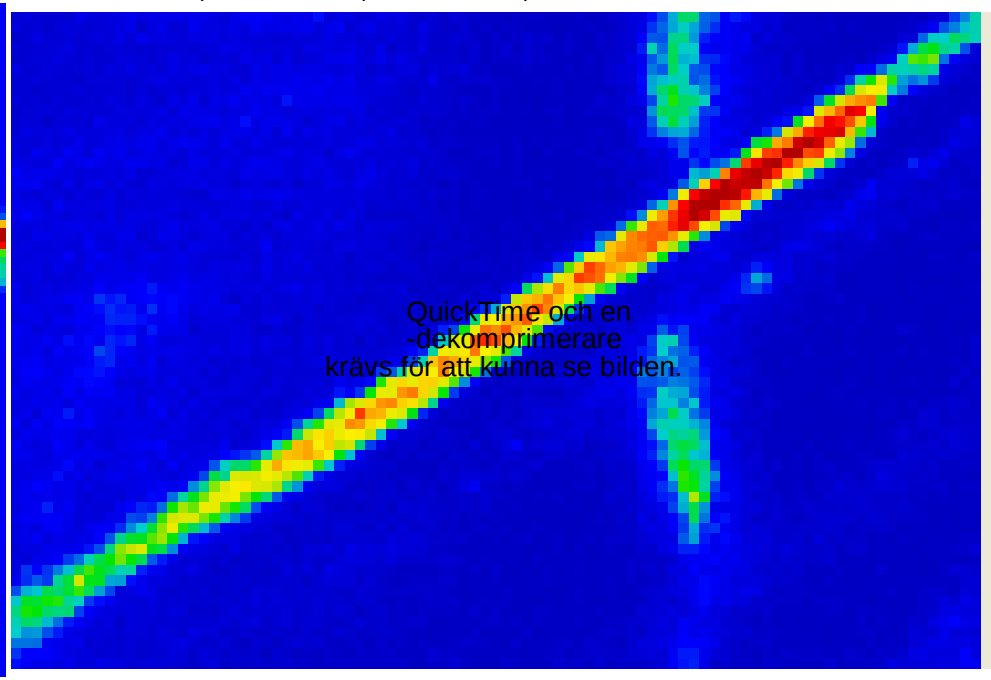
Rolling-up





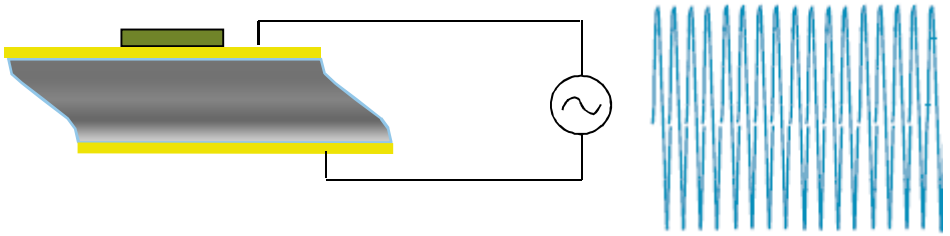




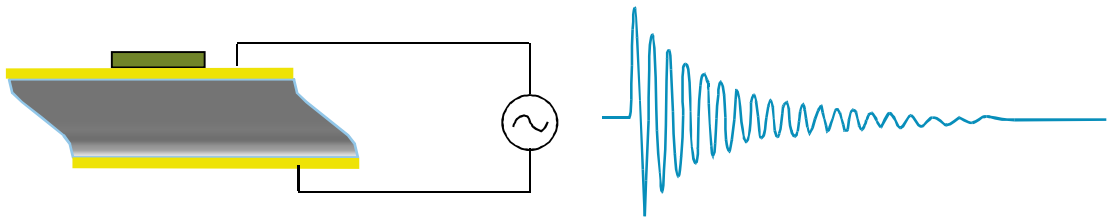


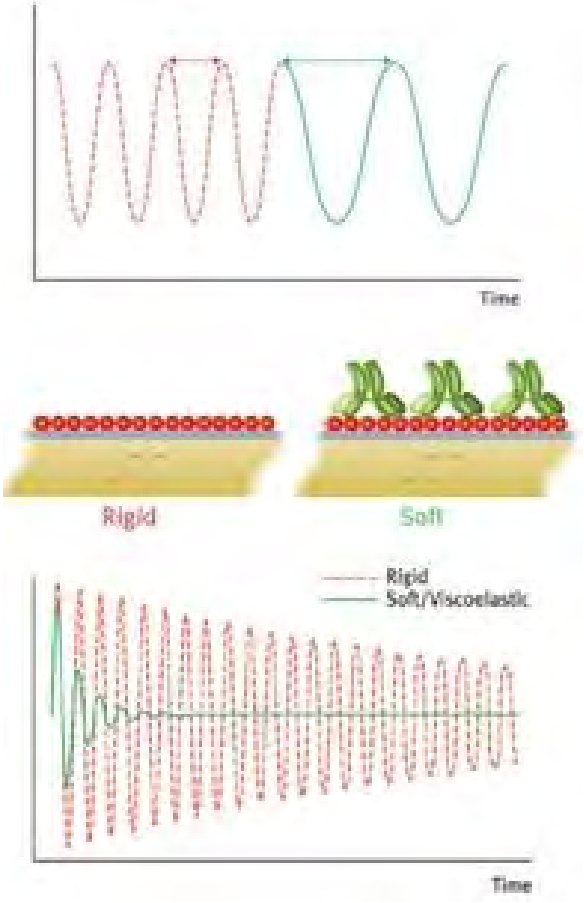
Principles of the QCM-D Technology

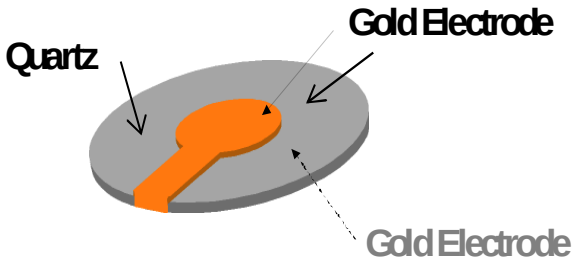
- Apply voltage, cause the crystal to oscillate



- Stop voltage, allow crystal to come to rest







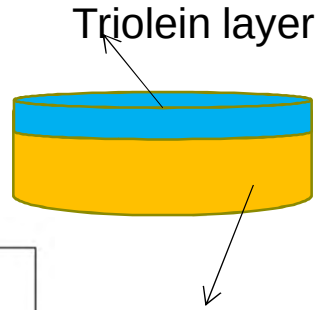
- Change in mass on sensor → change in frequency (Df)
- Change in viscoelastic properties → change in Dissipation (DD)
- Layer viscosity, elasticity and thickness can be calculated.

