# Neutron Scattering

## as a tool to characterize

# fibrous structure formation

## of

# Calcium caseinate

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TU Delft

# Context

Human overpopulation affects the environment adversely

The production of meat is highly inefficient:

- 3-10kg of grain is needed to produce 1kg of meat[1]
- Animal wastes are major source of greenhouse gasses, water pollution[2]

Call for a more sustainable food production approach

**One solution is to produce high quality meat analogue**

1: Tilman, Caassman et al . 2002
2: Food and Agricultural Organization

# A Brief history of meat analogue

**965, tofu, China**

**1587, Yuba, Japan**

**1815, Tempeh, Indonesia**

⋮

**1911 Dec.** – The first commercial meat alternative is made by the Food Factory in Nashville, Tennessee.

**1960s and 1970s** – There is a big increase in the number of vegetarians in Europe and the United States.

**2002 March.** – Burger King is the first major U.S. fast food chain to put a veggie burger on its menu.[1]

1: History of meat alternatives. http://www.soyinfocenter.com/pdf/179/MAL.pdf

# Nowadays

# How are they made?

| Ingredients | + | Techniques | + | Conditions |
|---|---|---|---|---|
| - Soy protein | | - Extrusion | | - Pressure |
| - Wheat gluten | | - Spinning | | - pH |
| - Lupine | | - Simple Shear | | - Heat |
| - Milk protein | | | | - Enzyme |
| - Others | | | | |

# What are the challenges?

Find out a more sustainable way of production

- High temperature heating (>100°C) is rather energy consuming

Maintain better quality control

- Fibre length, water holding capacity, color, etc

Understand how fibre is formed

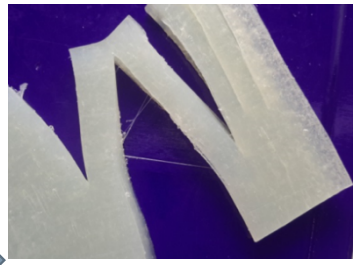
- All the apparatus are essentially a **black box**

