X-ray Micro Computer Tomography Microstructure in Ice cream, sorbet and frozen products and desserts

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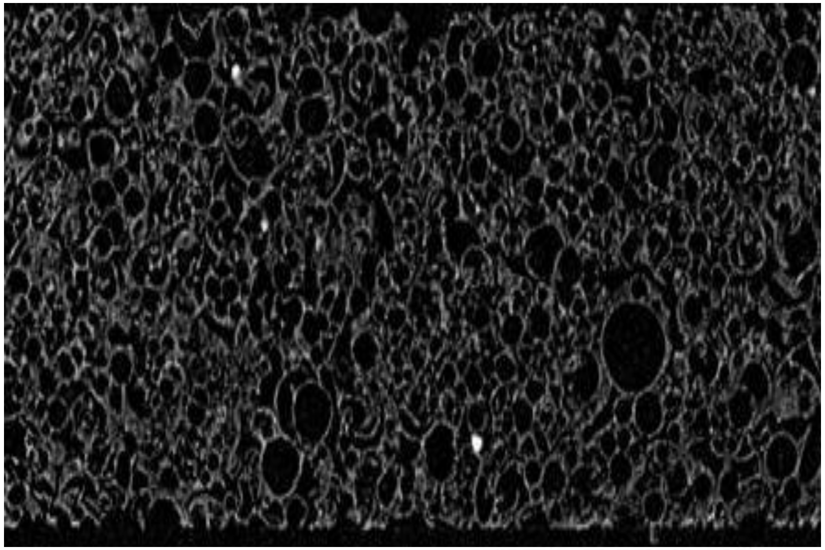
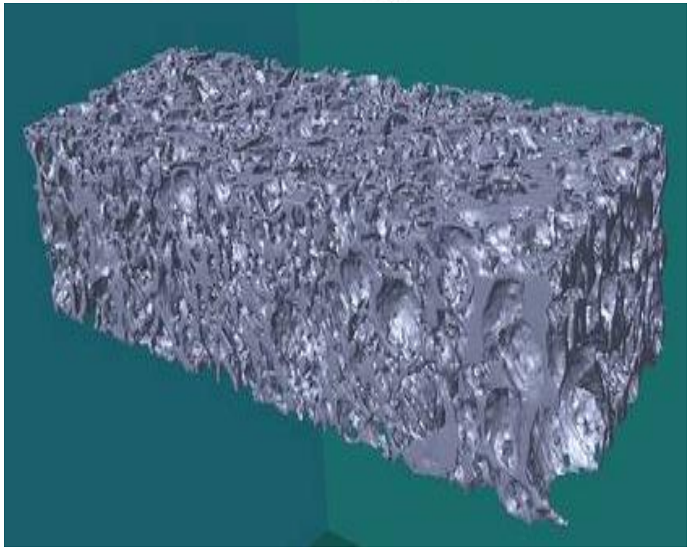
X-ray tomography (XRT)

micro/nano computed tomography (µCT/nCT)

- Non-invasively measurement of the 3D structure of objects at spatial resolution below 1 µm



2D shadow images
Pixel: → X-ray transmission



3D volumetric data :
Voxel → local density

X-ray tomography (XRT) micro computer tomography

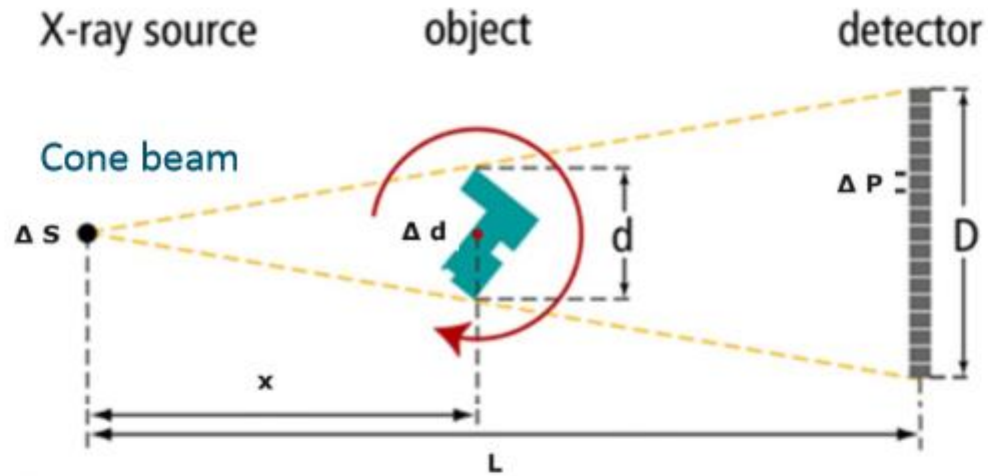
- Synchrotron
  polychromatic or monochromatic beam, parallel beam
  resolution <0.5 µm, measurement time ~ 1 min

- Lab scale,
  resolution <1 µm, measurement time > 15 min

    - source/detector rotates
      (medical scanners)

    - sample rotates

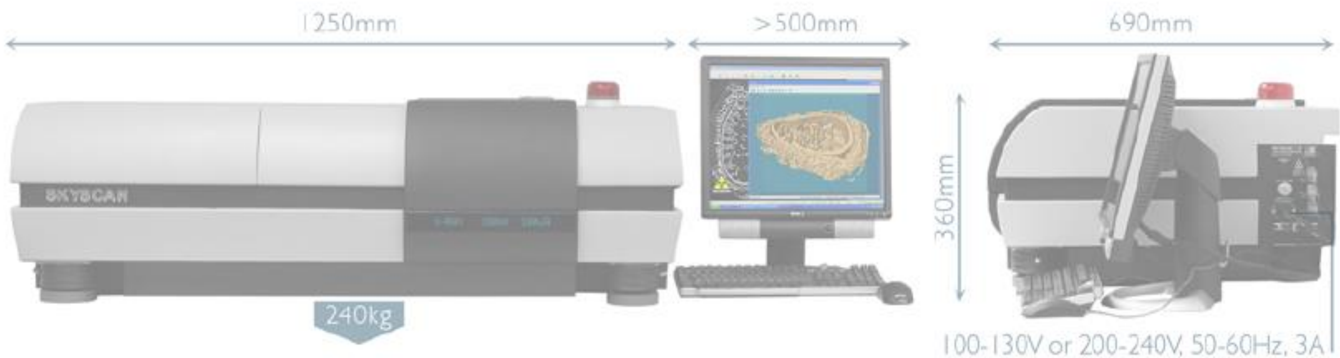
Technical characteristics lab scale scanner



- Spatial resolution / magnification (below 5 µm)
- Low acquisition time (down to 10 min scale)
- Good contrast and signal to noise ratio

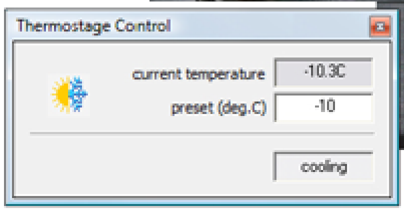
Micro CT used

KU Leuven's facilities



Heating and Cooling Stages

The heating and cooling stages allow micro-CT scanning under controlled object temperature above or below ambient. The heating stage can keep an object at any temperature up to +85°C. The cooling stage can keep an object at sub-zero temperature down to 30-40°C below ambient. An internal



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P. Verboven, Bart Nicolaï

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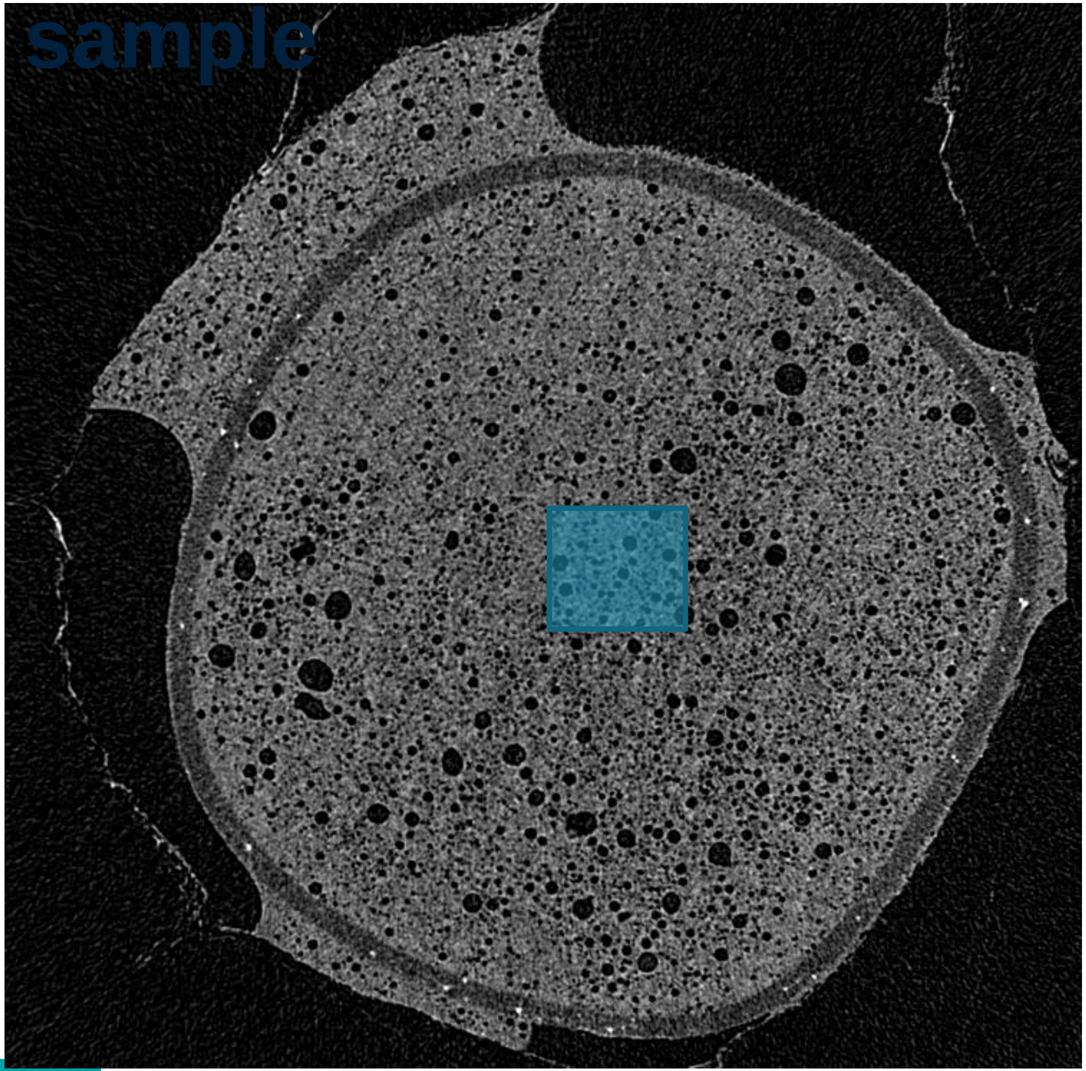
X-ray micro CT Ice cream sample



Monitoring microstructure
of Ice cream samples
3phases (air bubbles,
Ice crystals, fat globules)

- Size distribution
- microstructure
- fat destabilisation

- SkyScan 1172
- heating cooling stage

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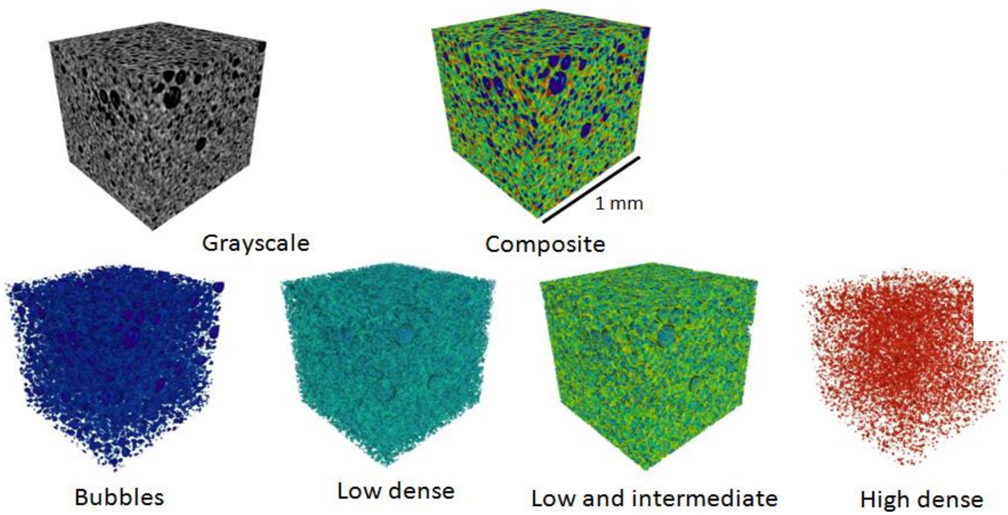
Computing facilities

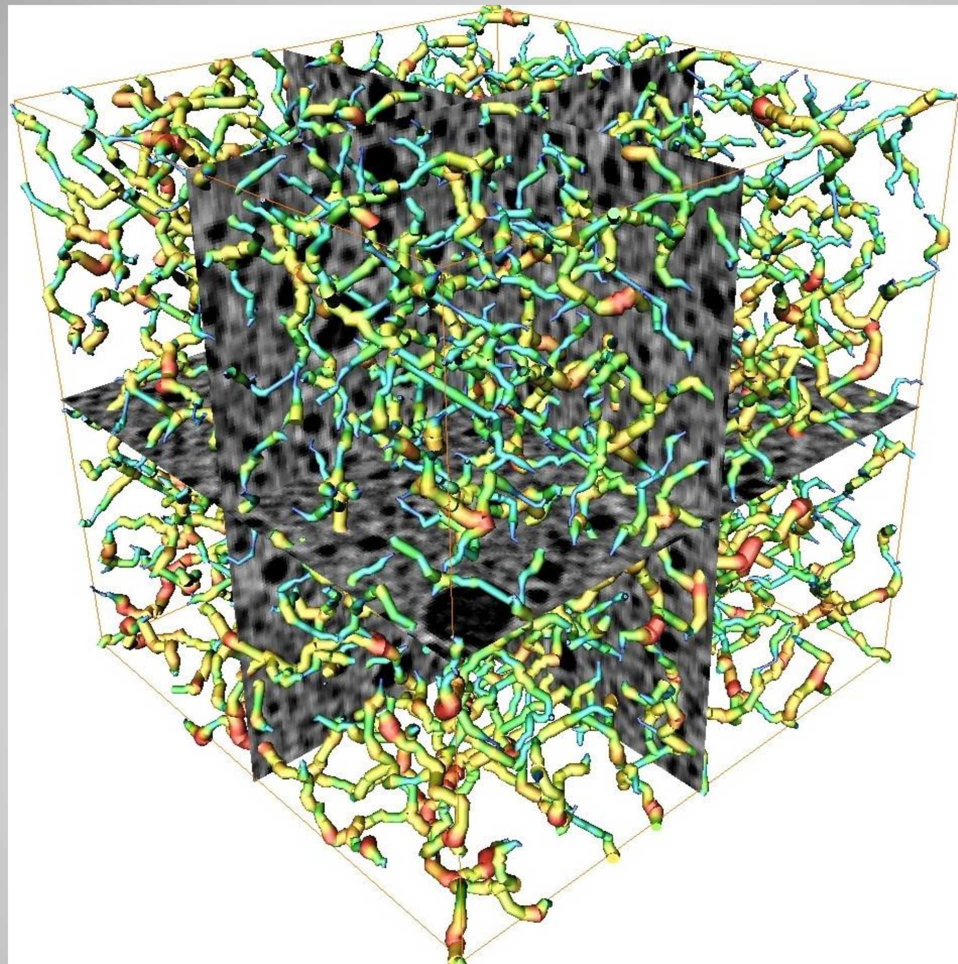
- Dedicated high performance workstations with maximal graphic power and memory
  - Fast tomography reconstruction software with special correction algorithms.
  - State of the art software for visualisation, image analysis,