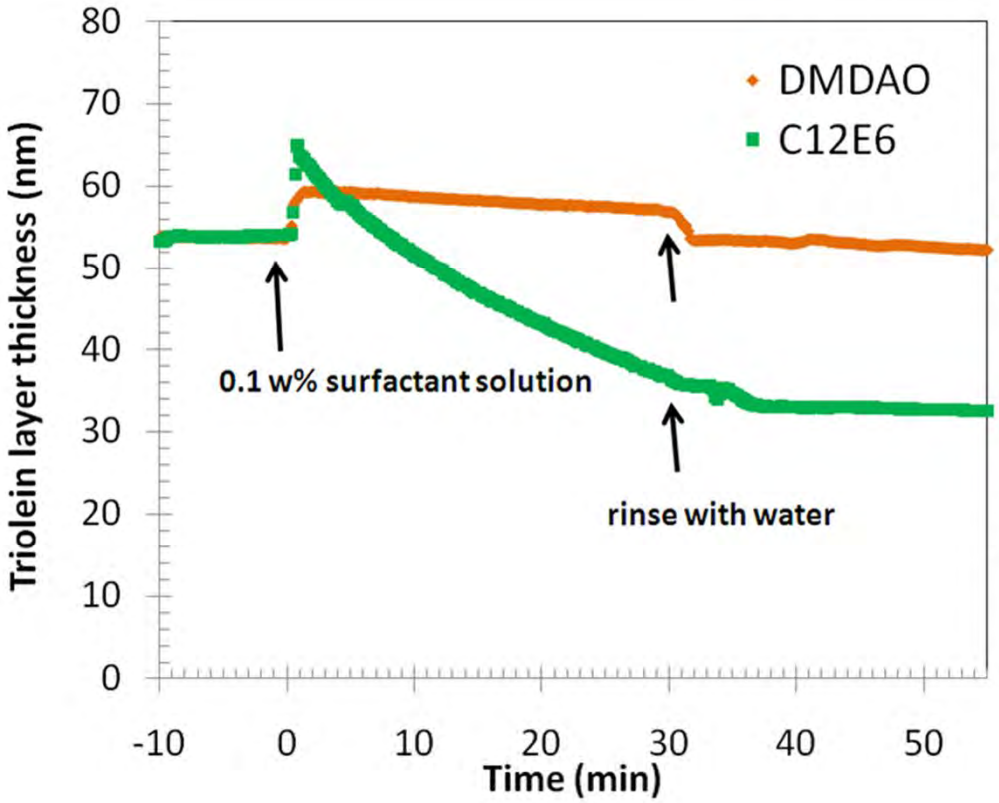
$$Dm = -\frac{CDf}{n}$$

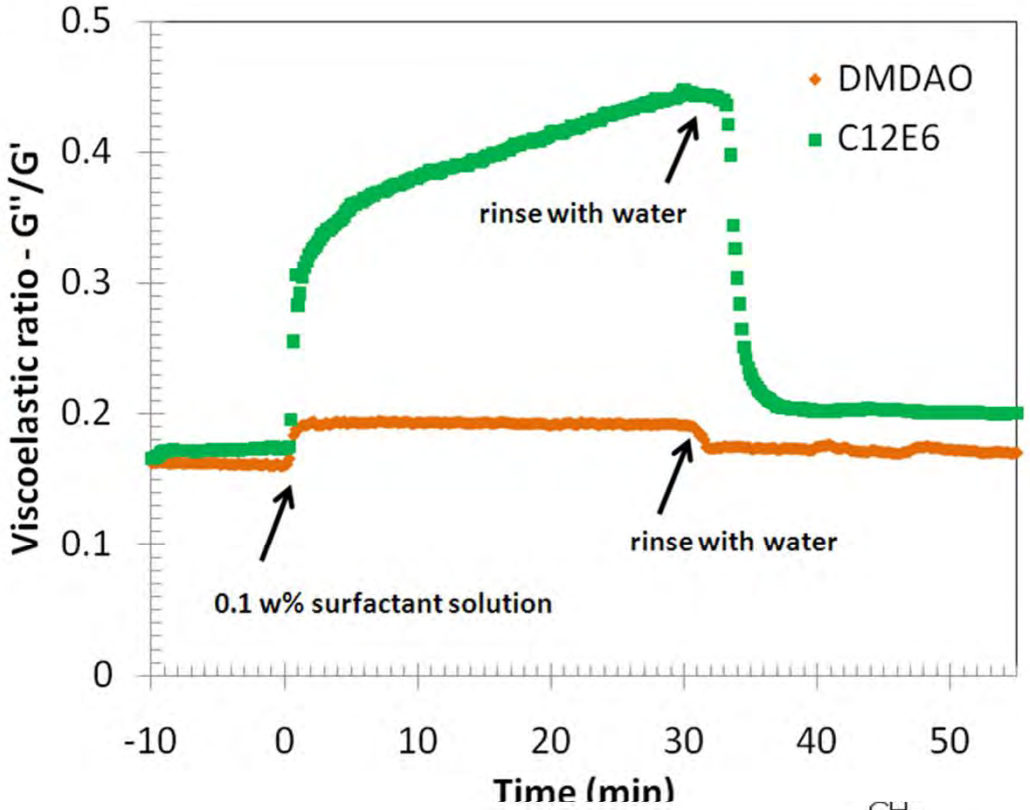
$$C = \frac{r_q t_q}{f_0} = 17.8 ngcm^{-2}Hz^{-1}$$

Quantifying oily soil removal and understanding cleaning mechanisms

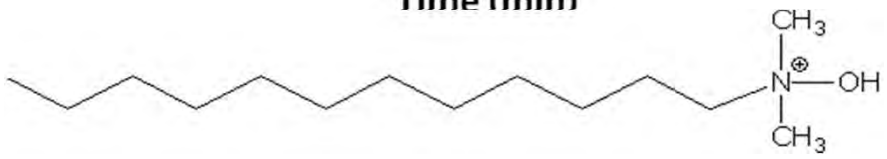
The quartz crystal surface is covered with a layer of model dirt (triolein) and then mounted into the QCM-D apparatus. The cleaning solution is then added and the amount dirt removed is continuously monitored as a function of time.





