**ACE 1-** Science Concept Review

*June 14, 2011*

# Birth, structure and evolution of depletion gels in μ-gravity

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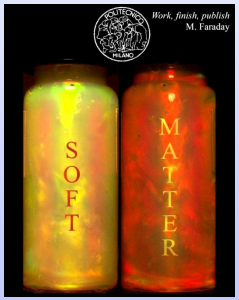

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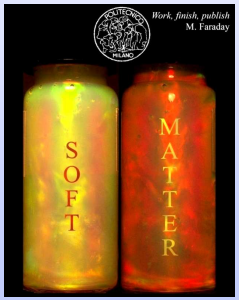
# Birth, structure and evolution of depletion gels in μ-gravity

*Roberto Piazza*

*Stefano Buzzaccaro*

*Luca Cipelletti*


POLITECNICO MILANO

UNIVERSITÉ MONTPELLIER 2
SCIENCES ET TECHNIQUES

# SUMMARY

A) MOTIVATIONS, AIMS, AND STRATEGY

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B) BRIEF SCIENCE OVERVIEW/REVIEW OF GROUND EXPERIMENTS

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E) PRELIMINARY TEST MATRIX

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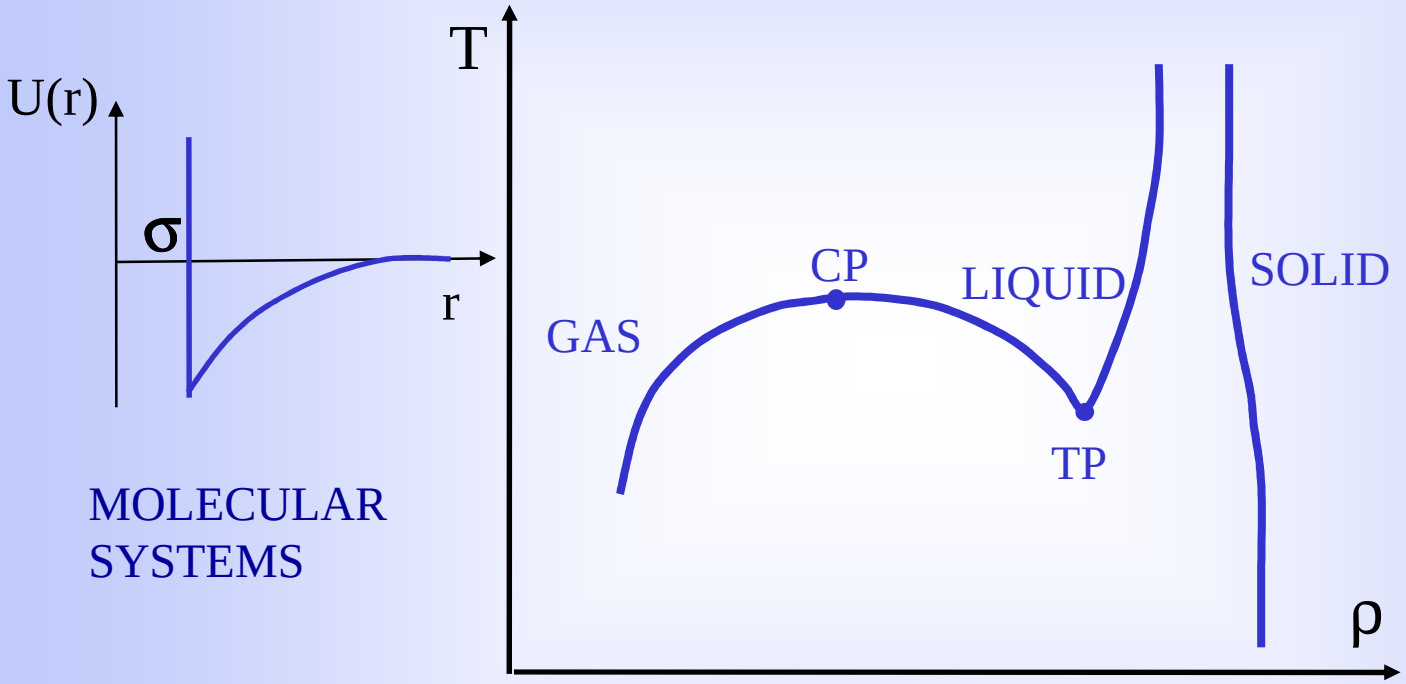
- Depletion interactions due to the addition to a colloid of high molecular weight additive are a well studied tool to tuneshort-range attractive interactions.