Volume size: 200 x 200 x 200 pixels

Resolution: 5 um/ pixel

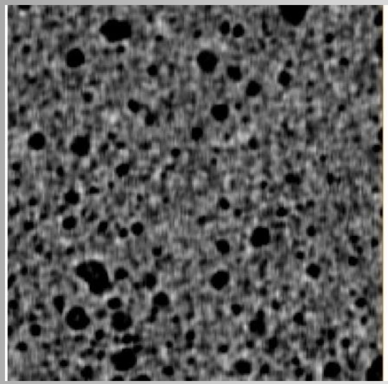
Color bar in terms of grayscale intensity



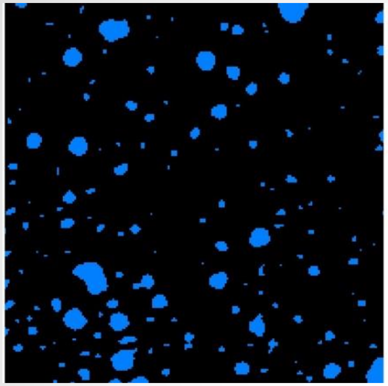
Solid phase skeleton

Narrow Medium Wide

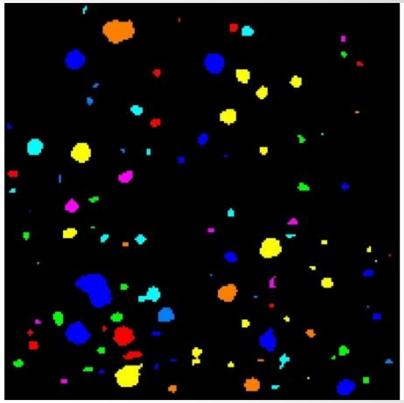
Bubble segmentation



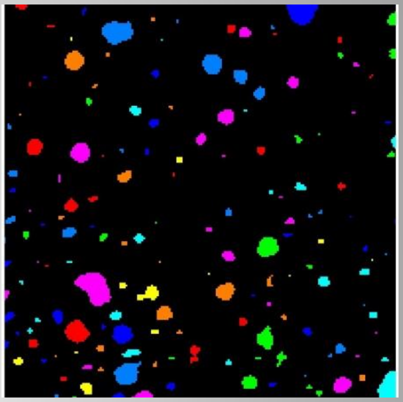
Grayscale



Threshold

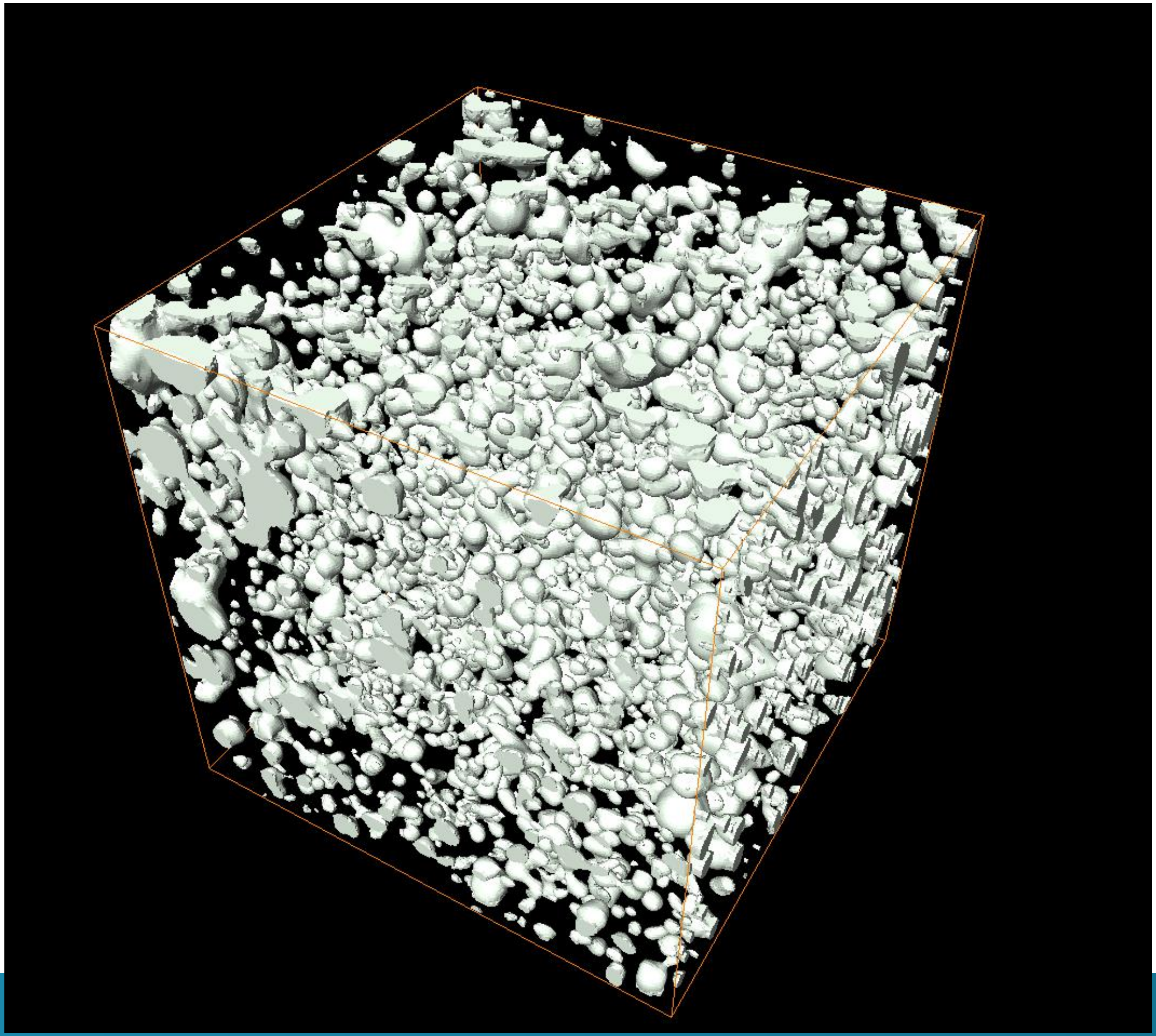


Label

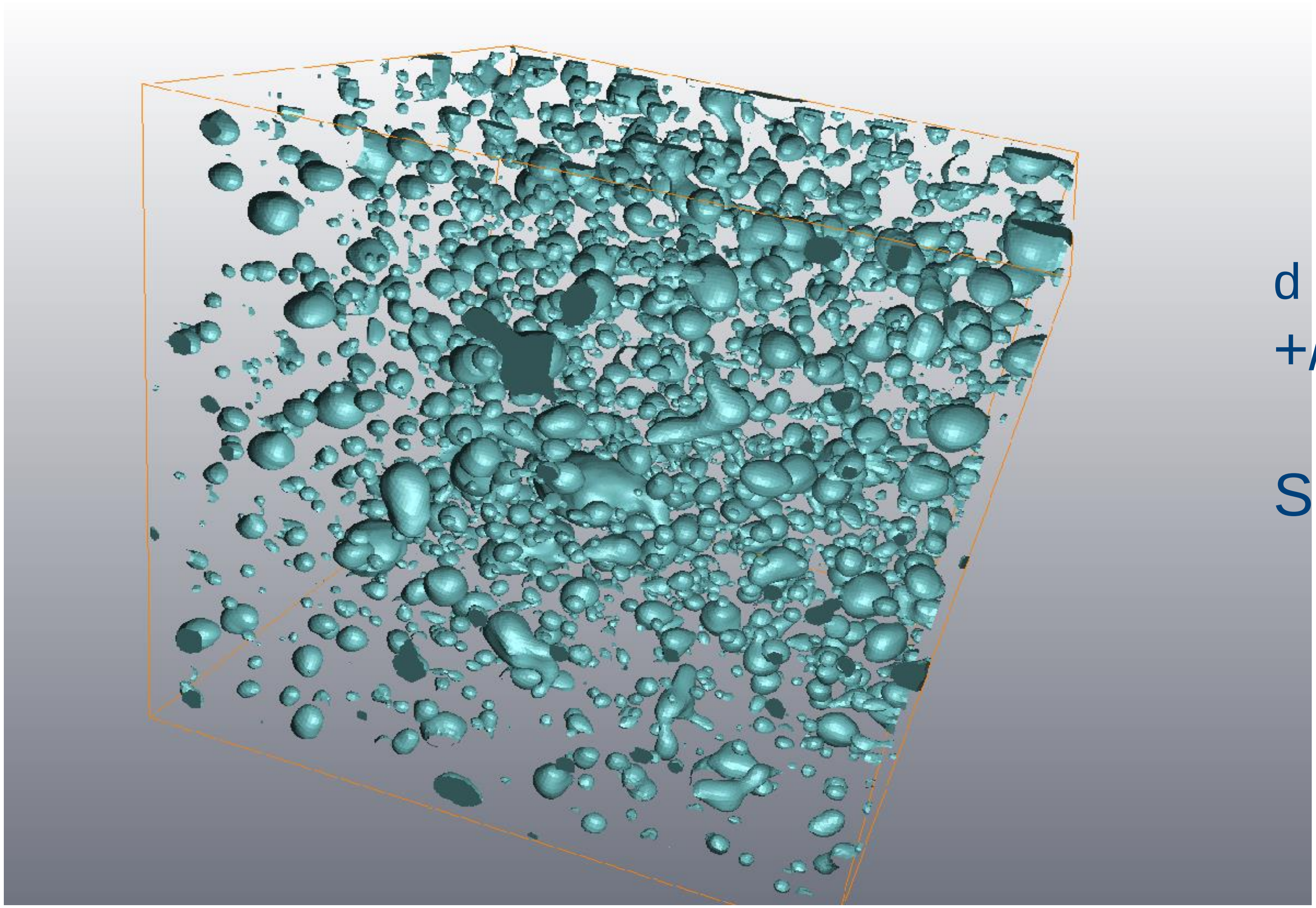


Label filtered

Air bubbles sample 12%fat



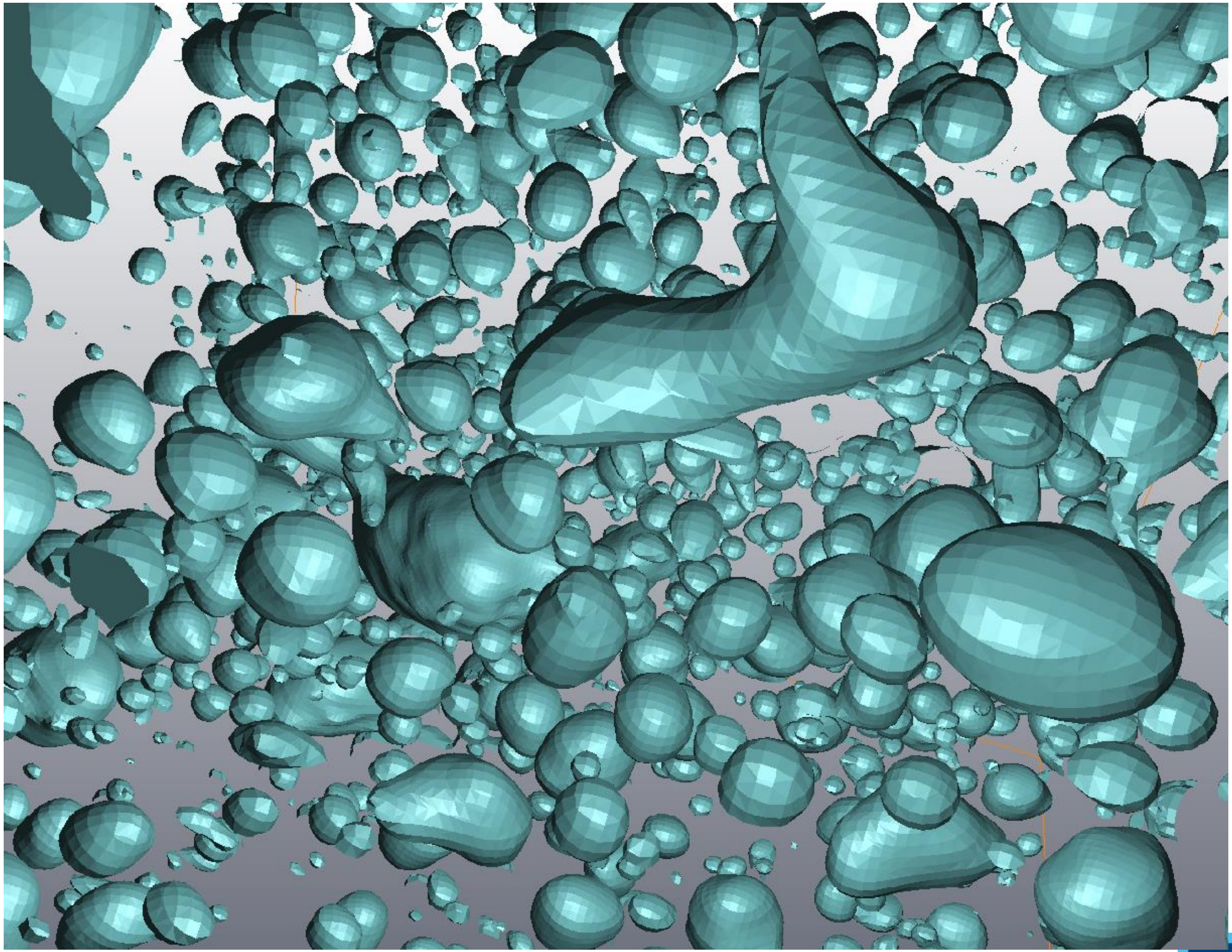
Air bubbles 1000RPM 12% fat



d moy 12µ
+/- 10µ

Spher 0.82

Air bubble inside 12%fat)