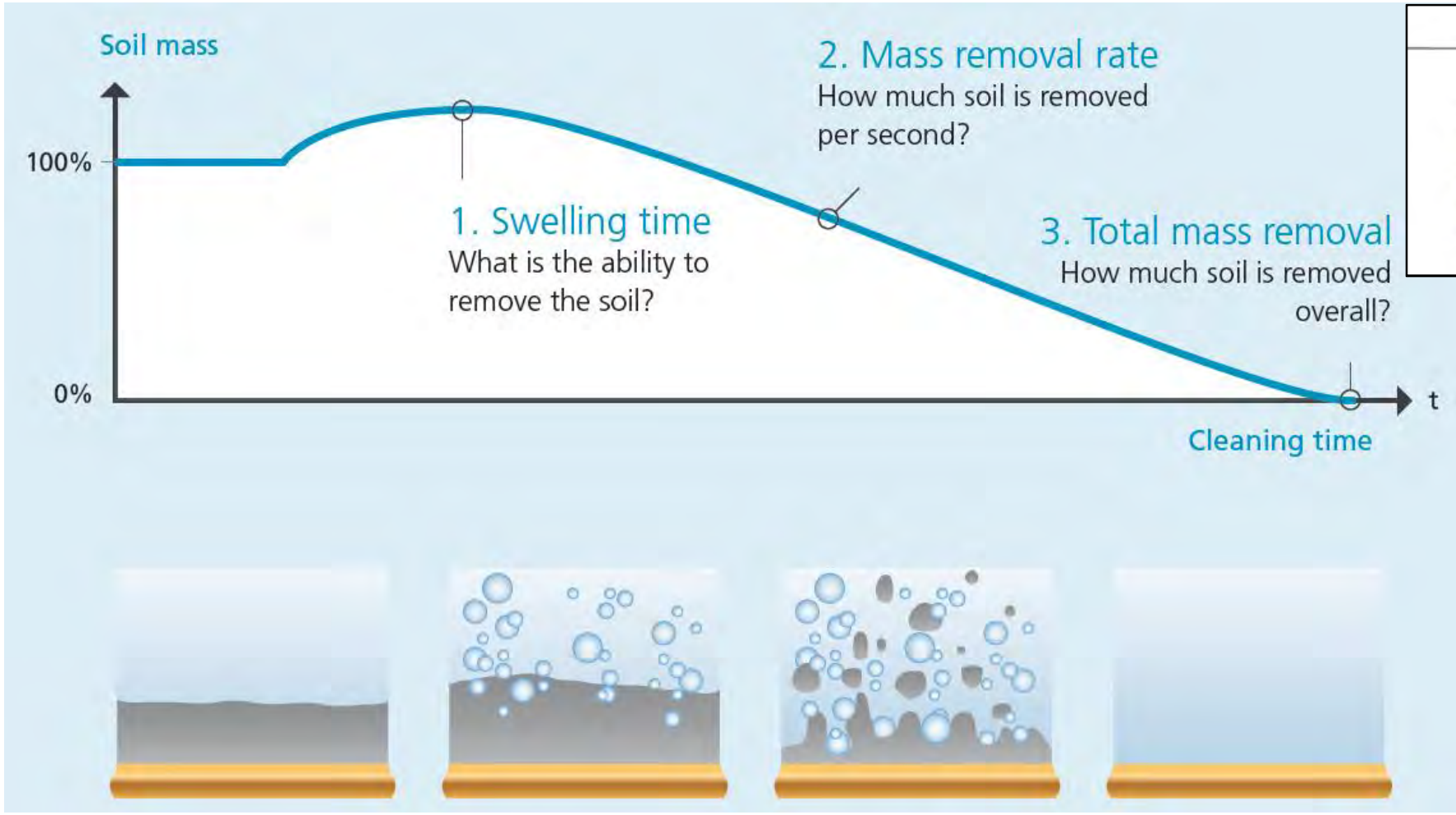


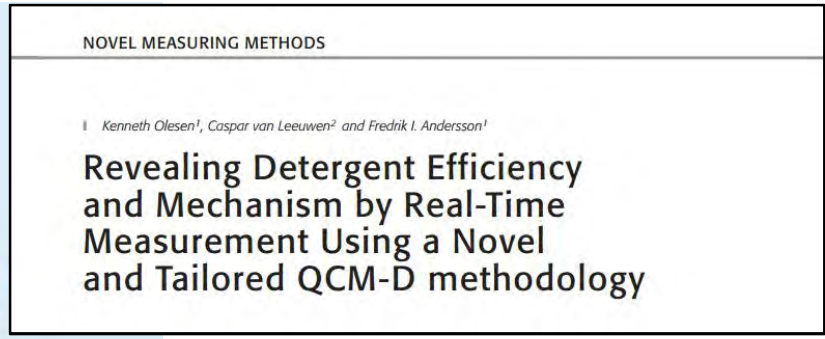
$C_{12}E_6$



DMDAO

The Cleaning profile at a glance. Development at Biolin.




NOVEL MEASURING METHODS

Kenneth Olesen[1], Caspar van Leeuwen[2] and Fredrik I. Andersson[1]

Revealing Detergent Efficiency and Mechanism by Real-Time Measurement Using a Novel and Tailored QCM-D methodology

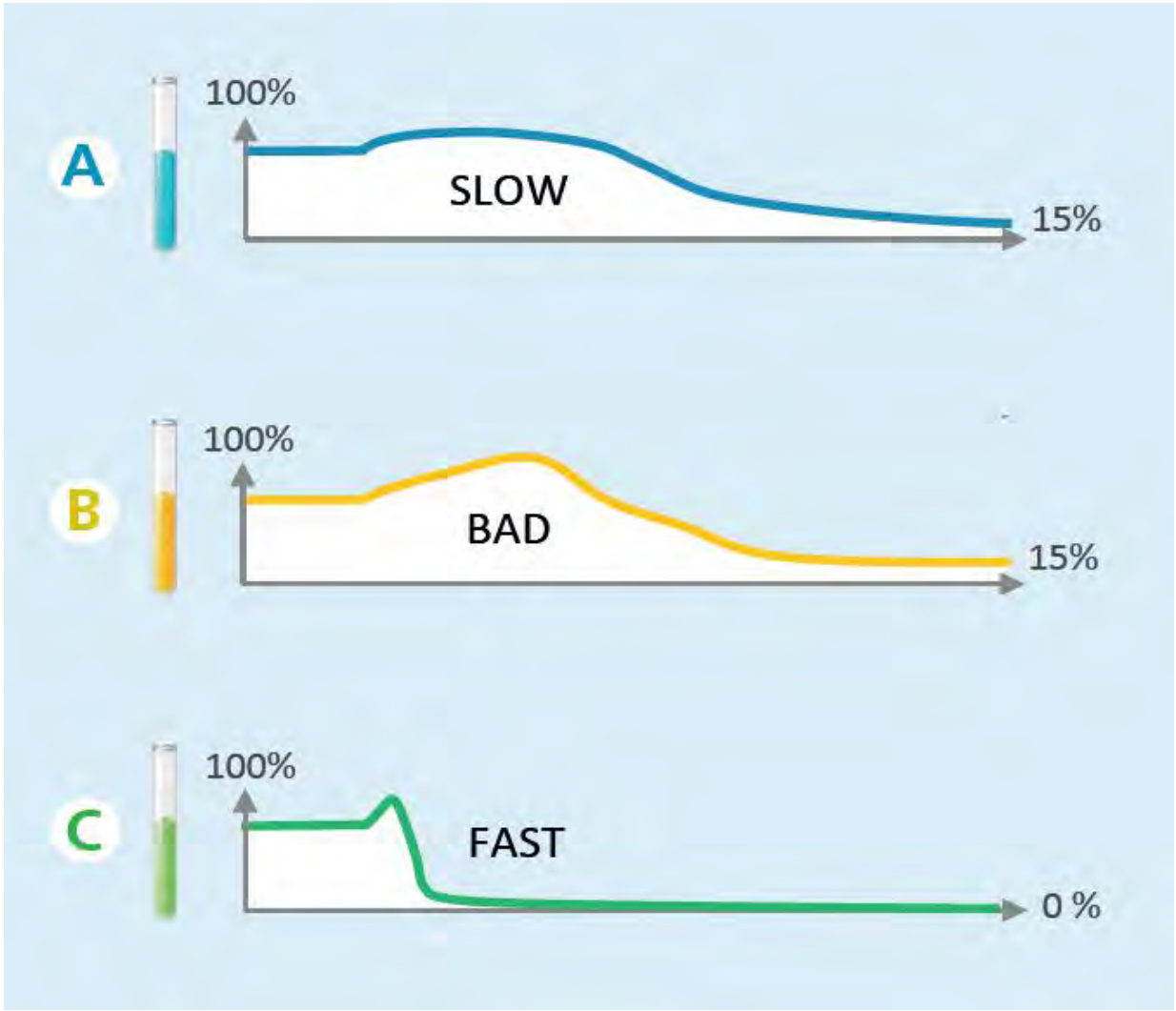
Nano scale soil

Compound flown over sensor swells soil

Soil dissolves and leaves surface

Final cleaning result

Comparing different cleaning profiles



Cleaning profile A: SLOW

- 15% of the soil remains
- Cleaning process still ongoing
- Longer wash cycle required

Cleaning profile B: BAD

- 15% of the soil remains
- Cleaning process inactive
- Bad final result

Cleaning profile C: FAST

- 100% of the soil removed
- Cleaning process inactive
- Shorter wash cycle or less efficient candidate possible

How well suited your candidate's cleaning profile is depends on the target wash cycle, the cost and efficiency targets for your specific detergent.

