engineering for a better world

Formation of powders – from single particles to full scale

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FORMULA X, Manchester, June 24-27 2019

Outline

- RISE and GEA Process Technology
- Spray-drying
- Particle formation – internal and external structure
- Levitated particle dring vs spray-drying
- Modelling and use of levitated particle drying data
- Spray-freeze-drying vs spray drying

RISE in brief

- Governmentally owned, not-for-profit research institute
- Present across the whole of Sweden.
- 2,700 employees, 30 % with a PhD.
- Turnover approx. SEK 3 billion (2018).
- Research and innovation in collaborative projects, and confidential industrial projects
- A large proportion of customers are SME clients, accounting for approx. 30 % industry turnover.
- Runs 100s of test and demonstration facilities, open for industry, SMEs, universities and institutes

With our broad range of competencies and unique expertise, we create added value

Bioeconomy	Fire and safety	Cement and concrete	Certification	Circular economy
Design	Electronics	Energy and fuels	Packaging	Glass
Health and Care	ICT and telecoms	Agriculture and food	Chemistry, materials and surfaces	Life Science
Maritime	Mechanical engineering	Mechanics	Metrology and measurement technology	Paper and Pulp
Process development	Built environment	Safety	Mobility	Wood
Water	Production	Corrosion	Work environment	Composites
Manufacturing processes	Metals	Additive manufacturing	Casting	Textiles

engineering for
a better world

- **GEA Group**
  - German company
  - 18.500 employees
  - Process technology equipment supplier
    - Production plants solutions
    - All industries: Food, Dairy, Pharma and Chemical
  - GEA Søborg (Denmark)
    - 650 employees
    - Global Technology Center for Drying & Powder Processing
    - Core technolgies
      - Spray drying, freeze drying, solid feed drying
      - Powder transport and powder packaging

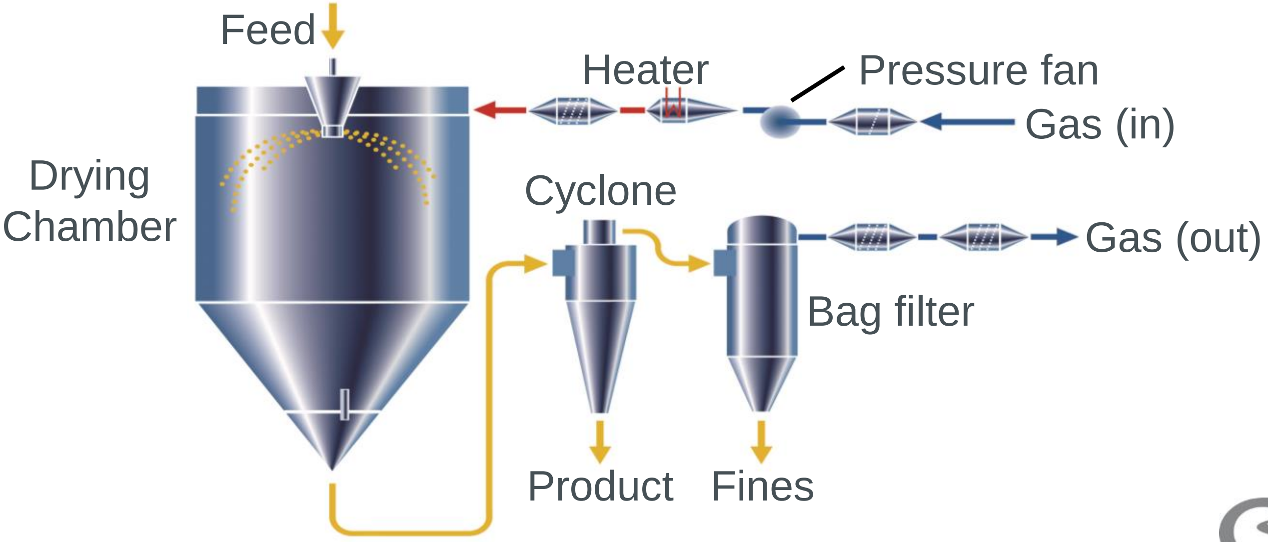
Powders

- Ubiquitous as ingredients and end products
- >80% of all products were a powder at some point
- Used in all types of industries
- Manufacturing methods
  - Spray-drying
  - Freeze-drying
  - Spray-freeze drying
  - Crystallisation
  - Milling
  - …

Spray Drying

- Spray drying
  - Conversion of a solution or a suspension into a dry powder product

1. Impact of drying on structure and functionality
2. Scale up



Scale up

The DRYING KINETICS ANALYZER™ (DKA)

The DRYING KINETICS ANALYZER™

– Process conditions

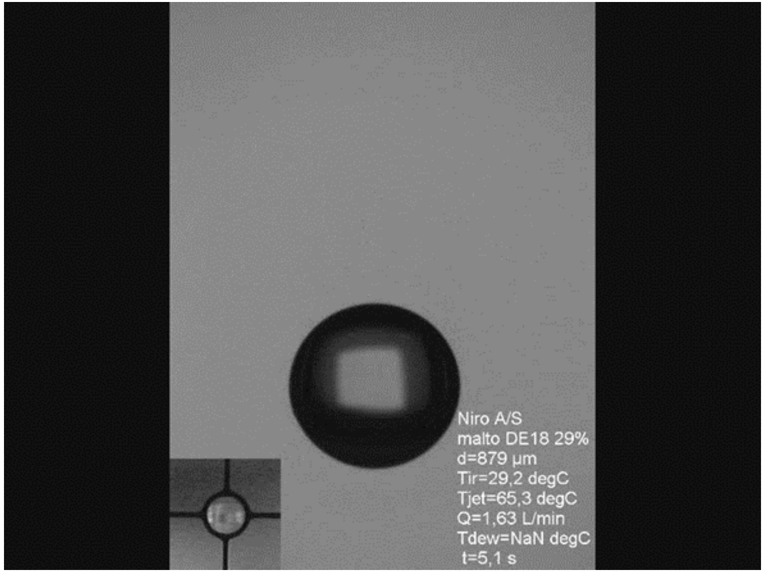
- Temperature 20-105°C
- Relative humidity 0-95%
- Droplet size 50-2000 µm
  - Initial droplet size usually 800-900 µm

– Feed materials

- Any compound
- Any solvent
- 25 ml feed required

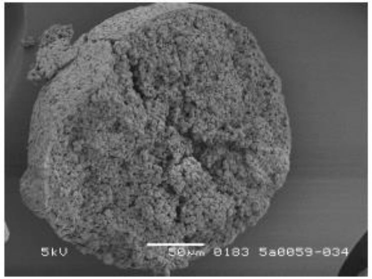
The DRYING KINETICS ANALYZER™

- Drying of maltodextrin DE18
  - T_{air} = 65°C
  - RH = 0 %

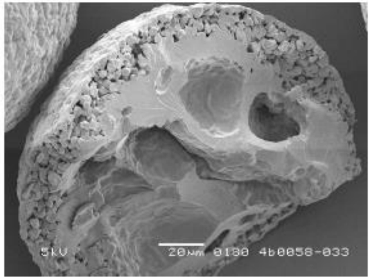


Structure and Functionality

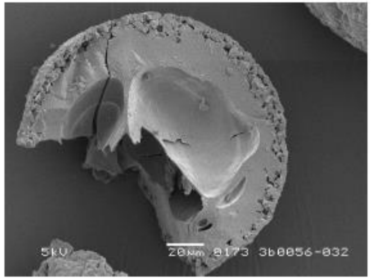
- Effect of drying gas properties
  - E.g. temperature
  - Rice starch dried at different temperatures