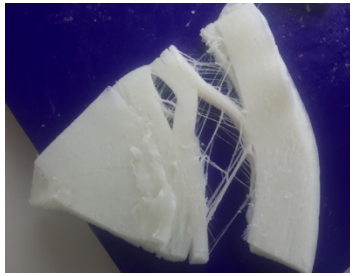
# Defining the system

30% Calcium caseinate + Simple shear + Enzyme / Heat 50°C

Roller dried

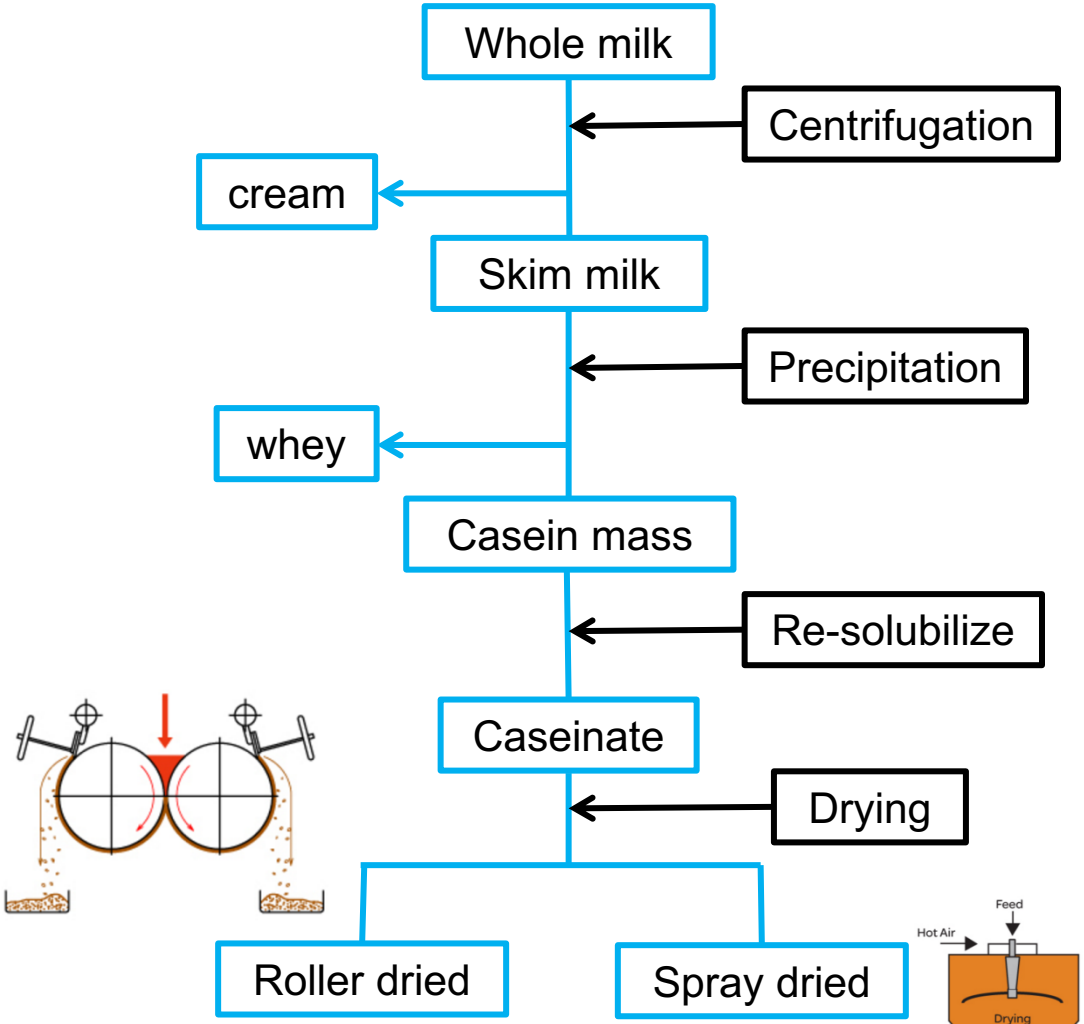
Spray dried





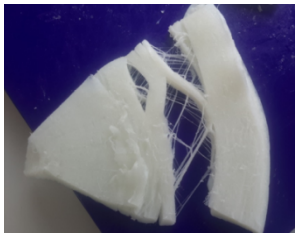
Pictures in courtesy of Zhaojun Wang from Wageningen University