Comparing candidates in varying conditions

- Composition
- Concentration
- Temperature dependence
- Water quality
- Wash cycle time

- Pre-program and run up to 8 samples in one go using the Q-Sense Pro.
- Get high precision, real-time reproducible data

Evidence-based ranking of surfactants/formulations

Study by Biolin Scientific in collaboration with Center for Testmaterials BV(Netherlands)

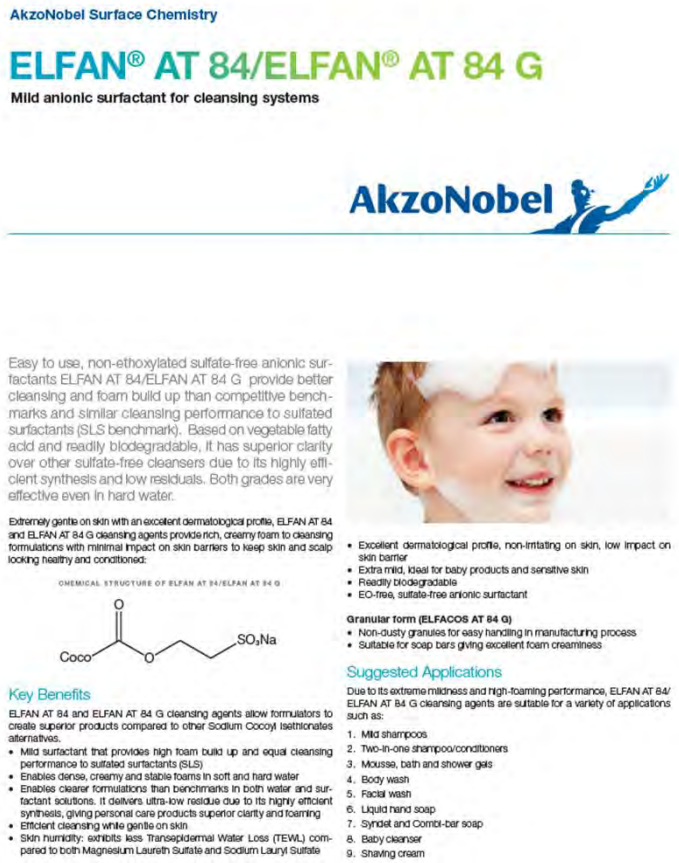
- Swell time, mass removal rate and total mass removal tested for 9 commerically available formulations
- Rapid screening and ranking of the formulations was easily achieved
- Significant correlation found when comparing to Center for Testmaterials 'macro scale' set-up

http://www.hanser-elibrary.com/doi/pdf/10.3139/113.110445

AkzoNobel – an application example

In Q2 2017 AkzoNobel revealed new results for their ELFAN AT 84 G surfactant based on QSense Cleaning Profile

- Helped differentiate products
- Used both during and after the development phase
- QSense Cleaning Profile added to the ELFAN product data sheet



AkzoNobel Surface Chemistry

ELFAN® AT 84/ELFAN® AT 84 G

Mild anionic surfactant for cleansing systems

AkzoNobel

Easy to use, non-ethoxylated sulfate-free anionic surfactants ELFAN AT 84/ELFAN AT 84 G provide better cleansing and foam build up than competitive benchmarks and similar cleansing performance to sulfated surfactants (SLS benchmark). Based on vegetable fatty acid and readily biodegradable, it has superior clarity over other sulfate-free cleansers due to its highly efficient synthesis and low residuals. Both grades are very effective even in hard water.

Extremely gentle on skin with an excellent dermatological profile, ELFAN AT 84 and ELFAN AT 84 G cleansing agents provide rich, creamy foam to cleansing formulations with minimal impact on skin barriers to keep skin and scalp looking healthy and conditioned:

CHEMICAL STRUCTURE OF ELFAN AT 84/ELFAN AT 84 G

Coco SO_3Na

Key Benefits

ELFAN AT 84 and ELFAN AT 84 G cleansing agents allow formulators to create superior products compared to other Sodium Cocoyl Isethionates alternatives.

- Mild surfactant that provides high foam build up and equal cleansing performance to sulfated surfactants (SLS)
- Enables dense, creamy and stable foams in soft and hard water
- Enables clearer formulations than benchmarks in both water and surfactant solutions. It delivers ultra-low residue due to its highly efficient synthesis, giving personal care products superior clarity and foaming
- Efficient cleansing while gentle on skin
- Skin humidity: exhibits less Transepidermal Water Loss (TEWL) compared to both Magnesium Laureth Sulfate and Sodium Lauryl Sulfate

- Excellent dermatological profile, non-irritating on skin, low impact on skin barrier
- Extra mild, ideal for baby products and sensitive skin
- Readily biodegradable
- EO-free, sulfate-free anionic surfactant

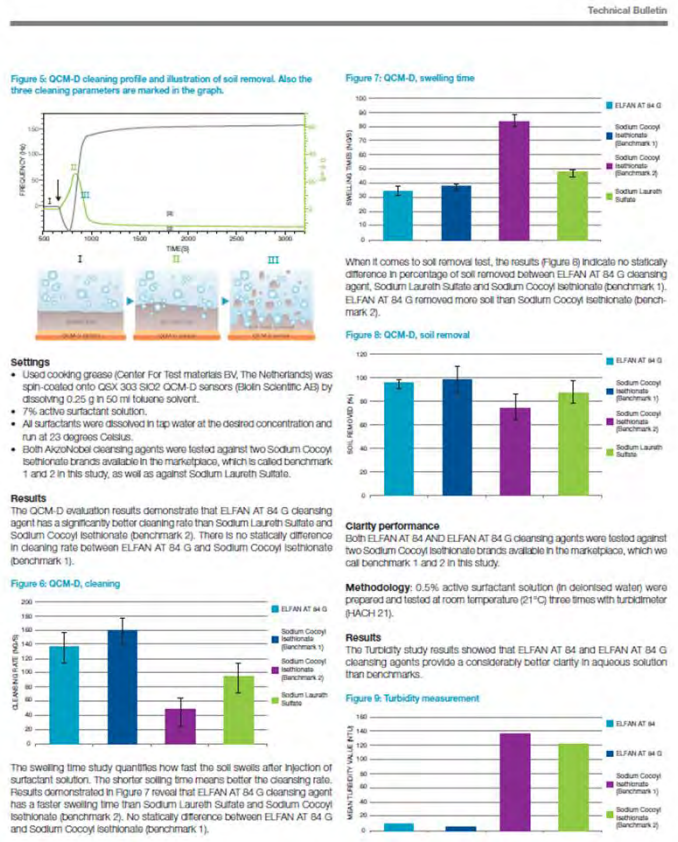
Granular form (ELFACOS AT 84 G)

- Non-dusty granules for easy handling in manufacturing process
- Suitable for soap bars giving excellent foam creaminess

Suggested Applications

Due to its extreme mildness and high-foaming performance, ELFAN AT 84/ELFAN AT 84 G cleansing agents are suitable for a variety of applications such as:

1. Mild shampoos
2. Two-in-one shampoo/conditioners
3. Mousse, bath and shower gels
4. Body wash
5. Facial wash
6. Liquid hand soap
7. Syndet and Combi-bar soap
8. Baby cleanser
9. Shaving cream



Technical Bulletin

Figure 5: QCM-D cleaning profile and illustration of soil removal. Also the three cleaning parameters are marked in the graph.

Figure 7: QCM-D, swelling time

When it comes to soil removal test, the results (Figure 8) indicate no statically difference in percentage of soil removed between ELFAN AT 84 G cleansing agent, Sodium Laureth Sulfate and Sodium Cocoyl Isethionate (benchmark 1). ELFAN AT 84 G removed more soil than Sodium Cocoyl Isethionate (benchmark 2).

