Granulometric analysis of maltodextrin particles observed by scanning electron microscopy

Geosto days, Dijon, 2023

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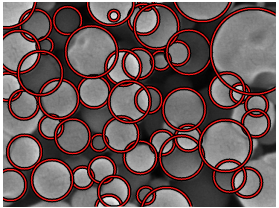
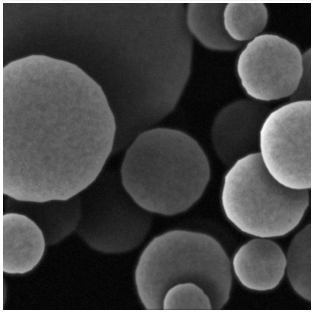
Organization of the talk

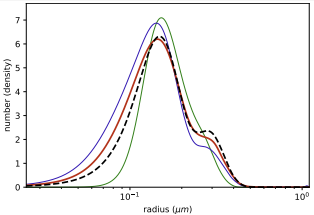
A bit of image processing



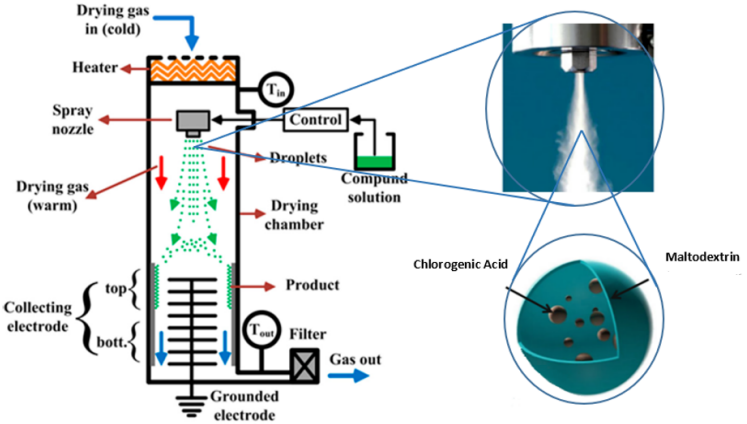
A bit of simulation



Validation/Comparison



Context: pharmaceutics



- Particles of maltodextrin
- Encapsulation of Chlorogenic Acid (CGA) 5-O-caffeoylquinic acid (5-CQA)
- Spray-Drying technique
- Applications: food, pharmaceutics, cosmetics

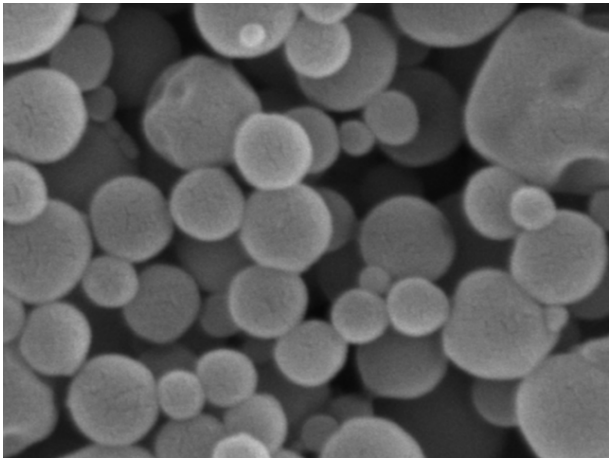
Observation: Scanning electron microscopy

Image observation

- Spheres (or near)
- SEM characteristics:
  - 3D like effect
  - Depth effect

Objectives

- Particles Size Distribution (PSD)
- Linked to delivery properties of the active molecule



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3 segmentation methods

- Stochastic Watershed
- Circular Hough Transform
- Curvature Analysis Method

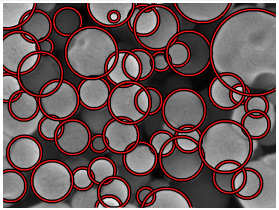
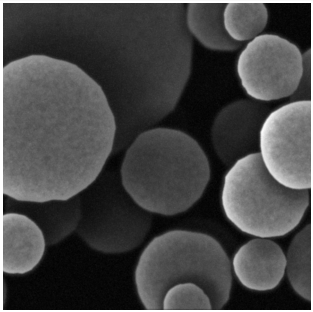


Image simulation and model

- Simulate SEM images
- Specific PSD



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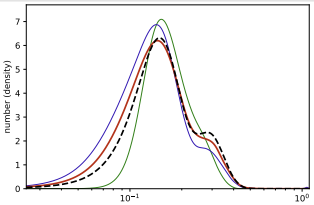
- PSD from Laser Diffraction
- PSD of simulated images



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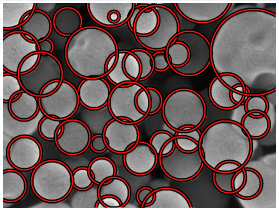

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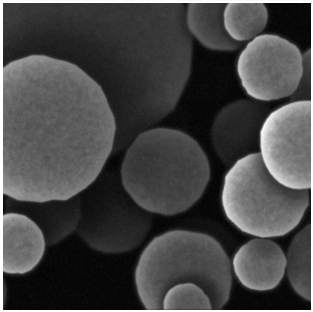

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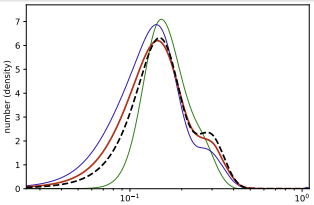


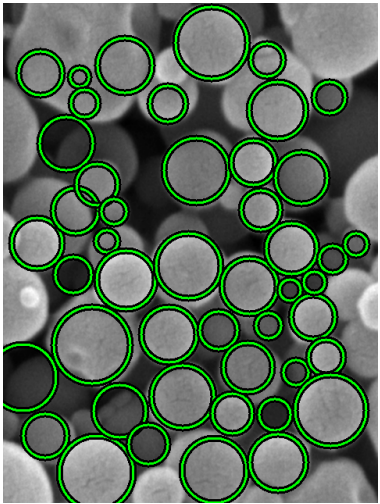
Stochastic Watershed (SW)

Principles

- Watershed (mathematical morphology)
- Constrained by random markers
- Repeat the process
- Accumulate the results
- Distance transform, local maxima and circles computation

Drawbacks

- Number of markers
- Spatial distribution ?