150°C



200°C

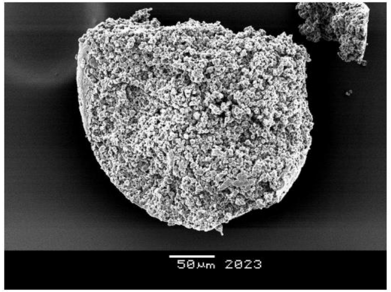


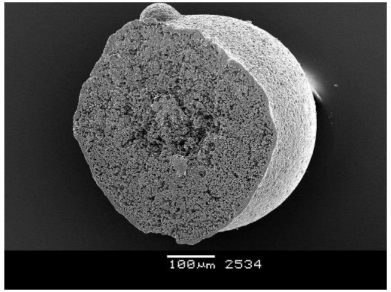
250°C

Structure and Functionality

- Effect of feed formulation
  - Small change in the formulation can give great morphology variation
  - Rice starch and different amounts of maltodextrin

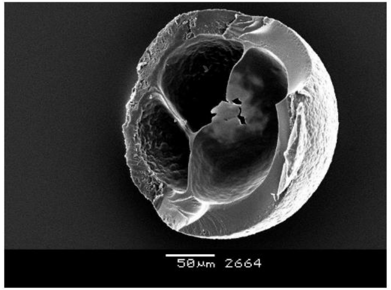
0 wt%

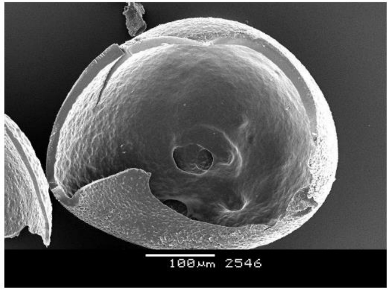




0.51 wt%

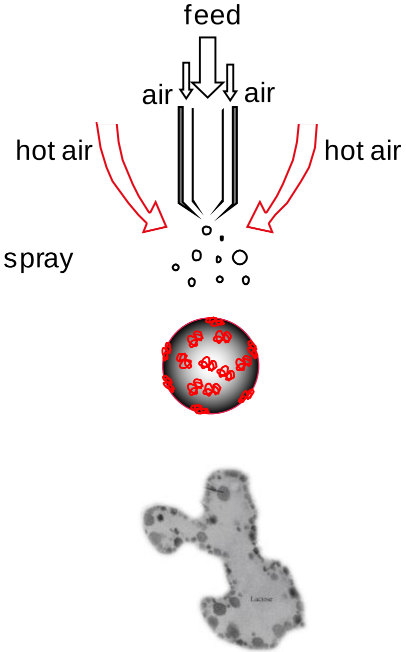
3.24 wt%

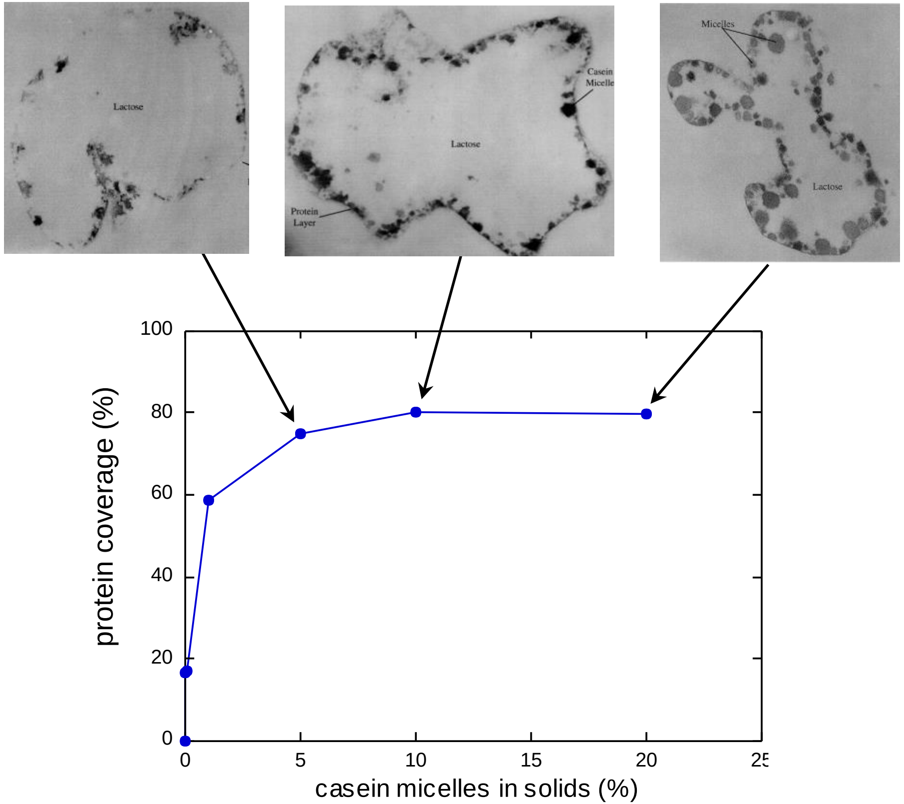




6.54 wt%

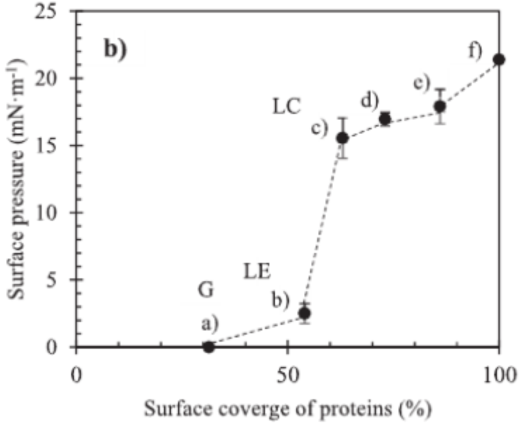
Particle structure formation in spray-drying