Figure 8: QCM-D, soil removal

Settings

- Used cooking grease (Center For Test materials BV, The Netherlands) was spin-coated onto QSX 303 SiO2 QCM-D sensors (Biolin Scientific AB) by dissolving 0.25 g in 50 ml toluene solvent.
- 7% active surfactant solution.
- All surfactants were dissolved in tap water at the desired concentration and run at 23 degrees Celsius.
- Both AkzoNobel cleansing agents were tested against two Sodium Cocoyl Isethionate brands available in the marketplace, which is called benchmark 1 and 2 in this study, as well as against Sodium Laureth Sulfate.

Results

The QCM-D evaluation results demonstrate that ELFAN AT 84 G cleansing agent has a significantly better cleaning rate than Sodium Laureth Sulfate and Sodium Cocoyl Isethionate (benchmark 2). There is no statically difference in cleaning rate between ELFAN AT 84 G and Sodium Cocoyl Isethionate (benchmark 1).

Figure 6: QCM-D, cleaning

The swelling time study quantifies how fast the soil swells after injection of surfactant solution. The shorter soiling time means better the cleansing rate. Results demonstrated in Figure 7 reveal that ELFAN AT 84 G cleansing agent has a faster swelling time than Sodium Laureth Sulfate and Sodium Cocoyl Isethionate (benchmark 2). No statically difference between ELFAN AT 84 G and Sodium Cocoyl Isethionate (benchmark 1).

Clarity performance

Both ELFAN AT 84 AND ELFAN AT 84 G cleansing agents were tested against two Sodium Cocoyl Isethionate brands available in the marketplace, which we call benchmark 1 and 2 in this study.

Methodology: 0.5% active surfactant solution (in deionised water) were prepared and tested at room temperature (21°C) three times with turbidimeter (HACH 21).

Results

The Turbidity study results showed that ELFAN AT 84 and ELFAN AT 84 G cleansing agents provide a considerably better clarity in aqueous solution than benchmarks.

Figure 9: Turbidity measurement

Develop cleaning formulations with low glass etching

§ Challenge

To rapidly determine glass etching instead of performing between 100-1000 dishwashing cycles.

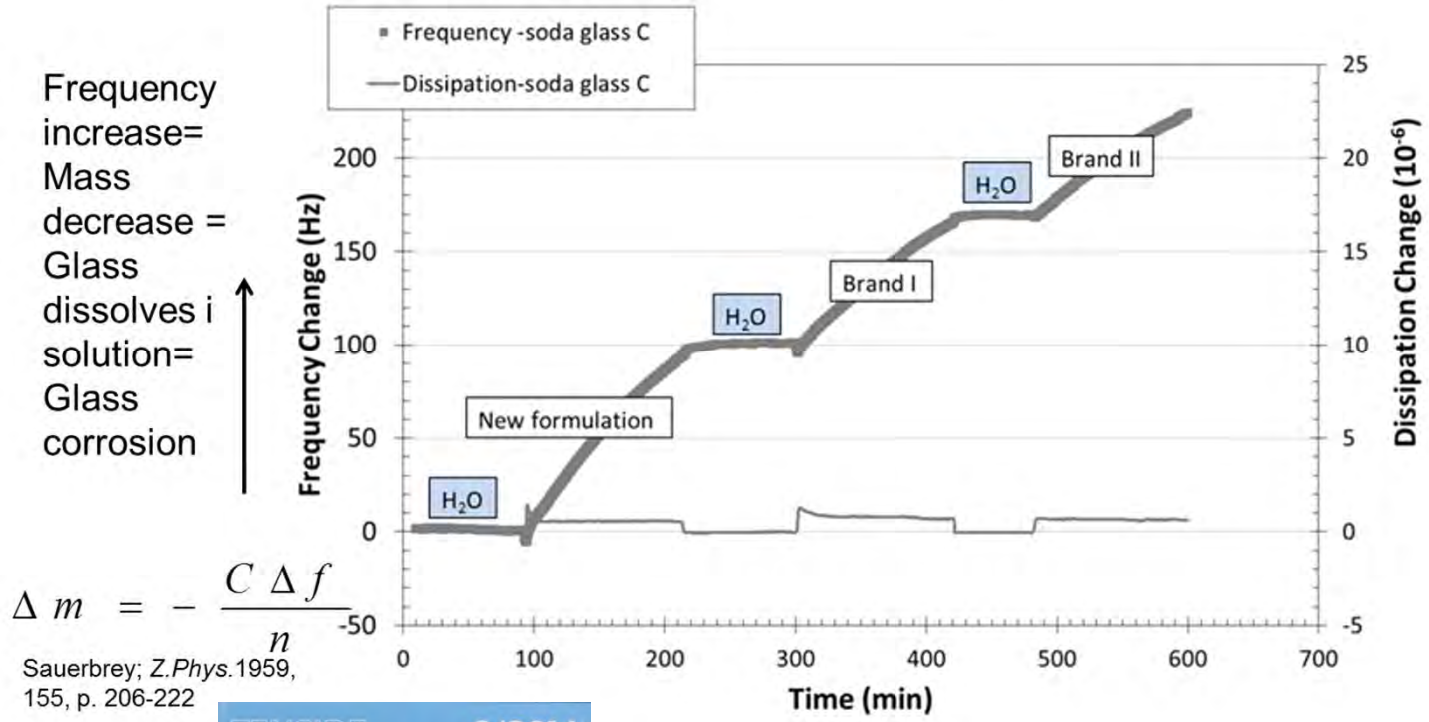
§ Solution

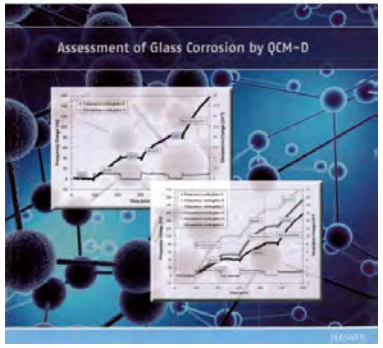
Use the QCM-D, which has the ability to measure glass removal of nm thicknesses. Four formulations can be evaluated in parallel. Different glass types can be used.

§ Results

The etching effect of the formulation is completed in 1 hour and enables the screening of many formulations. The best formulations can then be evaluated in full scale at GLAFO (Swedish glass research institute).