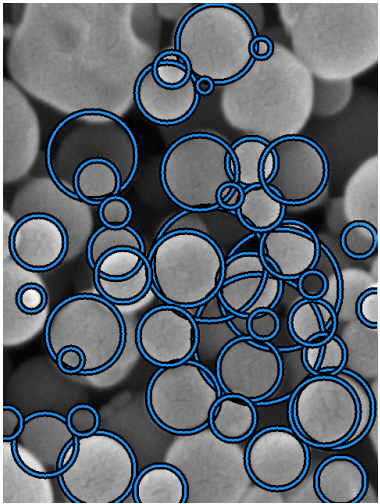
Circular Hough Transform (CHT)

Principles

- Contours detection (gradient and binarization)
- Hough Transform for circles detection

Drawbacks

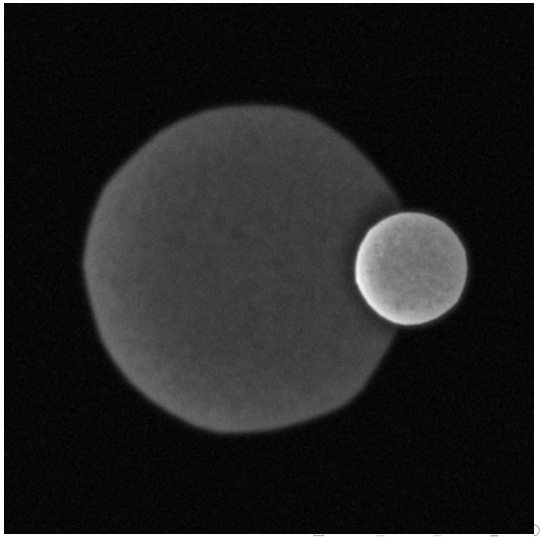
- Many overlapping circles
- Partially occluded circles not detected
- Different contours are mixed



Our proposition: Curvature Analyse Method (CAM)

Algorithm details: minimum MSE map

Original image



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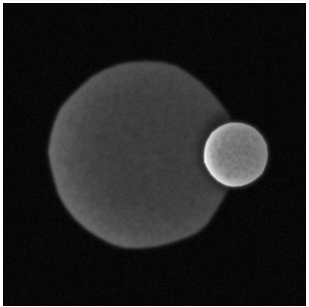
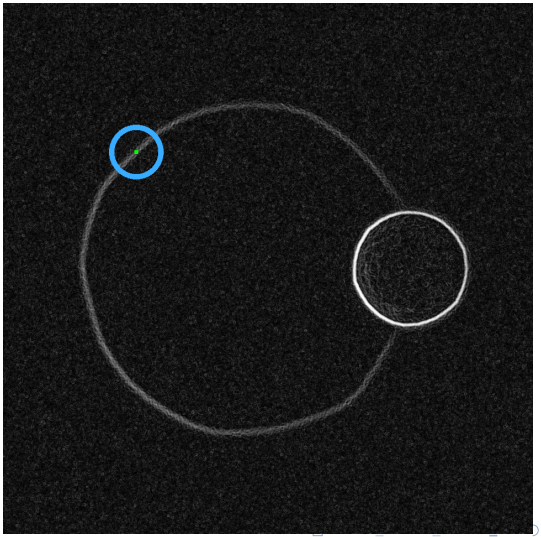
Gradient magnitude





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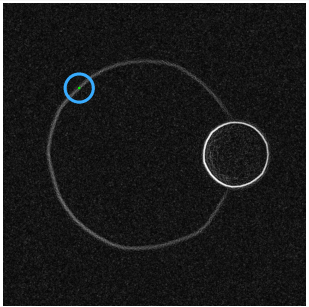
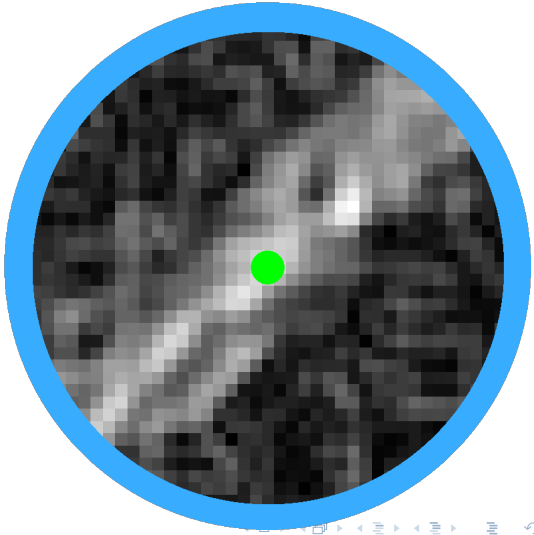
Zoom en blue circular window.
Centered at point p_c.





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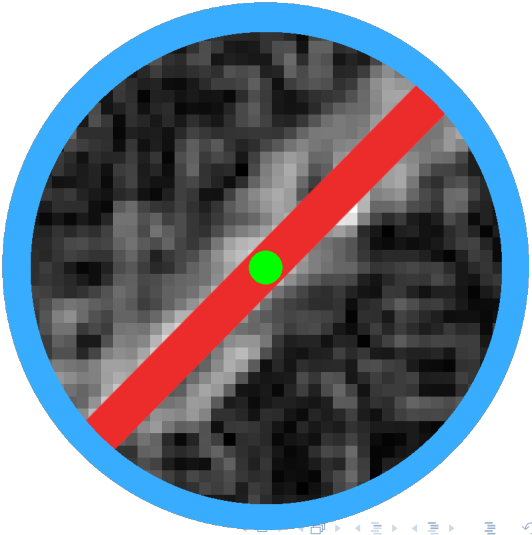
Line obtained by minimizing E on grayscale points.

$$E_{p_c}(\alpha) = \frac{1}{\sum_{i=1}^{n} \nabla(p_i)} \sum_{i=1}^{n} d(p_i, L_{p_c,\alpha})^2 \nabla(p_i)$$

$$d(p_i, L_{p_c,\alpha}) = \frac{(y_i - y_c) - \alpha(x_i - x_c)}{\sqrt{1+\alpha^2}}$$



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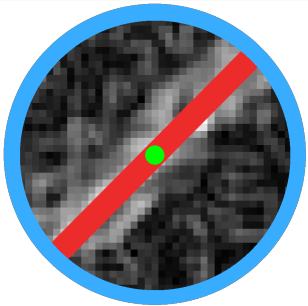
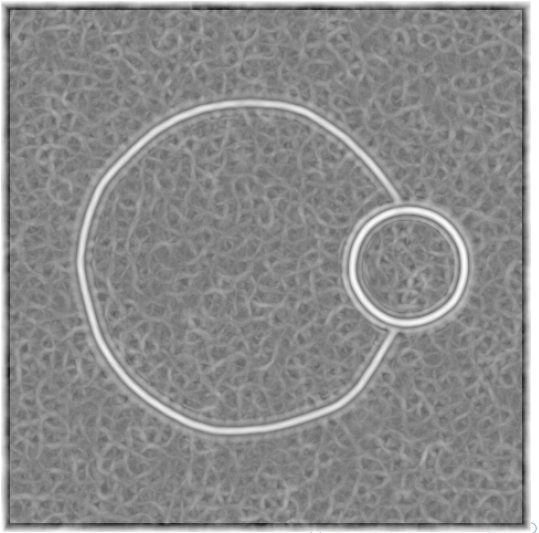
Final minimum MSE map.

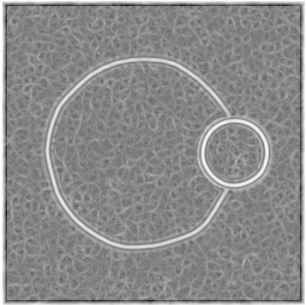


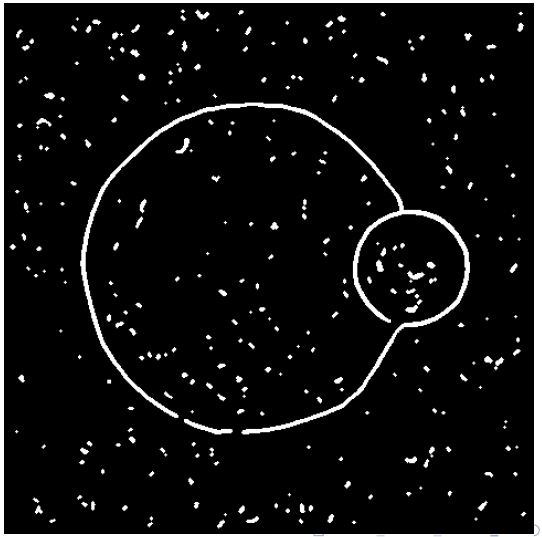


Our proposition: Curvature Analyse Method (CAM)

Algorithm details: extraction of arcs

Binarization.





