



Faculty of Science



**DFM**

Danish National Metrology Institute



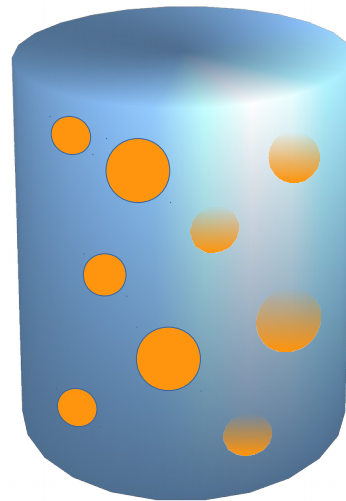
# Revealing the 3D nanostructure of extended colloidal networks in food emulsions using ptychographic X-ray computed tomography

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Danish Fundamental Metrology A/S,  
Lyngby, Denmark



## The structure of food emulsions

Food emulsion



Liquid

(Semi)-solid



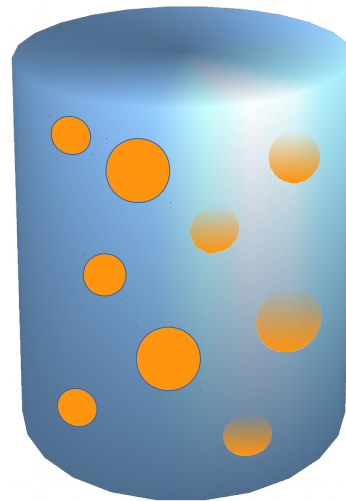
# The structure of food emulsions

Food emulsion

Water  
Fat



Picture: Bearas - Wikimedia Commons



Picture: Merete Bøgelund Munk

Liquid

(Semi)-solid



## Effect of addition of emulsifier

*Effect of addition of emulsifier*

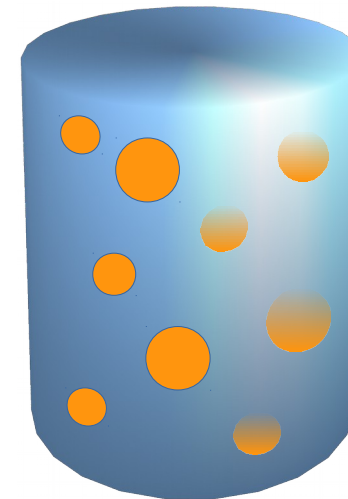
### Product composition

~25 w% lipid phase:

- 25 w% *Palm kernel oil*

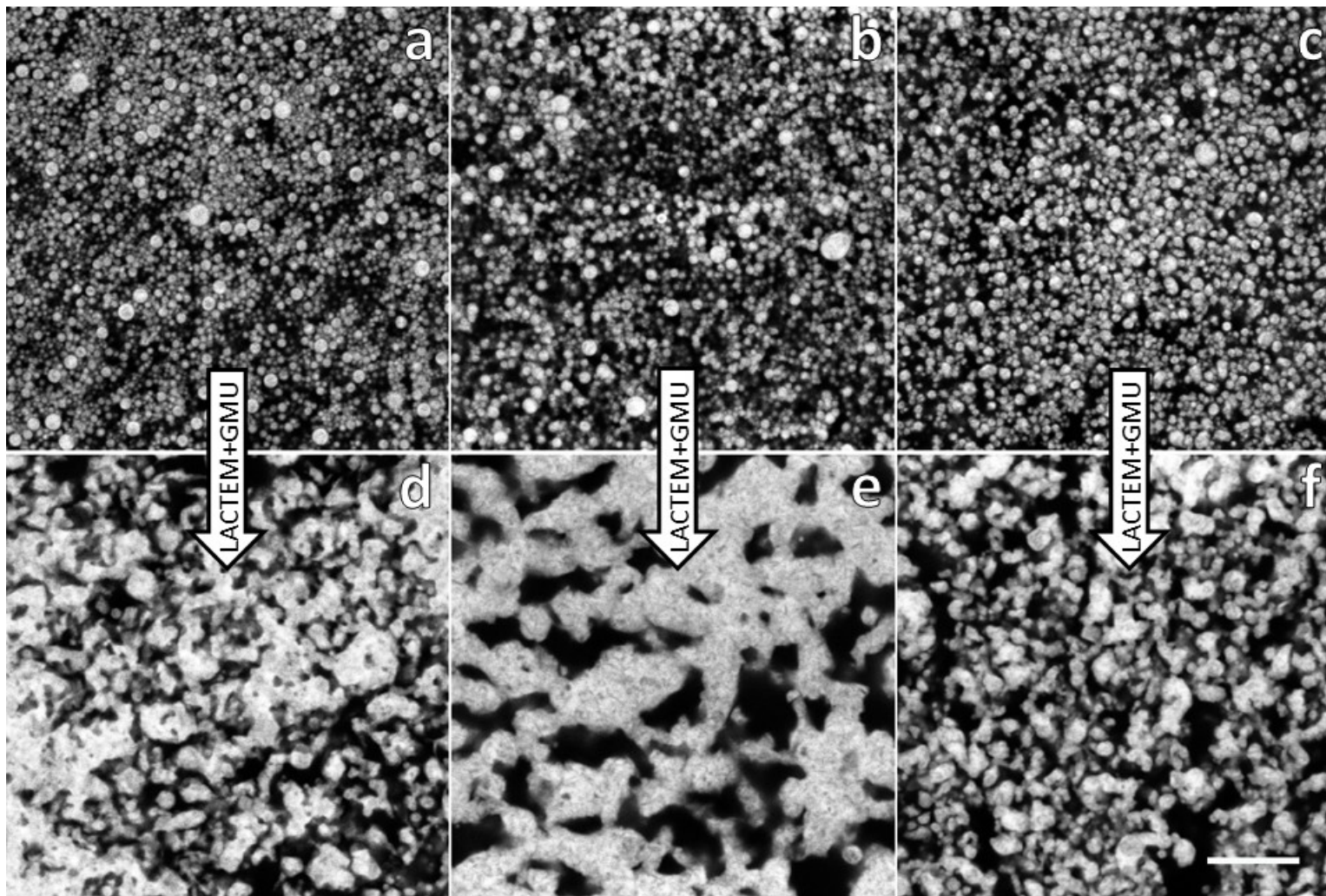
~75 w% water phase:

- 10 w% *sugar (sucrose)*
- 62 w% *water*
- 0.6 w% *sodium caseinates*
- 0.6 w% *stabilizers*
- 1 w% *emulsifier*