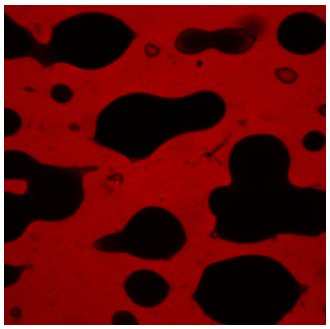
# How caseinate is obtained

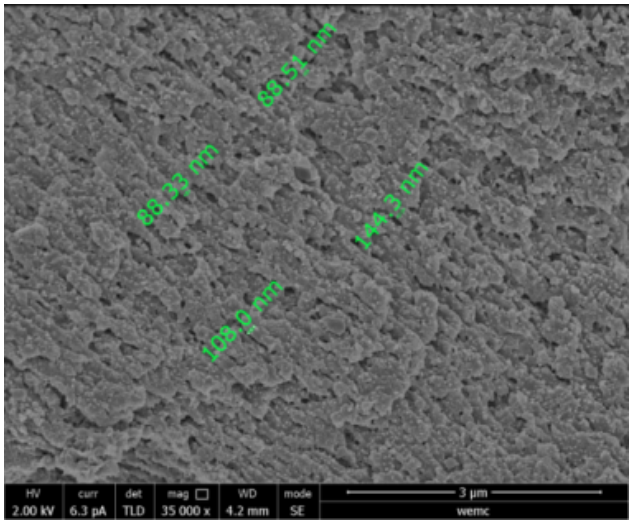


# Microscopy

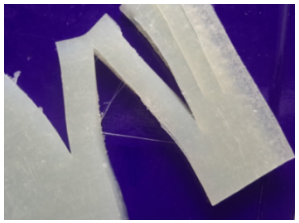
**Spray dried**

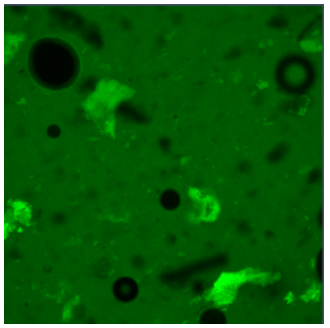


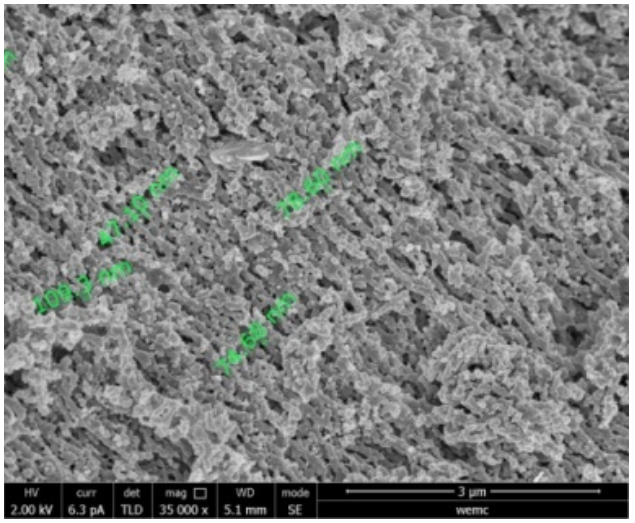





**Roller dried**







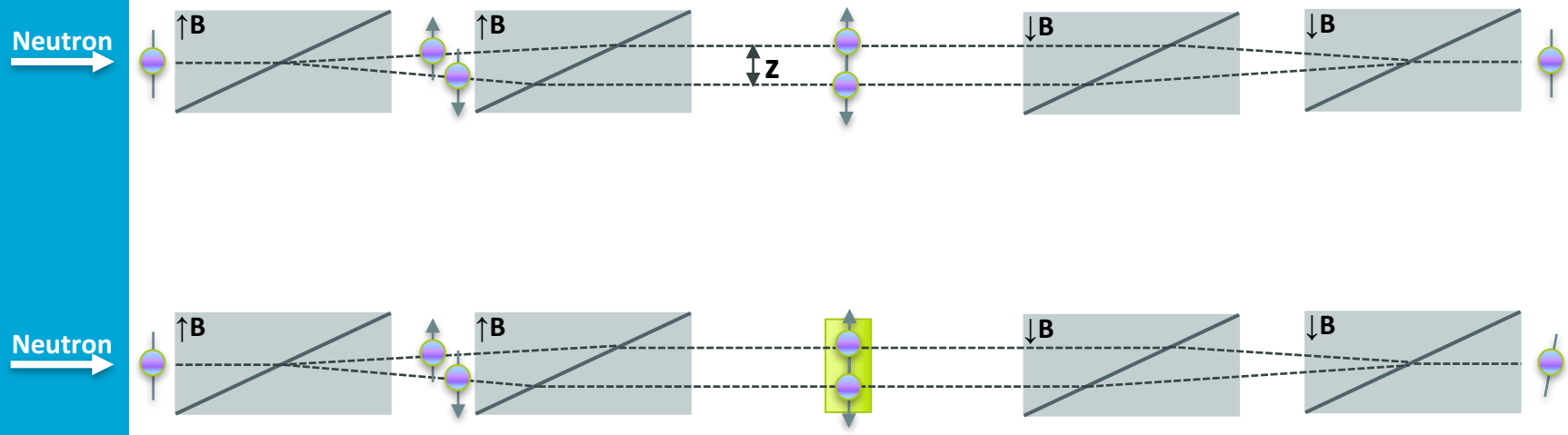

Size of the air bubbles (10~100 µm)