Our proposition: Curvature Analyse Method (CAM)

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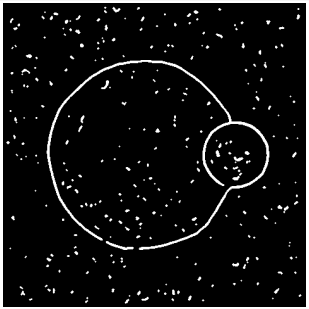
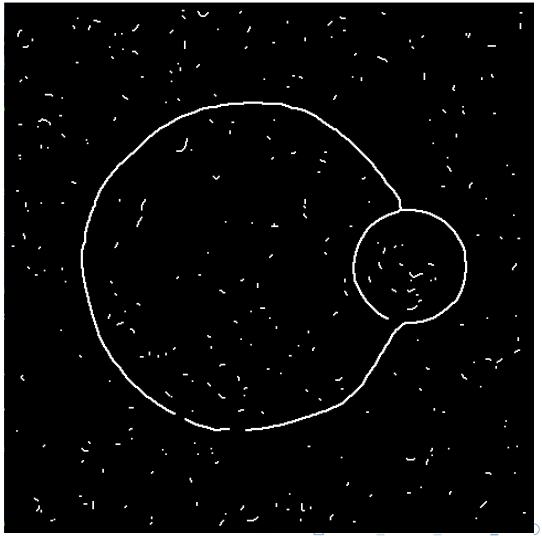
Skeleton.





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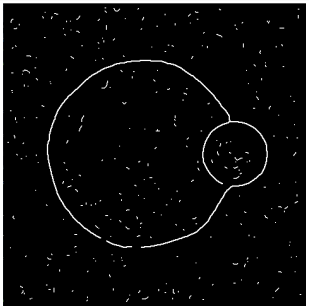
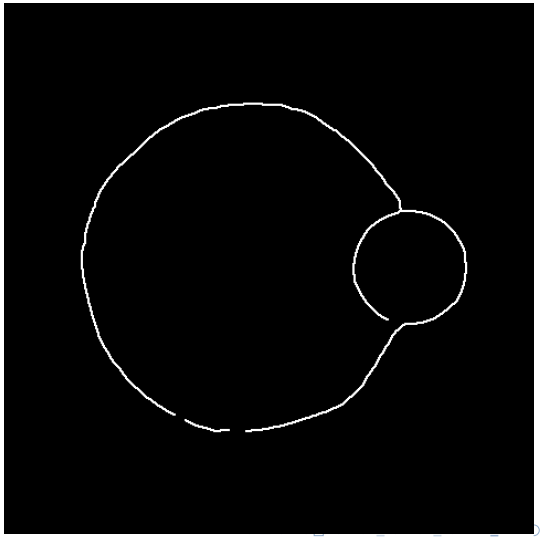
Cleaning.





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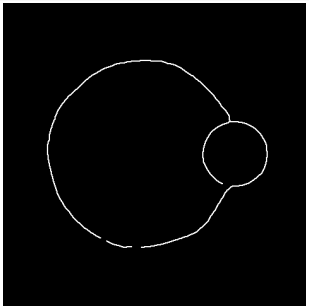
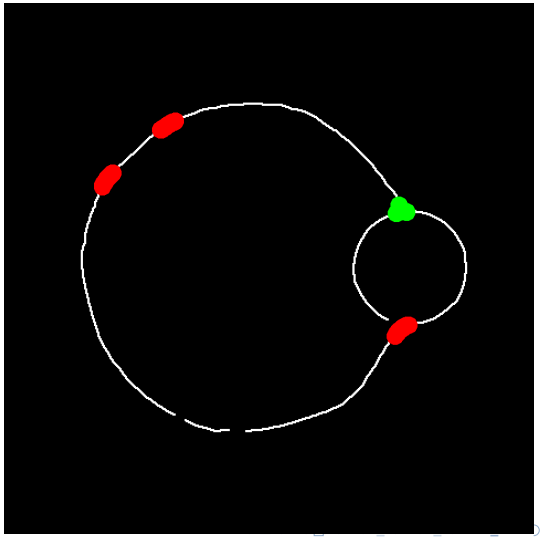
Algorithm details: extraction of arcs

Intersection areas and **curvature irregularities**.





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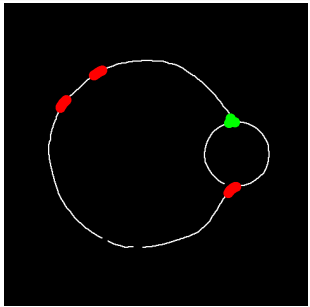
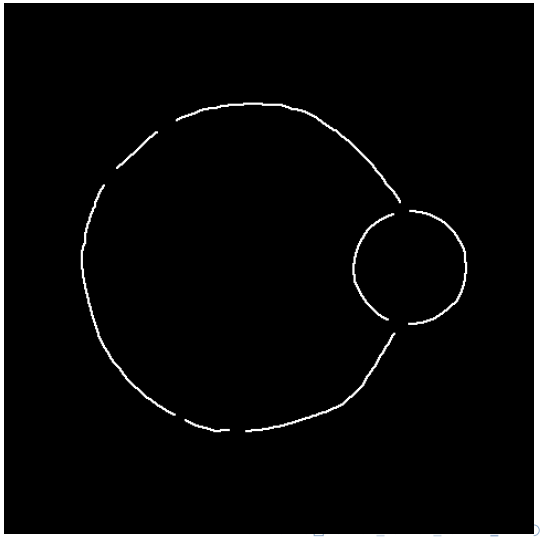
Split arcs.



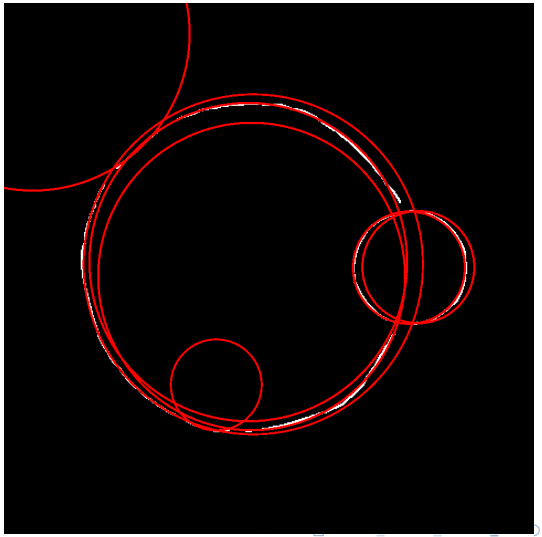


Our proposition: Curvature Analyse Method (CAM)

Algorithm details: circles association and rearrangement

Minimization process for circles detection

$$E_{\text{circle}}(p_c, r) = \frac{1}{n}\sum_{i=1}^{n}((x_i - x_c)^2 + (y_i - y_c)^2 - r^2)^2$$