## Effect of addition of emulsifier

*Confocal laser scanning microscopy*

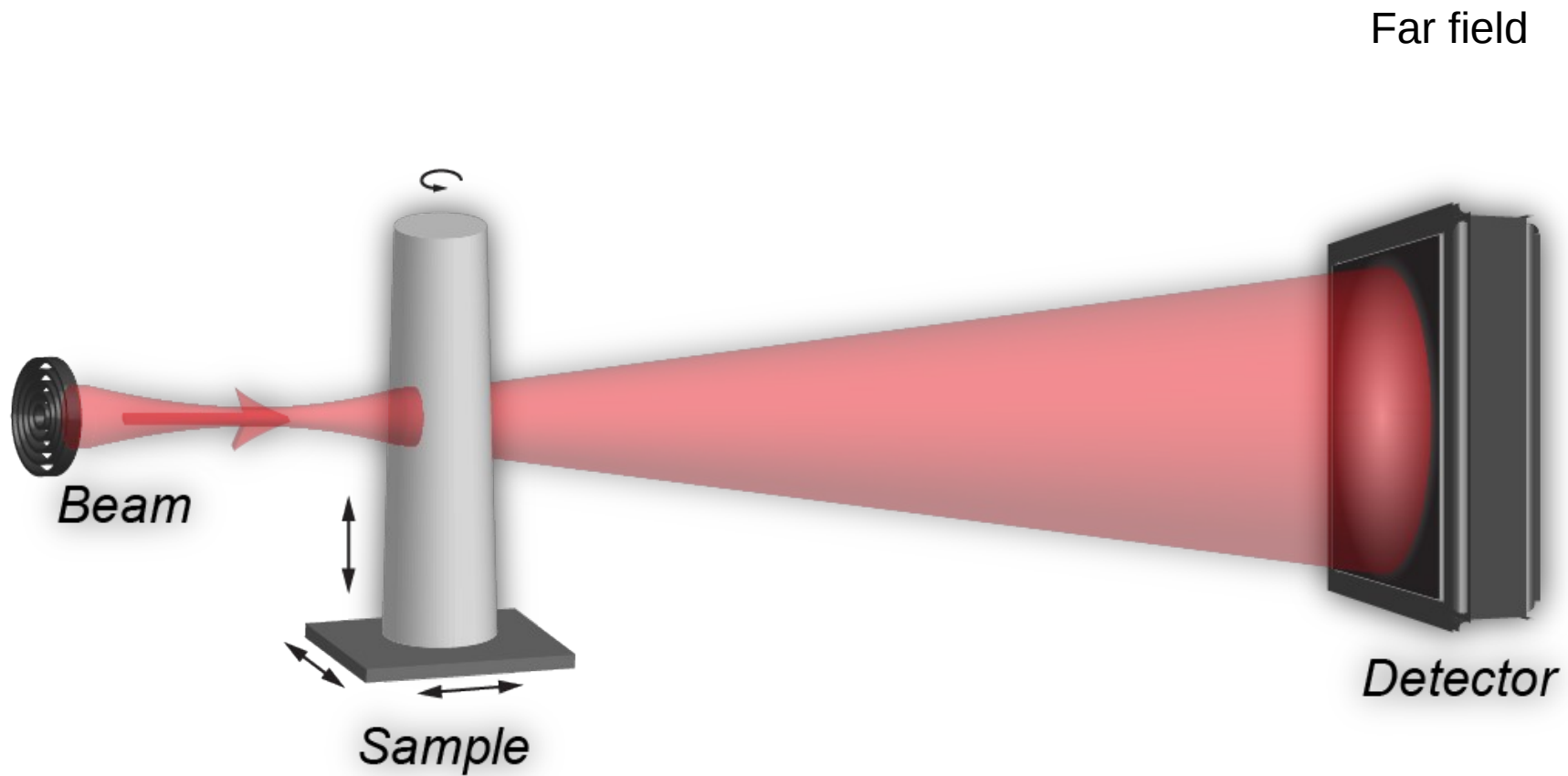


Scalebar: 10 microns

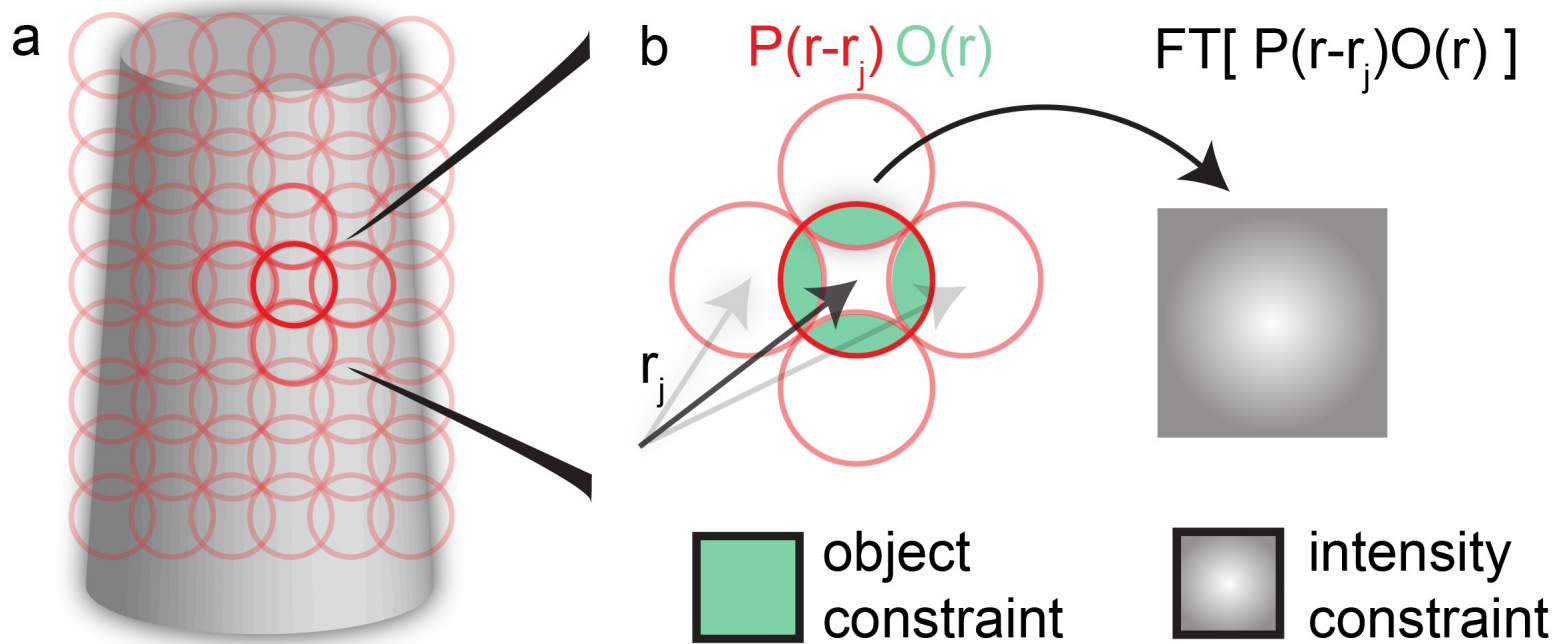
# X-ray ptychography



# X-ray ptychography



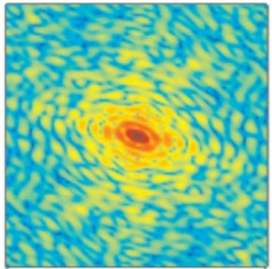
## Ptychographic scan





## From recorded patterns to an image

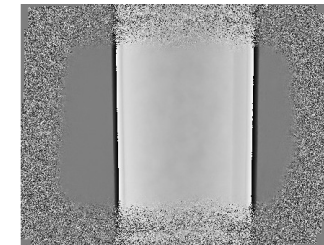
Recorded pattern  
(Reciprocal space)



Measured  
intensity

$\mathcal{FT}^{-1}$

Reconstructed image  
(Real space)



Go to new  
scan point

Intensity  
constraint

Pattern from  
updated image

$\mathcal{FT}$

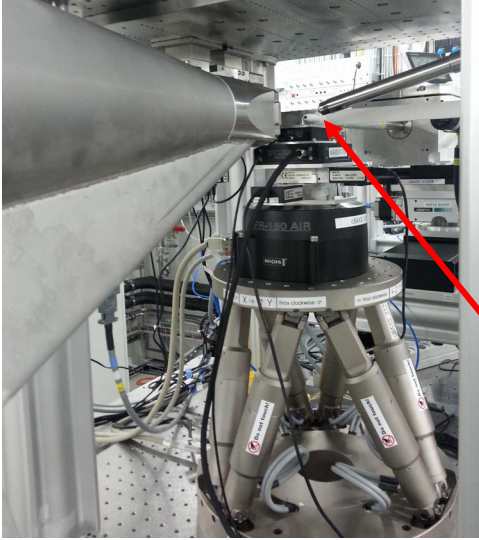
Overlap  
constraint

Updating  
image with  
overlap

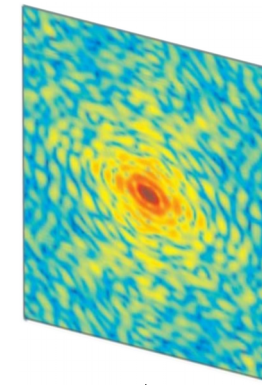


## Experimental setup

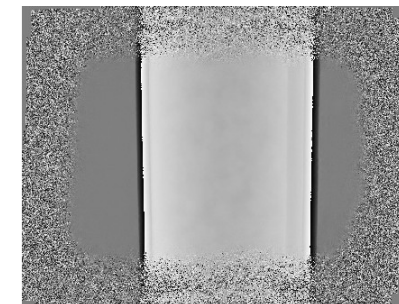
Setup at cSAXS beamline



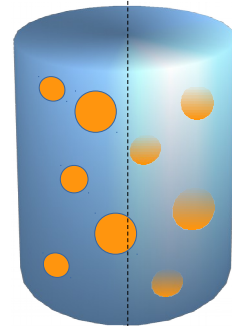
*Ptychographic X-ray  
computed tomography*



Reconstruction

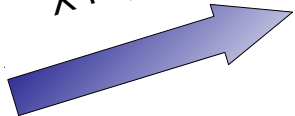


$\omega$



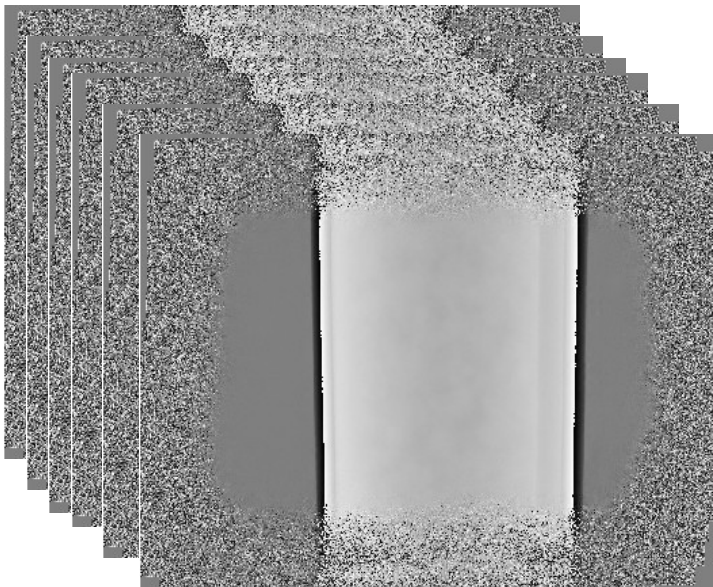
Food emulsion

X-rays



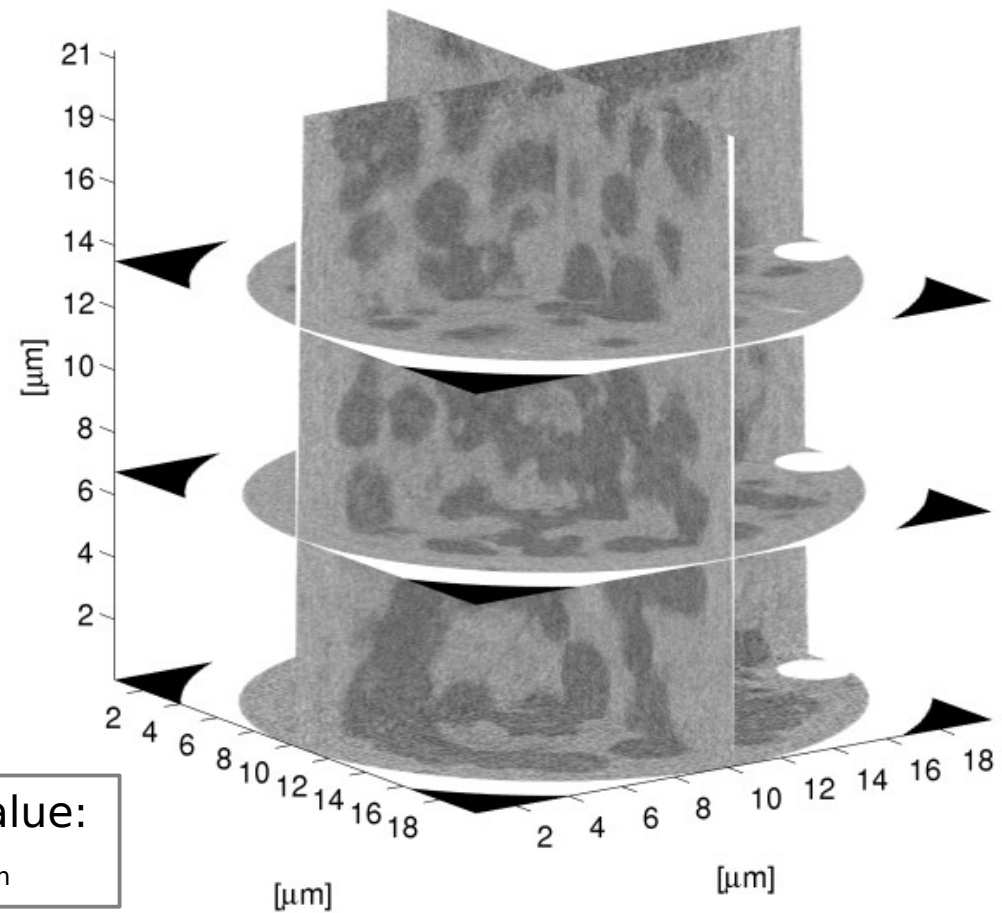
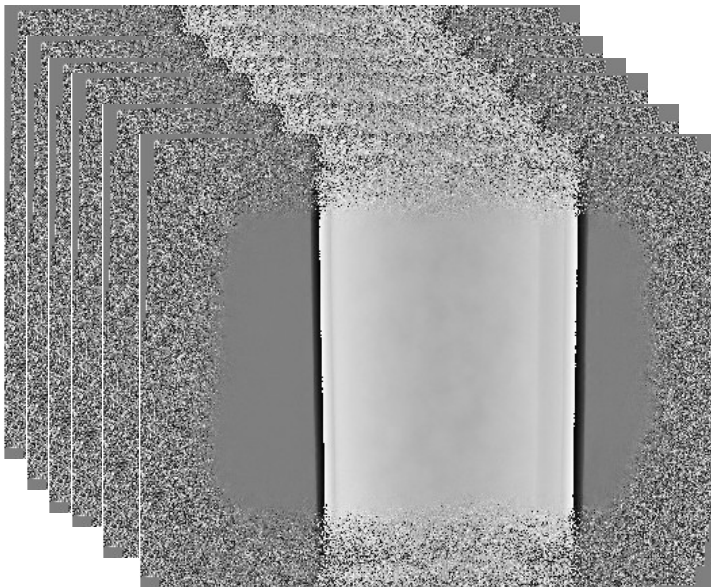
# Tomographic reconstruction