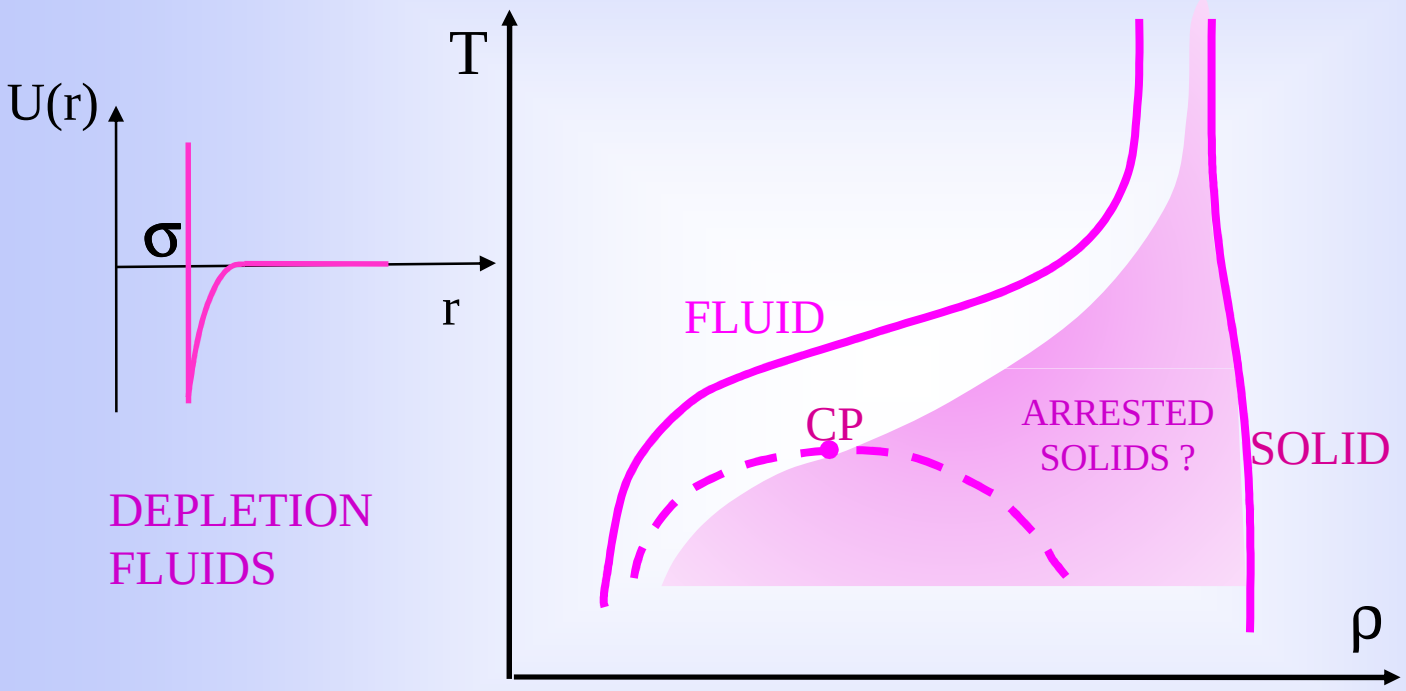
- The key strategy of this proposal is using a system where depletion interactions can be carefully and quantitatively controlled. Temperature can be used as a tuning parameter allowing to homogenously drive a system from an equlibrium suspension state to a long-lived metastable gel.