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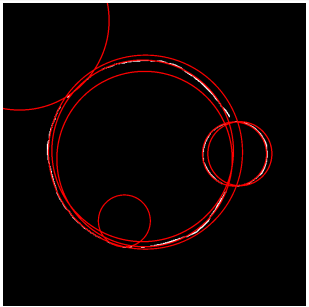
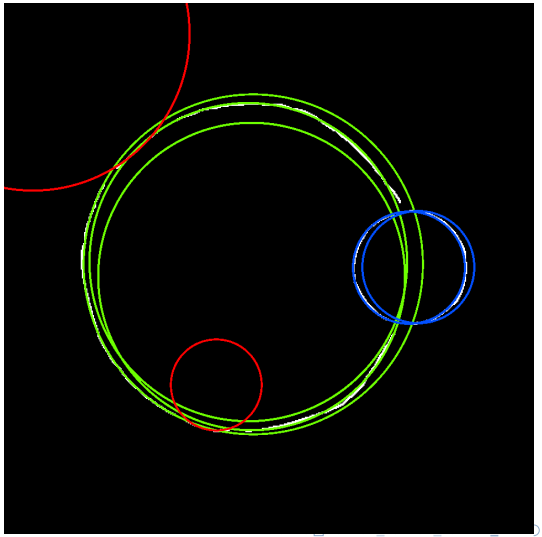
Algorithm details: circles association and rearrangement

Merging close circles **green** and **blue.**





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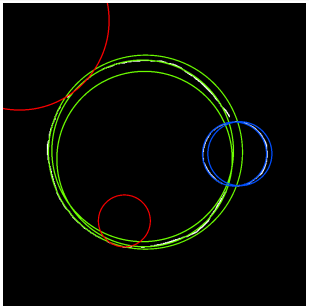
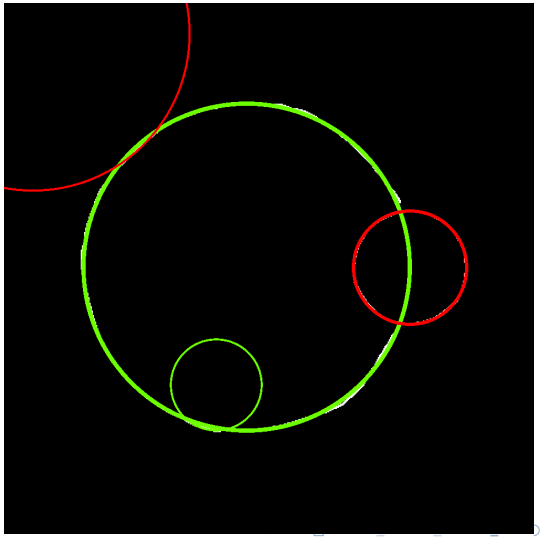
Merging **arc-sharing** circles.





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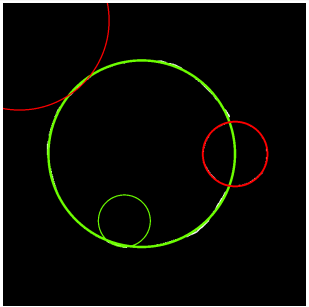
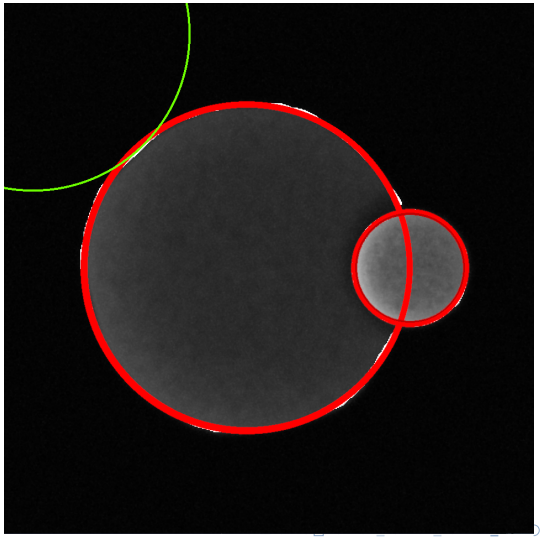
Algorithm details: circles association and rearrangement

Remove circles with a grayscale criterion.





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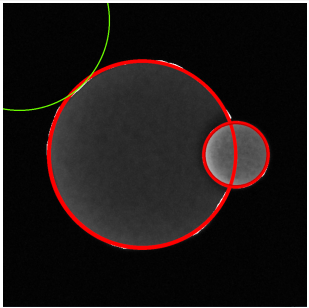
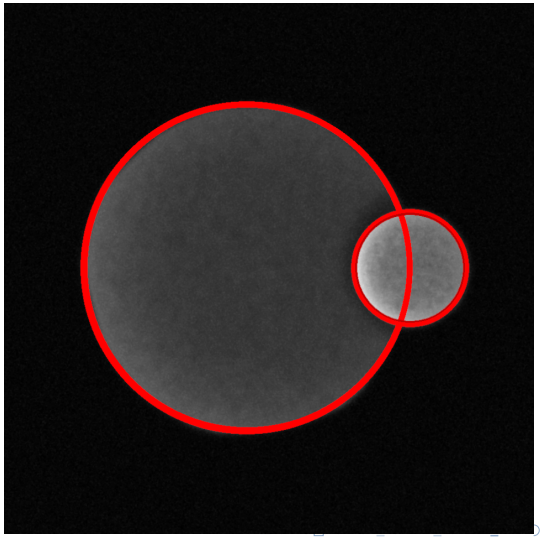
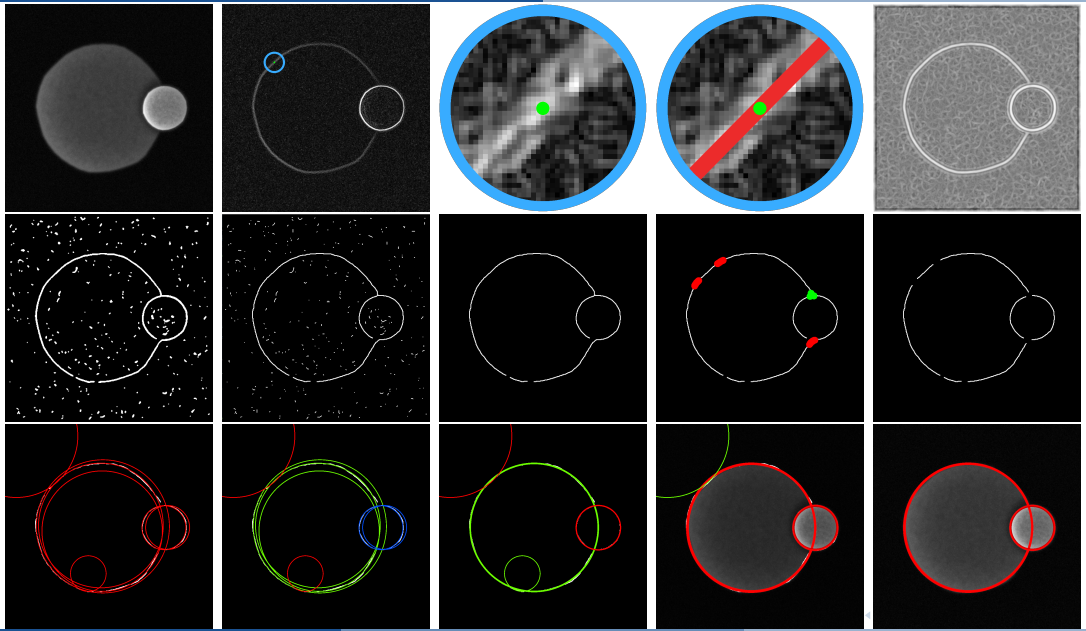
Final result.