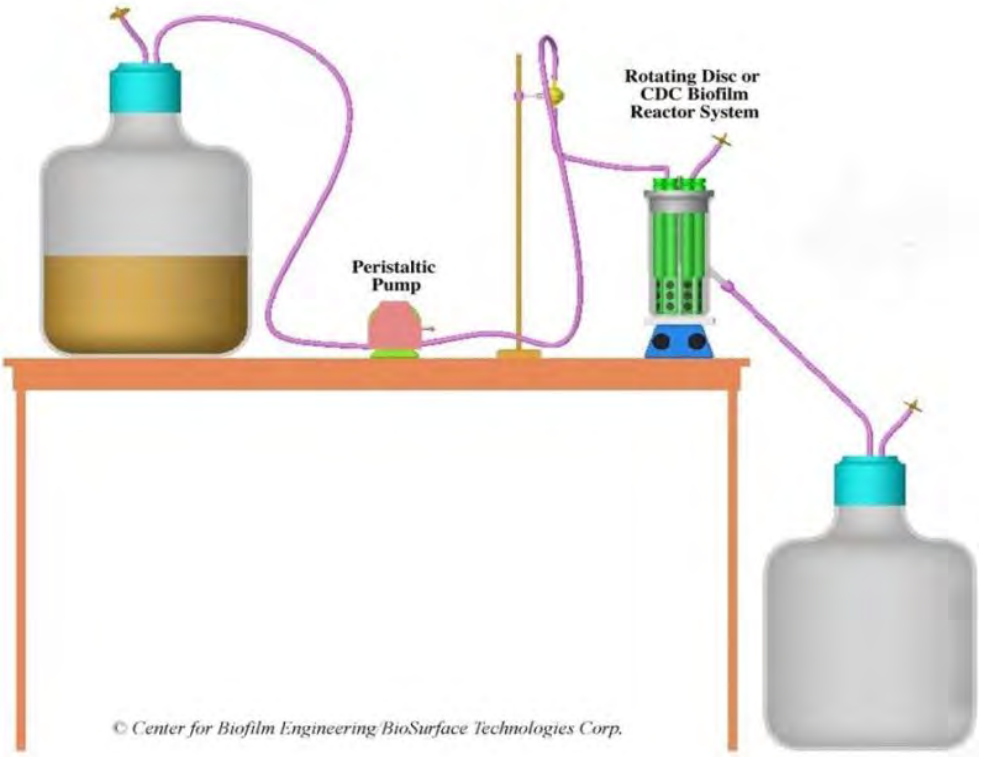
Macakova et al, Tenside, Surfactants, Detergents(2014), 51(6), 484,486-490

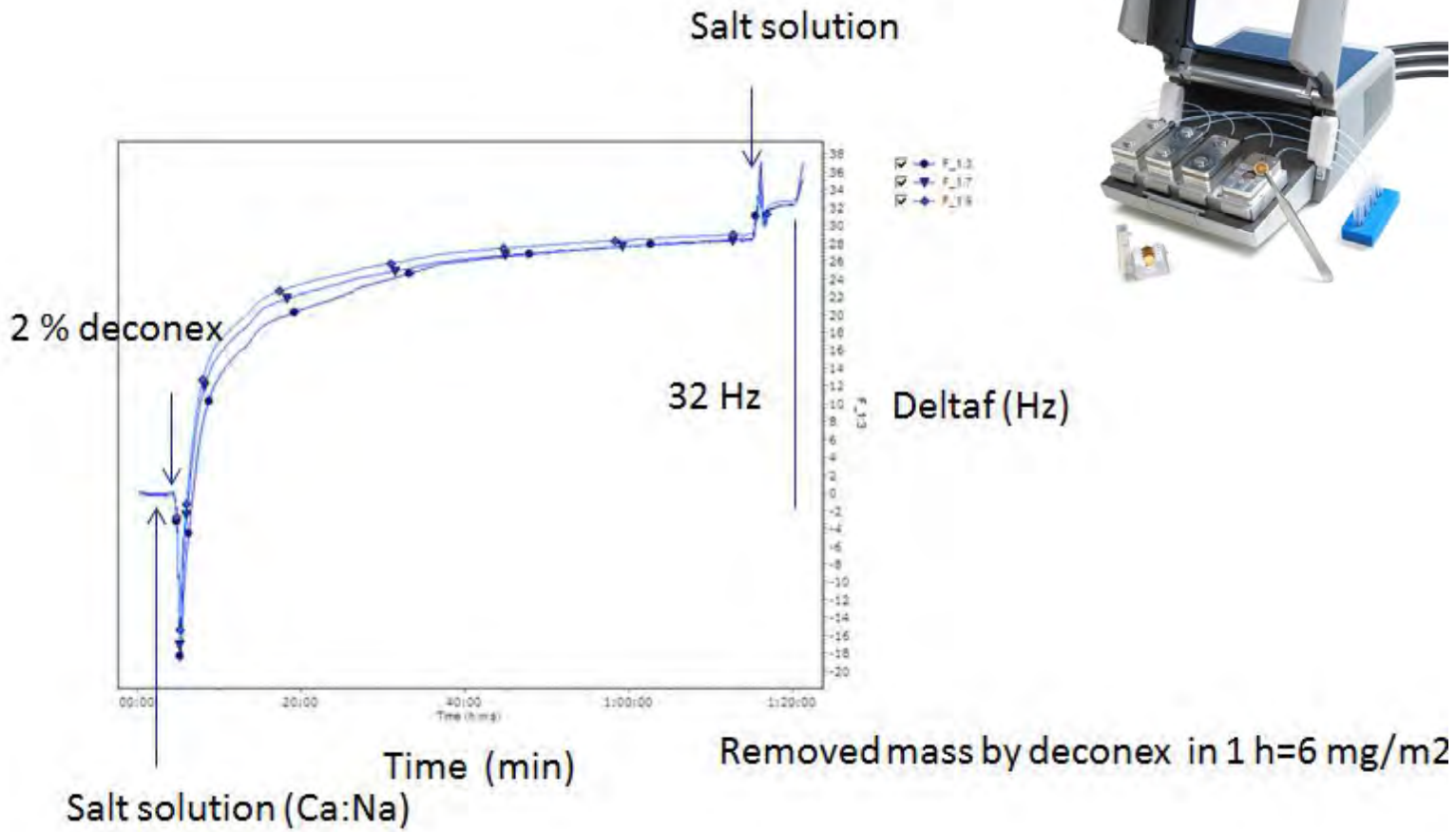




Follow desorption of biofilm/desinfection by QCMD technique

CBR 90 CDC Biofilm reactor **- Controlled growth of biofilm (temperature and flow)**





Measured parameters:

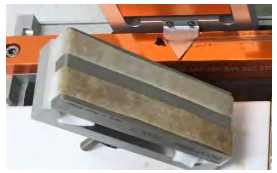
Resonance frequency, Deltaf (Hz) - (increase = desorption)

Osc.energy dissipation (low – stiff layer, high – fluffy, viscoelastic layer) (not shown in picture above)

Measuring cleaning effect of different ingredients

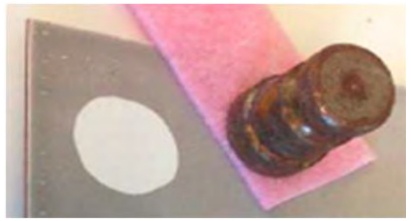
Methods

Elcometer 1720



Drop equilibration

Drop is allowed to equilibrate on soil surface and wiped off with cloth and weight at selected time for optimum result.



Materials

Surfactants
#1 2% Nonionic C10-AGP1
#2 2% Nonionic alcohol ethoxylate
#3 2% Nonionic C10-APG2
#4 2% Anionic ethyleneoxide sulfonate
#5 2% Nonionic C14-APG
#6 2% Sodium Dodecyl Sulfate

Builder/complexing agent
B: 1 wt% Sodium citrate
C: 1 wt% Potassium carbonate
D: 1 wt% Sodium silicate
E: 1 wt% Strong complexing agent

pH
pH=8
pH=11
pH=13

Surfaces

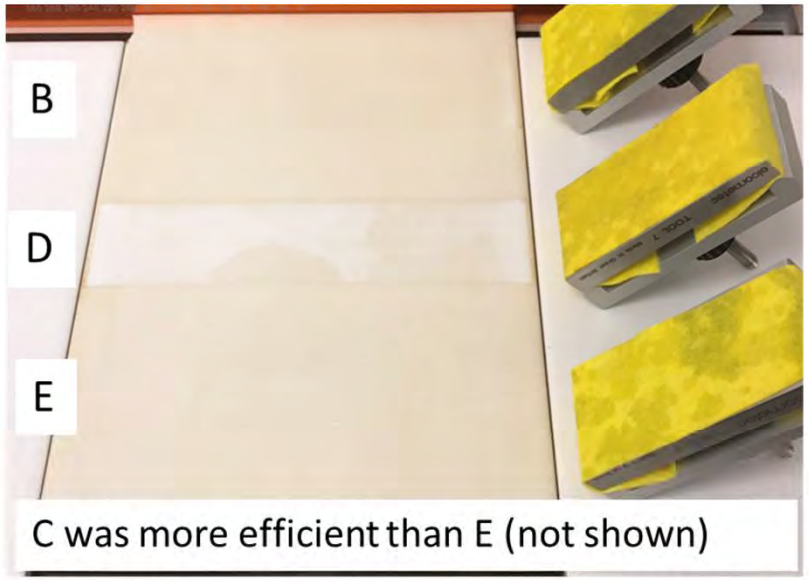
In-house prepared wood-tar surfaces on ceramic tile

- Amount of tar, oven baking time, drying time can be varied for optimum result.