*Combining projections from  
rotation of sample*



# Tomographic reconstruction

*Combining projections from rotation of sample*



Bonus info box:

Reconstruction algorithm:  
Filtered back-projection

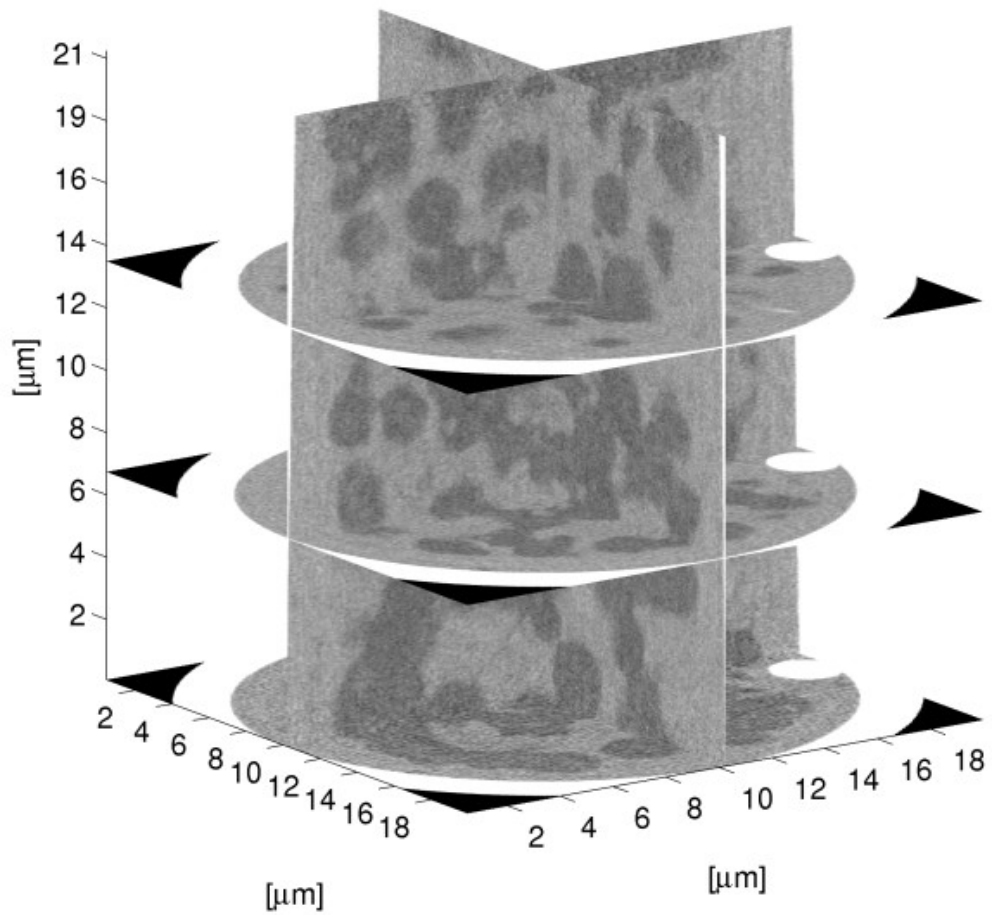
Reconstructed value:

$$\rho_e = N_A \cdot Z/M \cdot \rho_m$$



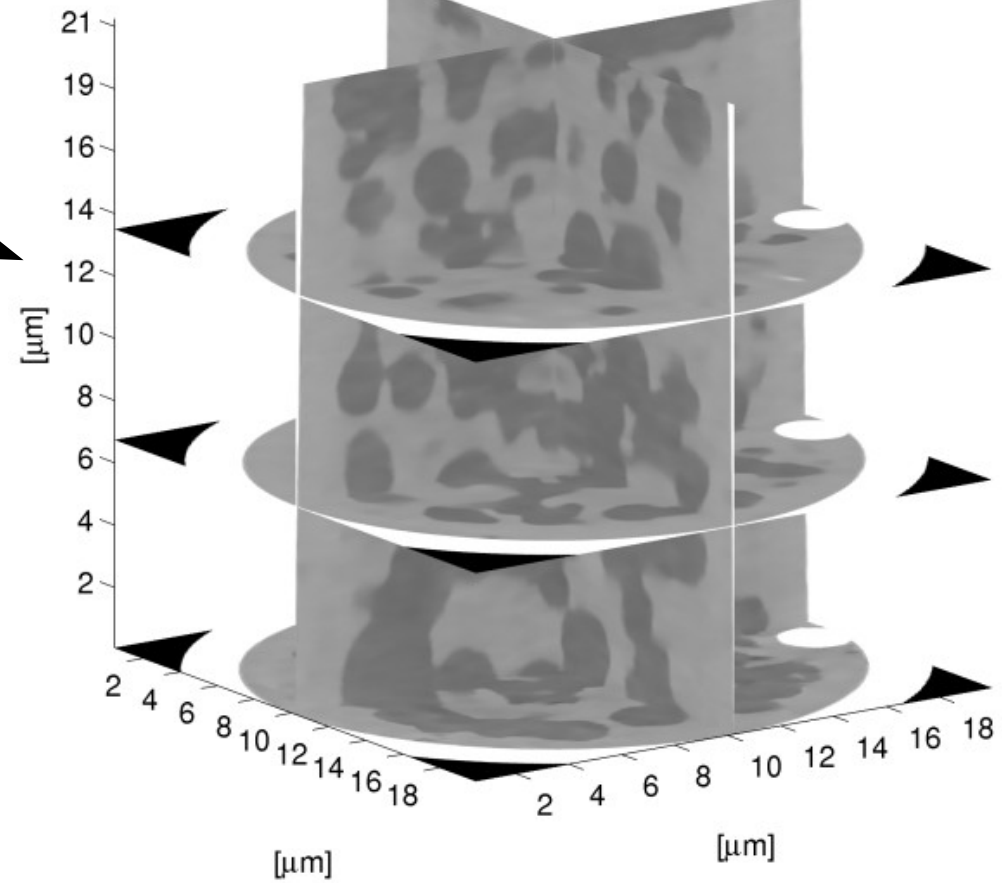
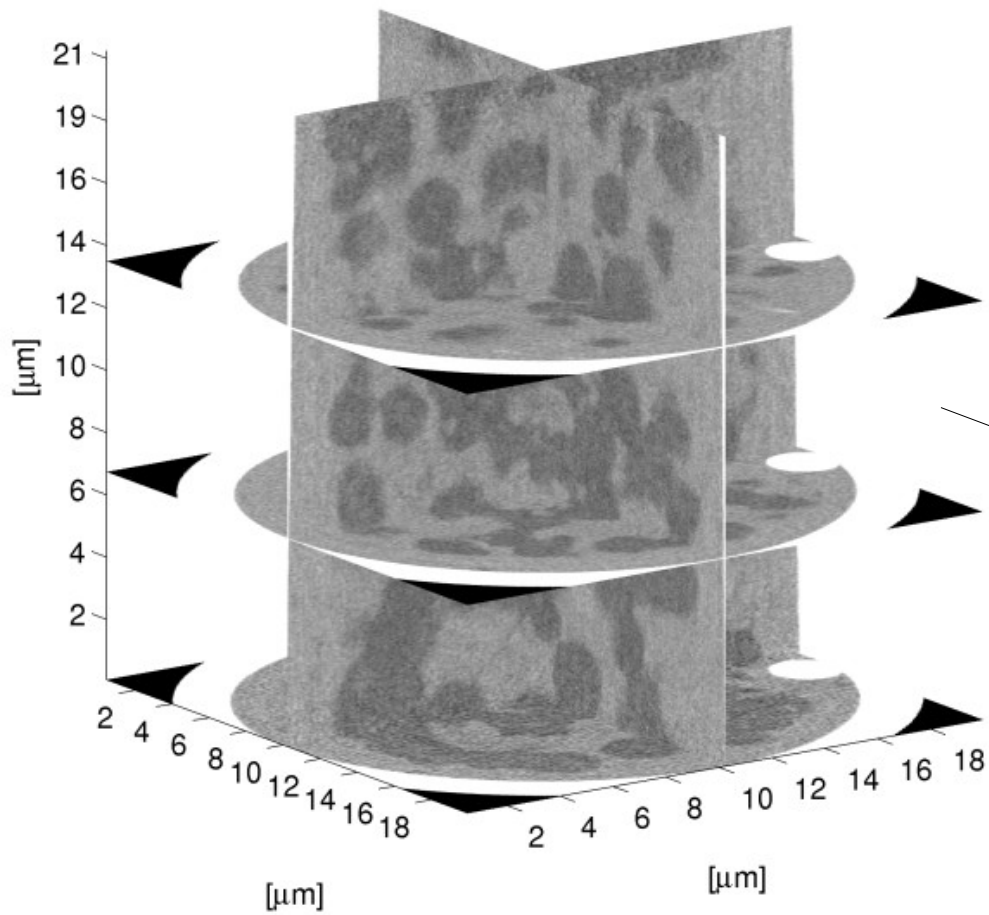
## Denosing