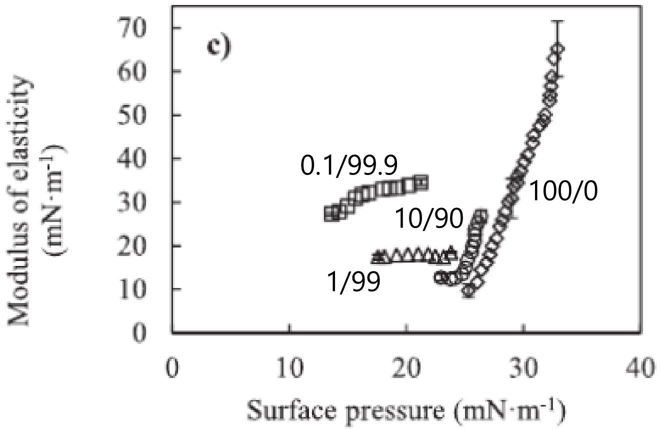


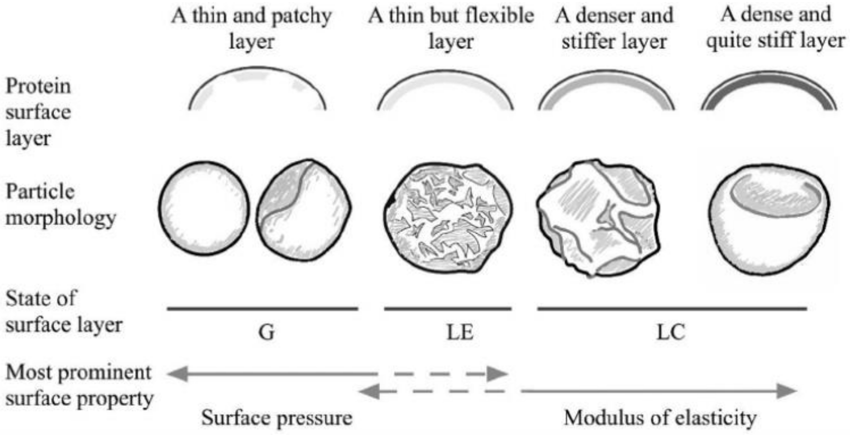


Particle formation Morphology

- Milk serum proteins / lactose
- Spray-dried
- Protein adsorption to surface
- Surface rheology of surface layer
- Packing of protein influences the stiffness of the surface layer and hence morphology

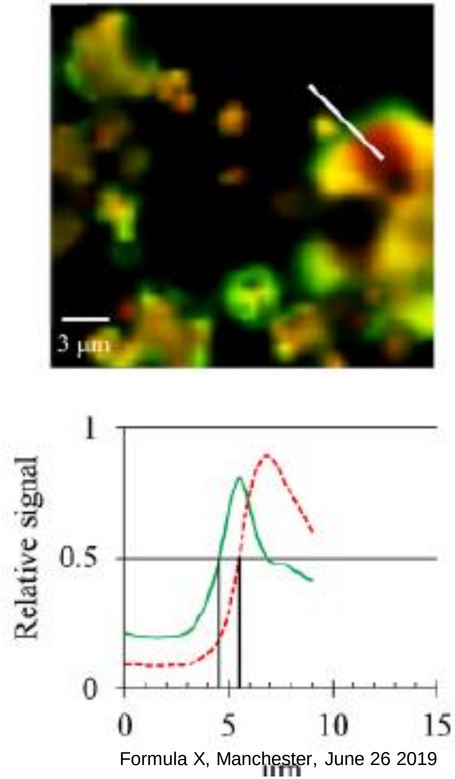


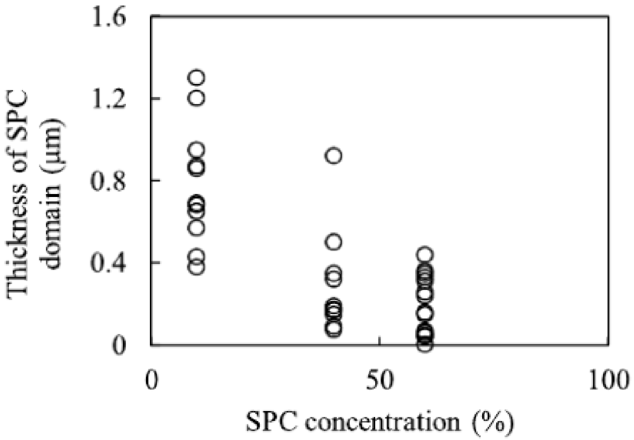


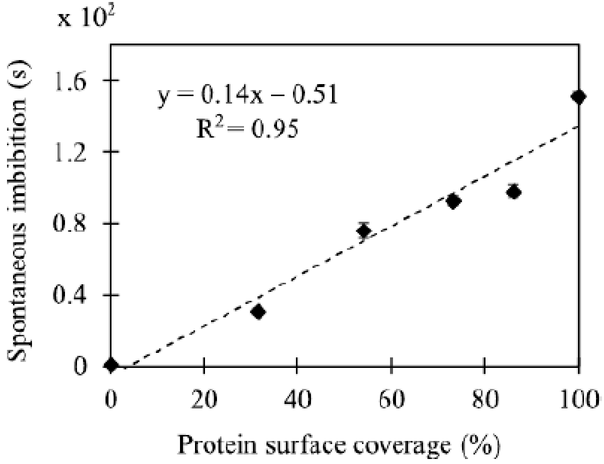


Andersson et al, 2018, Coll Surf A561, 395-404

Surface composition in spray-dried whey protein powder, relation to wetting





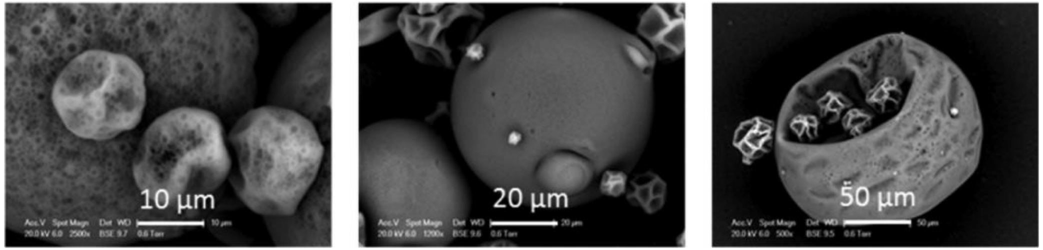


Andersson et al, 2018, Coll Surf A561, 395-404
Andersson et al, 2018, Int Dairy J, 85, 86-95

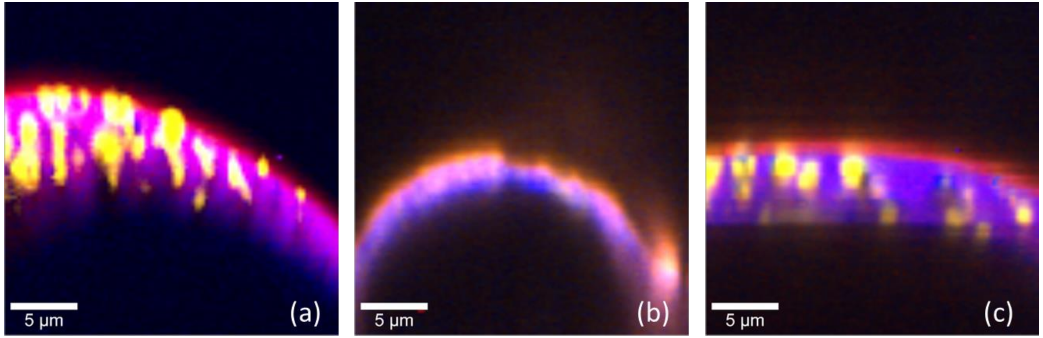
Particle surface formation and internal structure – Spray dried emulsion