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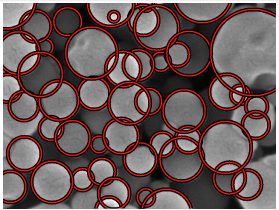

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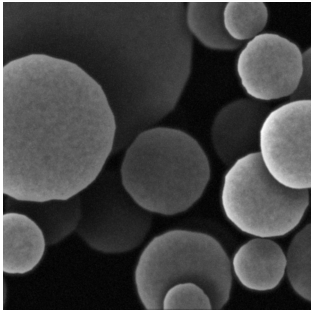

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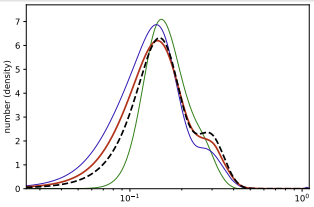

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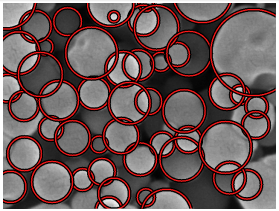

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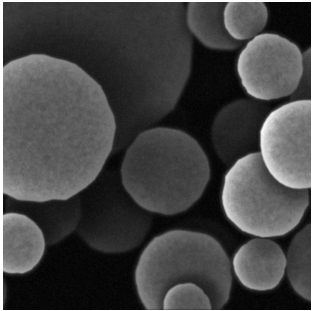

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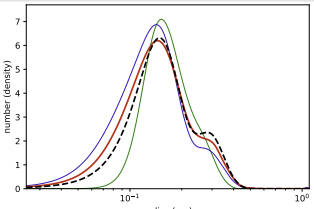


Image simulation

Algorithm details

Binary random (circular) shape.

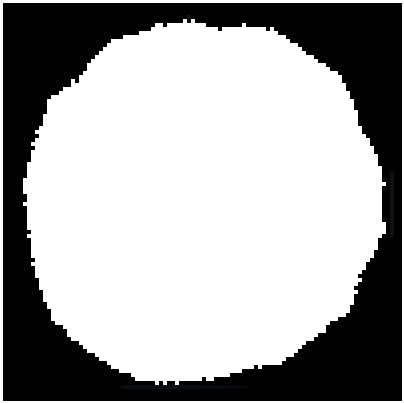


Image simulation

Algorithm details

Illumination effects (orientation).

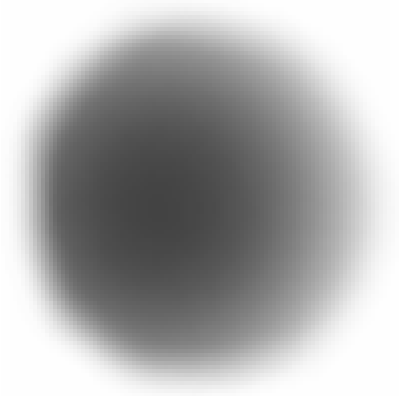


Image simulation

Algorithm details

Shadow effects.

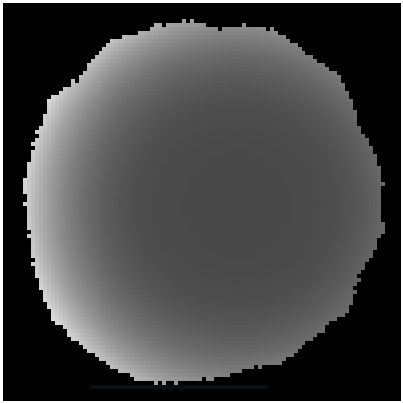
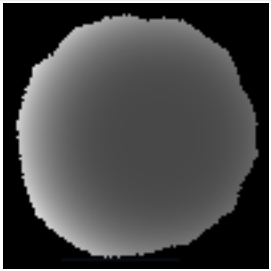


Image simulation

Algorithm details

Several grains, with depth effect.



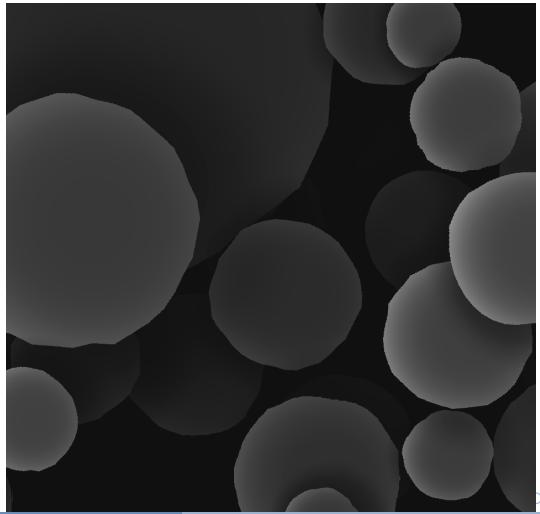
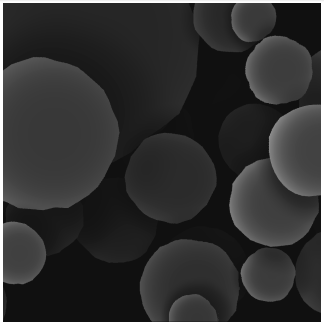
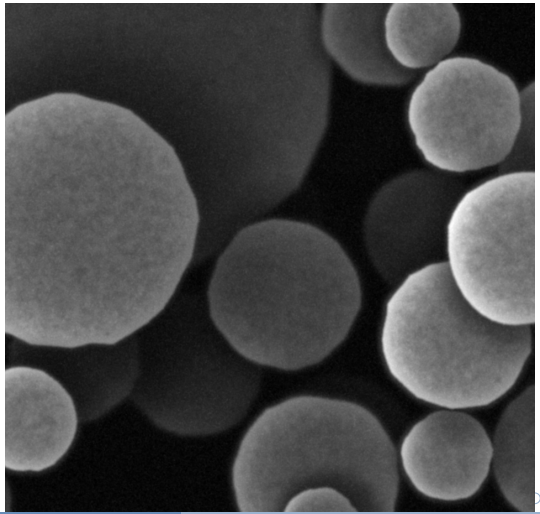


Image simulation

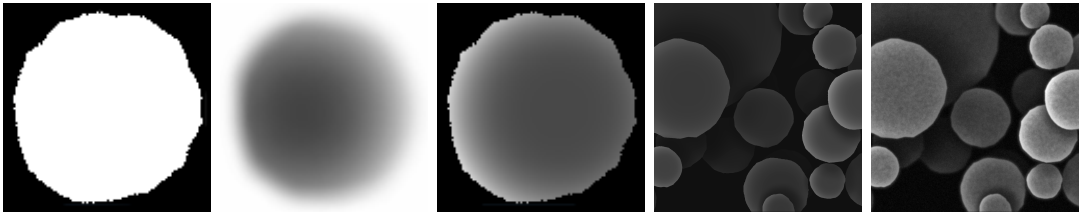
Algorithm details

Final result, with noise and blur.