Fibre thickness/length (~100nm/ ~ few µm)

→ Neutron

Pictures in courtesy of Zhaojun Wang from Wageningen University

# Spin Echo SANS (SESANS)



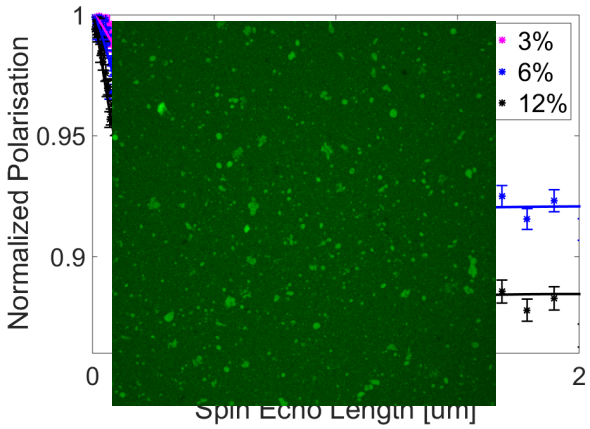

- z: spin-echo length (distance between the shift of two eigenstates)
- Polarisation P(z) is directly related to the correlation function G(z)

$$P(z) = e^{(G(z)-G(0))}$$

# SESANS – dilute system

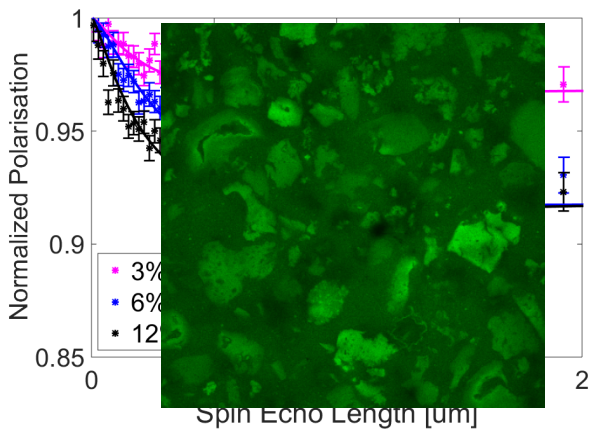
## Poly-dispersed sphere system

**Spray dried Calcium caseinate**




| | Radius [nm] | Absorbed water | Poly-dispersity |
|---|---|---|---|
| 3% | 250(70) | 13.6(0.9) | 0.74 |
| 6% | 270(45) | 8.3(0.3) | 0.7 |
| 12% | 235(63) | 4.5(0.1) | 0.88 |

**Roller dried Calcium caseinate**



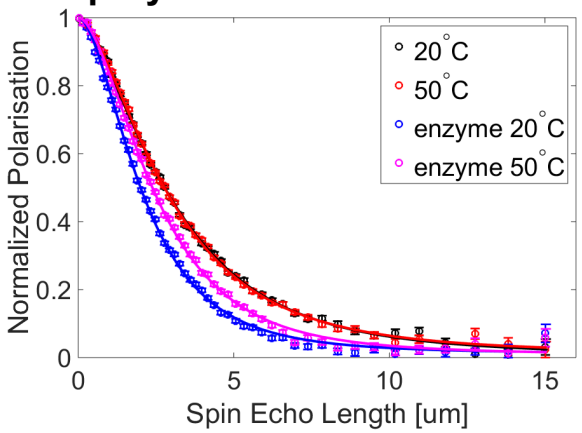

| | Radius [nm] | Absorbed water | Poly-dispersity |
|---|---|---|---|
| 3% | 240(240) | 15(5) | 0.9 |
| 6% | 507(47) | 9.7(1.1) | 0.95 |
| 12% | 425(113) | 5.4(0.1) | 0.97 |