Surfaces from Center for Testmaterials (CFT)

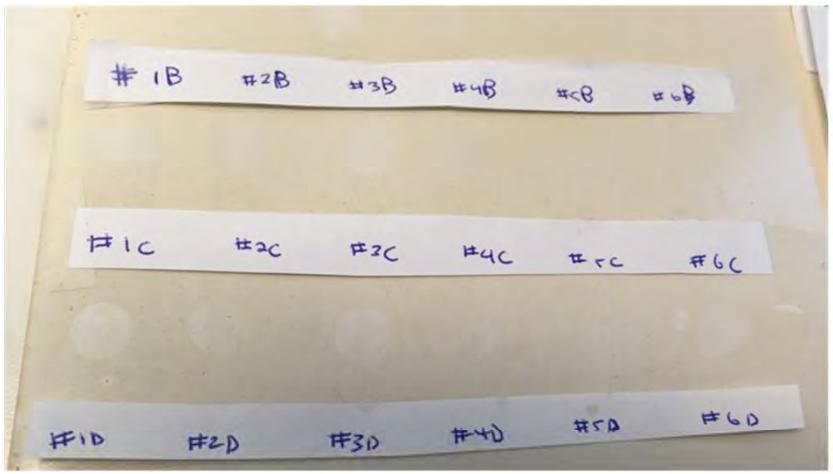
- DM 40 All-purpose cleaner soil, on melamine tile
- DM 80 All purpose cleaning soil on Melamine
- DS 50 Corn Oil on Stainless extra smooth steel
- DS 80 All purpose cleaning soil on Stainless Steel

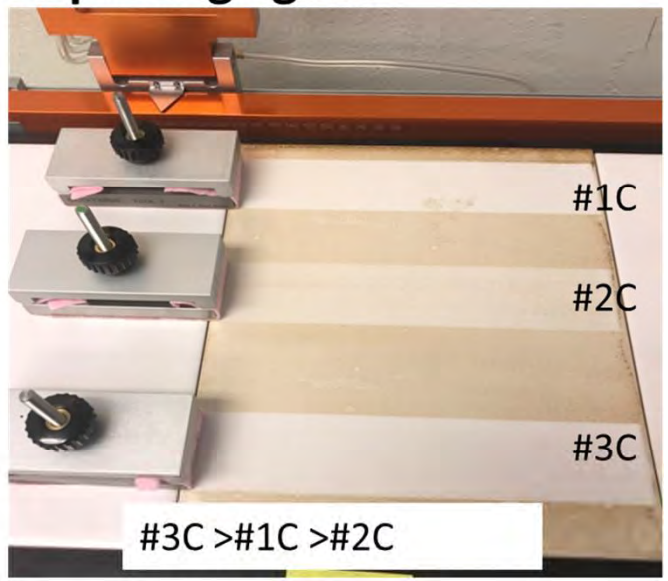
Effect of complexing agent alone, pH=13



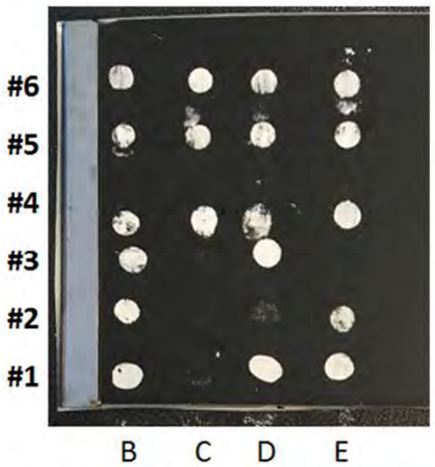
C was more efficient than E (not shown)