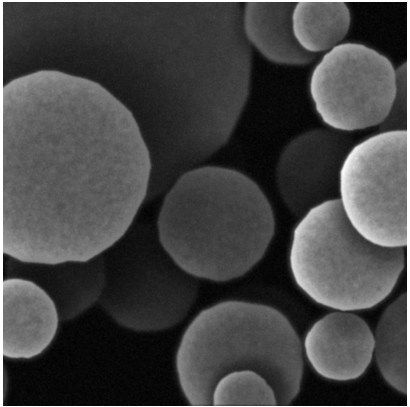


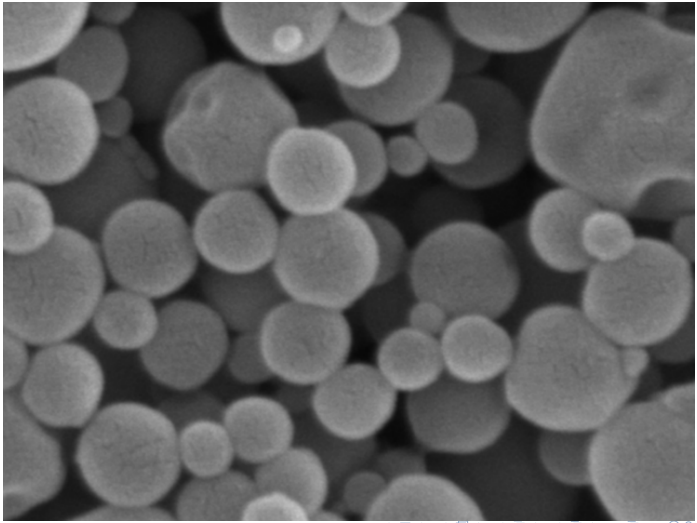


Summary: grains generator



Simulation vs real image





Organisation of the talk

3 segmentation methods

- Stochastic Watershed
- Circular Hough Transform
- Curvature Analysis Method

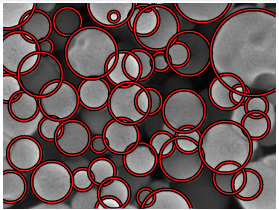
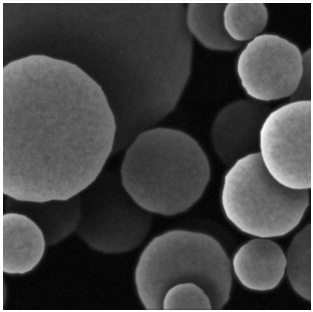


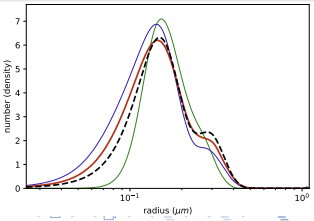
Image simulation and model

- Simulate SEM images
- Specific PSD



Validation/Comparison

- Segmentation results
- PSD from Laser Diffraction
- PSD of simulated images



Organisation of the talk

3 segmentation methods

- Stochastic Watershed
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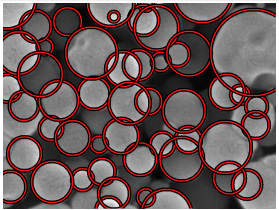

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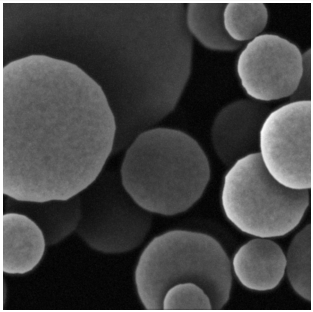

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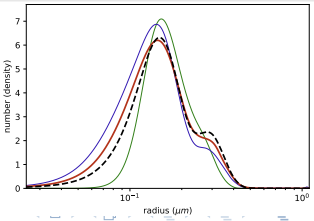
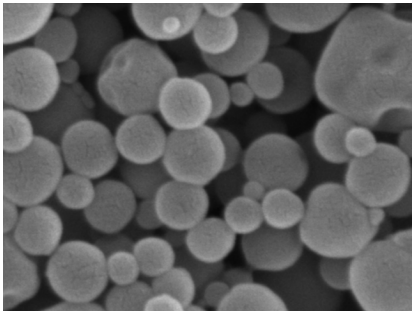
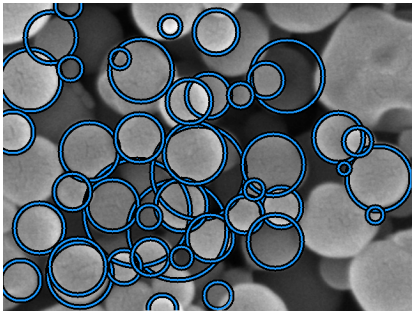
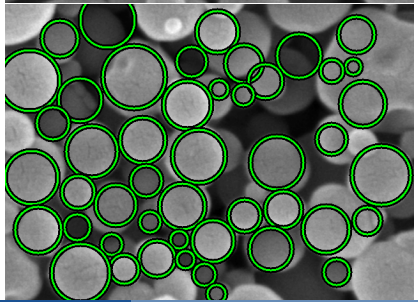


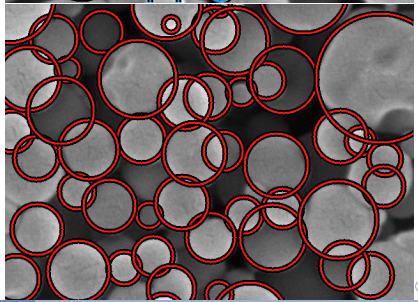
Segmentation results

- **CHT**
- **SW**
- **CAM**







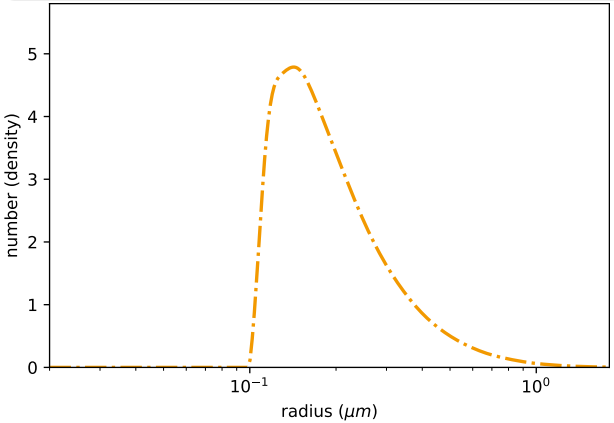


Particle Size Distribution: Laser Diffraction technique

Objective: PSD

- Delivery properties of Active Ingredient
- Abscissa: size (radius) in μm
- log scale
- Ordinates: number or volume (density)