# A QUICK LOOK TO THE STRANGE WORLD OF VERY SHORT-RANGED POTENTIALS




MOLECULAR SYSTEMS

# A QUICK LOOK TO THE STRANGE WORLD OF VERY SHORT-RANGED POTENTIALS




The liquid-gas transition becomes metastable with respect to freezing .
Nonetheless, the latter:

- Has huge effect on crystal nucleation (ten Wolte &Frenkel,1997)
- Is strongly related to the formation of structurally arrested phases (glasses/gels)

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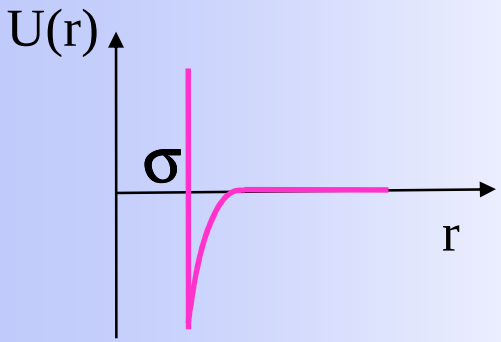
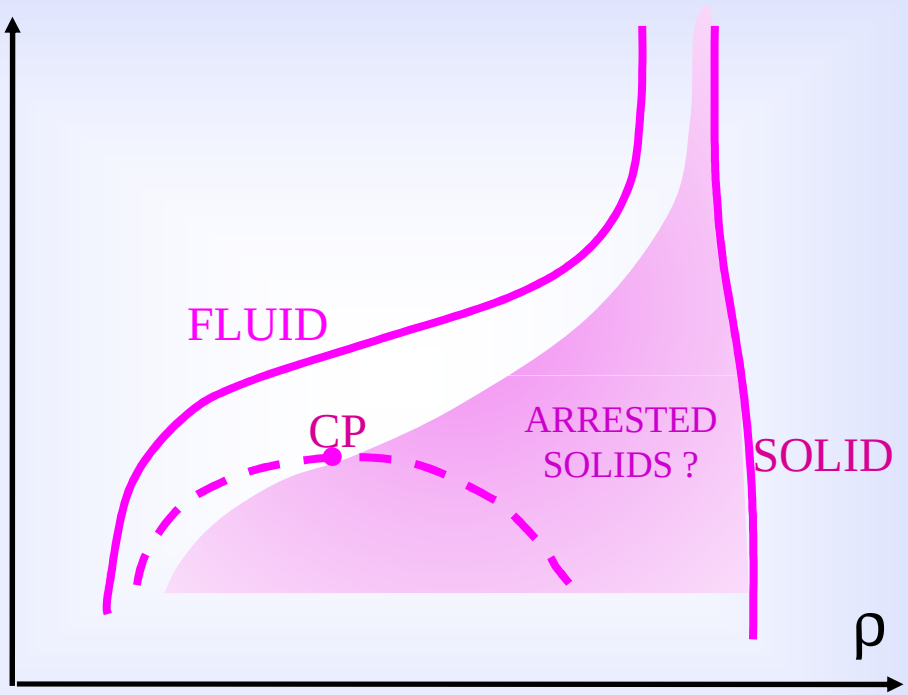
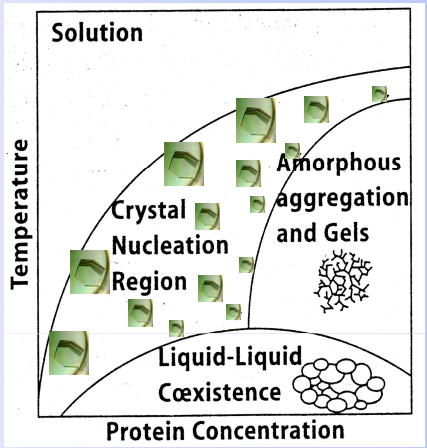



DEPLETION FLUIDS




*E.G.: PROTEIN CRYSTALLIZATION*

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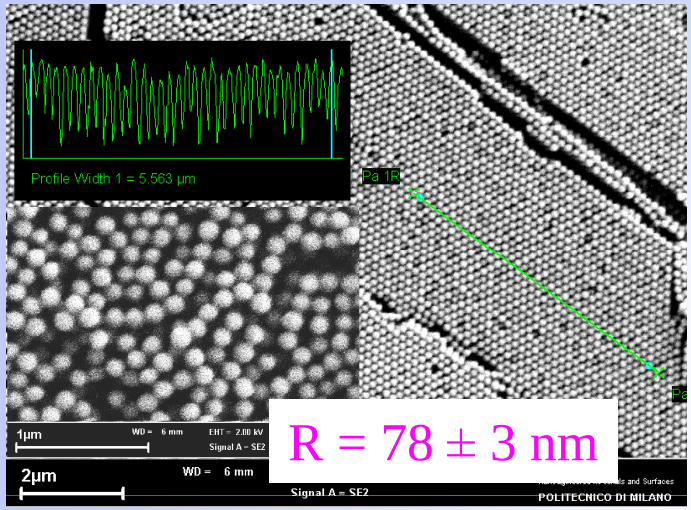