Emulsion:	1 µm droplets	0-1-0.2 µm droplets	1 µm droplets
Atomisation:	High shear	High shear	Low shear

Low vacuum SEM
Oil droplets in matrix



Confocal raman microscopy
Phase segregation in matrix



Sunflower oil   Acacia gum   Maltodextrin

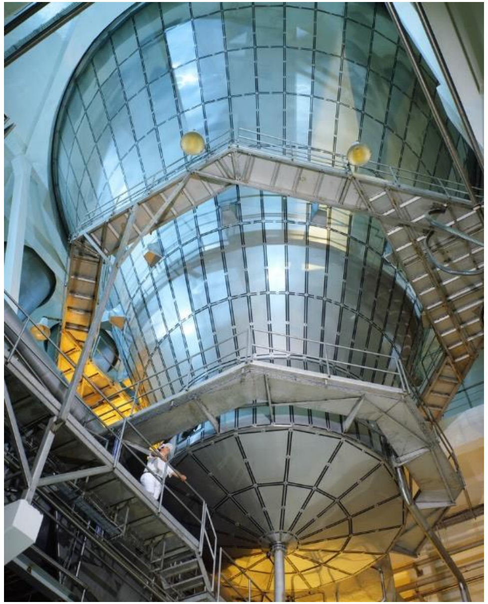
Munoz et al, Food Structure, 2016, 8, pp.16-24