Effect of surfactant+complexing agent

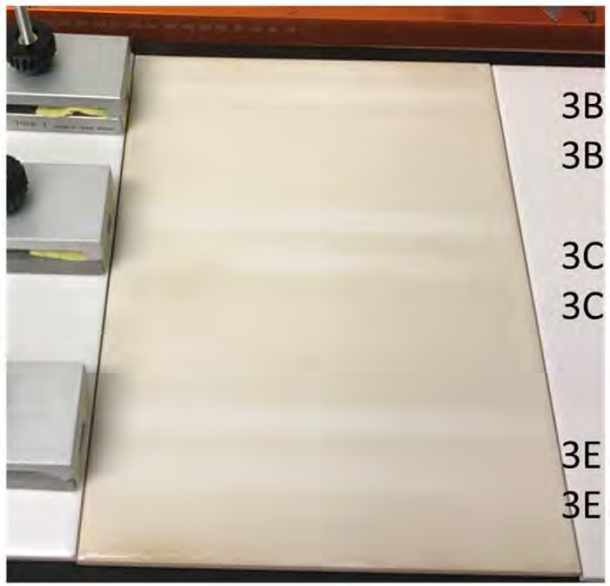




DM40, pH=13, t=30 min



pH effect

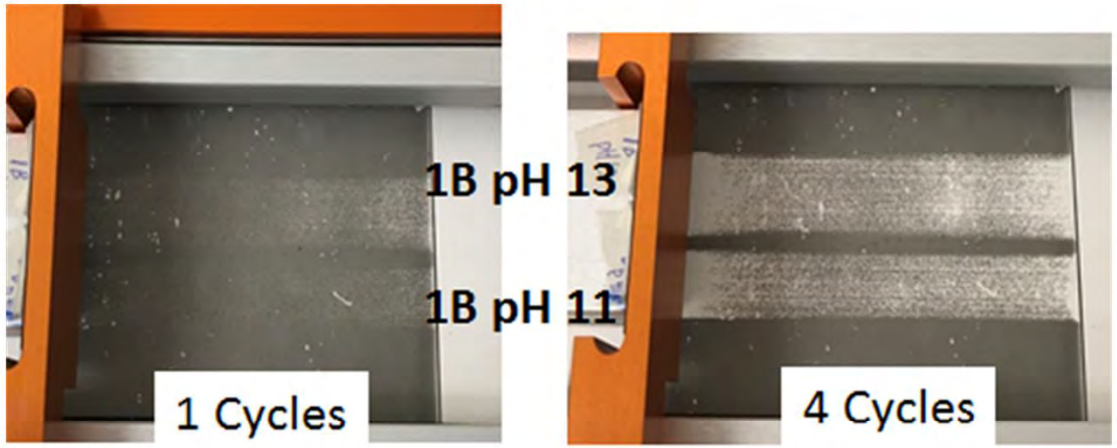


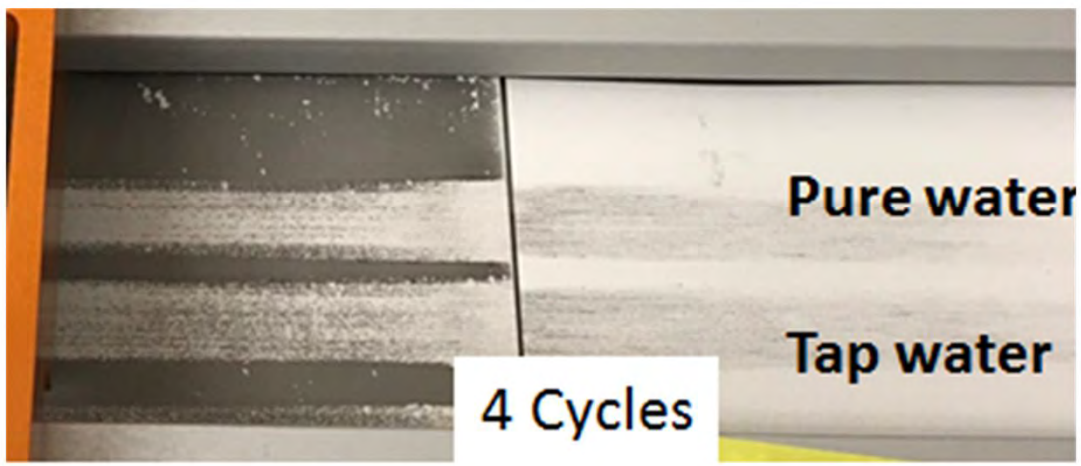
- Sodium silicate was most effective complex builder on tar surface.
- Surfactants #4-#6 showed best effect with all complex builders on DM40 surface
- #1 was very efficient with B, D, and E on DM40.
- pH13 works better than pH11 on DM40 and tar surface.

DS 80, all purpose soil

DS 50, corn oil

DM 80, all purpose soil

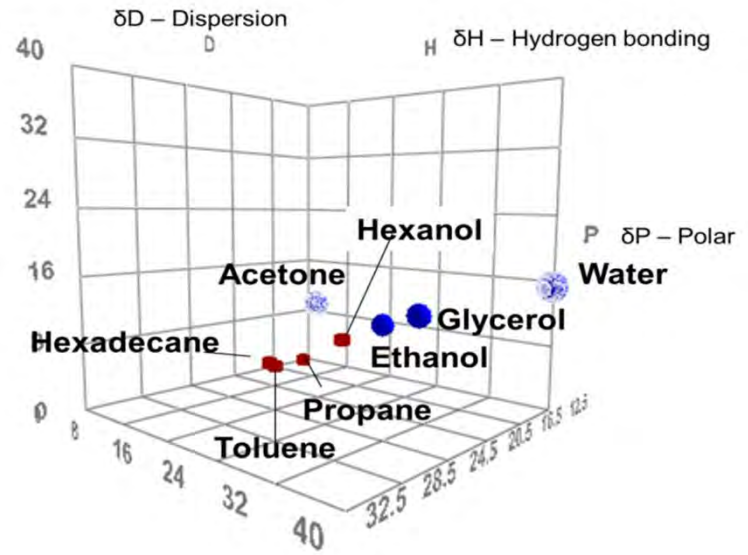




Mechanical abrasion is (too) significant for DM80. Note the tracks on the backing tray, which is not seen for surfactant solution.
Surfaces from CFT can efficiently by used in Elcometer 1720.

A Hansen solubility parameter approach to predict solubility and interaction of emollients, actives, polymers, solvents and skin

- All solvents, polymers, tissue and active ingredients have three Hansen solubility parameters
- HSPiP software and database
  - 1200+ solvents, 600+ polymers and 300 emollients in the database
  - Calculate HSP for emollient blends and for the full external phase in a formulation
  - Calculate HSP for unknown molecules or active ingredients using SMILES input
  - Calculate HSP for surfaces/skin (or use default values)
- Find which emollient mix / cream recipe is most compatible with the active
- Predict skin interactions of emollient mixes, actives and full formulation recipes
- Optimize recipe for maximum/minimum skin interaction and uptake



- δD – Dispersion
- δH – Hydrogen bonding
- δP – Polar

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AceForm 4.0

Value Chains in Formulation Manufacturing

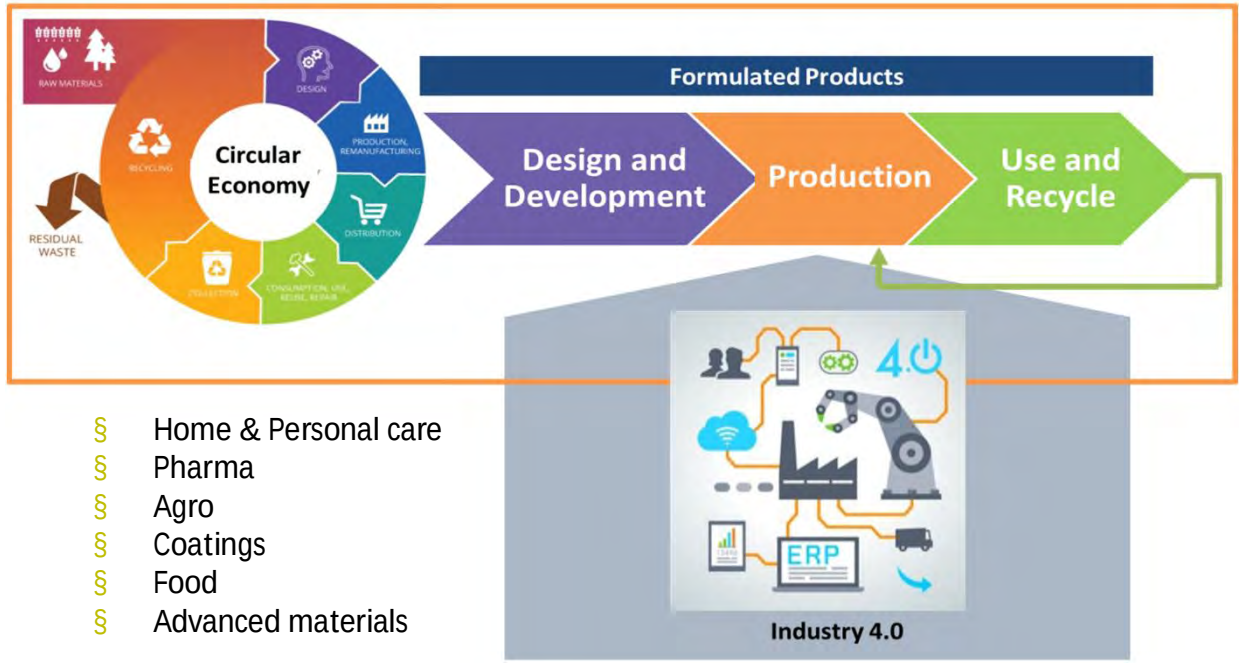
Horizon 2020 Coordination and Support Action (CSA) project
Oct 2016- Oct 2018

Objectives

- Establish a European Formulation Interest Group (EU-FIG)
- Identify common technical and industrial challenges for the European Formulated Products Industries
- Establish a common vision and roadmap
- Arrange knowledge exchange activities and facilitate new initiatives along the value chain

Key project output

- Influence the content of future EU calls
- EU-FIG community with >500 organizations
- > 10 new collaborative cross-sectorial initiatives



- Home & Personal care
- Pharma
- Agro
- Coatings
- Food
- Advanced materials

For more information visit the website at:
www.formulation-network.eu

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THANK YOU!

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Research Institutes of Sweden

Bioscience and Materials
Surface, Process and Formulation