CLSM pictures in courtesy of Zhaojun Wang from Wageningen University

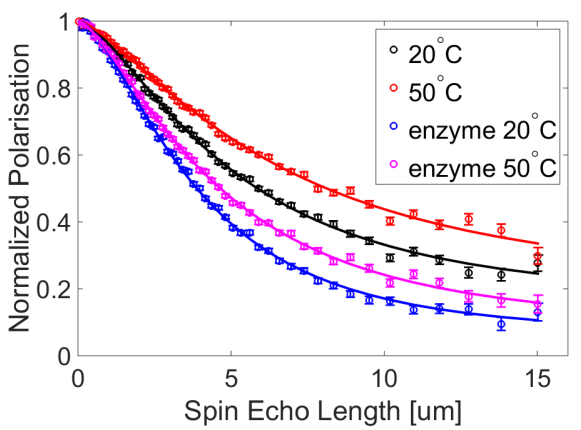
# SESANS – dense system

## Random two phase system

**Spray dried Calcium caseinate**




**Roller dried Calcium caseinate**



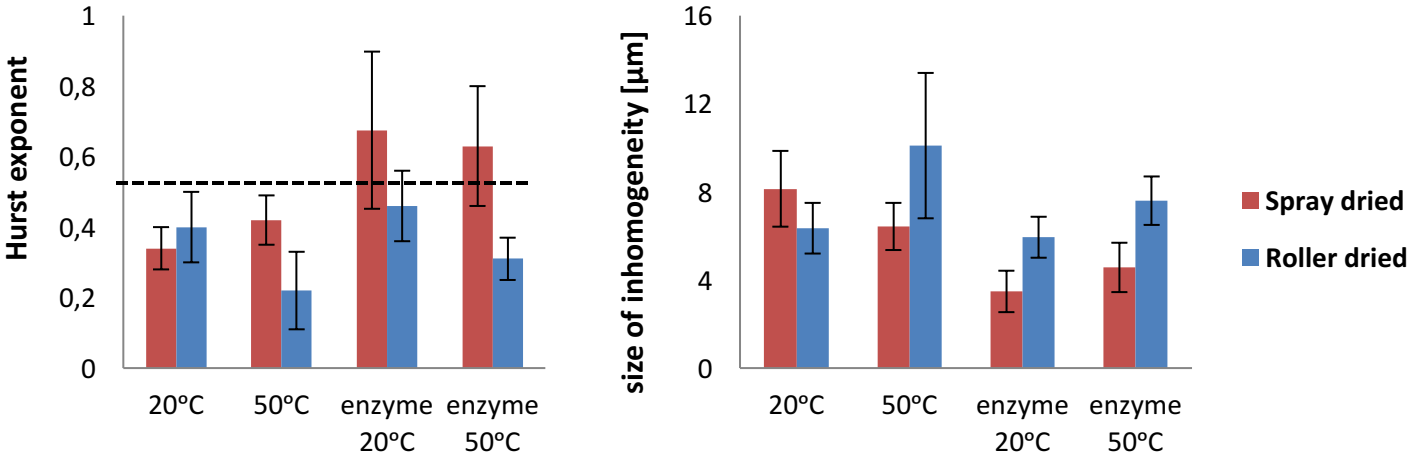

$$G(z) = \frac{2}{\Gamma(H+1/2)}\left(\frac{z}{2a}\right)^{H+1/2} K_{H+1/2}\left(\frac{z}{a}\right)$$