- PSD in number

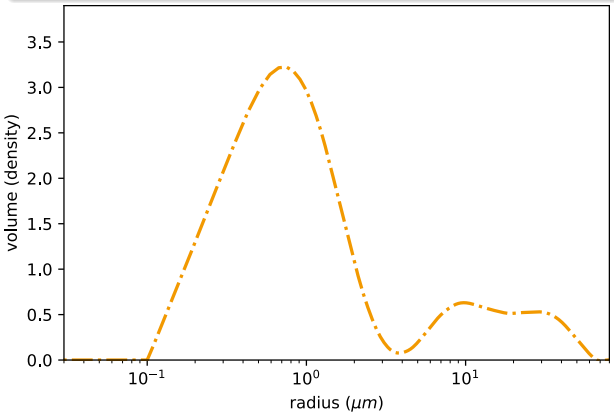


Particle Size Distribution: Laser Diffraction technique

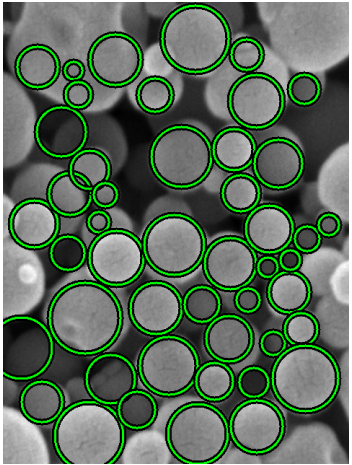
Objective: PSD

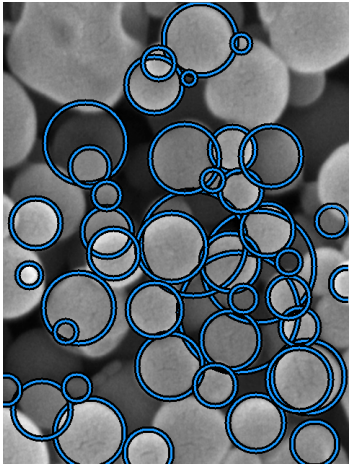
- Delivery properties of Active Ingredient
- Abscissa: size (radius) in μm
- log scale
- Ordinates: number or volume (density)

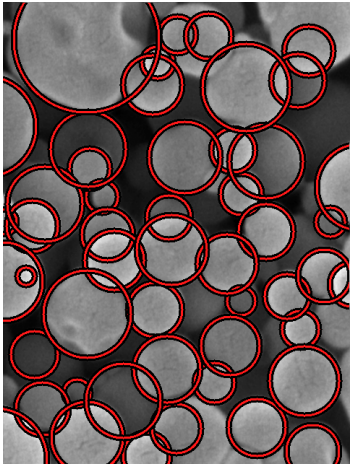
- PSD in volume



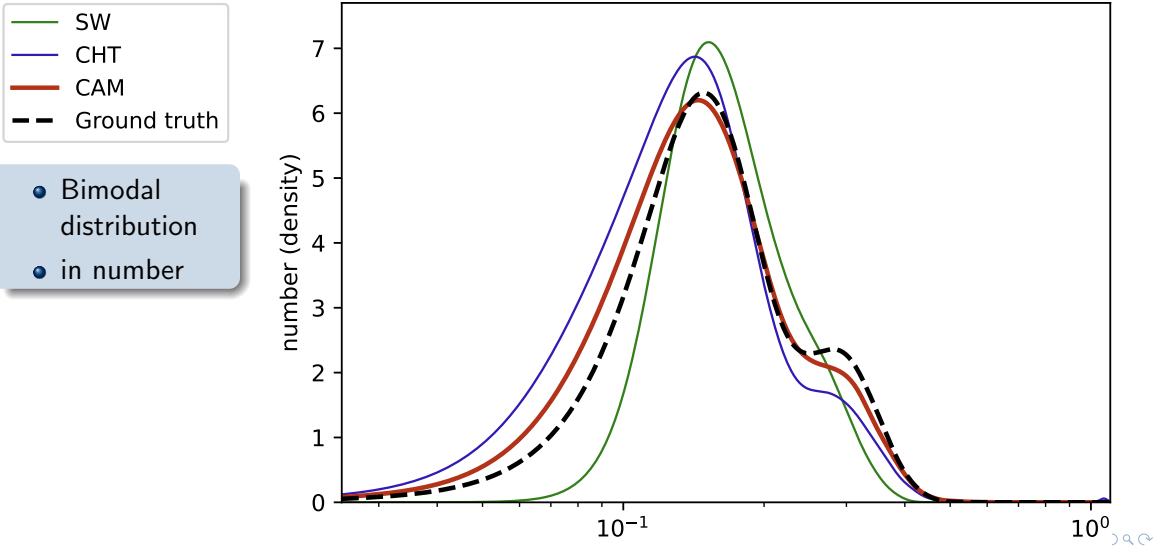
Comparison of SW/CHT/CAM on simulated images