Investigating whole milk powder particles

Single Particle

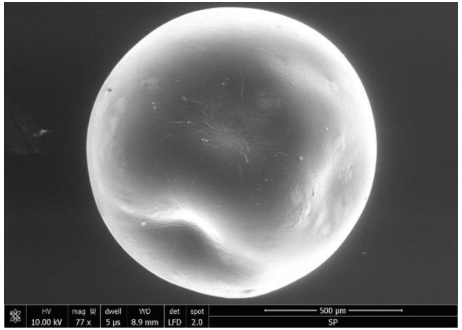
Pilot

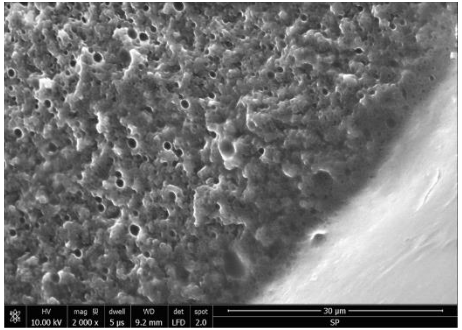
Full Scale



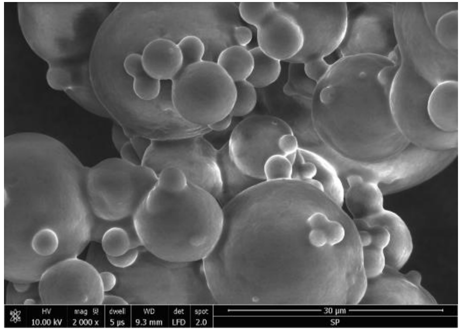
LV-SEM

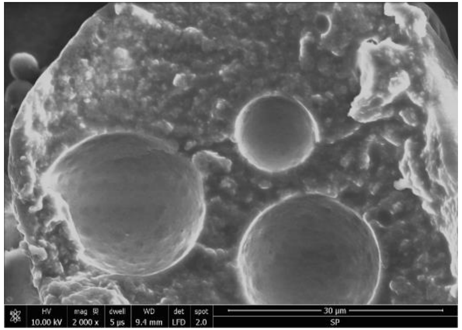
Single Particle



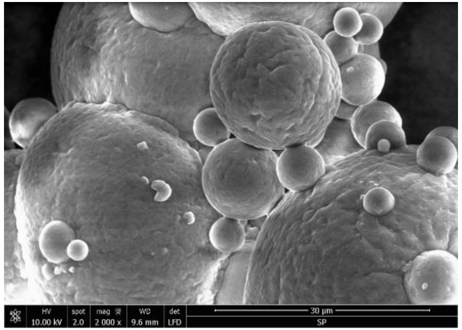


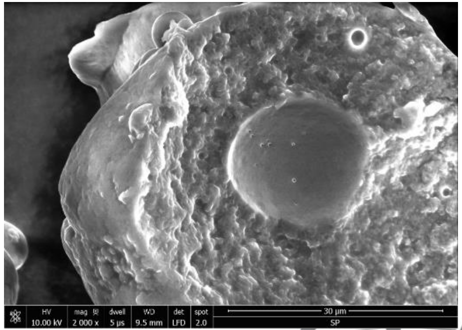
Pilot





Full Scale



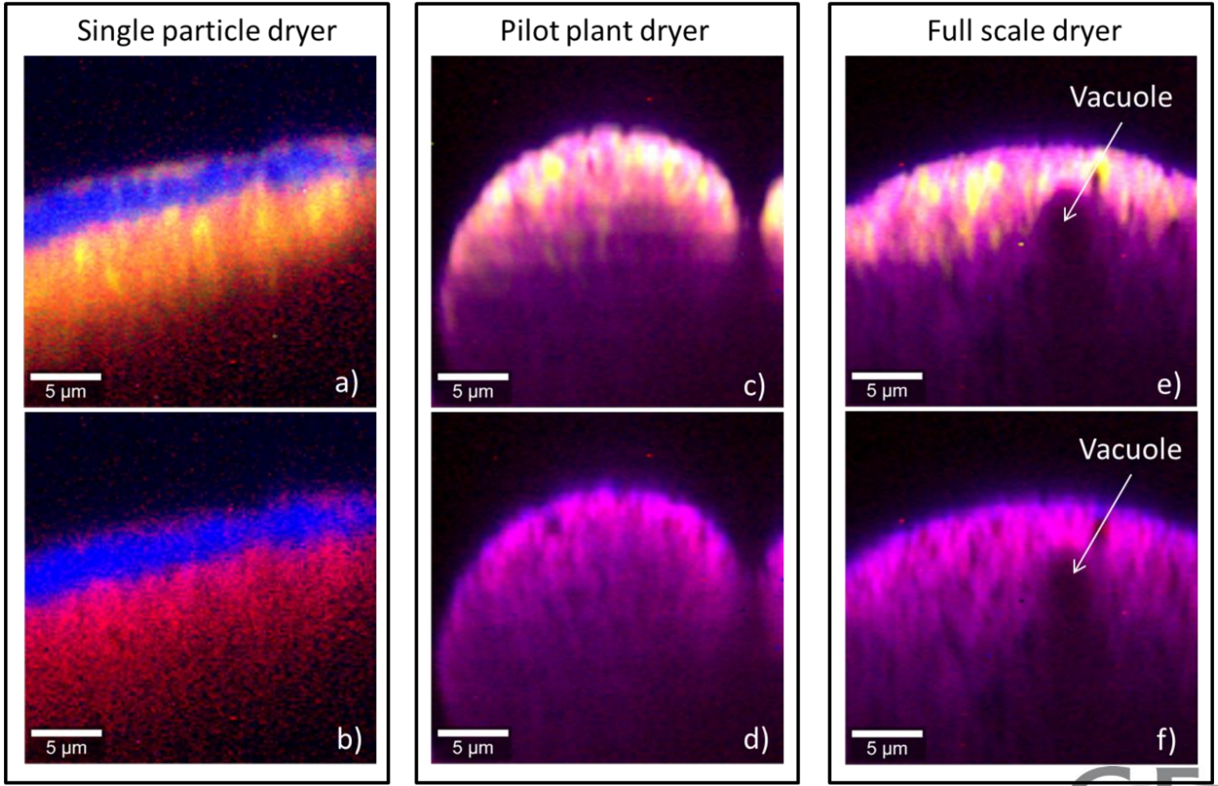


Nuzzo et al, Food Structure, 2017

Confocal Raman Images

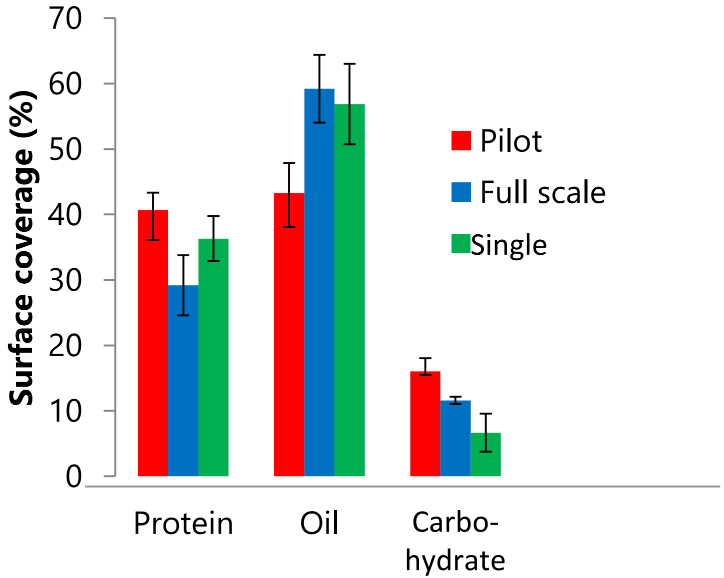
- Red (Protein)
- Blue (Carbohydrate)
- Yellow (Oil)