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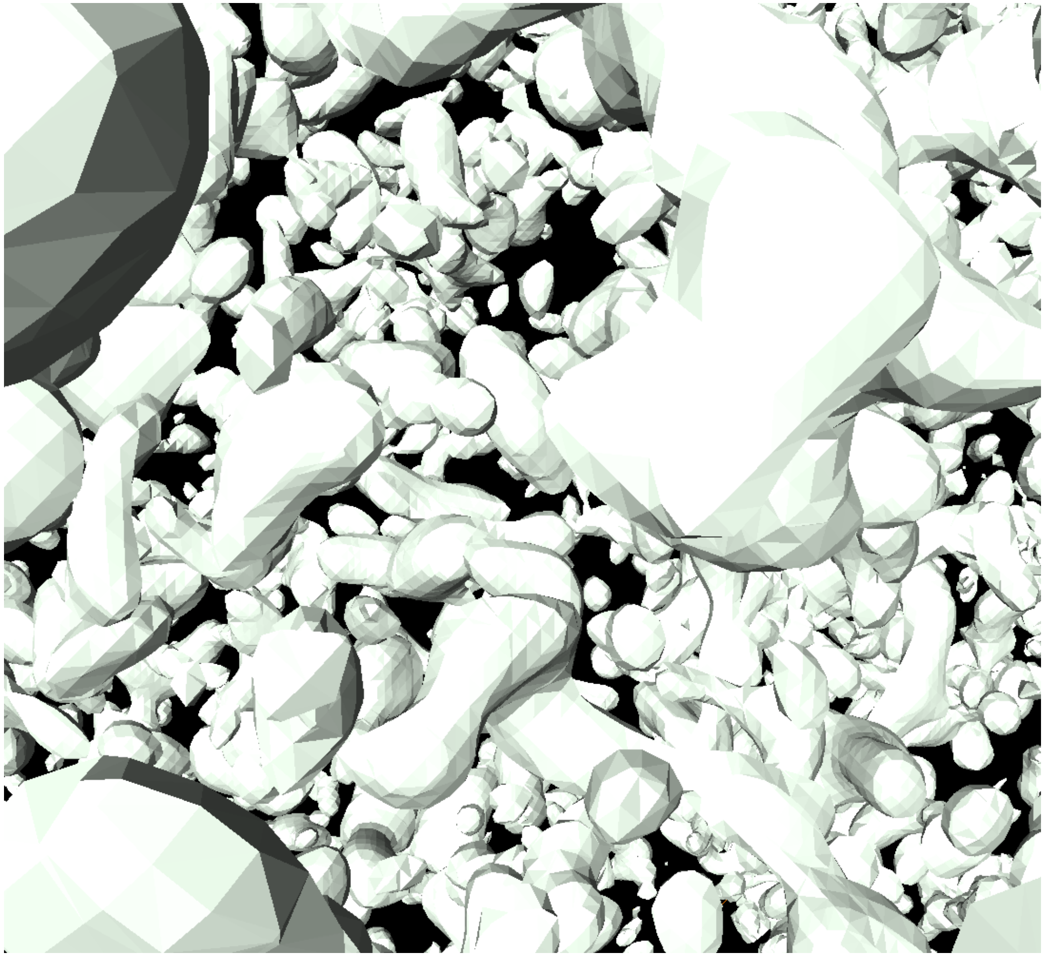
Ice crystals sample 12%f

d moy 24µ
+/- 11µ

Spher 0,53

Ice crystals inside the ice cream sample 12%f



d moy 24µ
+/- 11µ

Spher 0,53

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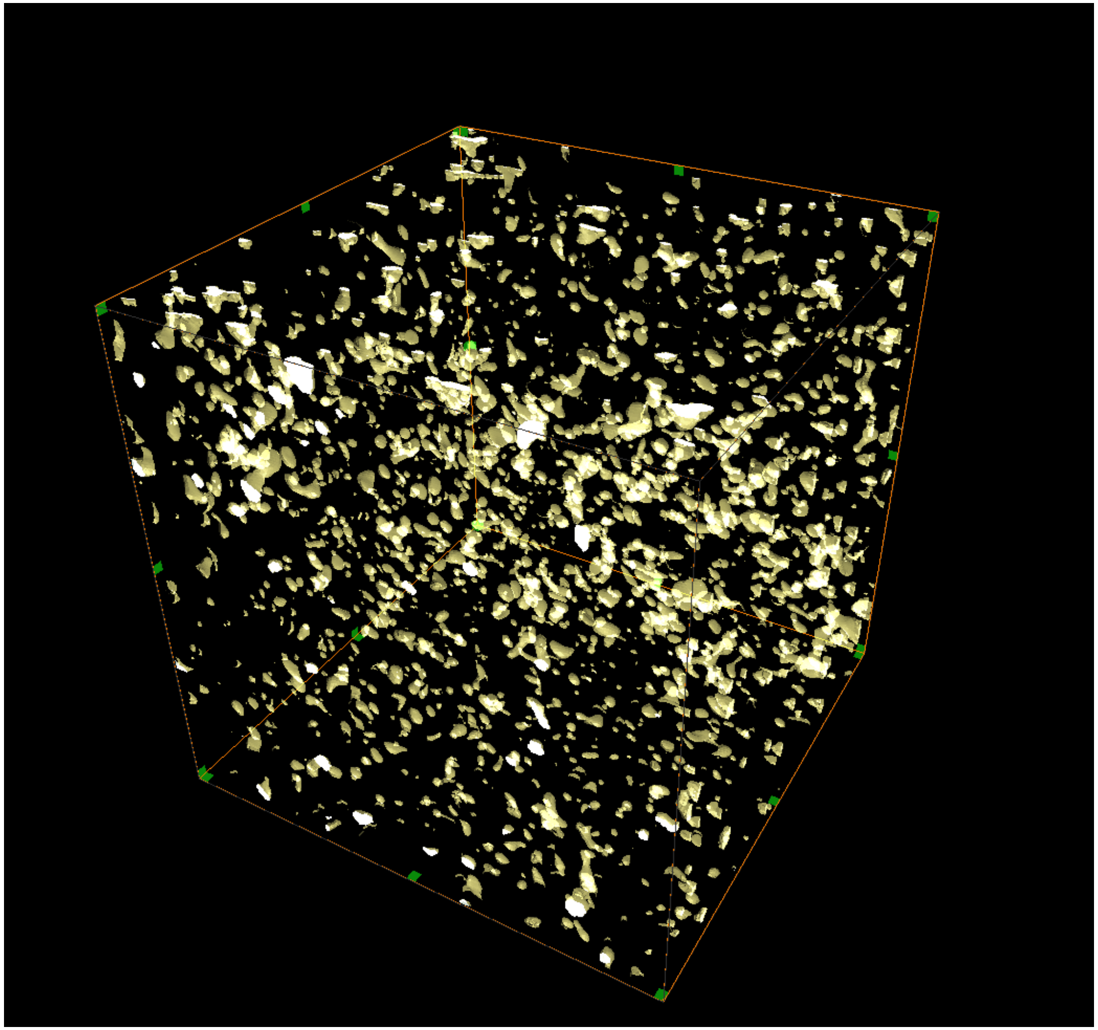
Fat in Ice cream sample 1000 12%fat



d moy 16µ
+/- 13µ

Spher 0,71

Freezing and storage microstructure studies

- Freezing
  - Decreases product T
  - Ice formation

  →
  - Preserve food quality
  - Prolong storage life

- Frozen chain
  - Set of refrigeration steps in handling of foods

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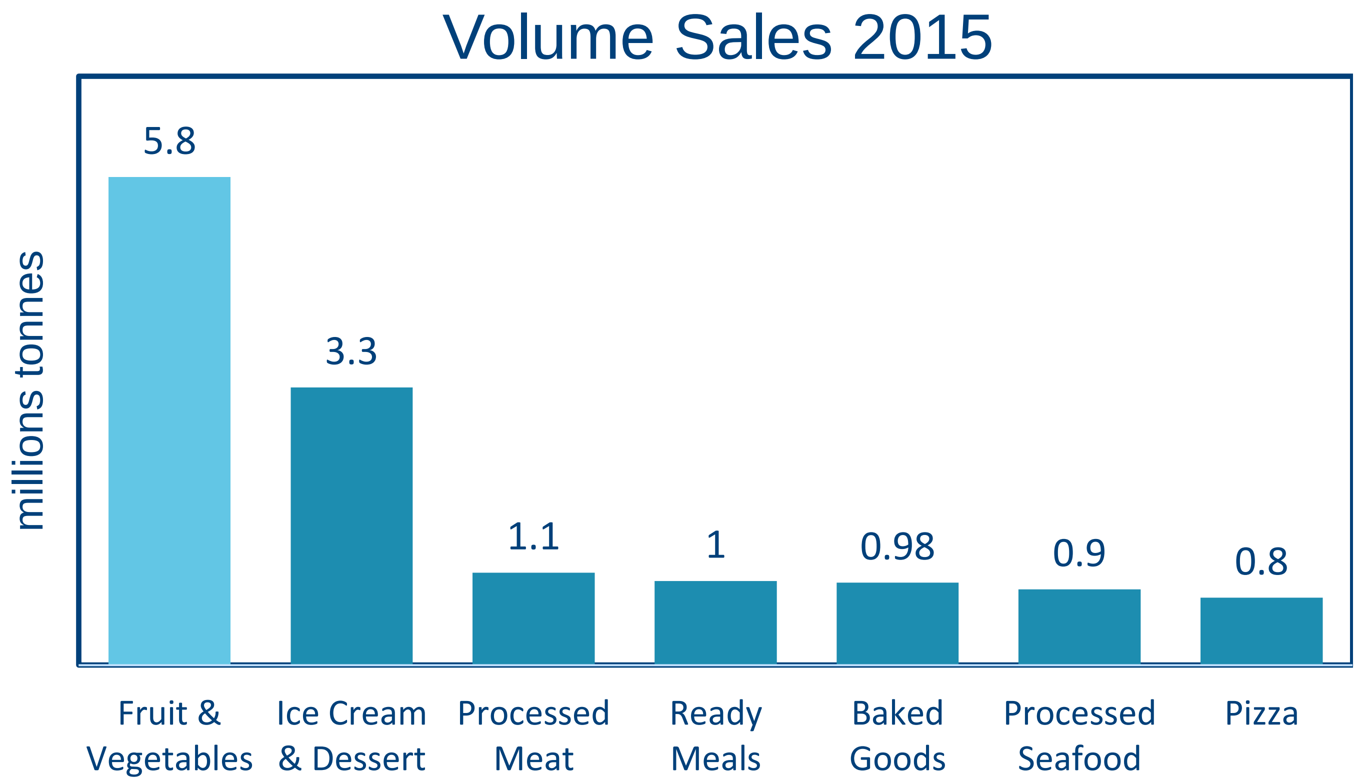
Frozen food market

- EU frozen food market



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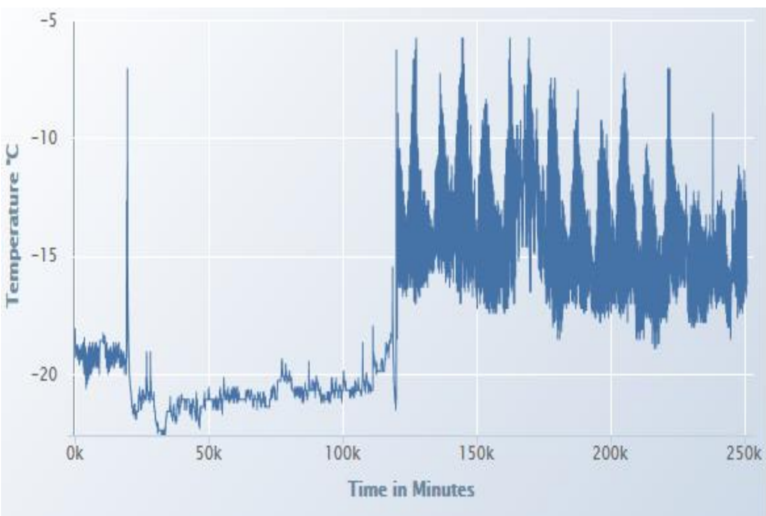
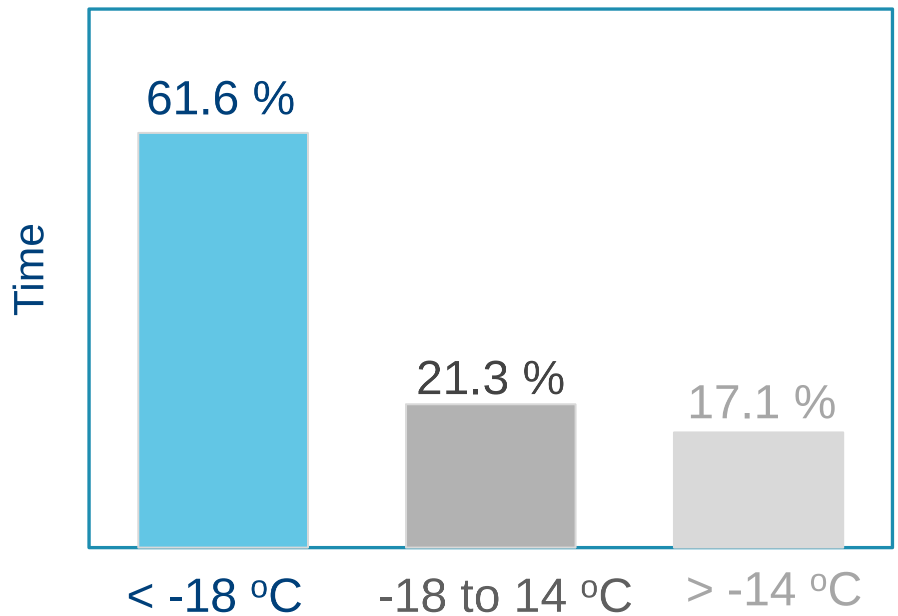
Temperature fluctuations

Frisbee: built with real t-T database along cold chain



30 foods in frozen chain for 174 d

t-T data



Quality changes in frozen foods

Instability of frozen water

Food microstructure: key parameter to defines food quality

Quality loss

Research questions

- Characterize microstructure of frozen foods ?
- Visualize the evolution of ice crystals ?
- Model ice recrystallization during storage ?
- Effects of fluctuating temperature on quality ?

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Objectives and outline

Develop and apply measurement and modeling tools to improve understanding of microstructural and quality changes during frozen storage

- Developing X-ray µCT method to characterize the 3D microstructure of frozen food
  - ↓ Tomographic imaging of ice crystal changes during frozen storage
  - ↓ Modeling ice recrystallization in food stored under dynamic temperatures
- Kinetics of food quality changes during frozen storage

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Objectives and outline

Develop and apply measurement and modeling tools to improve understanding of microstructural and quality changes during frozen storage

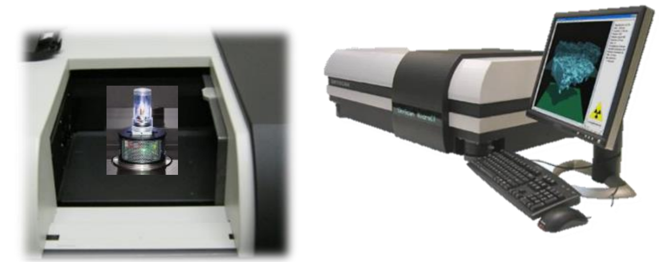
Developing X-ray µCT method to characterize the 3D microstructure of frozen product

Tomographic imaging of ice crystal changes in food during frozen storage

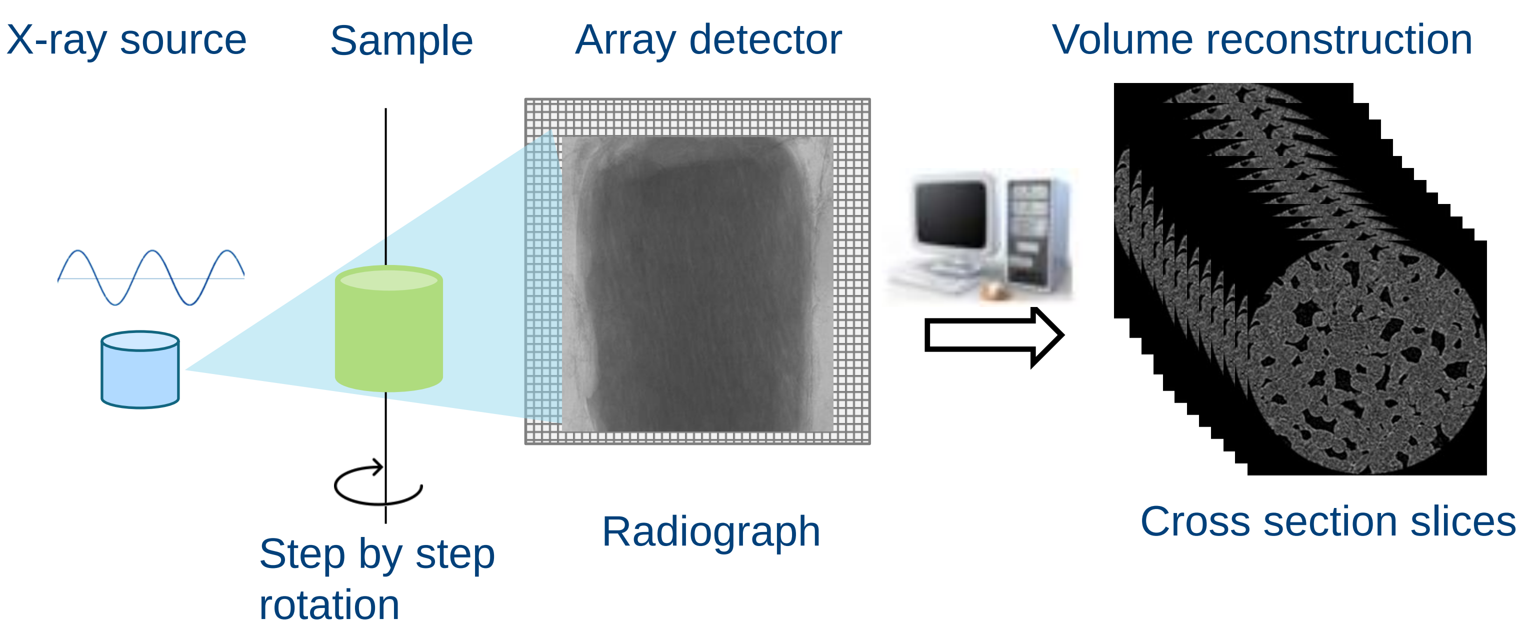
Modeling ice recrystallization in foodstored under dynamic temperatures