- a: size of inhomogeneity
- H: hurst exponent

# SESANS – dense system

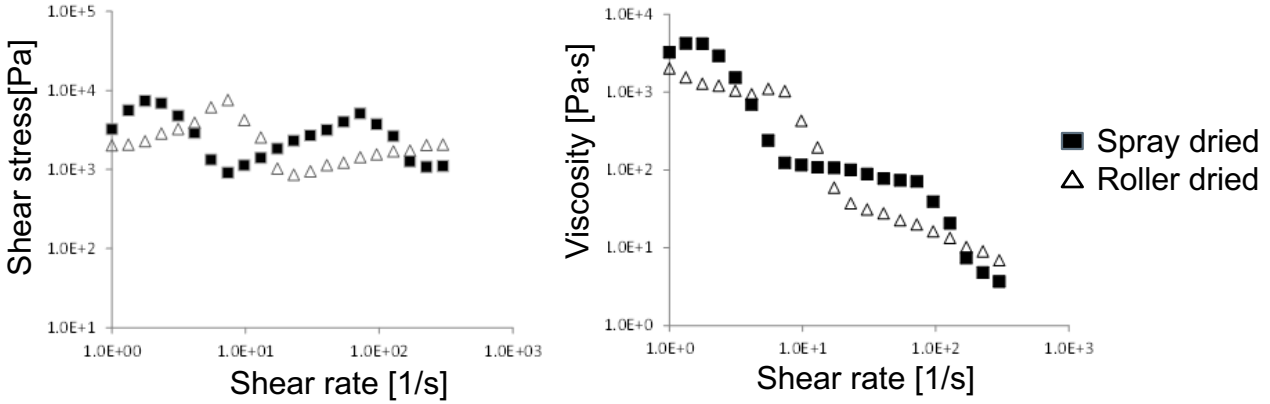
## Random two phase system

- for H > 0.5, the system is persistent, indicating smoothness and long-range correlations
- for H < 0.5, the system is anti-persistent, indicating roughness, short-range correlations



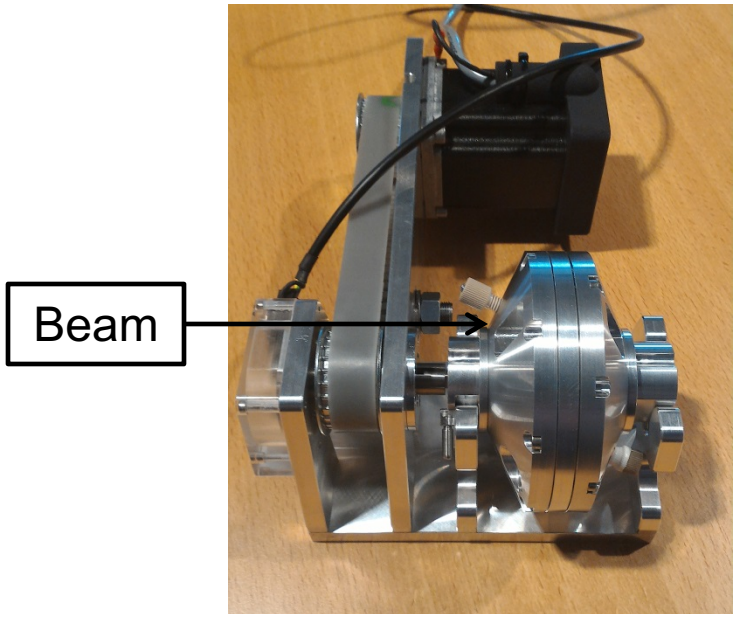
# Rheology

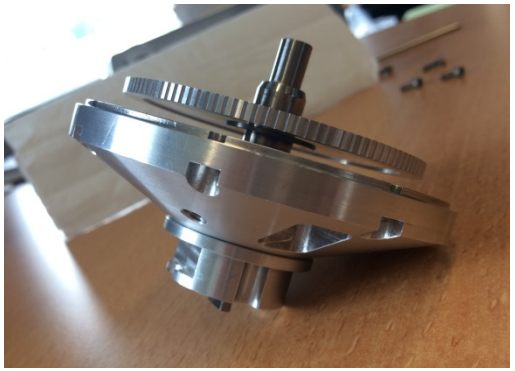
**Shear sweep:** non-monotonic behavior of the flow curve indicating shear-induced structure




**In situ experiment will be useful solving the 'black box' problem.**

# Next step: in-situ SANS



Questions to be answered:

- Dilute system: whether shear will elongate protein particle
- Dense system: how different shear rate induce shear banding

# To wrap up

- Neutron scattering is a novel and complementary tool to quantitatively describe the structure of calcium caseinate.