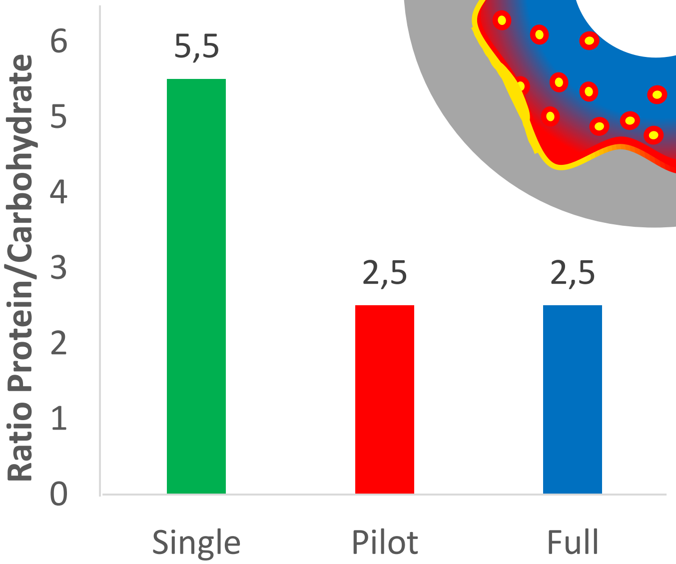
- Without oil

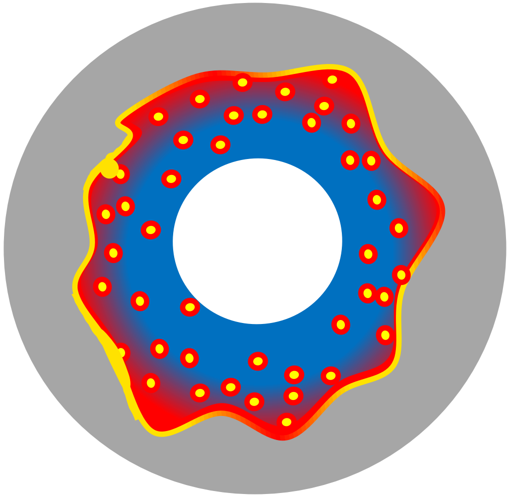


Nuzzo et al, Food Structure, 2017

Surface composition



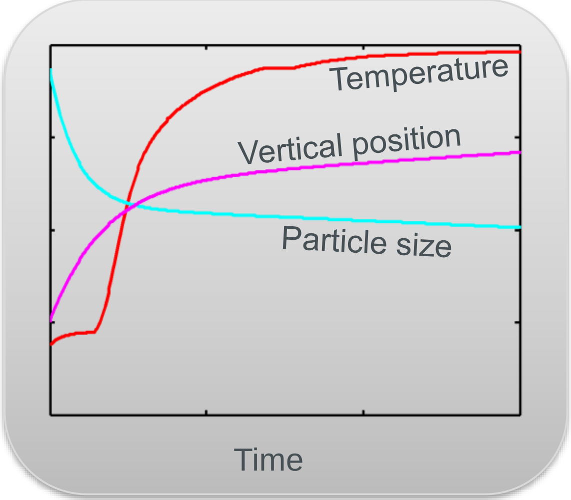




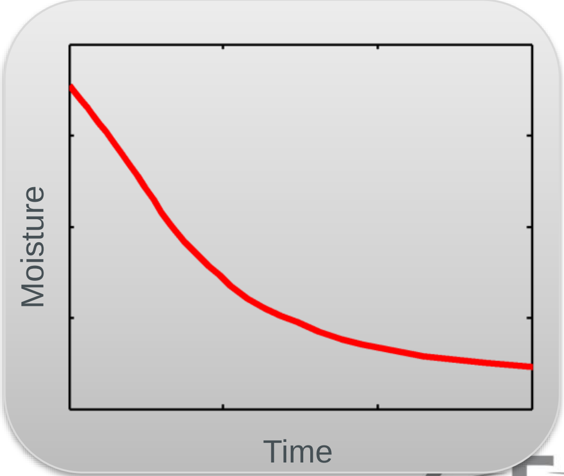
Nuzzo et al, Food Structure, 2017

Coupling DKA and CFD

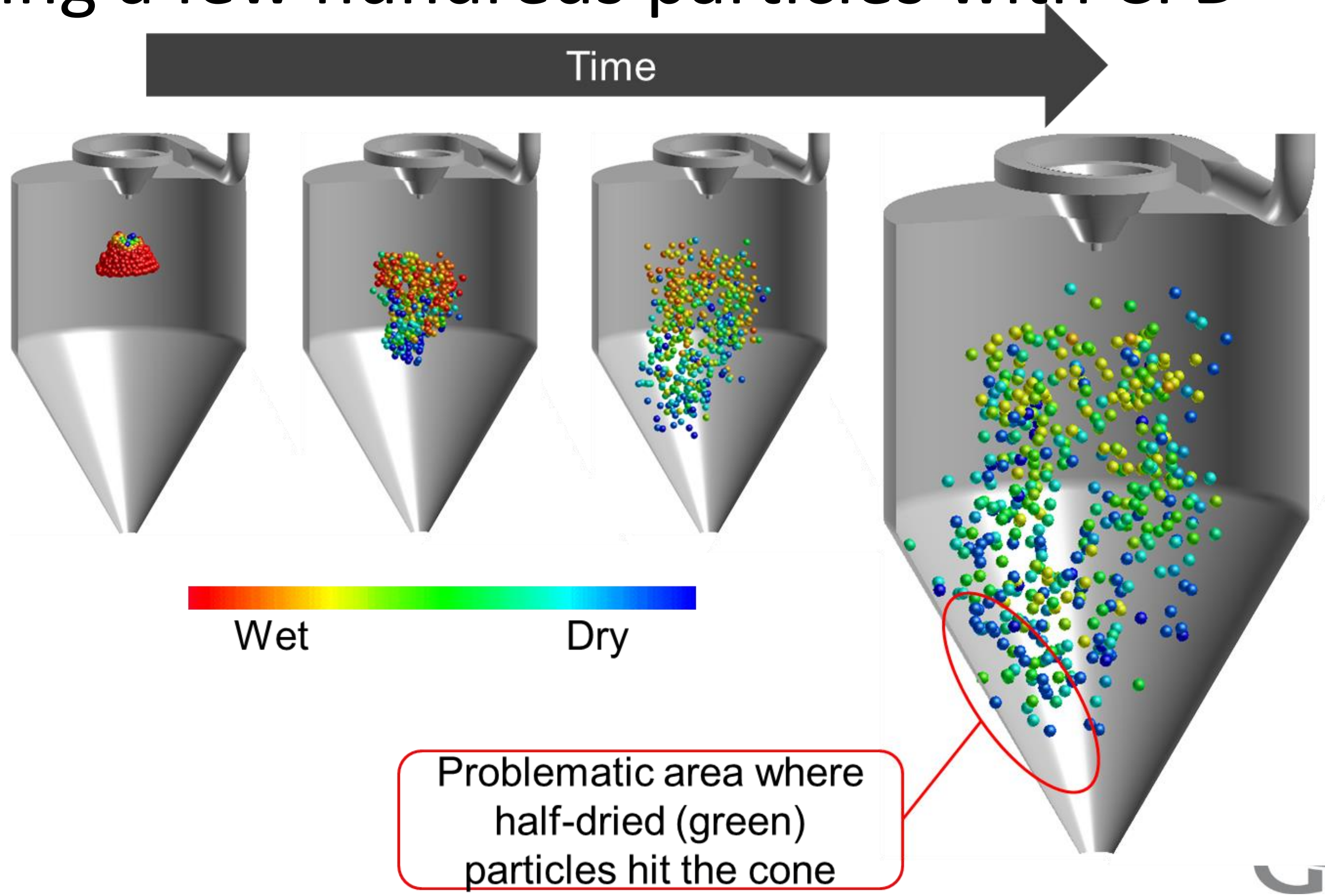
- The droplet drying process is implemented in Computational Fluid Dynamics (CFD) simulations
- Every product has unique drying properties
- Measured with the DKA



Modeling

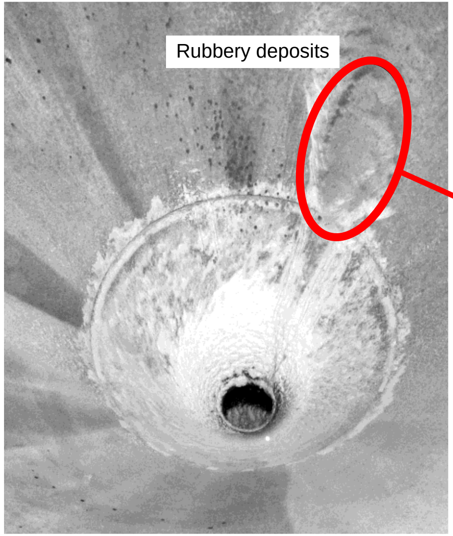


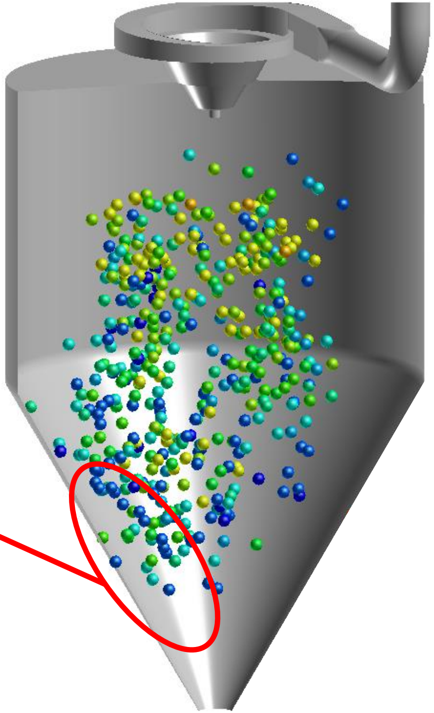
Tracking a few hundreds particles with CFD



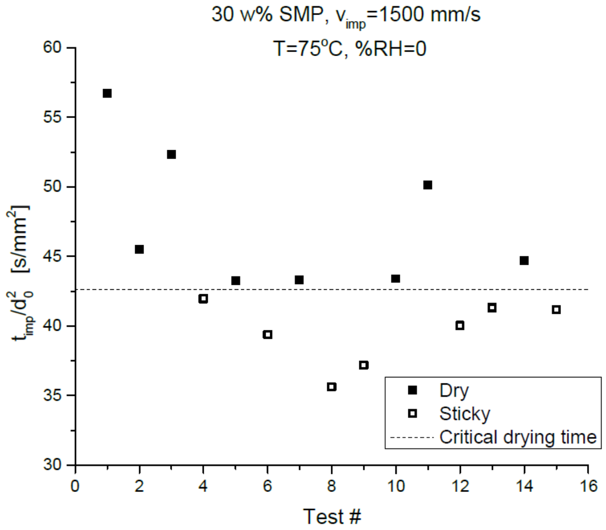
Experimental Validation

- Full scale experiments show the formation of deposits predicted by the CFD-simulations