Kinetics of foodquality changes during frozen storage

X-ray micro-CT

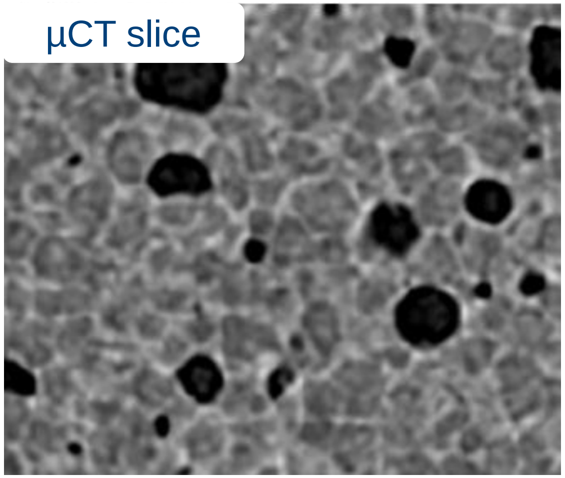


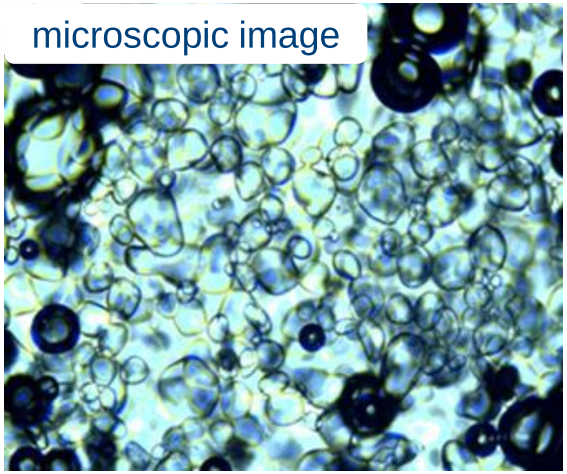
Cooling stage

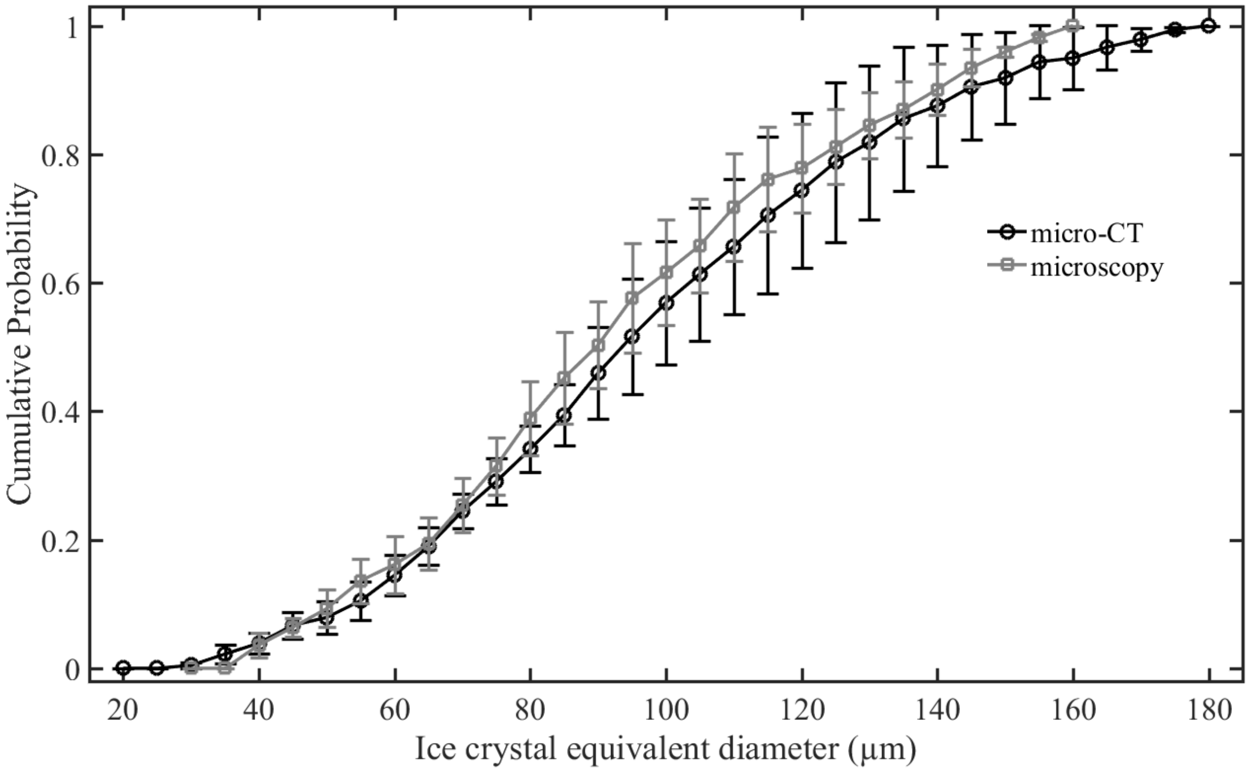


Vicent et al., 2017

Imaging calibration



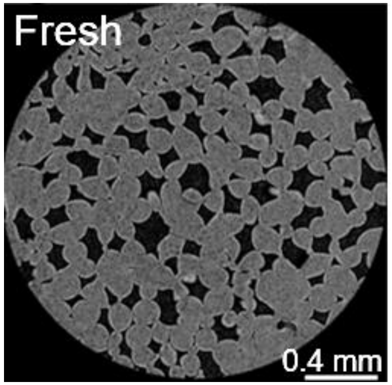


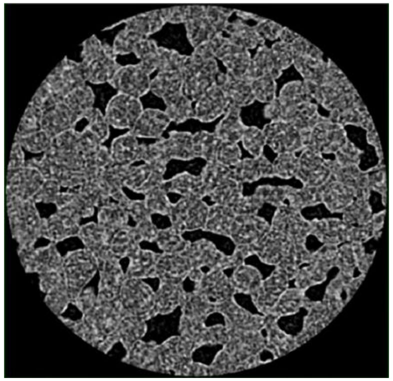


Apple sorbet

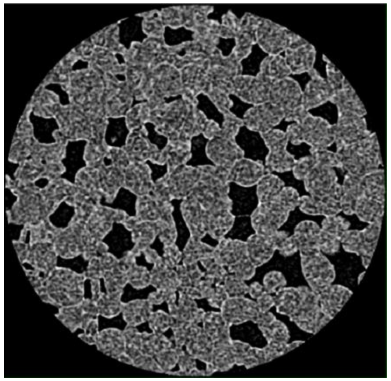
Vicent et al., 2017

Results: Freezing rate effects

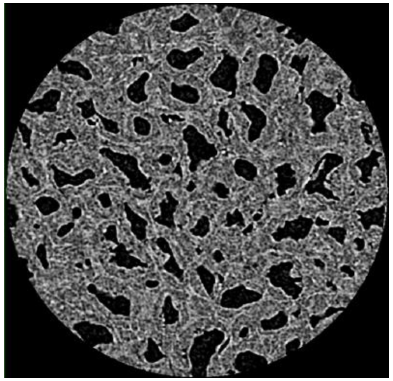




Fast freezing
(18.4 ºC/min)



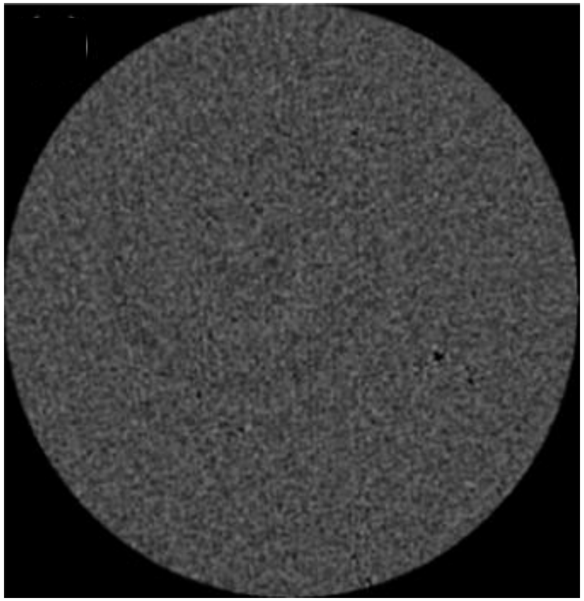
Intermediate freezing
(12.6 ºC/min)



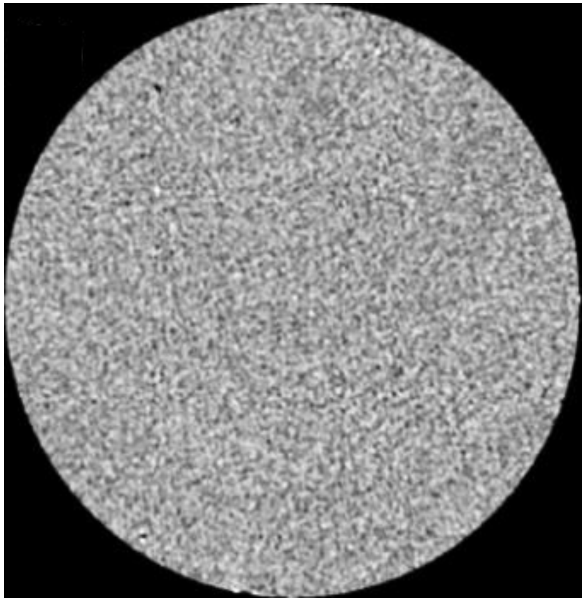
Slow freezing
(2.2 ºC/min)

Vicent et al., 2017

X-ray attenuation coefficient references



Frozen water



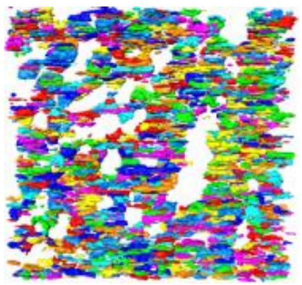
Jellified conc. solution

Vicent et al., 2017

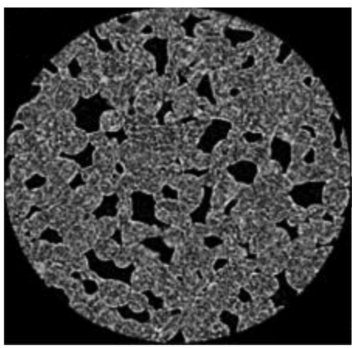
Image processing

Image acquisition

Individual analysis



Ice crystals



Original X-ray CT image

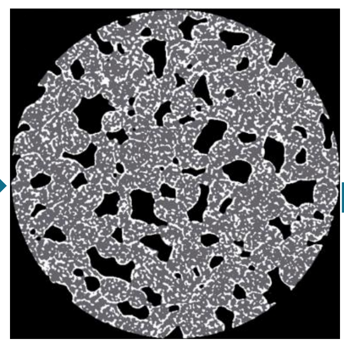
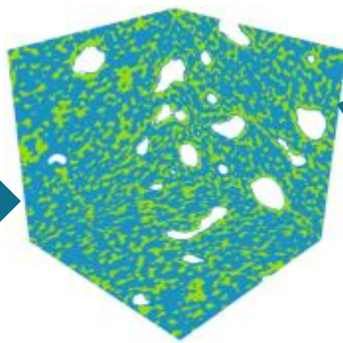