*Removing noise without blurring features*



# Denosing

*Removing noise without blurring features*



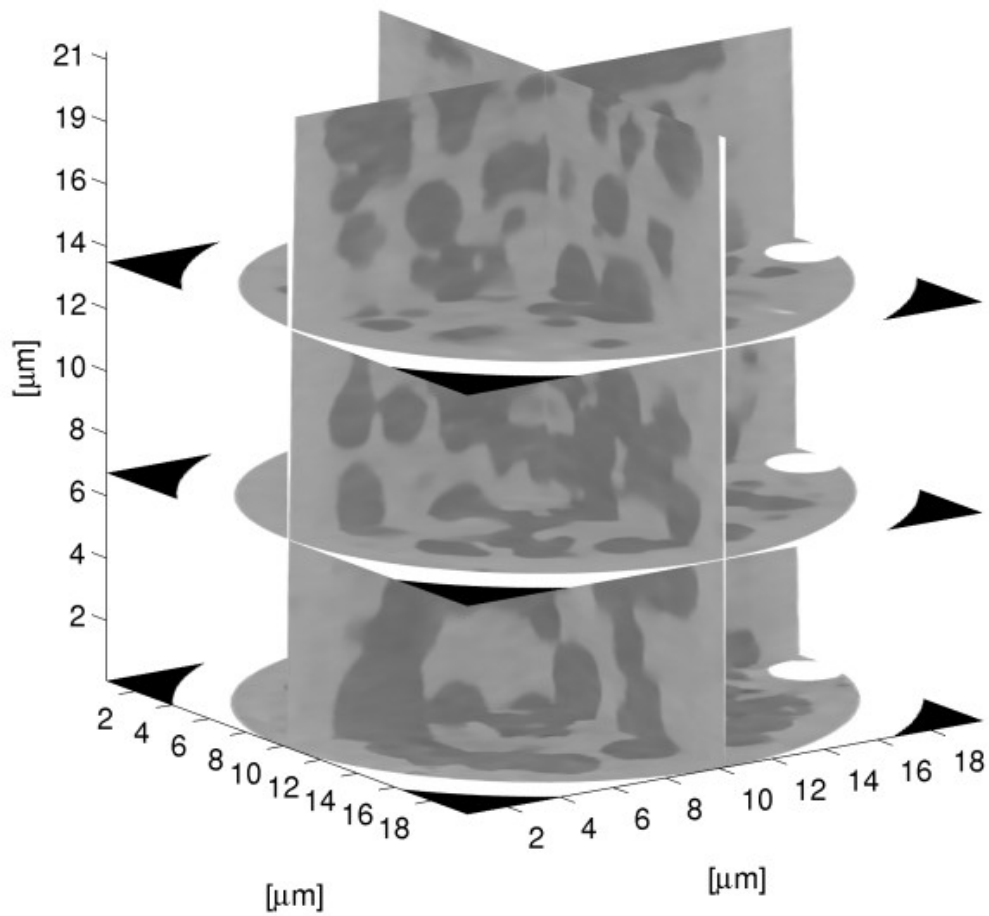
Bonus info box:

Denosing algorithm: Iterative non-local mean



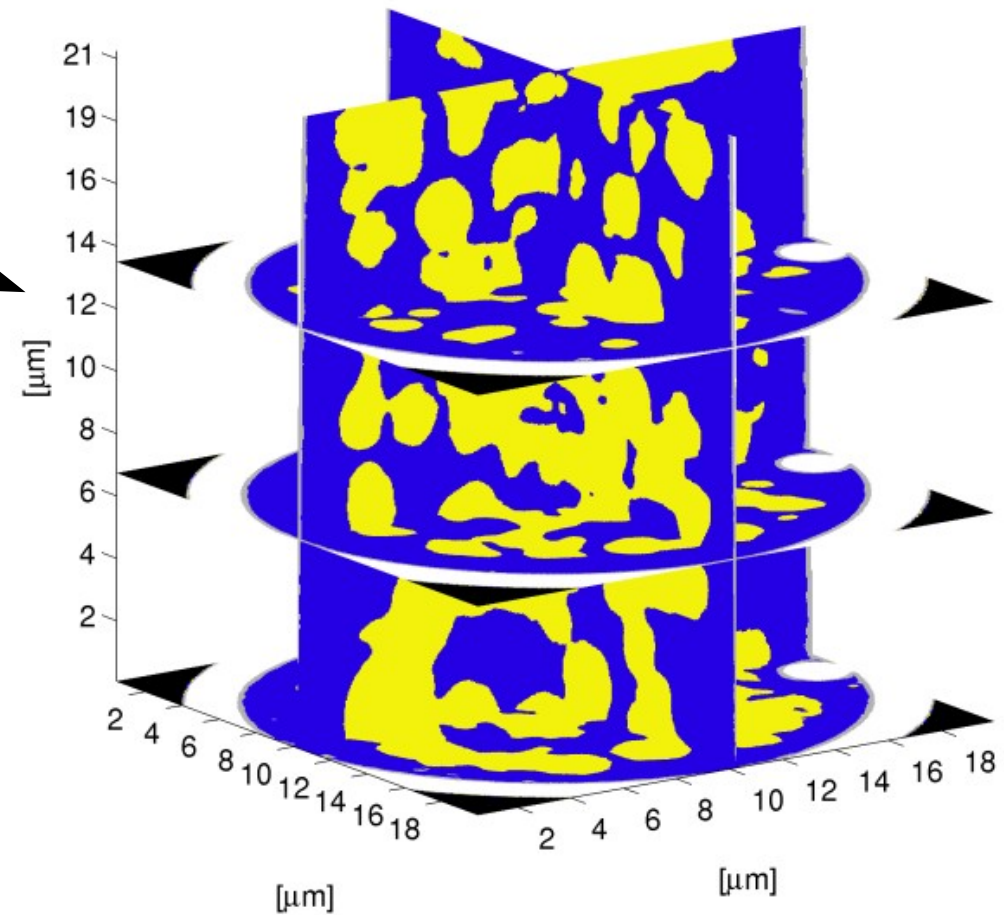
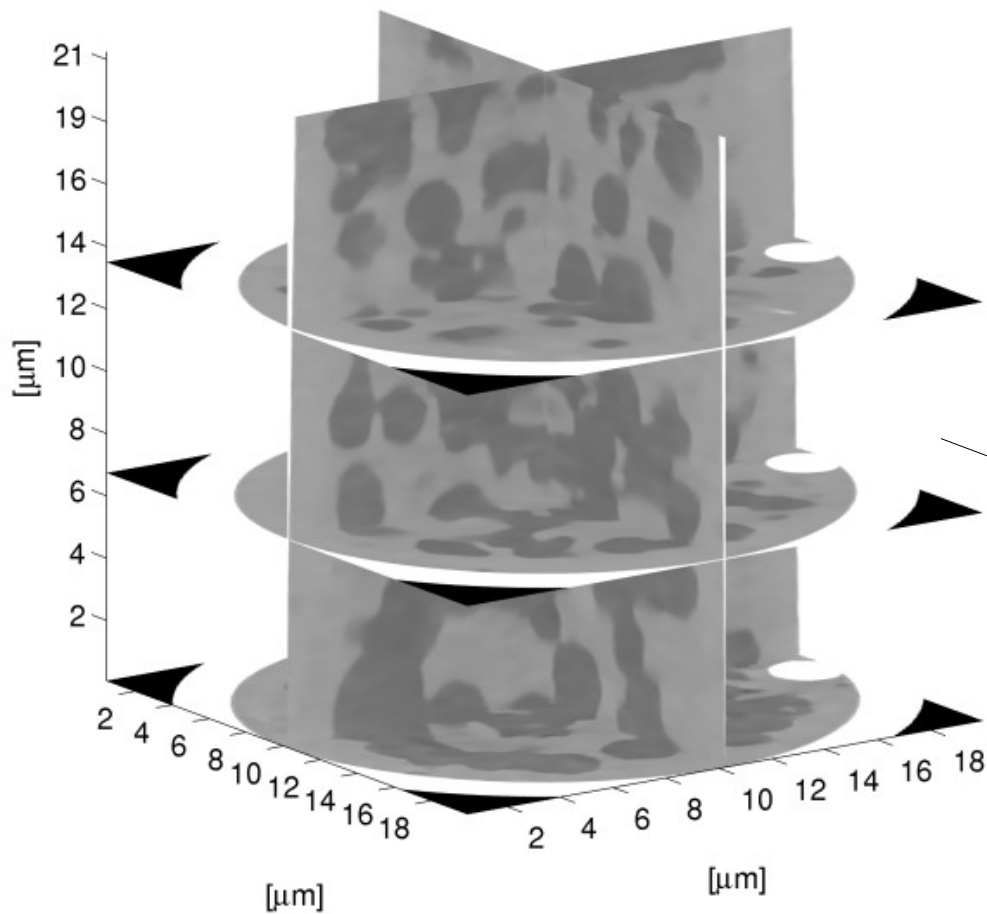
## Segmentation

*Segmentation of water and lipid phases*



# Segmentation

*Segmentation of water and lipid phases*



Bonus info box:

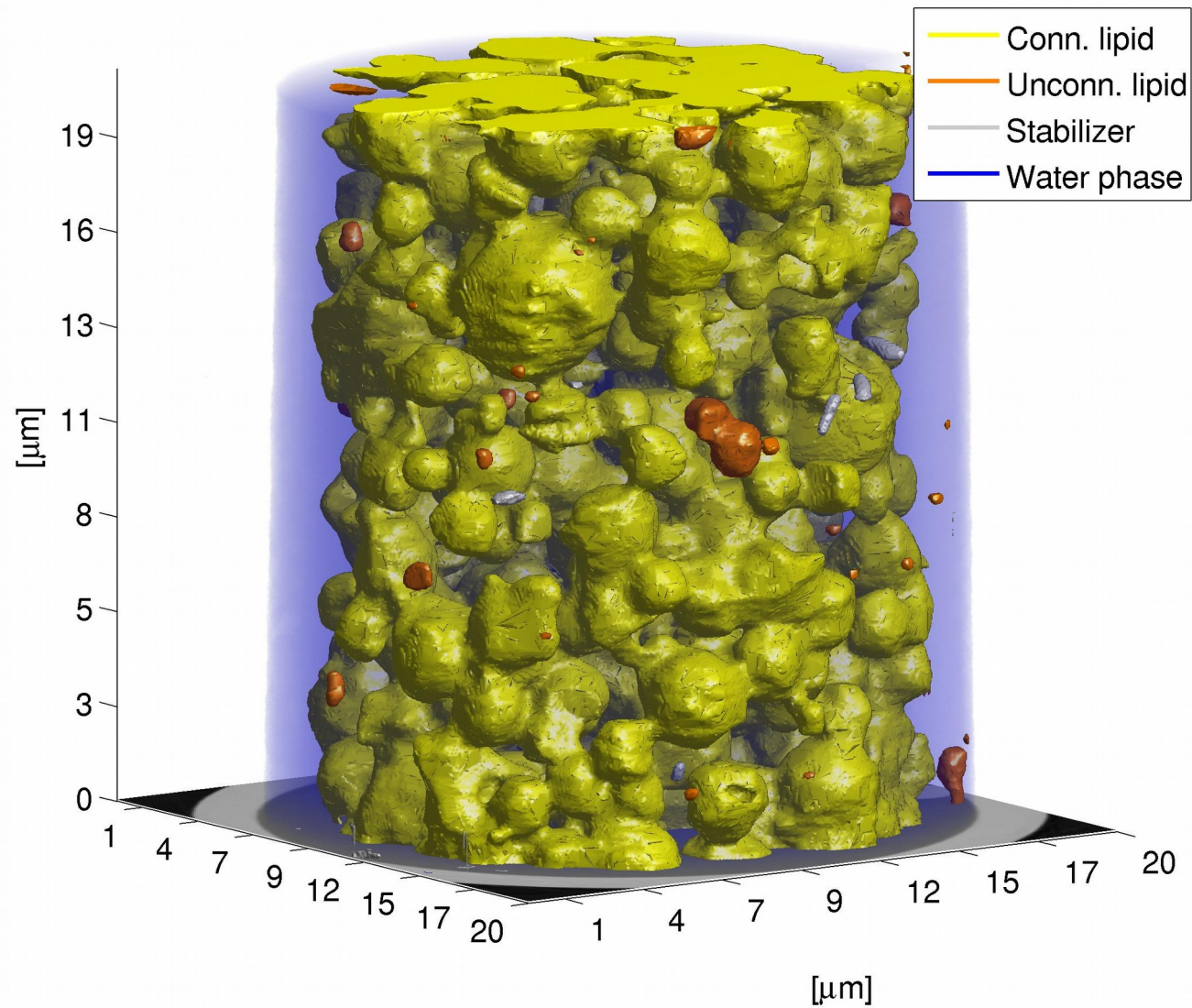
Segmentation algorithm: Markov random field segmentation with alpha-expansion

Slide 16



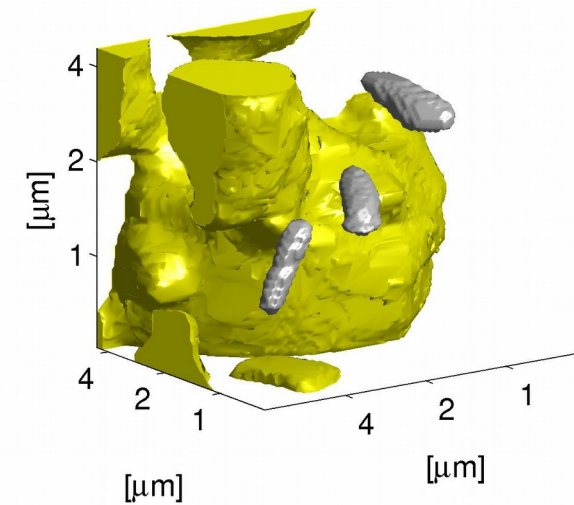
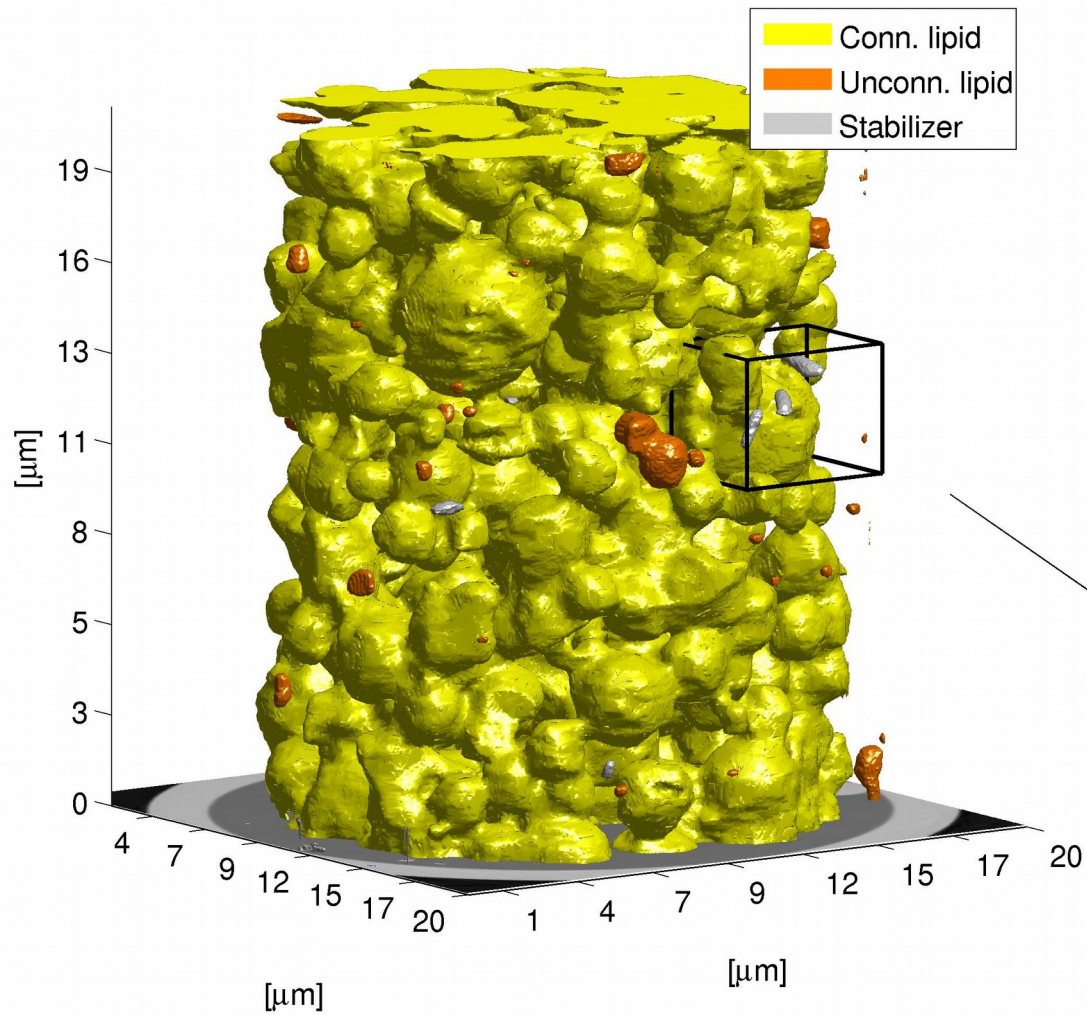