- In-situ (SE)SANS measurement will be promising in understanding how structure is formed under shear and mild heat.

- Other methods are needed to help interpret the results of neutron scattering.

# Acknowledgement

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**People contributing to this work:**

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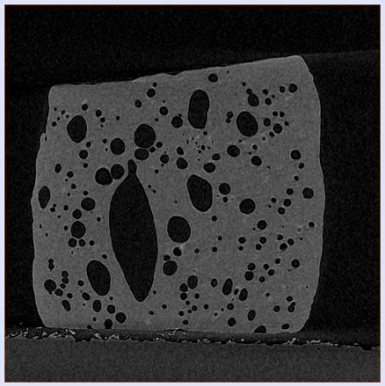
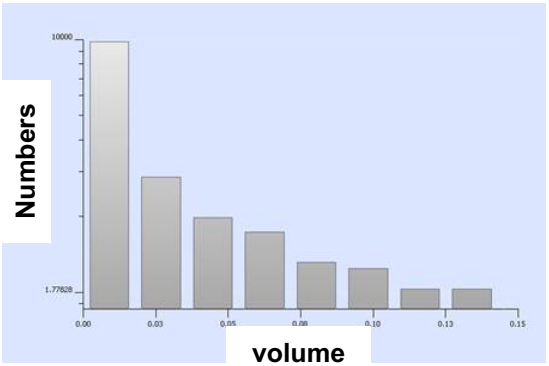
# Thank you for your attention