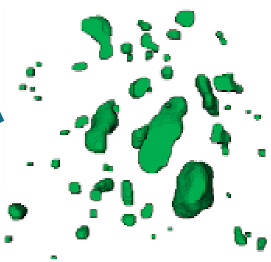
Image segmentation



3D visualization

Ice crystals

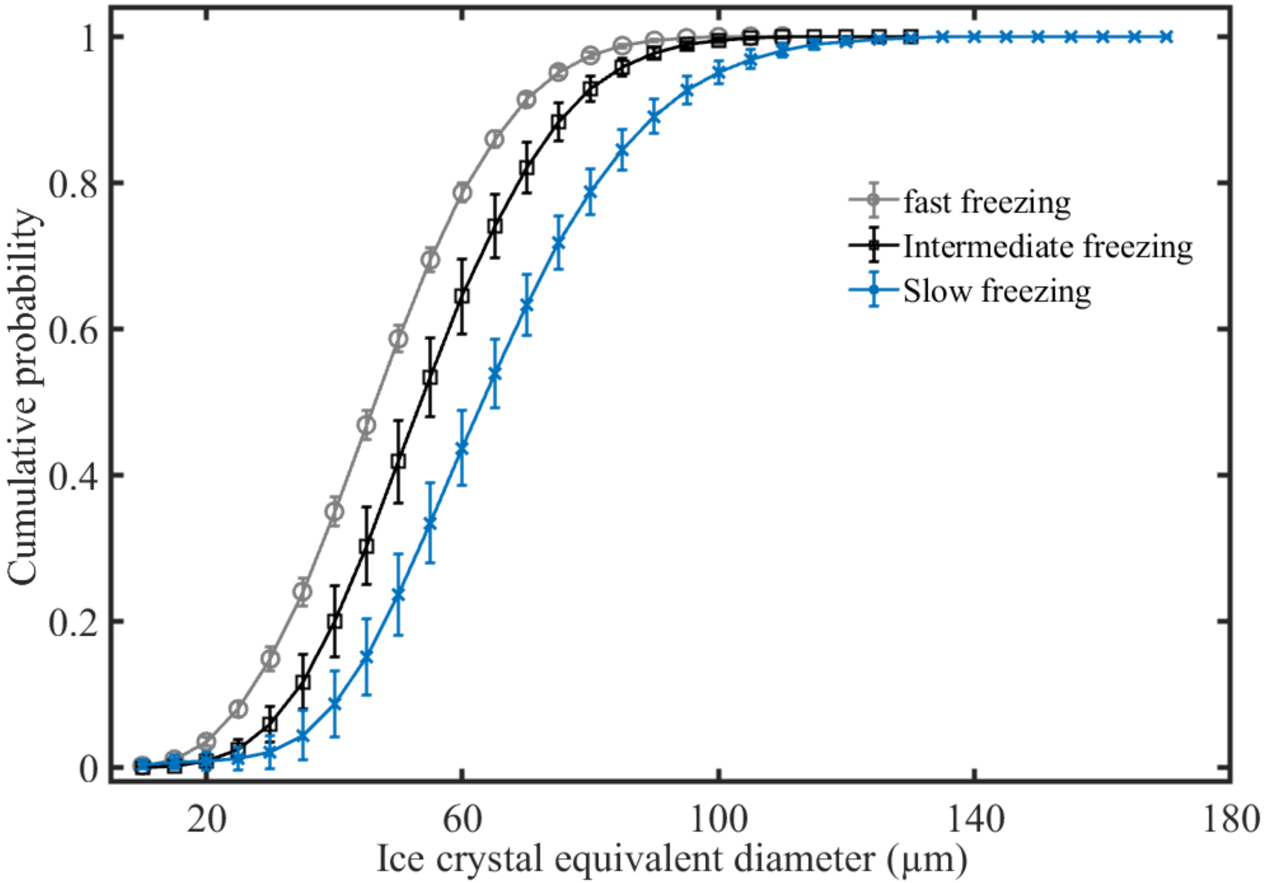
Air spaces



Airspaces

Vicent et al., 2017

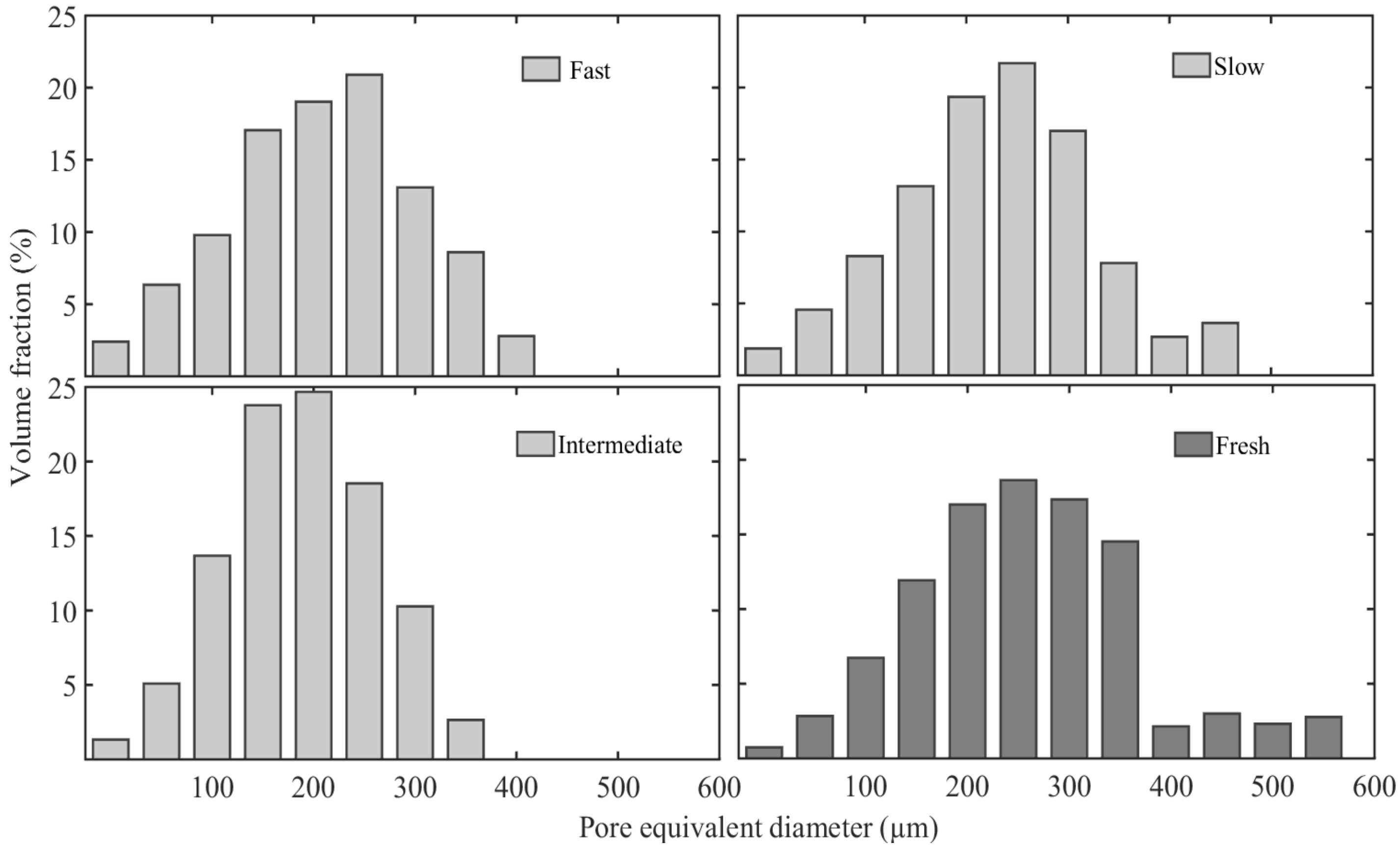
Ice crystal size distributions



$\bar{x}$ = 41 µm
$\bar{x}$ = 55 µm
$\bar{x}$ = 71 µm

Vicent et al., 2017

Pore size distributions



- Pore size: frozen tissue < fresh tissue
- Volumetric expansion of frozen water

Vicent et al., 2017

Objectives and outline

Develop and apply measurement and modeling tools to improve understanding of microstructural and quality changes during frozen storage

Developing X-ray µCT method to characterize the 3D microstructure of frozen apple

Tomographic imaging of ice crystal changes in products during frozen storage

Modeling ice recrystallization in stored under dynamic temperatures

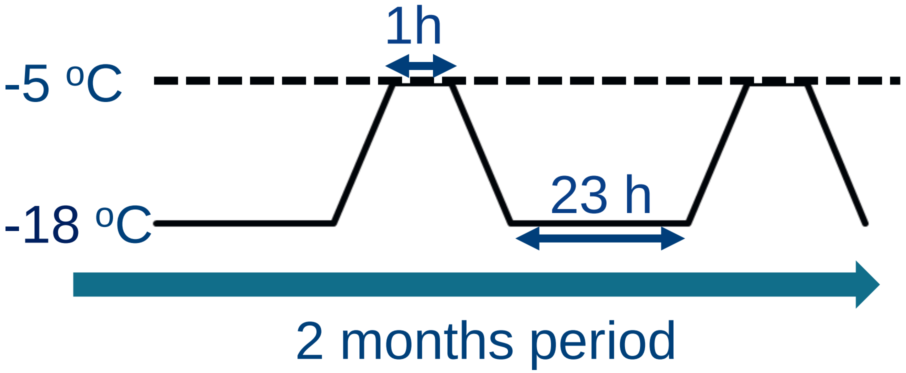
Kinetics of apple tissue quality changes during frozen storage

Sample preparation and storage

5 replicates

Straw

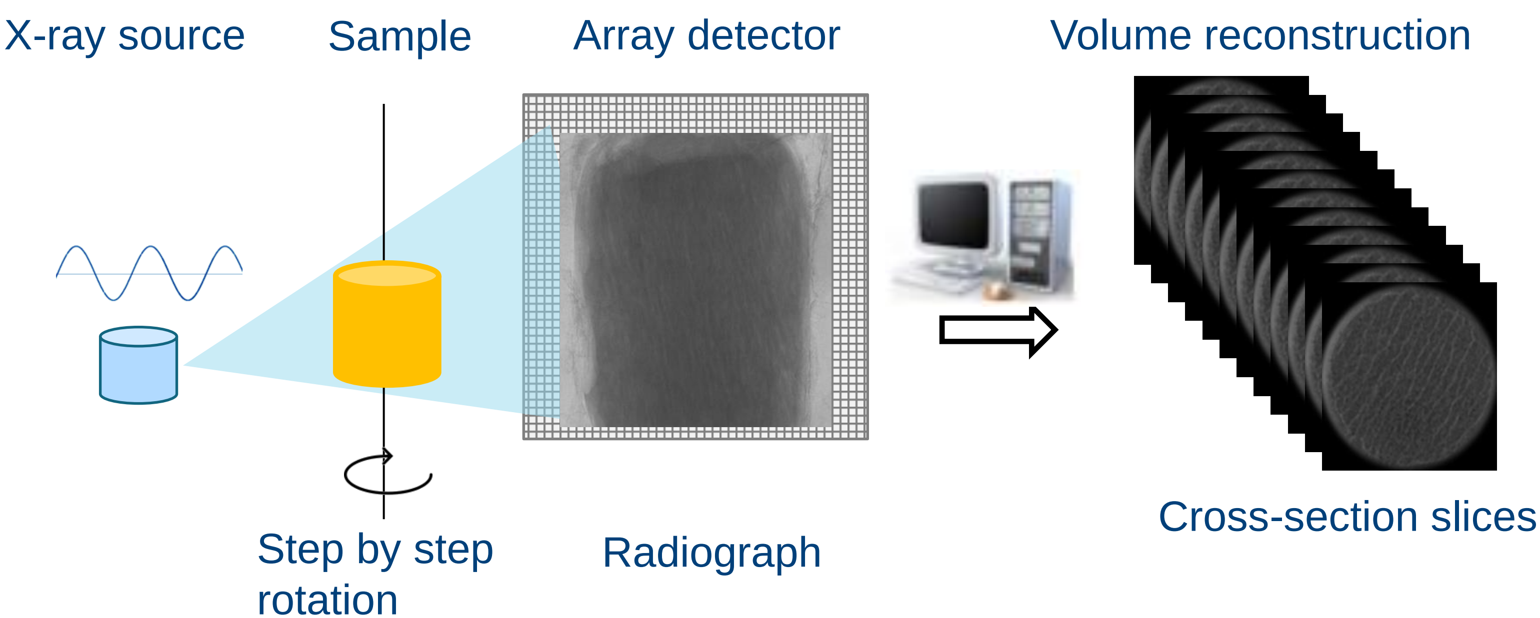
- Frozen to -18 ºC
- Dynamic temperature storage



Vicent et al., 2018a

X-ray µCT

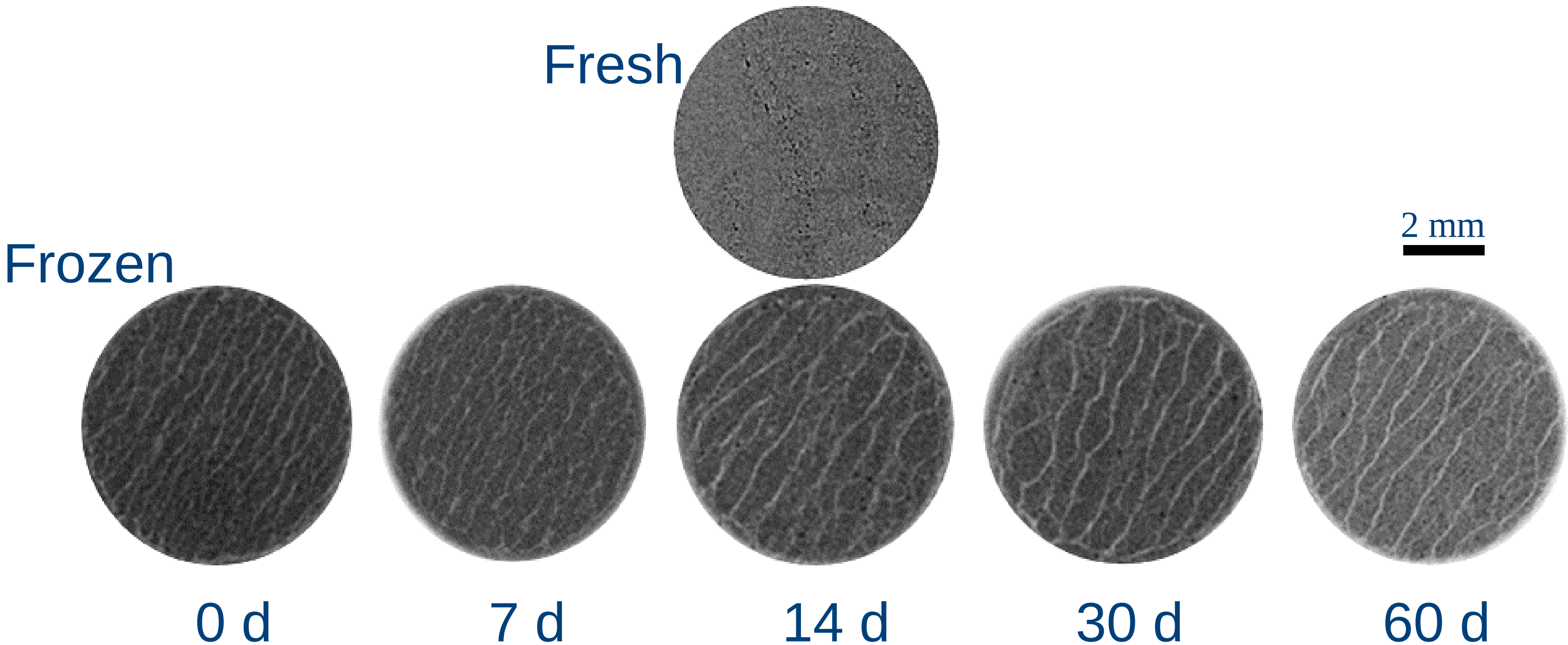
- DeskTom RX 130
  - Pixel resolution: 8.9 µm
  - PCM cooling stage