## 3d visualization of the emulsion

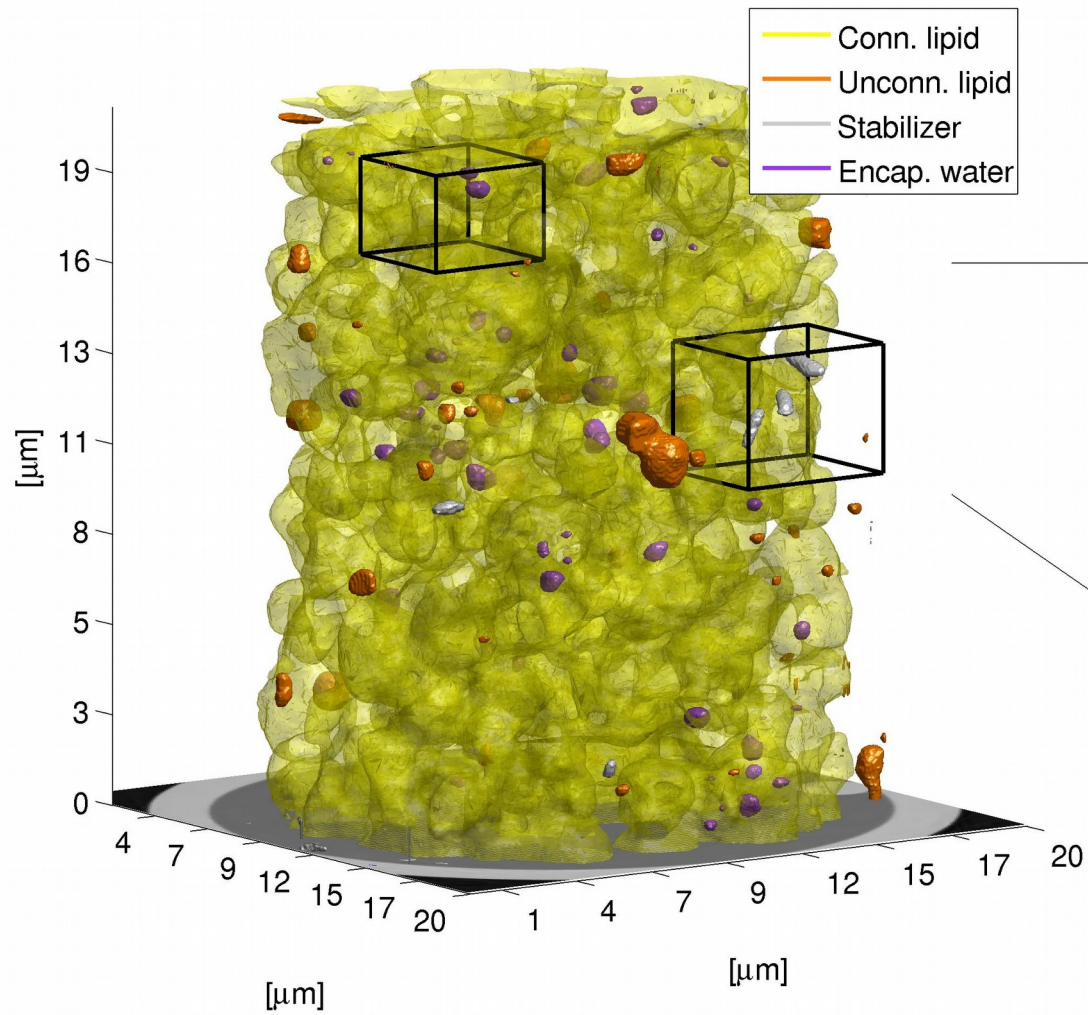


# 3d visualization of the emulsion

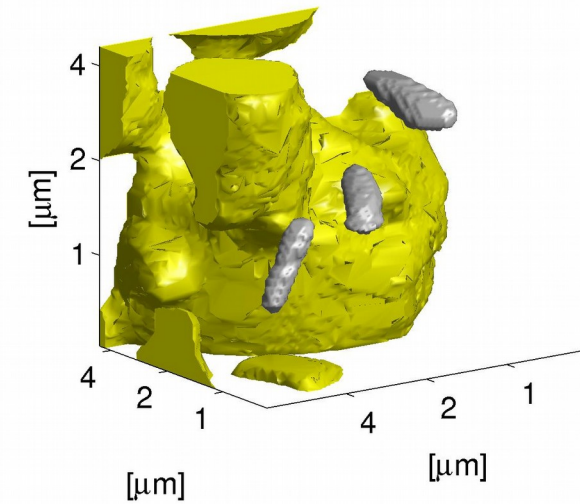
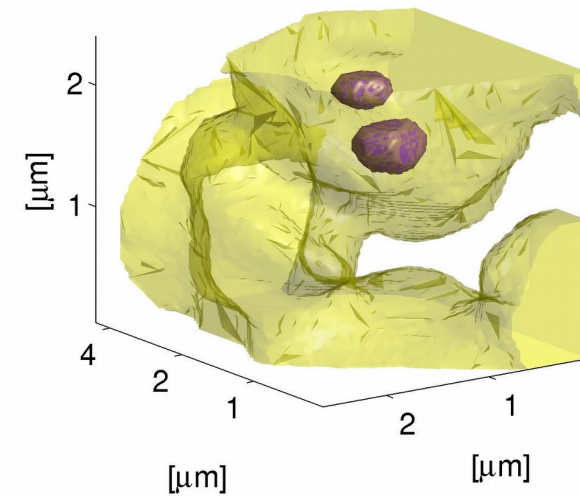
*Micro-cellulose stabilizers*



## 3d visualization of the emulsion



## *Encapsulated water in the lipid phase*

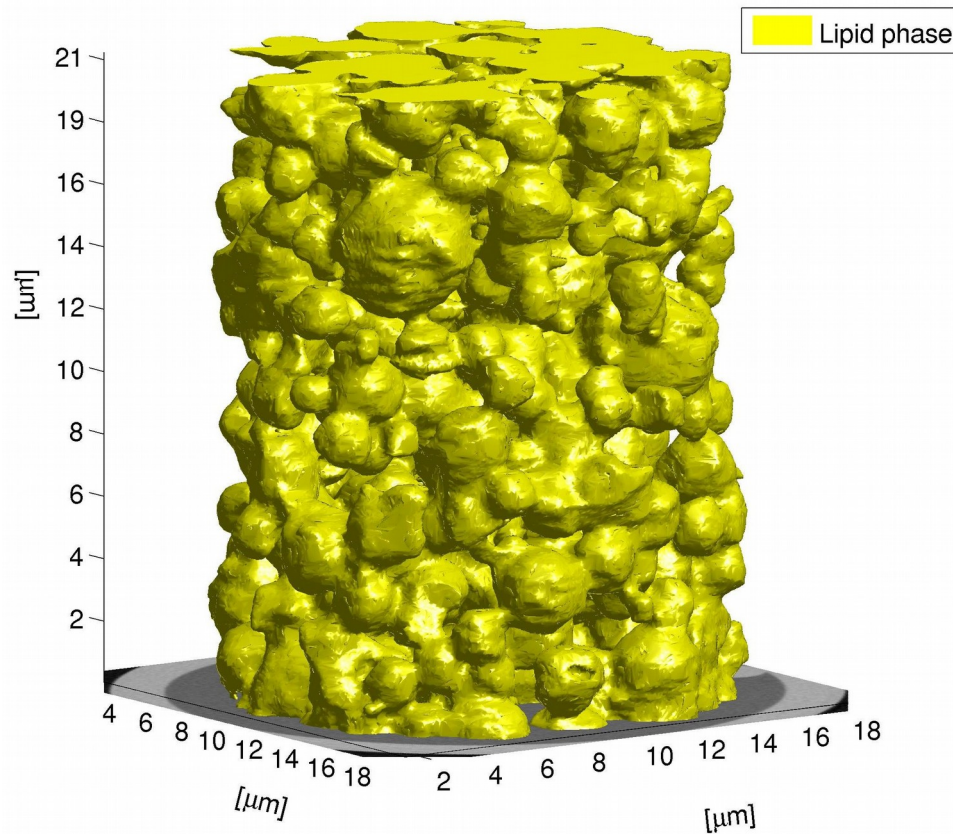


# Lipid network



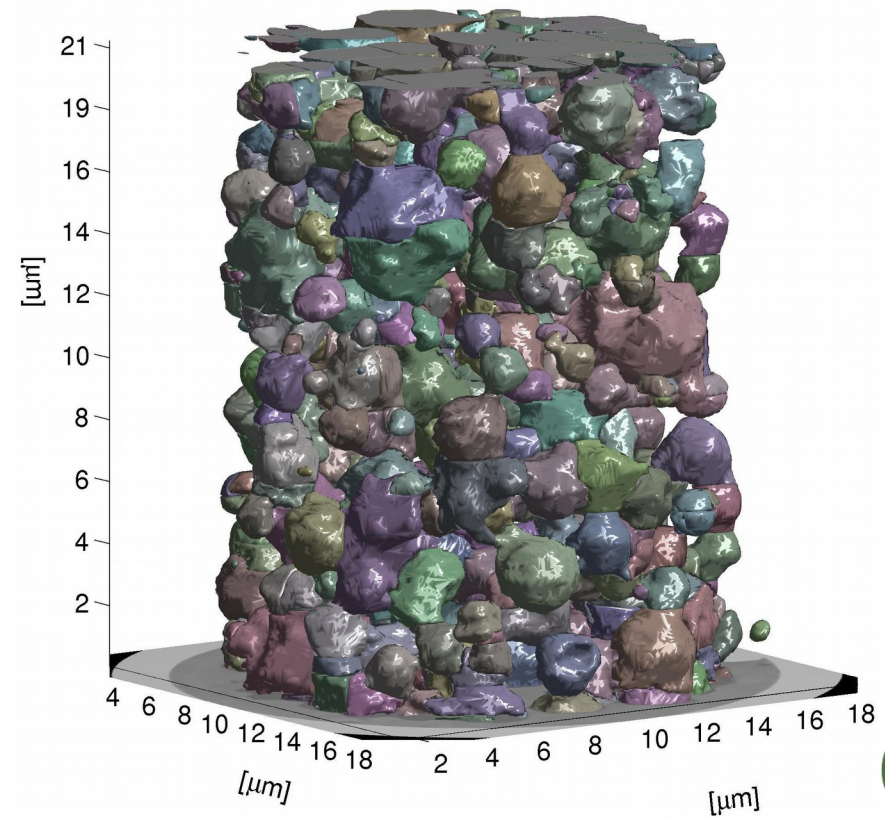
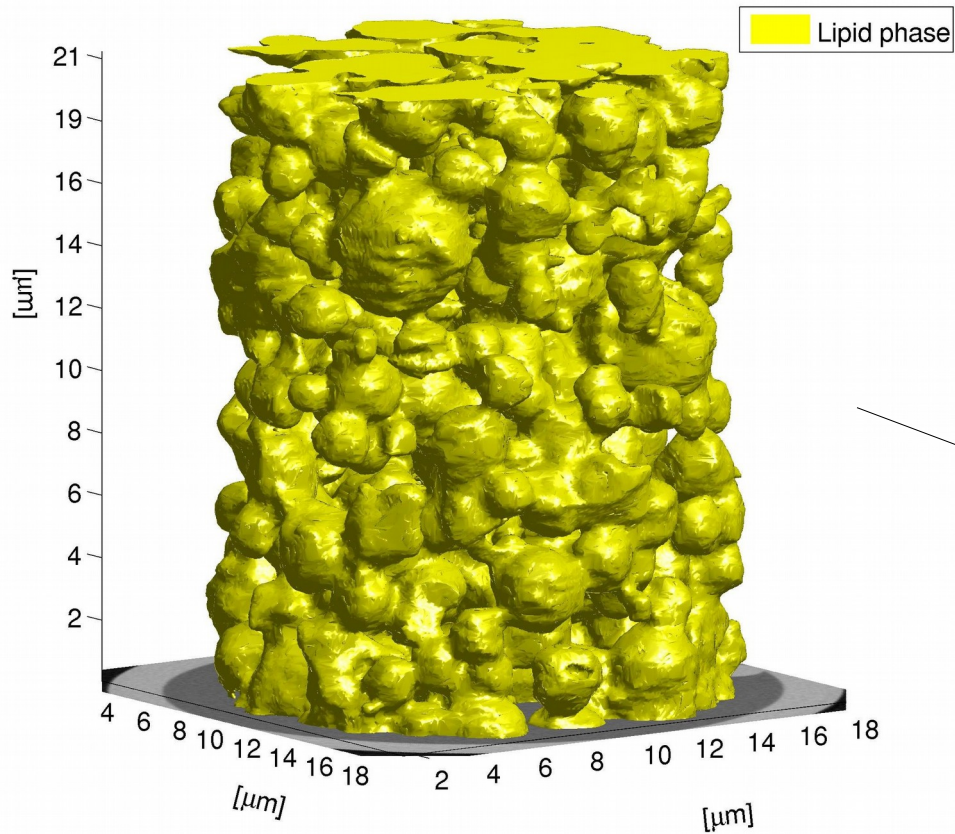
## Lipid network