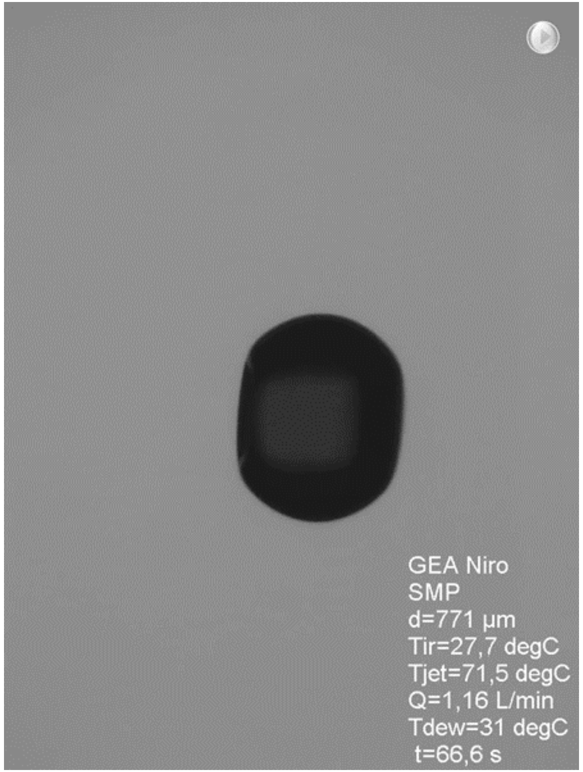


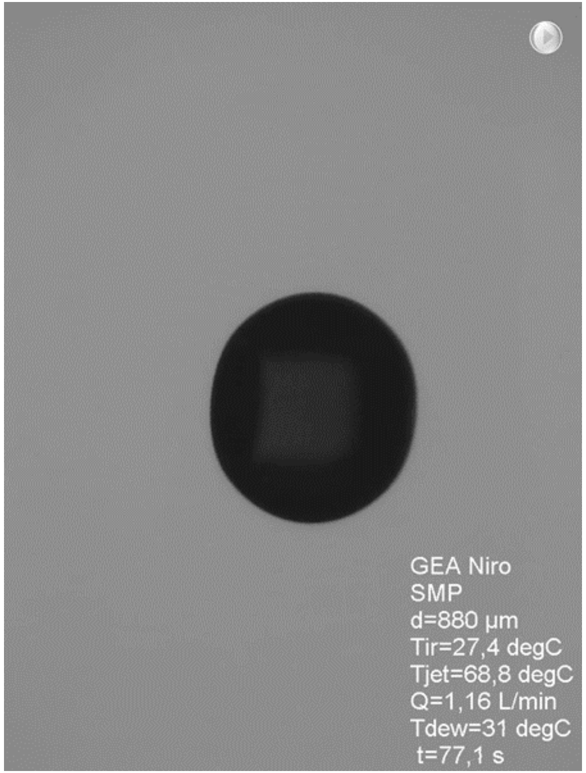
Measuring stickiness



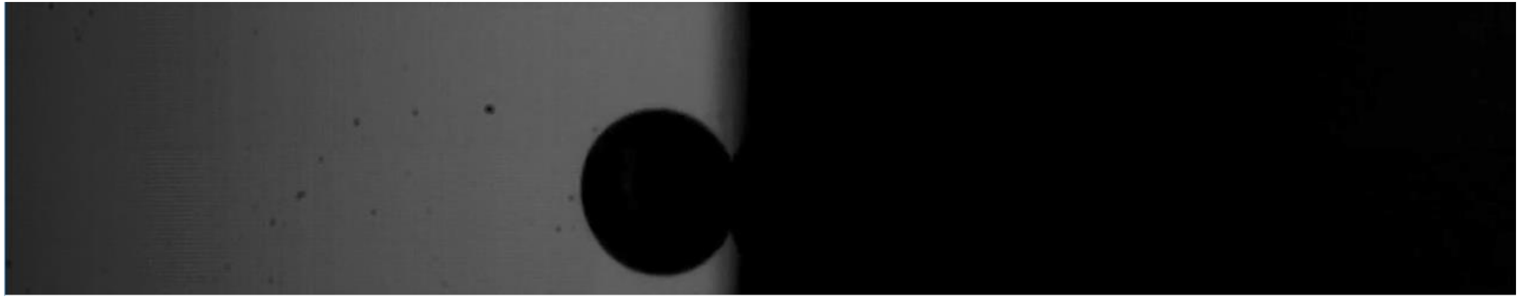
Work of PhD-student Thomas Petersen

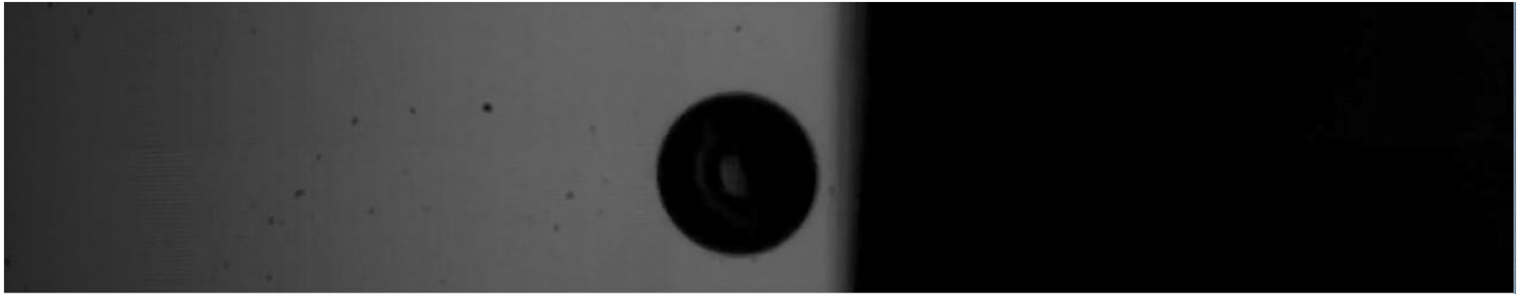
Examples


GEA Niro
SMP
d=771 µm
Tir=27,7 degC
Tjet=71,5 degC
Q=1,16 L/min
Tdew=31 degC
t=66,6 s


GEA Niro
SMP
d=880 µm
Tir=27,4 degC
Tjet=68,8 degC
Q=1,16 L/min
Tdew=31 degC
t=77,1 s