Vicent et al., 2018a

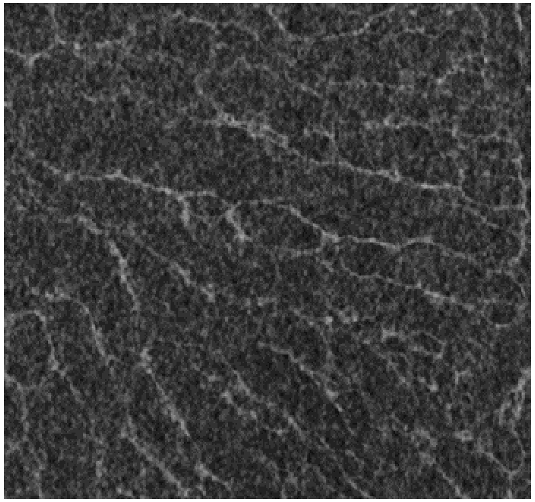
Results

CT slices of fresh and frozen product

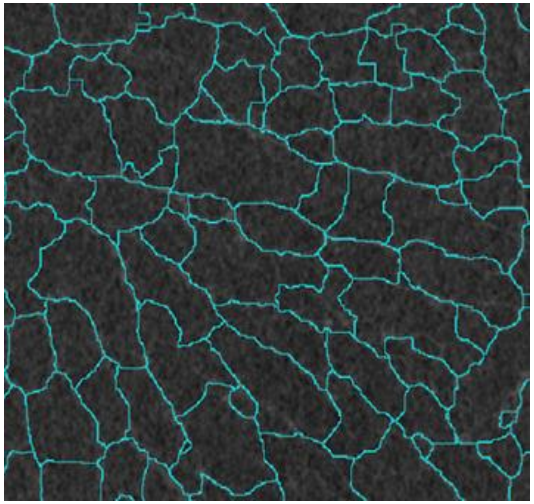


Vicent et al., 2018a

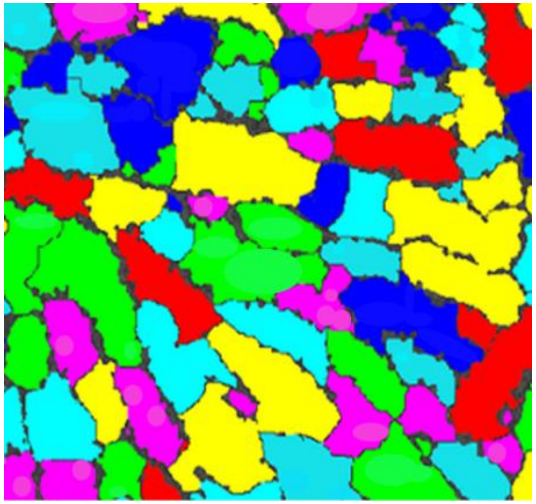
Image process workflow



ROI

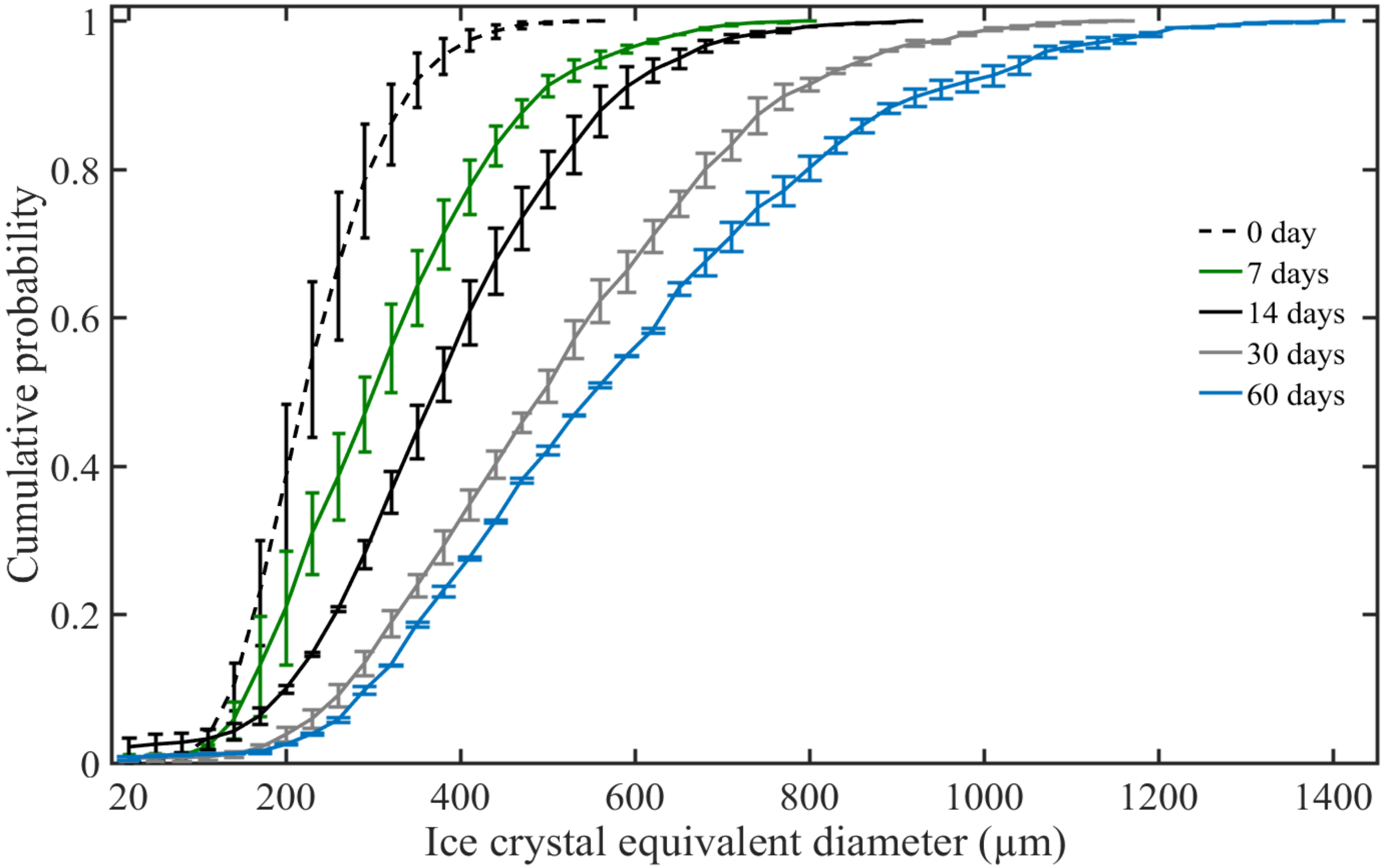


Ice crystal separation

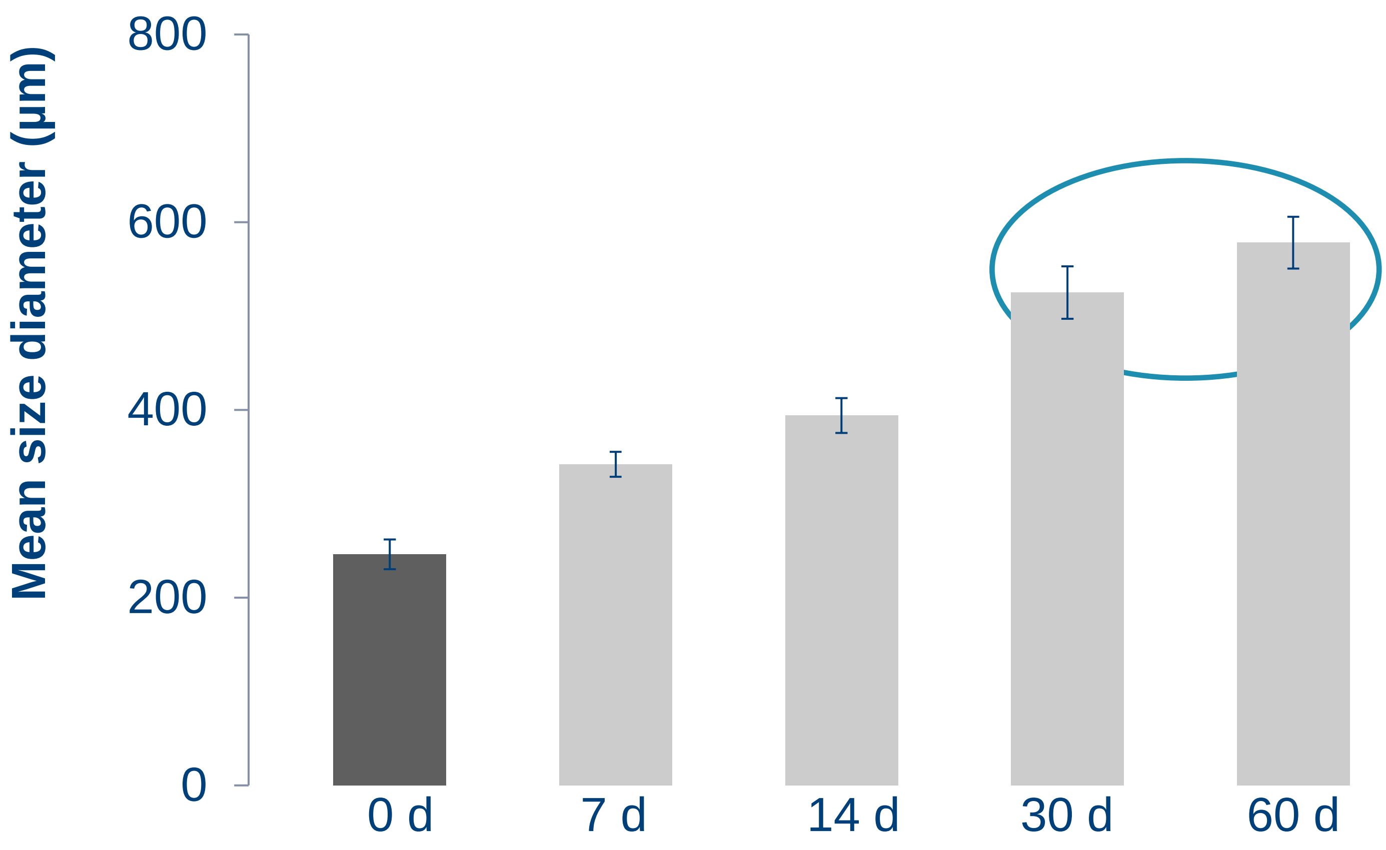


Crystal labeling

Ice crystal size distributions

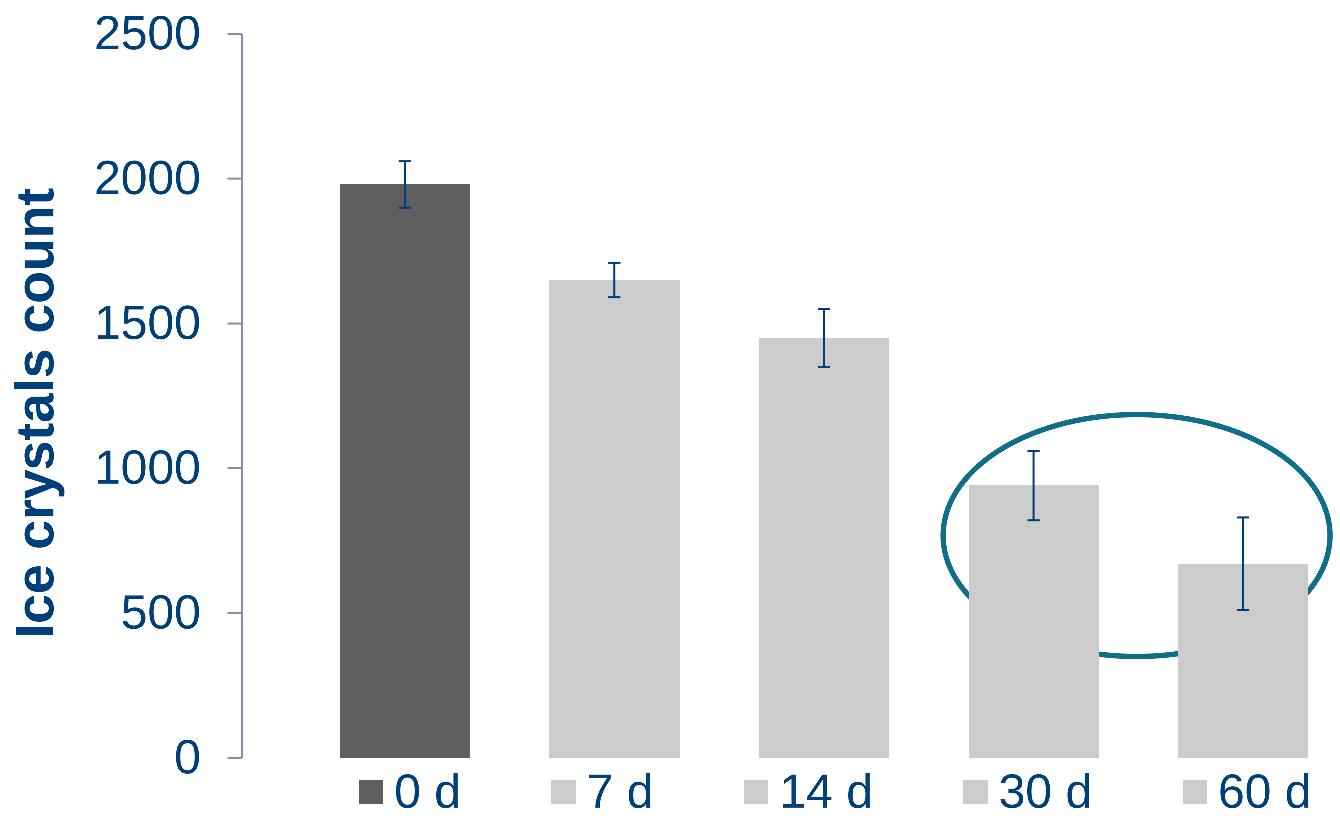


Mean crystal size



Vicent et al., 2018a

Number of ice crystals



Vicent et al., 2018a

Objectives and outline

Develop and apply measurement and modeling tools to improve understanding of microstructural and quality changes during frozen storage

Development of a 3D imaging method to visualize microstructure of frozen apple

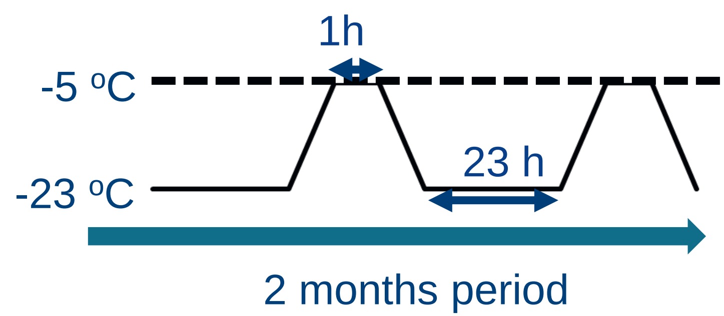
Tomographic imaging of ice crystal changes in carrot during frozen storage

Modeling ice recrystallization in product stored under dynamic temperatures

Modeling quality changes in food microstructure during frozen storage

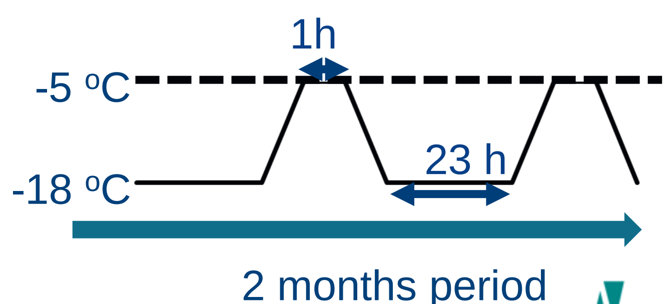
Materials and methods

Model calibration:



- Frozen at -18 ^{o}C
- Dynamic temperature storage

Model validation:



Model development

- Assumptions:
  - Ostwald ripening is a governing mechanism
  - Equivalent diameter, *L* can be used to describe ice crystal changes
  - Ice crystals < L_{crit} dissolve
  - No *T* gradients inside the product

- Evolution of ice crystal size distribution $n(L,t)$ was described using a PBE

$$\frac{\partial n}{\partial t} = -\frac{\partial (Gn)}{\partial L} + \frac{\partial (Dn)}{\partial L}$$

G and *D* are the growth and dissolution rates

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Model development

- Growth rate: $G = k_g \left(T_{eq} - T \right)$

k_g = growth coefficient
T_{eq} and T = equilibrium and product temperatures

$G \propto$ sub-cooling degree (ΔT)