*Partial coalescence of globules?*



# Lipid network

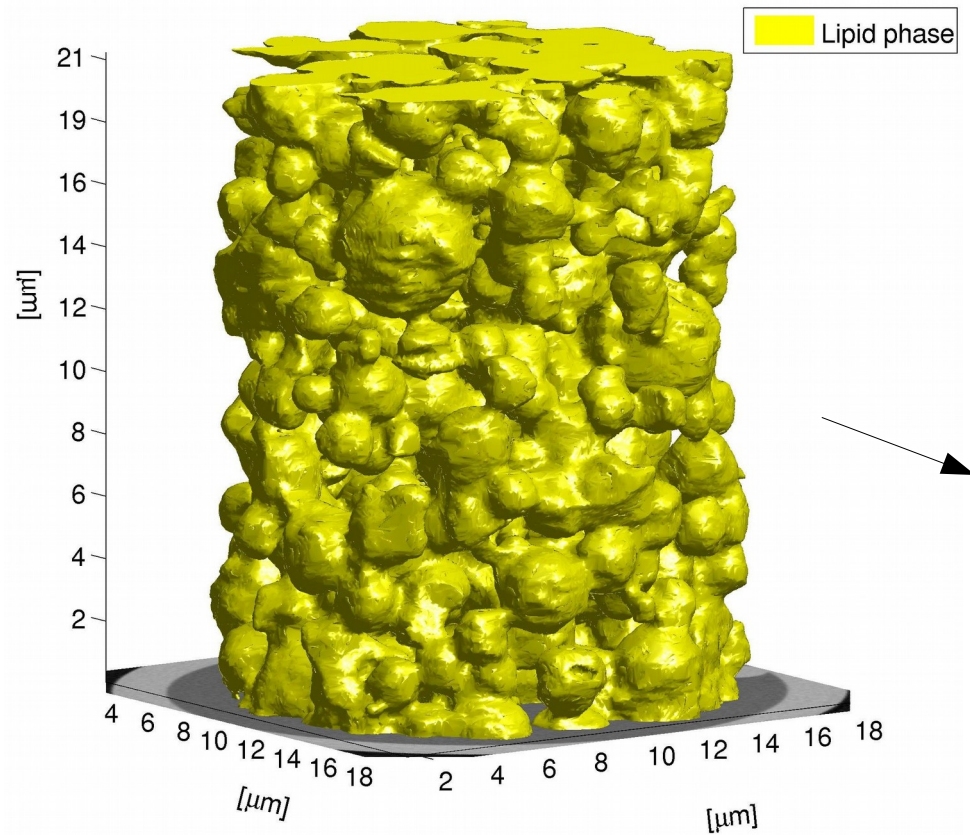
*Partial coalescence of globules?*



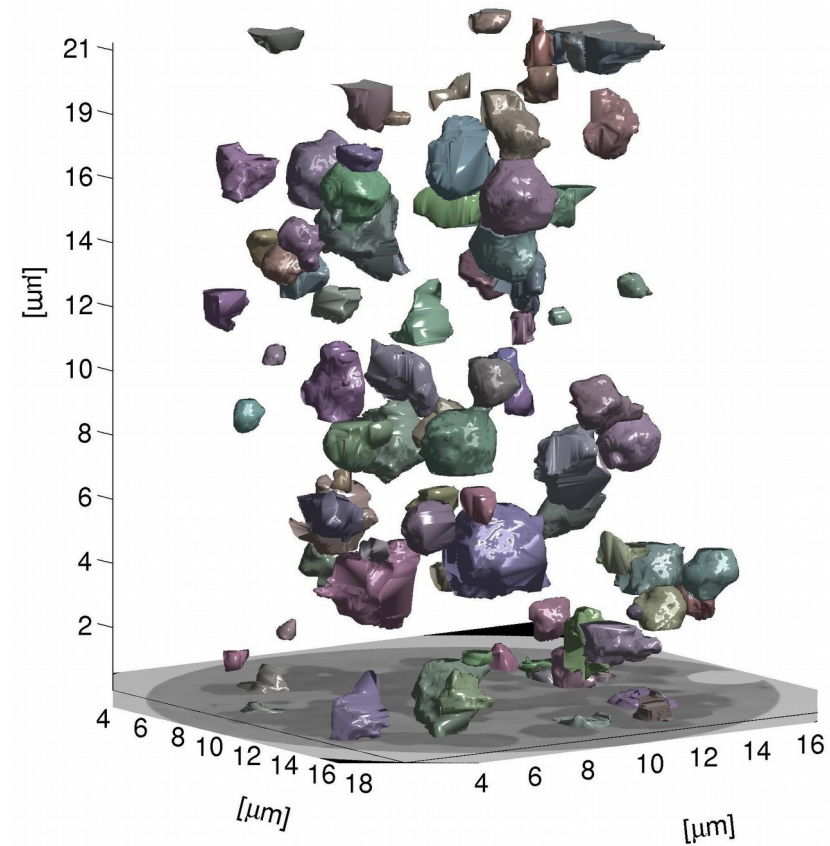
Watershed algorithm



# Lipid network



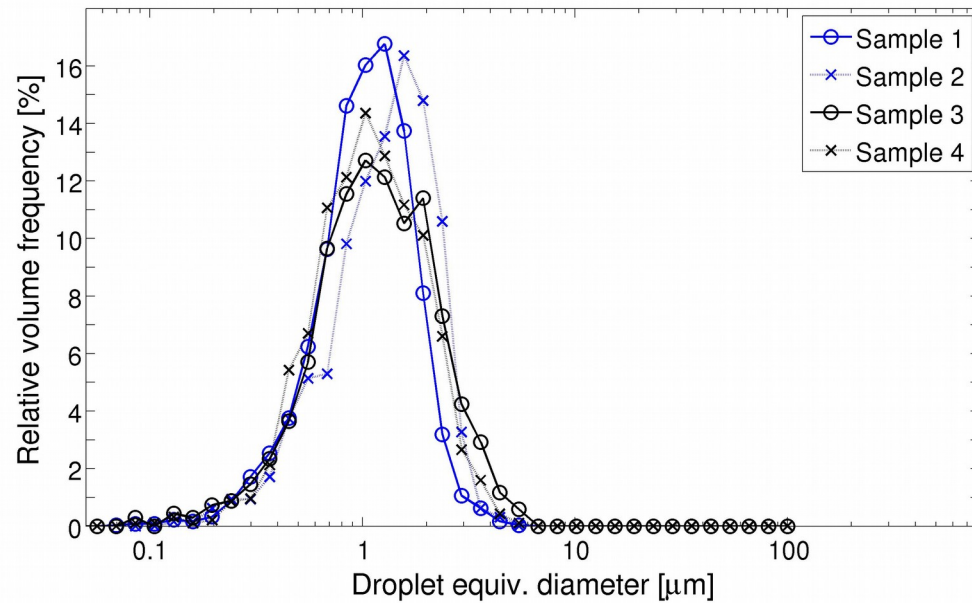
*Partial coalescence of globules?*



# Lipid network

*Lipid domain size distributions*

*Ptychographic X-ray CT*

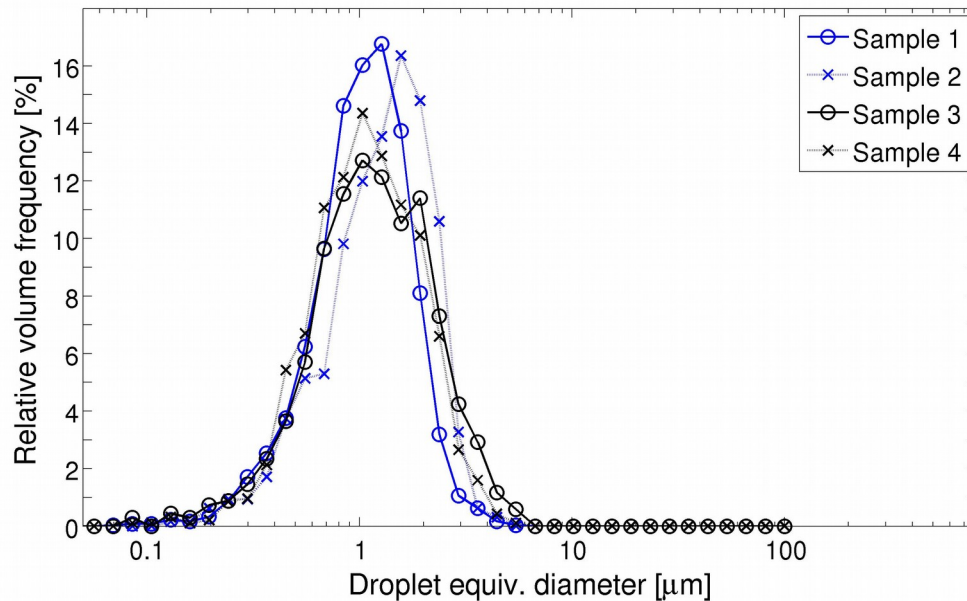




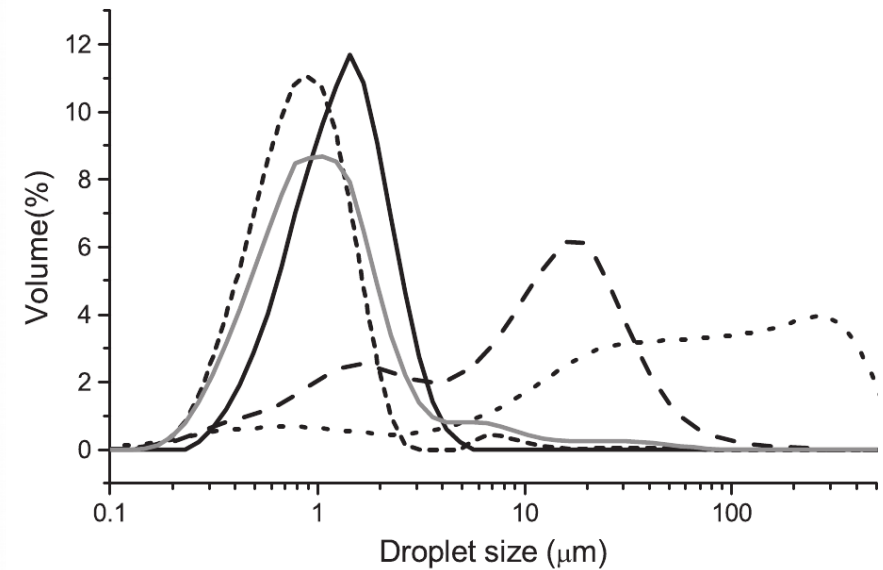
# Lipid network

## Lipid domain size distributions

*Ptychographic X-ray CT*



*Light scattering*



**Fig. 2.** The effect of monoglycerides on droplet size distribution in emulsions, — (solid black) control (solid black), - - - LACTEM (long dash), — The line style of "LACTEM + GMS" should be grey LACTEM + GMS (solid grey), - - - LACTEM + DATEM (short dash), ..... LACTEM + GMU (dot).