# XRT

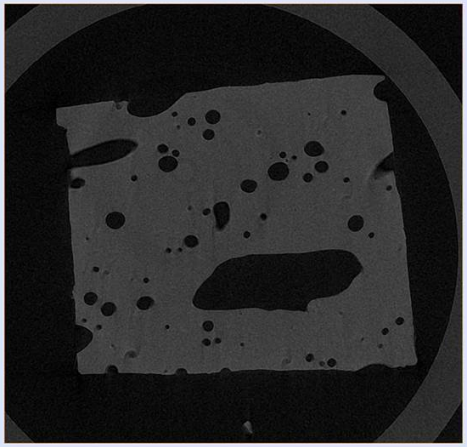
**Spray dried**

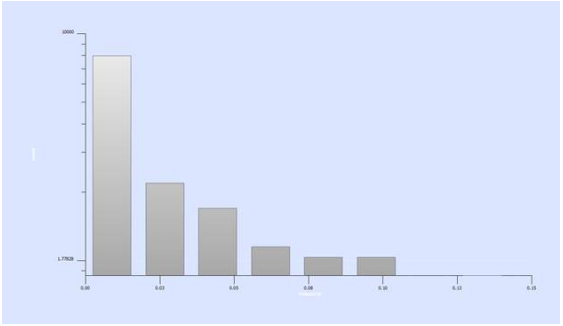





- More small air bubbles

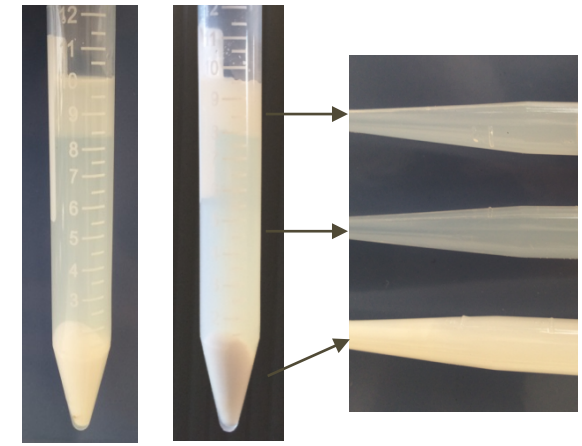
**Roller dried**



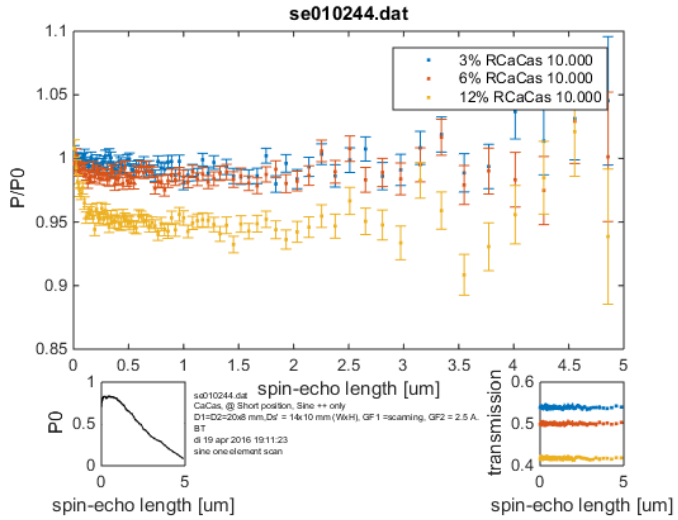


- Absence of large air bubbles

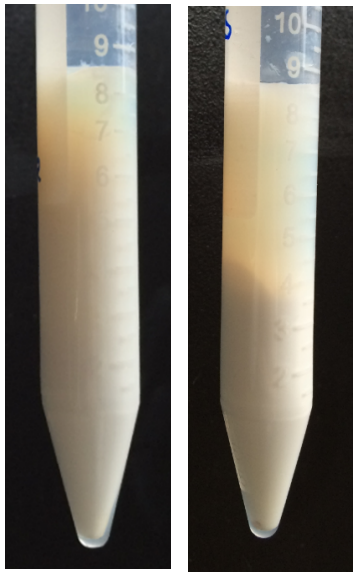
# SESANS
# --6&10% RCACAS/SCACAS



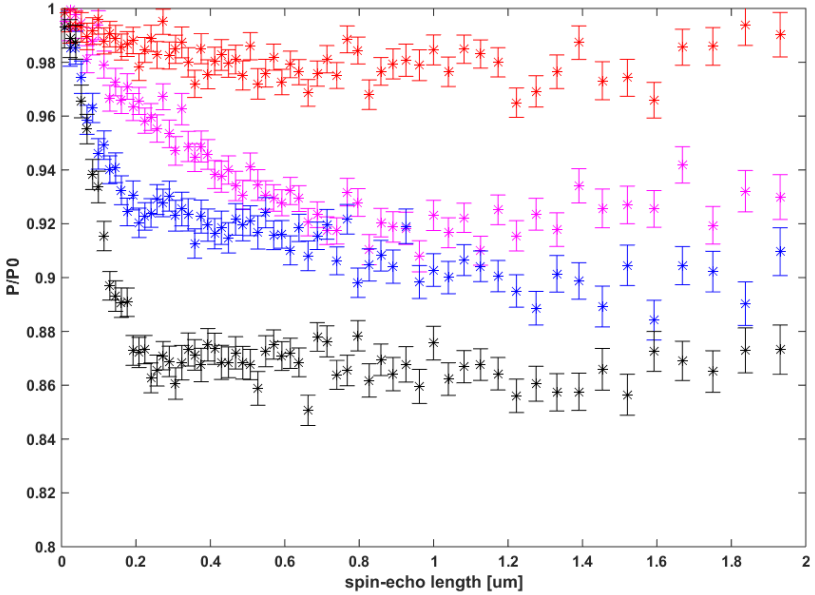
6% SCaCas 10% RCaCas



# SESANS
# --12% RCACAS/SCACAS



12% RCaCas 12% SCaCas




SCaCas Upper layer

RCaCas Upper layer

RCaCas Mid layer

SCaCas Mid layer