$G > 0$ when $T_{eq} > T$
$G < 0$ when $T_{eq} < T$

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Model development

- Dissolution rate: $$\begin{cases} D = \dfrac{k_d}{L_{crit} - L} \exp\left(\dfrac{-E_a}{RT}\right) & \text{for } L < L_{crit} \\ D = 0 & \text{for } L \geq L_{crit} \end{cases}$$

k_d = dissolution coefficient

D is inversely proportional to crystal size relative to L_{crit}

Model development

- Lumped energy equation to estimate energy transfer

$$\frac{d}{dt}\left[\boxed{V\rho_{app}C_pT}+\boxed{V_{ice}\rho_{ice}\Delta H_s}+\boxed{\gamma A_{ice}}\right]=\boxed{UA\left(T_a-T\right)}$$

sensible heat

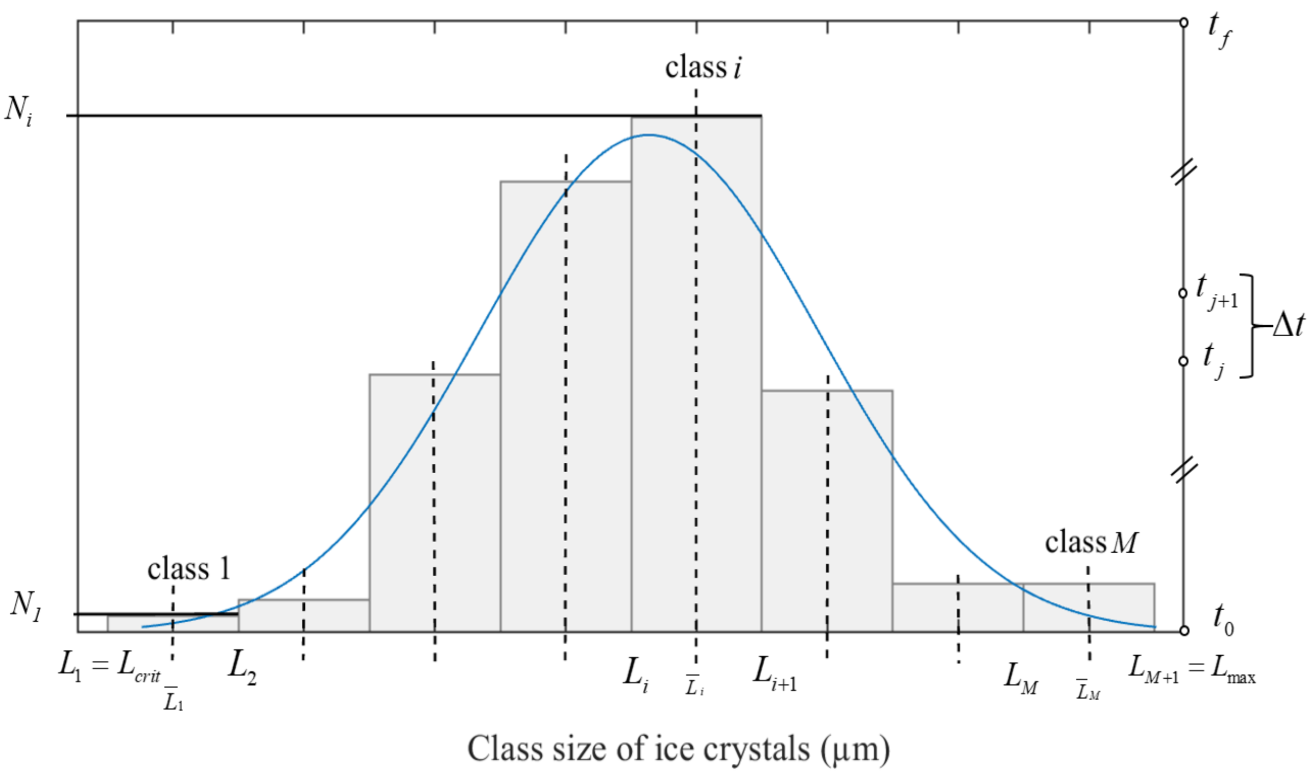
latent heat

surface energy

heat exchange between product & air

Discretization

- PBE was numerically solved using the classes method



Class size i: $\Delta L_i = L_{i+1} - L_i$

Mean class size i: $\bar{L}_i = \dfrac{L_{i+1} + L_i}{2}$

N of ice crystal i in interval $[L_i , L_{i+1}]$:

$$N_i(t) = \int_{L_i}^{L_{i+1}} n(L,t)\,dL$$

Upwind derivative scheme

- Set of equations to describe the discrete crystal $N_i(t)$

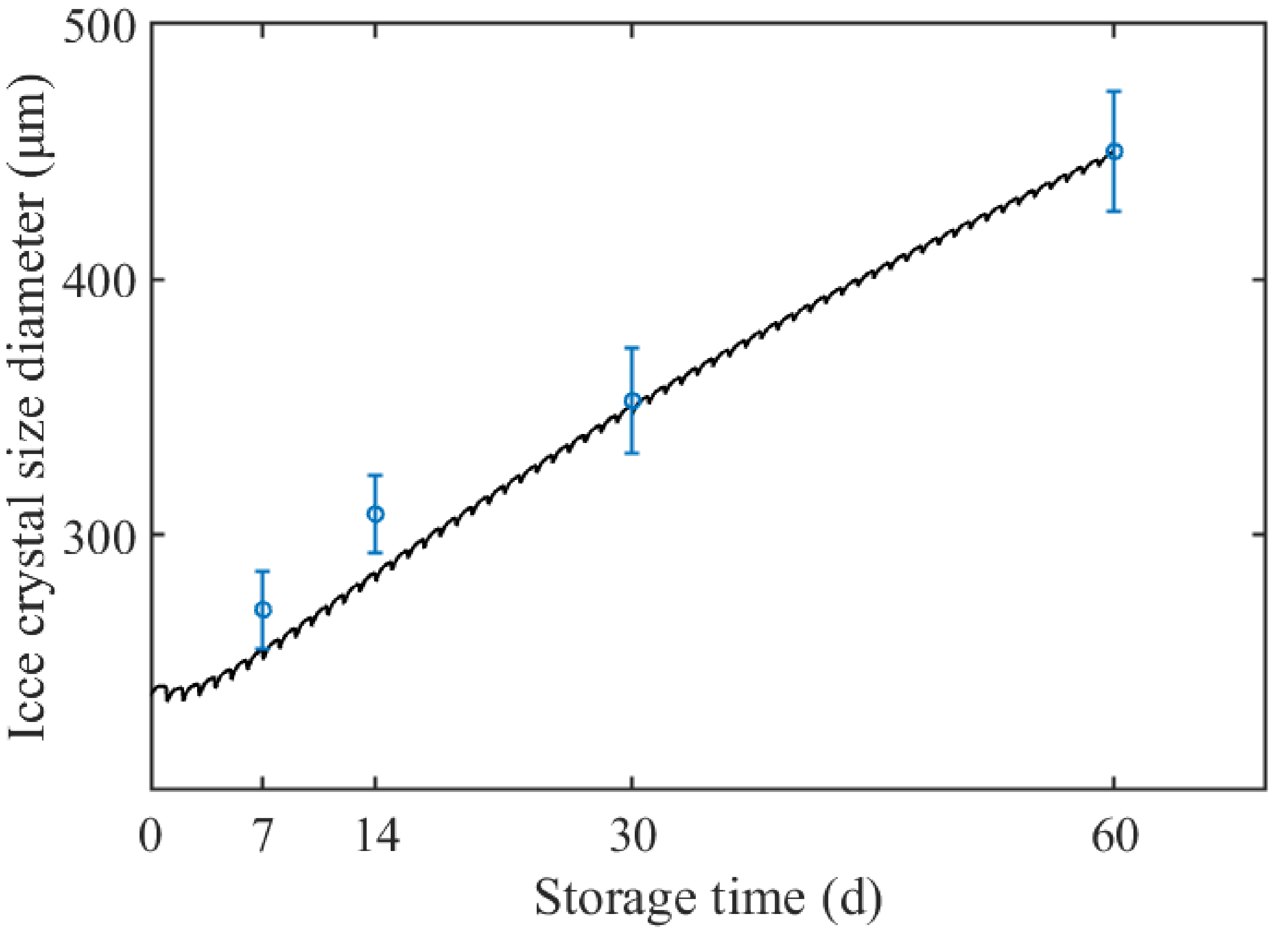
$$\frac{N_i\left(t_{j+1}\right)-N_i\left(t_j\right)}{\Delta t}=G\left(t_j\right)\frac{N_{i-1}\left(t_j\right)-N_i\left(t_j\right)}{\Delta L}+\frac{D\left(\overline{L}_{i+1},t_j\right)N_{i+1}\left(t_j\right)-D\left(\overline{L}_i,t_j\right)N_i\left(t_j\right)}{\Delta L}\text{ for }G>0$$

and

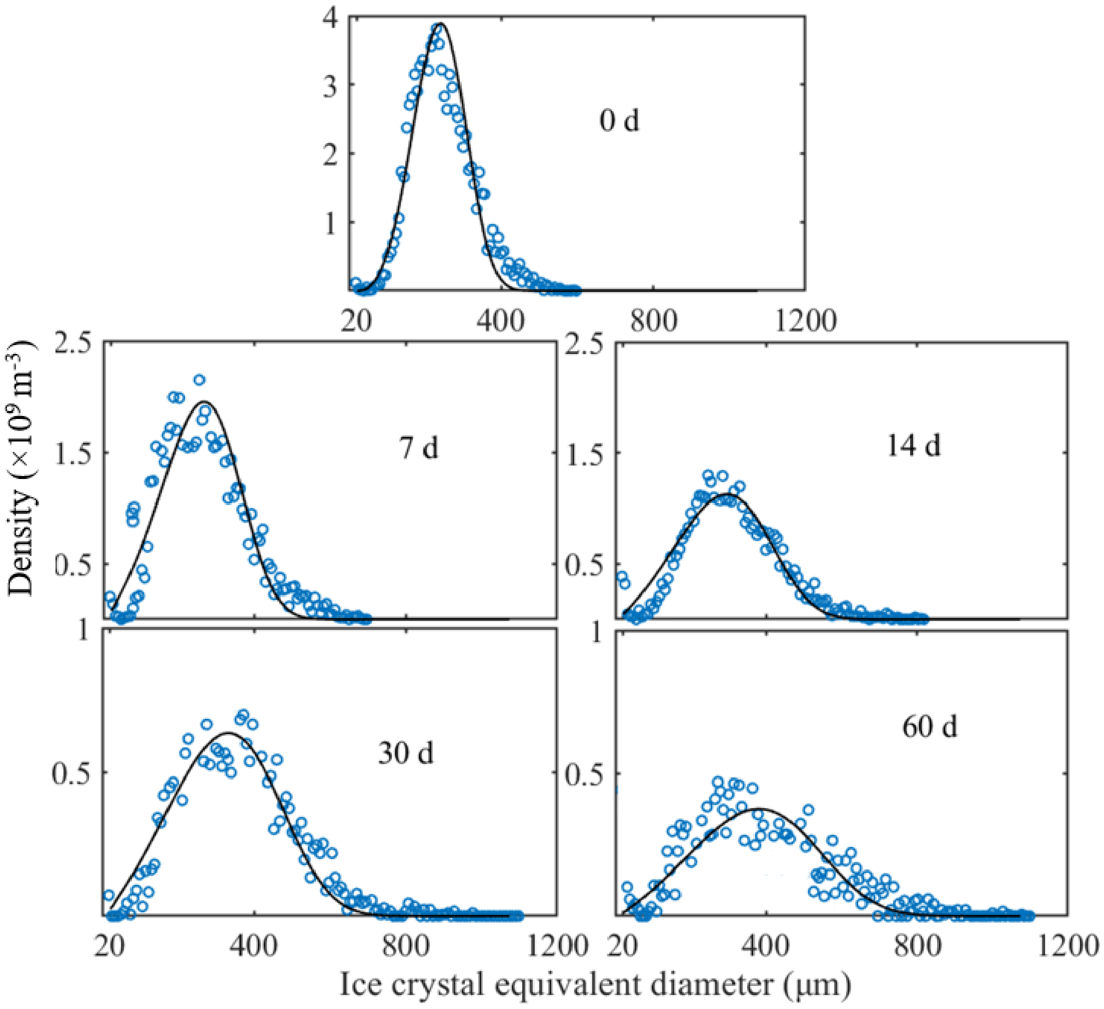
$$\frac{N_i\left(t_{j+1}\right)-N_i\left(t_j\right)}{\Delta t}=-G\left(t_j\right)\frac{N_{i+1}\left(t_j\right)-N_i\left(t_j\right)}{\Delta L}+\frac{D\left(\overline{L}_{i+1},t_j\right)N_{i+1}\left(t_j\right)-D\left(\overline{L}_i,t_j\right)N_i\left(t_j\right)}{\Delta L}\text{ for }G<0$$

Time step, Δt = 10 s

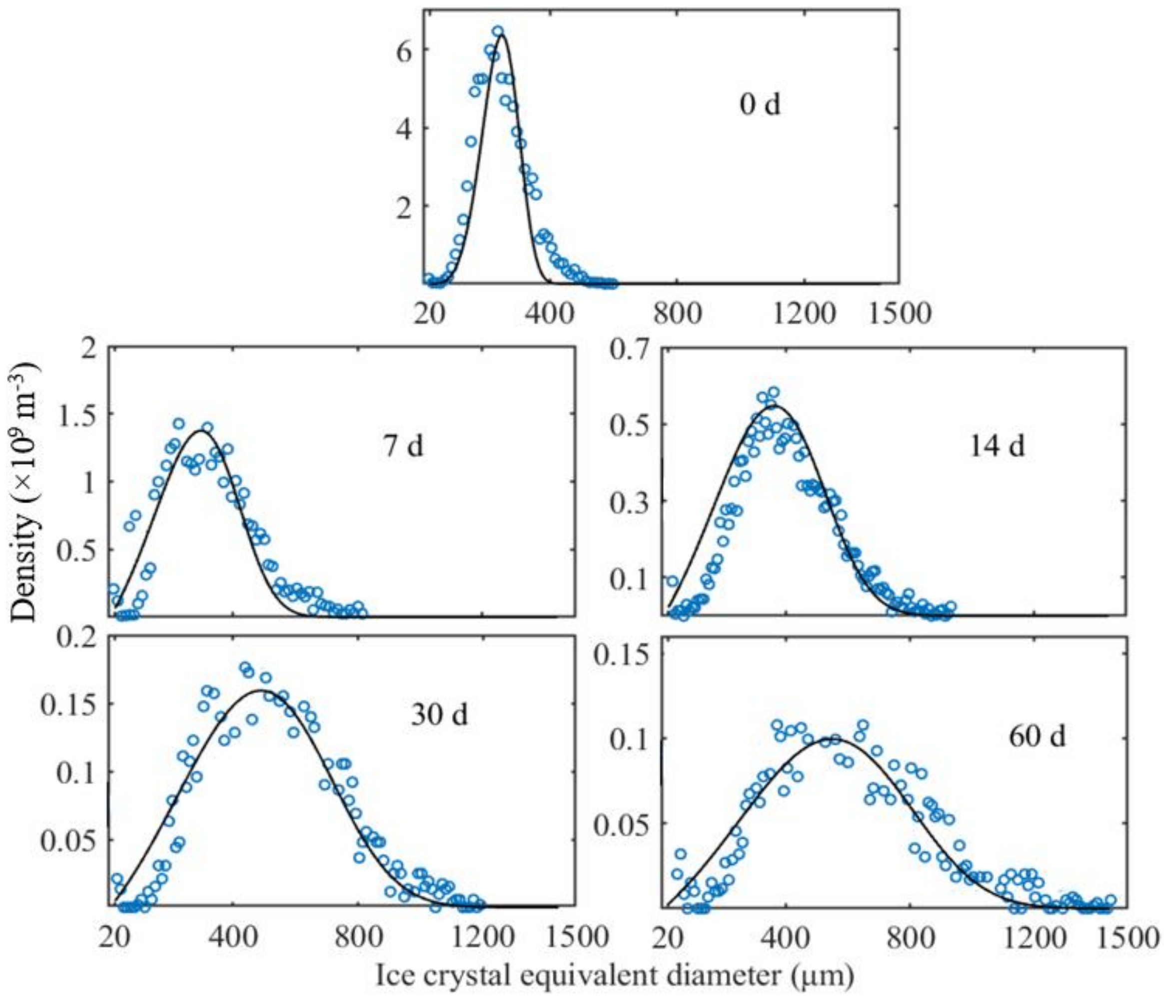
Results: Model calibration



Evolution of ice crystal size distribution



Model validation



Objectives and outline

Develop and apply measurement and modeling tools to improve understanding of microstructural and quality changes during frozen storage

Developing X-ray µCT method to characterize the 3D microstructure of frozen food

Tomographic imaging of ice crystal changes in food during frozen storage

Modeling ice recrystallization in food stored under dynamic temperatures

Kinetics of food quality changes during frozen storage

Quality modeling

- Kinetic models: $\frac{\mathrm{d}Q}{\mathrm{d}t} = -k_i$ and $\frac{\mathrm{d}Q}{\mathrm{d}t} = -k_i Q$