| Measurement   | DLS control | Sam1 | Sam2 | Sam3 | Sam4 |
|---------------|-------------|------|------|------|------|
| Mean diameter | 0.98        | 1.15 | 1.41 | 1.40 | 1.25 |

## Network analysis



*Fractal dimensions – a way forward?*

| Measurement       | Pty-1 | Pty-2 | Pty-3 | Pty-4 |
|-------------------|-------|-------|-------|-------|
| Fractal dimension | 2.67  | 2.42  | 2.44  | 2.48  |



## Network analysis



*Fractal dimensions – a way forward?*

| Measurement       | Pty-1 | Pty-2 | Pty-3 | Pty-4 |
|-------------------|-------|-------|-------|-------|
| Fractal dimension | 2.67  | 2.42  | 2.44  | 2.48  |

Table 2

Fractal dimension calculated via image analysis compared to fractal dimension calculated via rheology using the weak theory. Errors in  $D$  are standard errors of three replicates

| Fat system                          | Fractal dimension from image analysis | Fractal dimension from rheology (weak-link regime) | Percent deviation |
|-------------------------------------|---------------------------------------|--|-------------------|
| Cocoa butter #1                     | $2.31 \pm 1.7\%$                      | $2.37 \pm 4.0\%$                                   | 2.5               |
| NIE milkfat #4 (analysed using DMA) | $2.02 \pm 1.2\%$                      | $2.01 \pm 15.7\%$                                  | 1.5               |
| Palm oil                            | $2.82 \pm 0.6\%$                      | $2.82 \pm 0.6\%$                                   | 0.0               |
| Lard                                | $2.86 \pm 0.6\%$                      | $2.88 \pm 0.5\%$                                   | 1.0               |
| Tallow                              | $2.42 \pm 1.2\%$                      | $2.41 \pm 6.4\%$                                   | 0.4               |

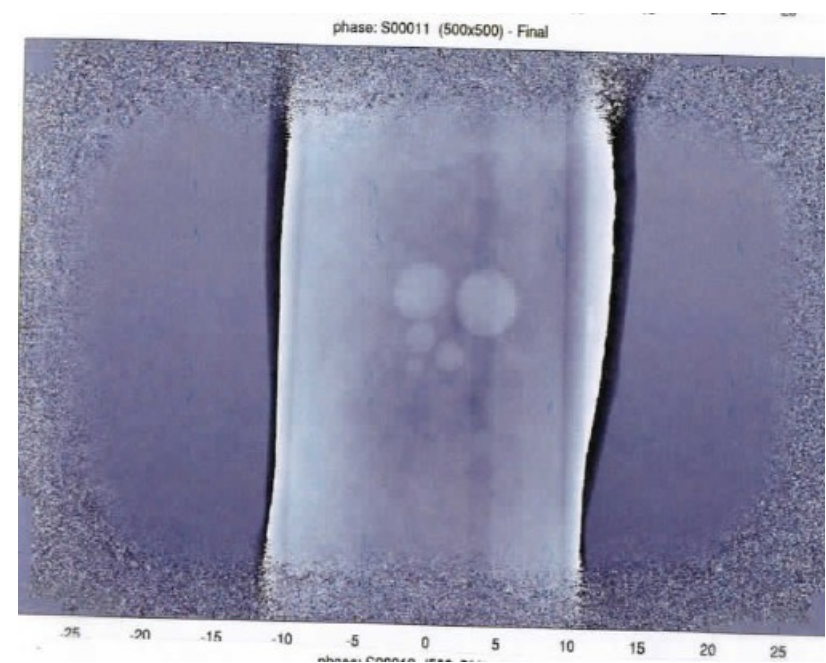
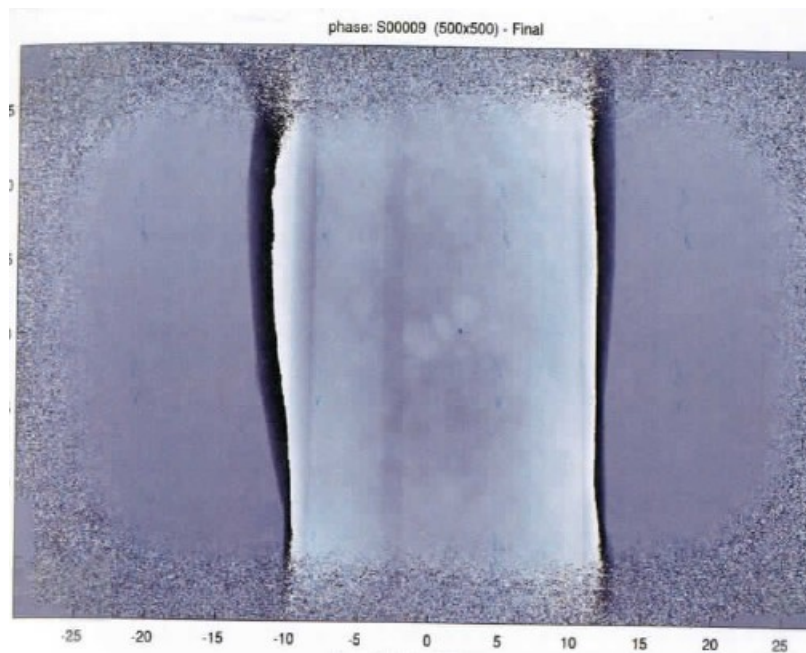


## Discussion and conclusion



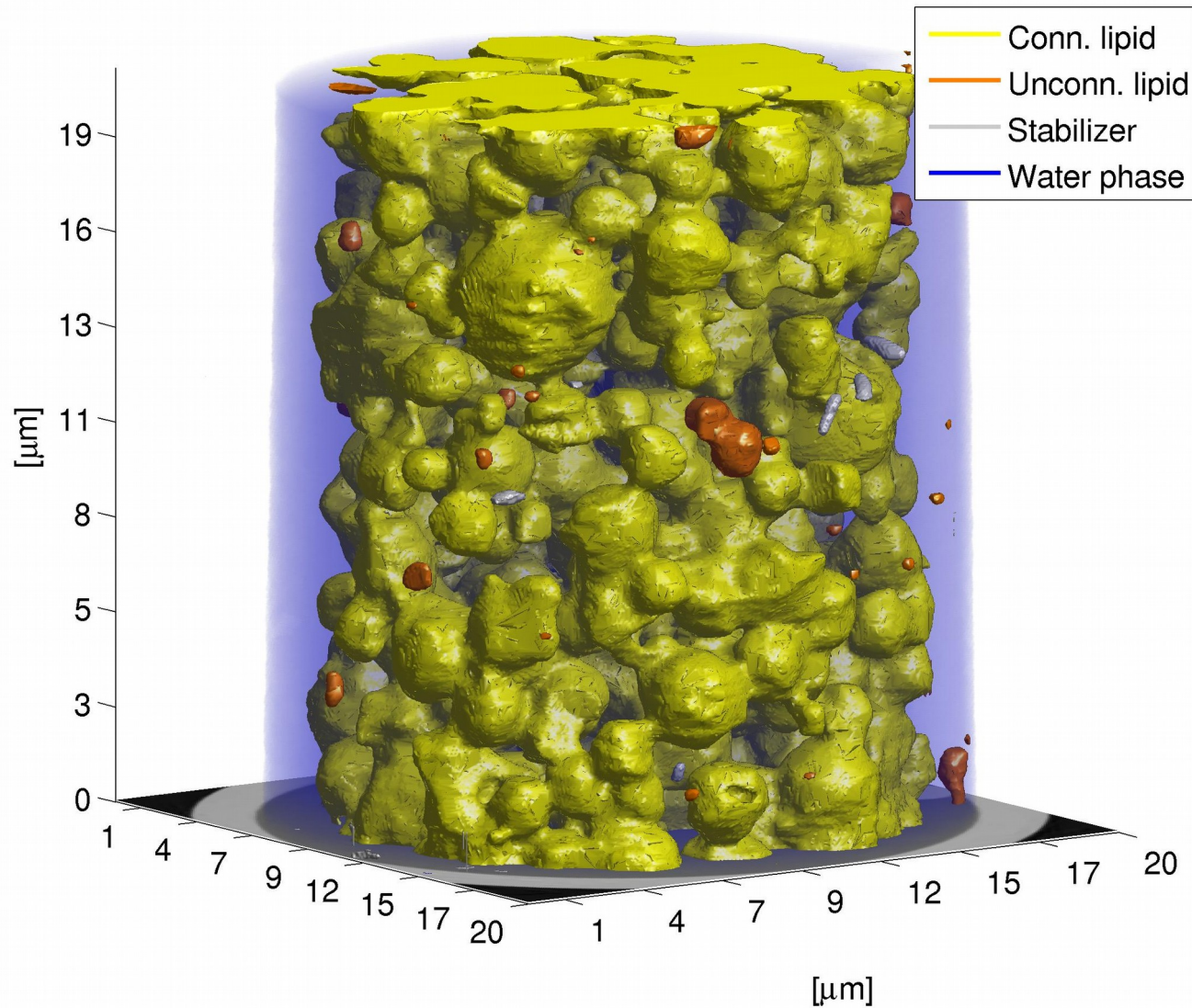
## Discussion - radiation damage

*High intensity of initial beam causes bubble formation*



## Discussion – sample container

*Small container effects the sample?*



## Conclusion and outlook

*The 3D nanostructure of a dairy-like emulsion*

### Conclusion

- The nanostructure of the food emulsion was imaged using ptychographic X-ray CT.
- Lipid-phase forms 3D network
- Consistent with (extreme) partial coalescence.

### Outlook

- From tomographic scans to viscoelastic properties - *Finite element modelling?*
- Increased availability of method – MAX IV



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