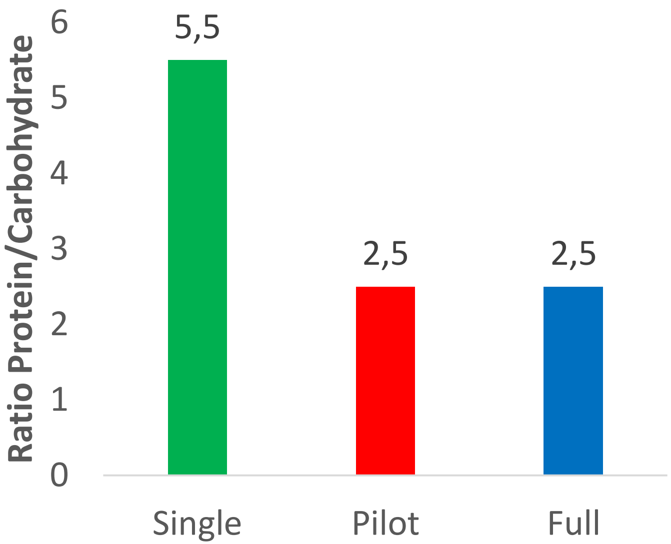
Examples





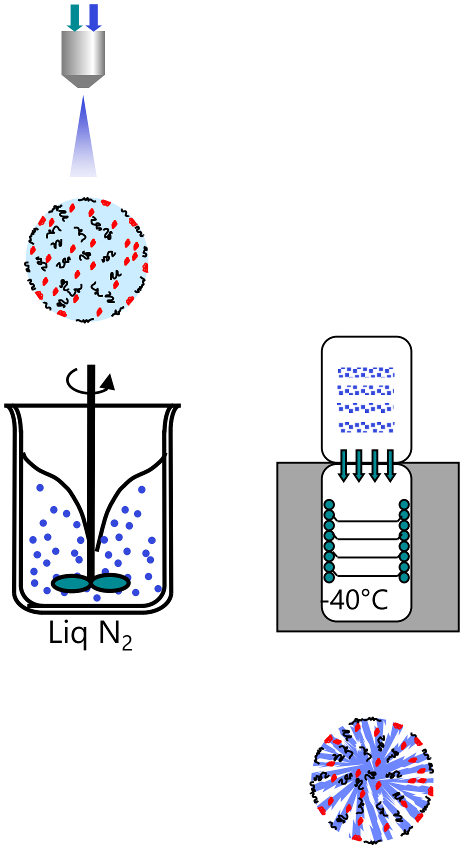
Critical drying times

Product	Critical time (s/mm^2)	Confidens int.
Whole Milk + 0% Glucose	75,5	±2,80
Whole Milk + 5% Glucose	72,5	±2,80
Whole Milk + 10% Glucose	76,5	±5,69



Spray-freeze drying

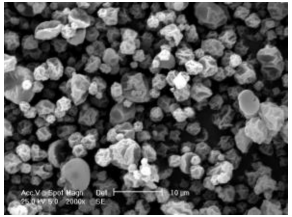
- Outer surface forms as in spray drying
- Droplets are instantly frozen in liq N2
- Frozen droplets transferred to freeze-drier
- Ice crystals are sublimated and pores are formed



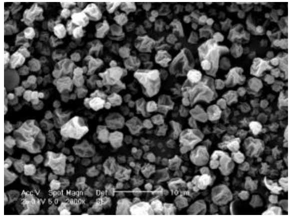
Effect of particle structure on dissolution

- Same formulation, different processing - *spray dry* or *spray-freeze dry*

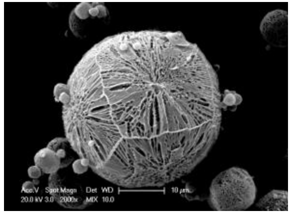
Structure analysis (SEM)



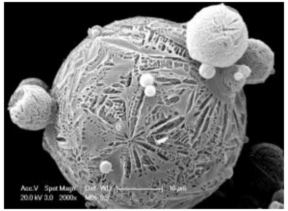
5% BSA



1% HPMC,
5% BSA

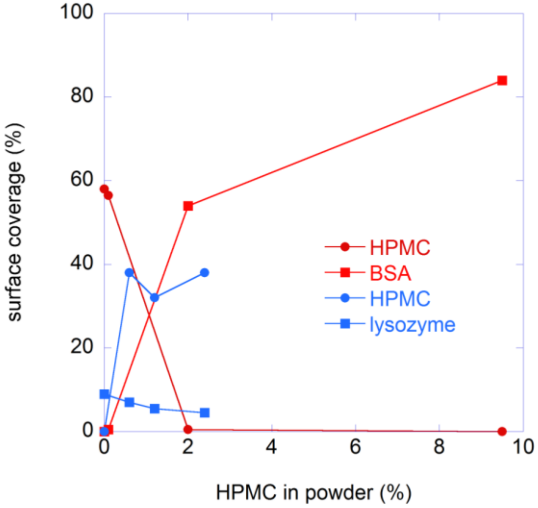


5% lysozyme



1.2% HPMC,
5% lysozyme

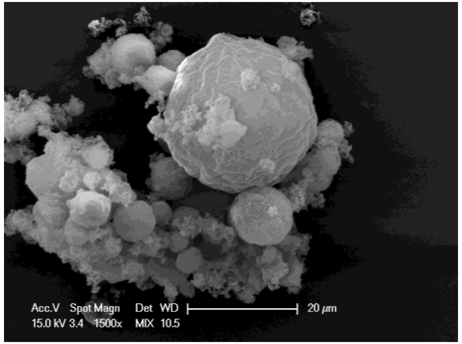
Surface chemical analysis (XPS)

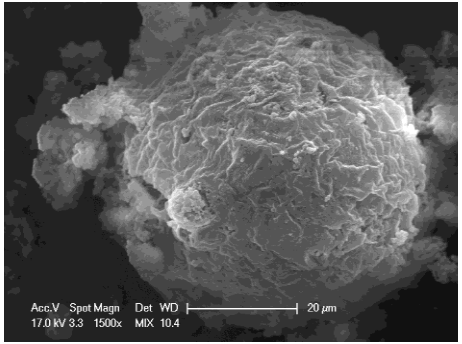


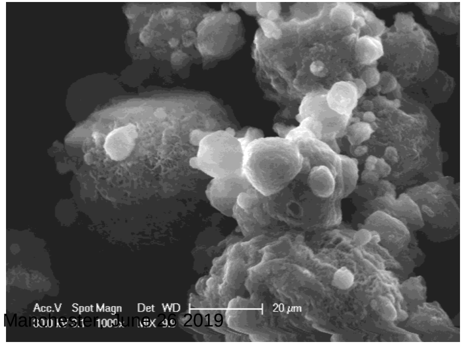
Dissolution time, 1-1.2% HPMC:
Spray-dried: 90 sec
Spray-freeze dried: < 10 sec

GEA RISE

SFD of milk and coffee cream







Sample	Dissolution	
	RT water	Hot water
Milk	Slow, remains at surface	Quick wetting Particles sink
Milk, hom.	Good	Quick wetting Particles sink
Milk, hom., evap.	Very good	Quick wetting Particles sink
Coffee cream	Good, some residual particles	Quick wetting OK after stirring
Coffee cream, hom.	Very good, some residual particles	Quick wetting Particles sink

Conclusions

- Particle structure is determined by:
  - Formulation
  - Drying technology
  - Drying conditions
- Particle morphology, surface properties and internal structure are similar in lab, pilot and full scale
- Single particle drying has different internal structure due to different scales of drying and dimensions
- DKA can be used to predict :
  - Morphology & Drying Kinetics
  - NOT Stickiness & Surface properties

Acknowledgements

- POWTECH (ITN project, ID: 264722)
- RISE/SP/YKI
- Arla Foods Amba
- Dr. Marine Nuzzo (RISE)
- Annika Dahlman, Hans Ringblom, Mikael Sundin, Marie Ernstsson (RISE)
- Ida-Marie Andersson (Lund University)
- Prof. Björn Bergenståhl (Lund University)
- Dr Marta Munoz-Ibanez (Agro Paris Tech; Unilever)
- Prof. Christelle Turchiuli (Agro ParisTech)
- Dr. Thomas Petersen (Danish Technological Institute)
- Prof. Ole Hassager (Technical University of Denmark)
- GEA Research & Development
- Dr. Anders Brask (MechInvent)


THANK YOU
FOR YOUR
ATTENTION

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