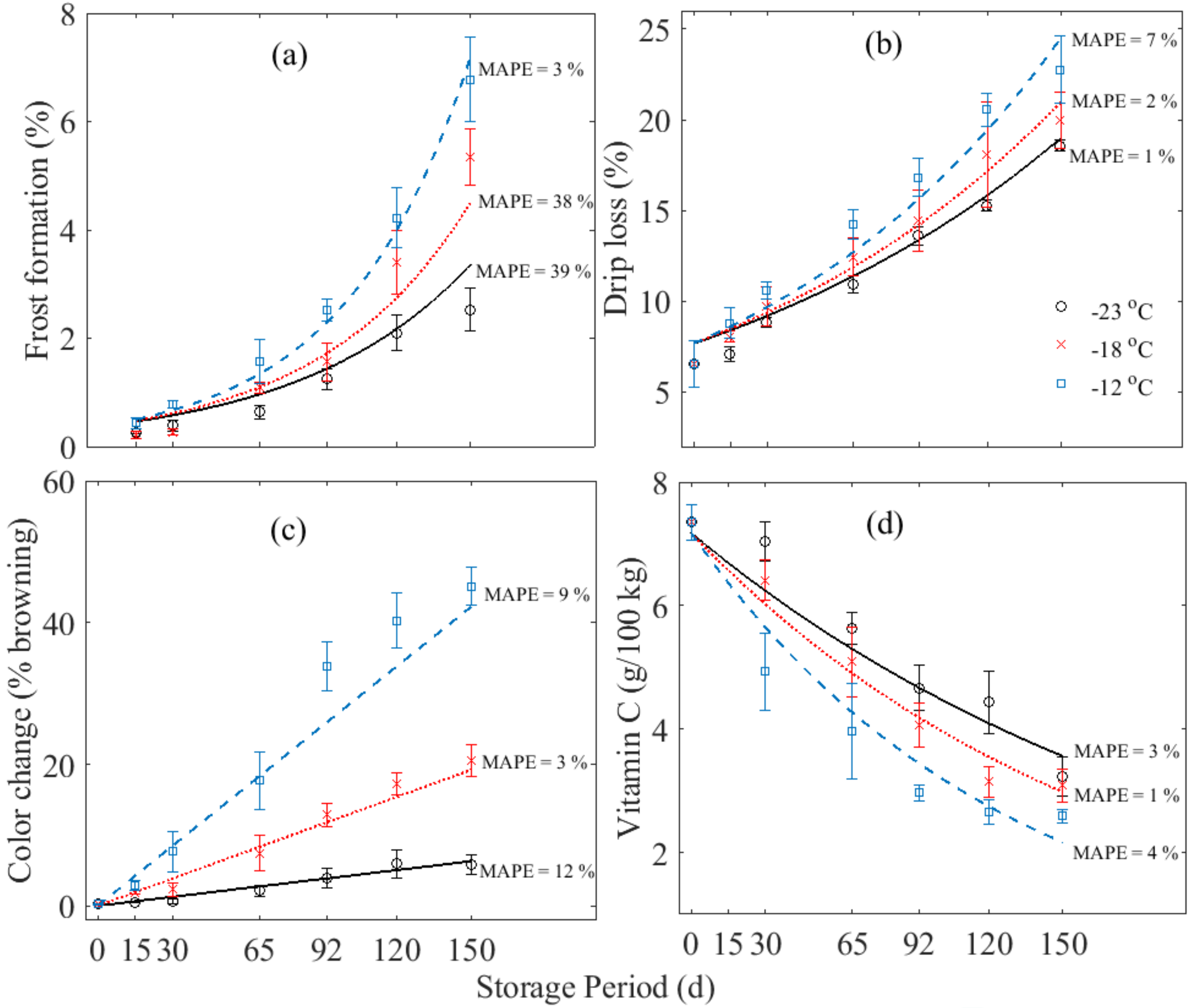
- Incorporating temperature effect by Arrhenius equation

$$k_i = k_{\mathrm{ref},i} \exp\left(-\frac{Ea_i}{R}\left(\frac{1}{T(t)} - \frac{1}{T_{\mathrm{ref}}}\right)\right) \qquad Q_0$$

- Physical model: $\frac{\mathrm{d}F_m}{\mathrm{d}t} = k_m A\left(a_w \rho_s - \frac{RH}{100}\rho_a\right)$

Vicent et al., 2018b

Kinetic model predictions



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Vicent et al., 2018b

AgroParisTech KU LEUVEN

General conclusions

- Tomographic imaging to characterize frozen food
  - 3D ice crystals
  - 3D microstructure
  - Mean size and number of ice crystals changes

- Modeling ice recrystallization
  - PBE describes well the ice crystal evolution
  - Broader ice crystal size distribution
  - Governing processes: *D* and *G*

New research

- Limited contrast in µCT images
  - Phase contrast tomography
  - Contrast-enhanced µCT

- Microstructure dynamics
  - 4D CT
  - Short exposure time or reduce projections.
  - Phase transition in real time

Use of enhance contrast agent for sorbet

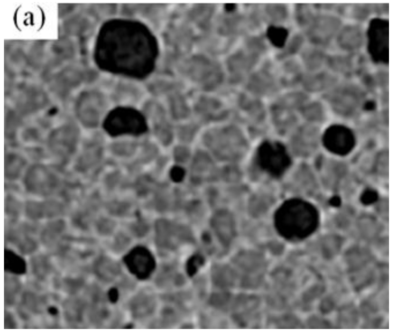
Microtomographie RX: mise au point de la méthode de traitement d'image

- Exemple de traitement pour le sorbet témoin

Logiciel de traitement d'image: Avizo 2019.1® (Thermo Fisher)

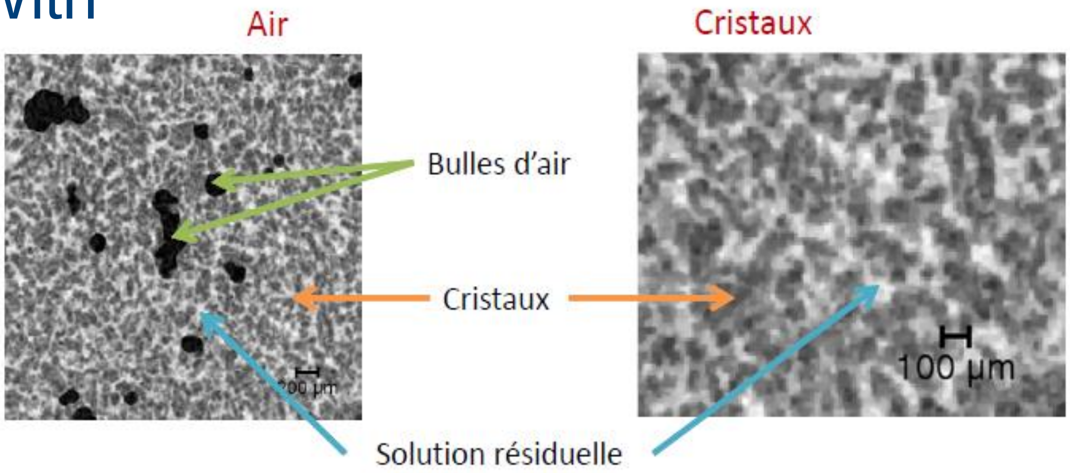
Traitement d'un sous-volume: **cube de 360 voxels de côté** ≈ 3 mm de côté

Without CA



With

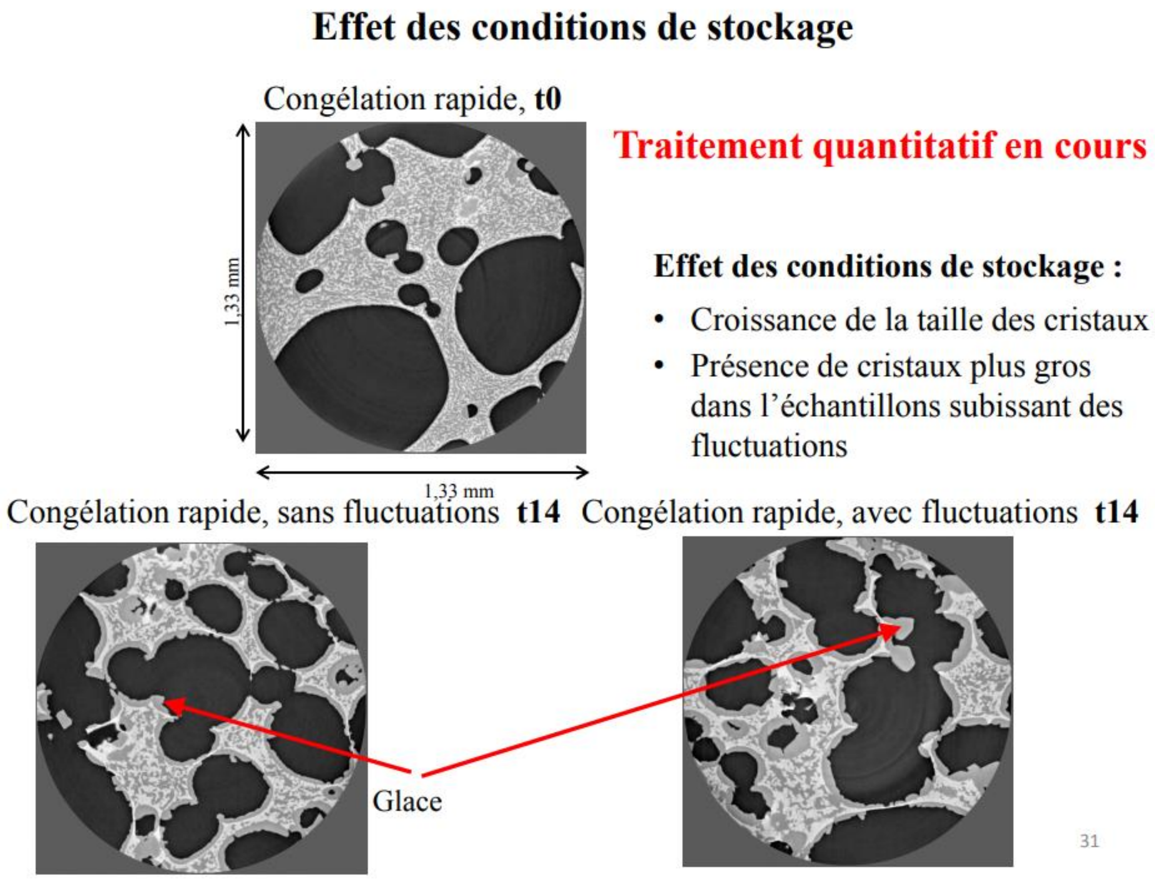
Images brutes



Masselot V. 2020

SynchotronTomography

- Case of frozen like cake product low water content



Amira Zennoune , H. Benkhelifa, T. Ndoye   2021 et al

What is next? Following dynamical phenomena : meltdown

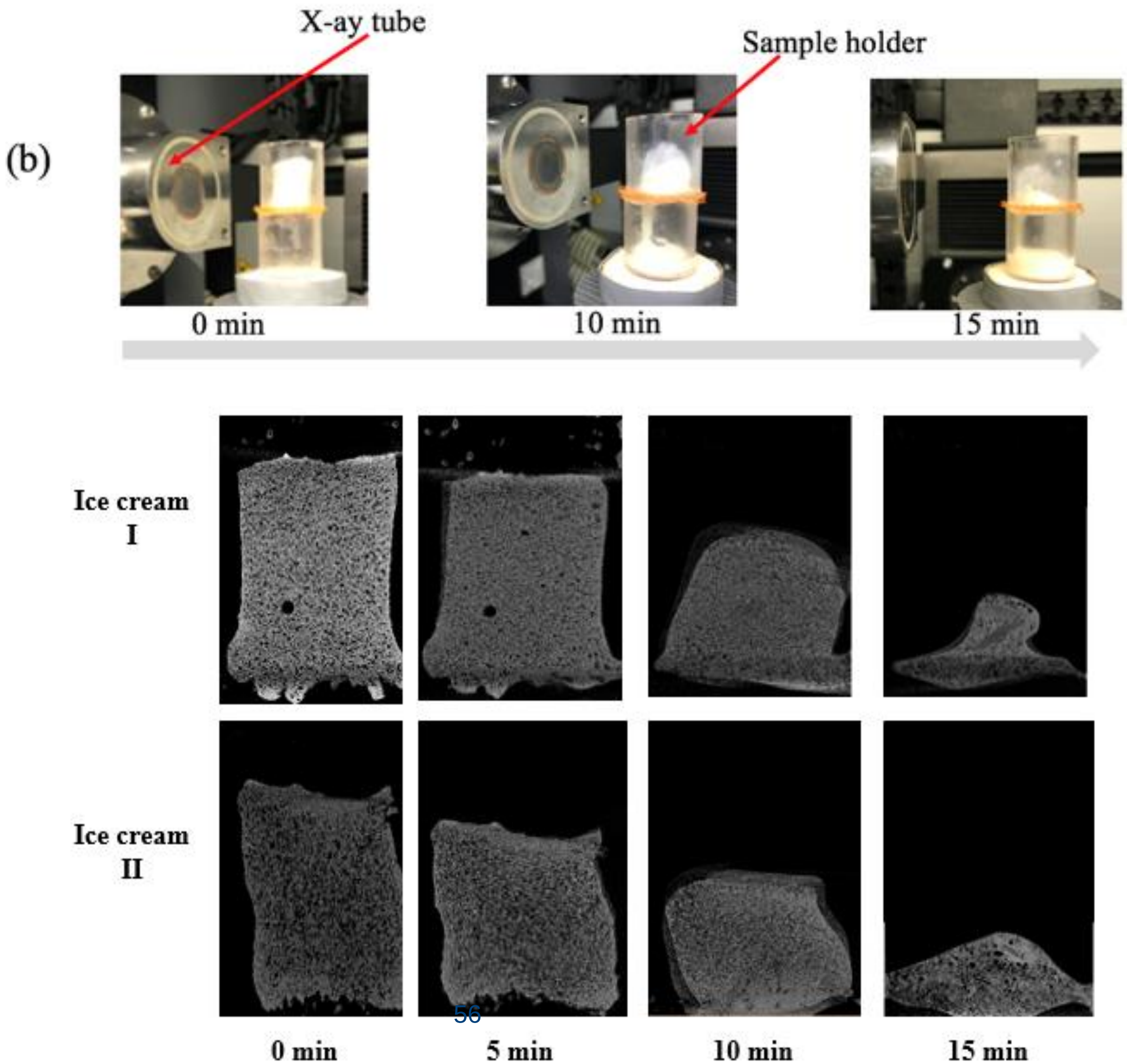
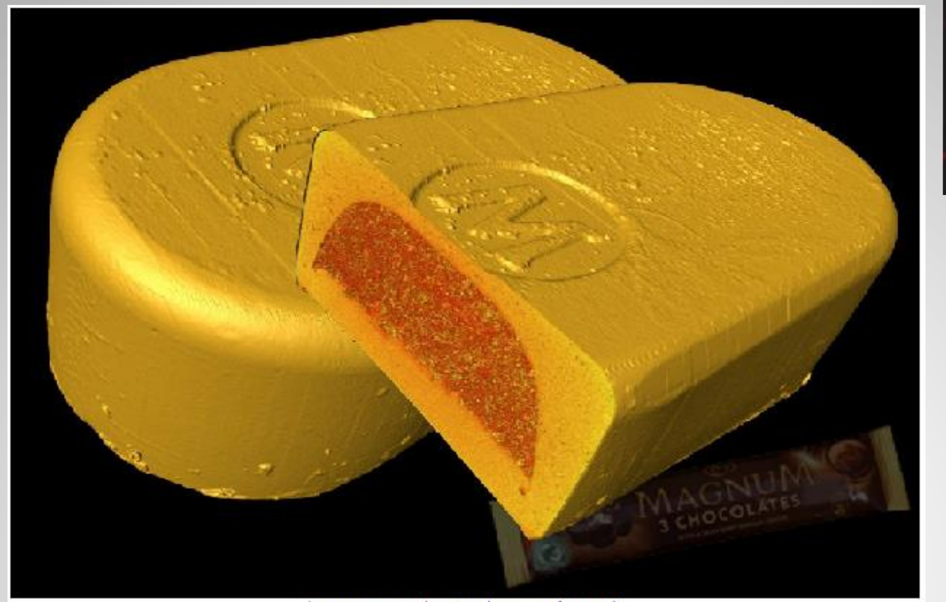


Fig. 4. CT cross-section slices of ice cream for each time-point during the meltdown



Thank you!

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