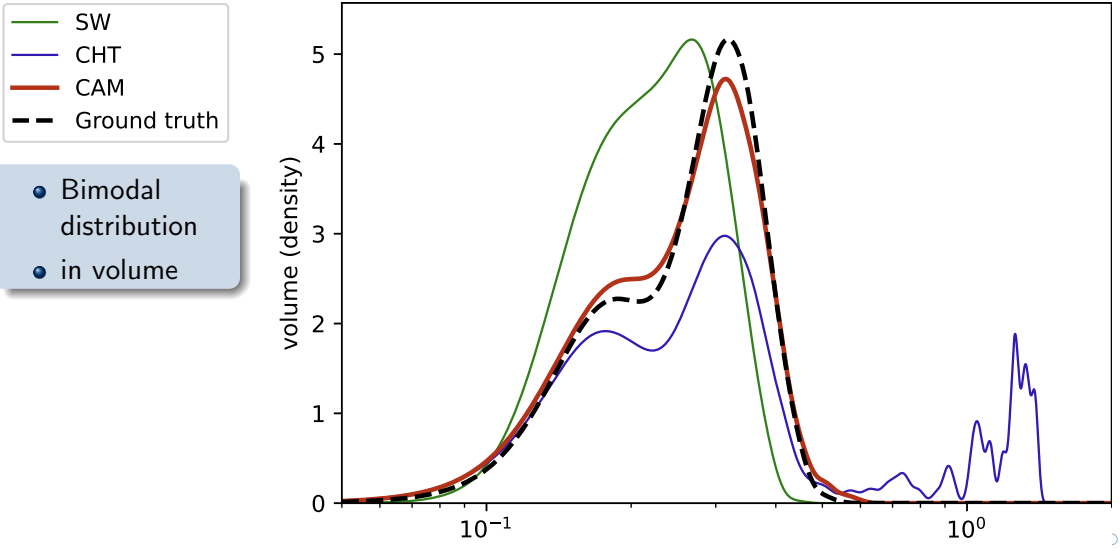


Comparison of SW/CHT/CAM on simulated images

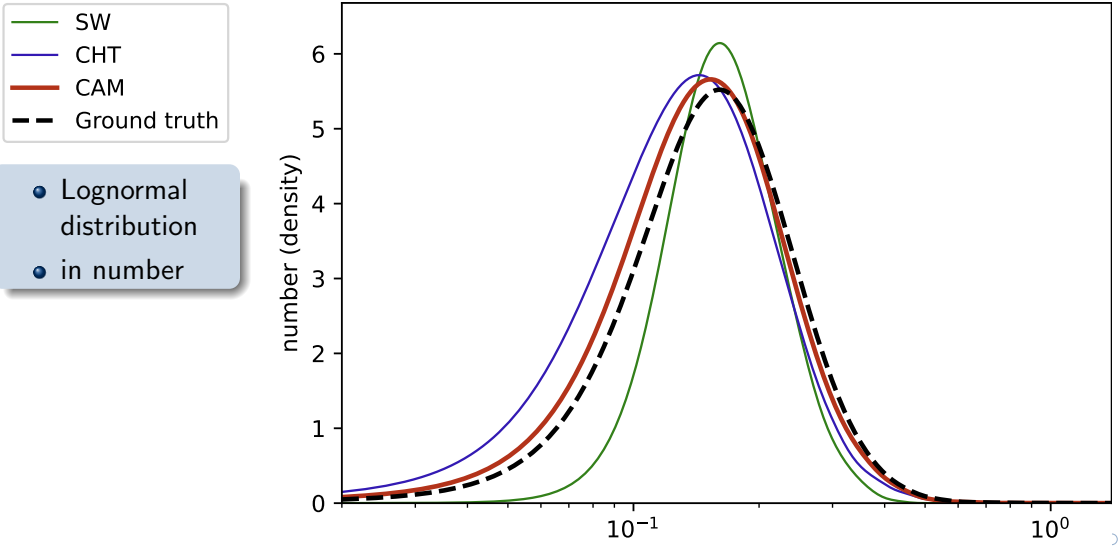


- Bimodal distribution
- in number

Comparison of SW/CHT/CAM on simulated images



Comparison of SW/CHT/CAM on simulated images

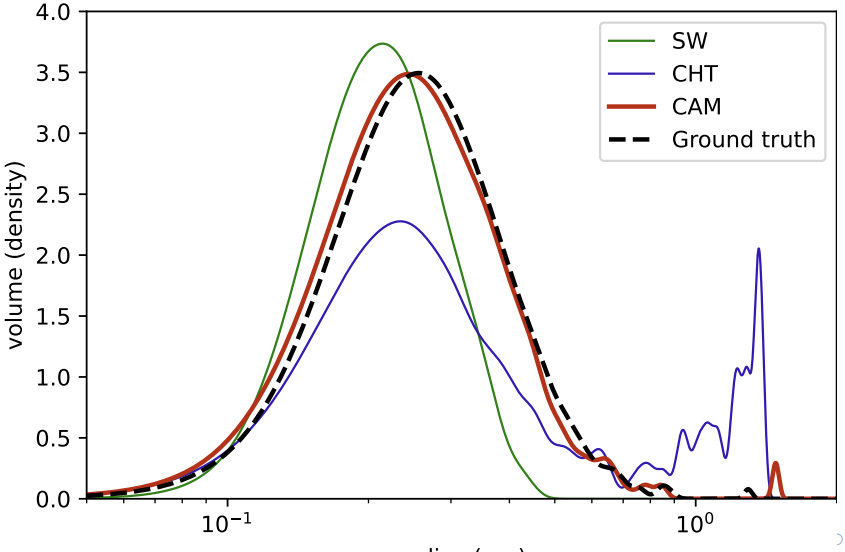


- Lognormal distribution
- in number

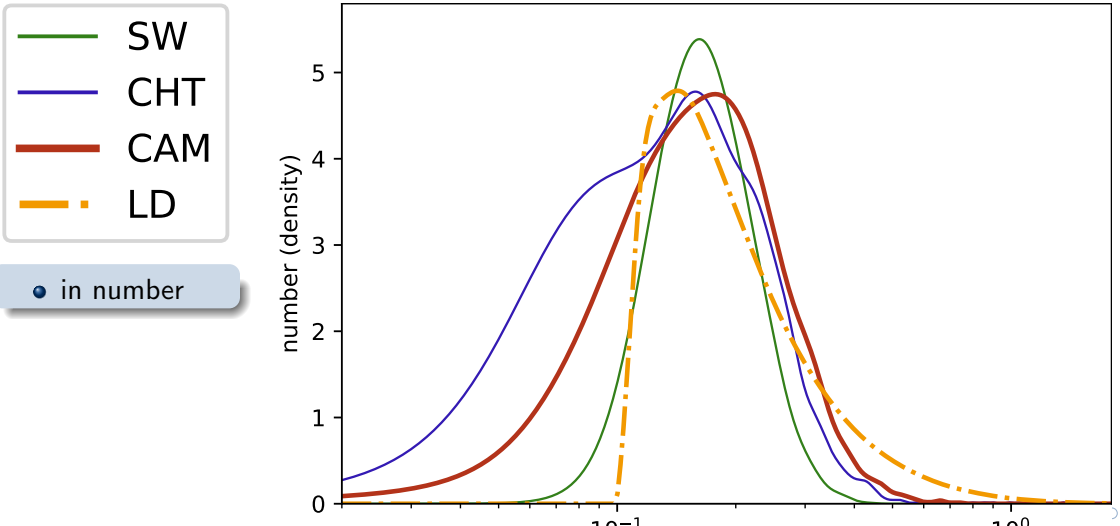
Comparison of SW/CHT/CAM on simulated images

SW
CHT
CAM
Ground truth

- Lognormal distribution
- in volume



Comparison of SW/CHT/CAM with Laser Diffraction LD

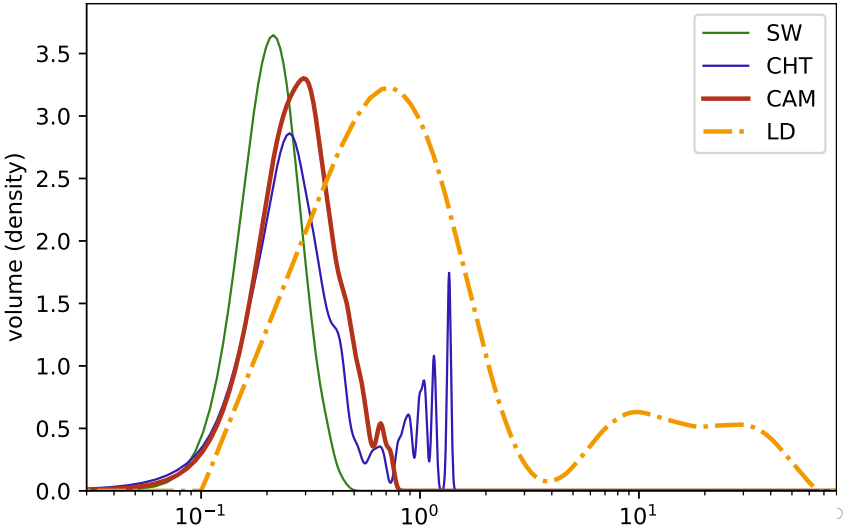


- in number

Comparison of SW/CHT/CAM with Laser Diffraction LD

SW
CHT
CAM
LD

- in volume



Discussion and perspectives

Discussion

- Image segmentation for CAM seems better
- Good agreement for CAM in bimodal or lognormal PSD
- Other methods (SW, CHT) shows a few drawbacks
- Laser Diffraction also presents a few drawbacks (aggregates)

Conclusion

- Method for simulating SEM images of spherical particles
- Method for segmenting these images
- Good agreement with particle size distribution

Perspectives

- Deep learning segmentation
- Synthetic database

We are hiring !

- Maître Assistant (Maître de Conf.) Associé (CDD)
- 3 years, in Saint-Etienne
- Subject: open to proposition, but mainly stochastic geometry, computational geometry, image processing, applied maths
- CNRS lab: process engineering and materials
- Preparation for HDR
- Supervision of PhD thesis + participation to ongoing projects
- Direct research projects with ORANO (nuclear company)
- sept.2024: Minkowski functionals and fuel cells (3D microstructures)
- Teaching: around 50h
- What's next: Maître Assistant position