R = 78 ± 3 nm

## SYSTEM

COLLOID: MFA (modified PTFE) particles

- Low refractive index (<n> = 1.35) → Low turbidity
- High density ($\rho > 2$ g/cm$^3$) → "Fast" sedimentation

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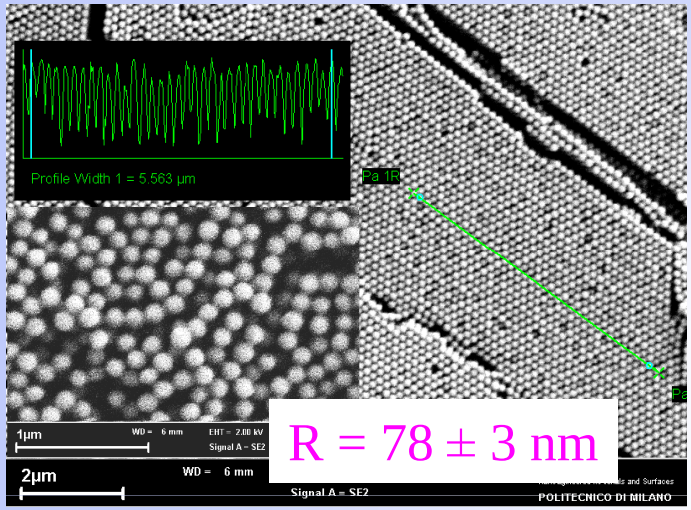


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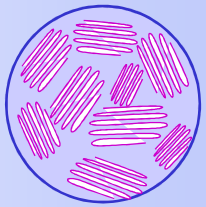
DISTINCTIVE OPTICAL PROPERTIES

PARTIALLY CRYSTALLINE

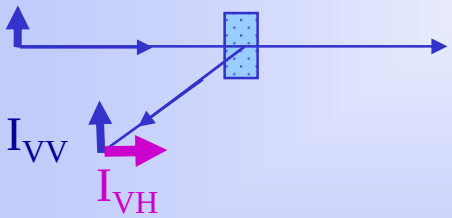


↓

OPTICALLY ANISOTROPIC

↓

DEPOLARIZED SCATTERING

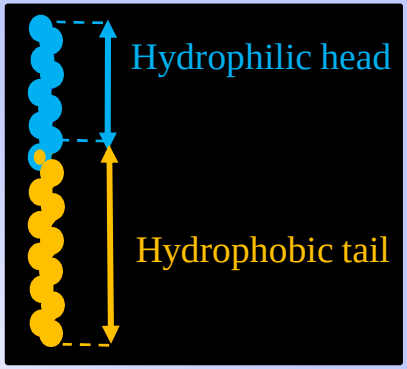



$I_{VH}$ IS INSENSITIVE TO PARTICLE INTERACTIONS !

(useful for sedimentation studies – see below)

# DEPLETANTS

## Triton X100/$C_mE_n$ (nonionic surfactants)




- Forms globular micelles in a wide conc. range
- Adsorb on MFA, leading to steric stabilization
- MICELLAR DEPLETION at larger surfactant concentration
- Phase separation with water by raising T (T-dependent micellar interactions)

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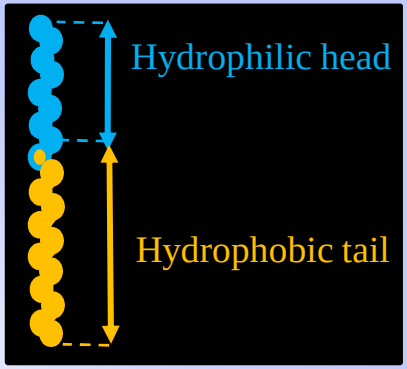


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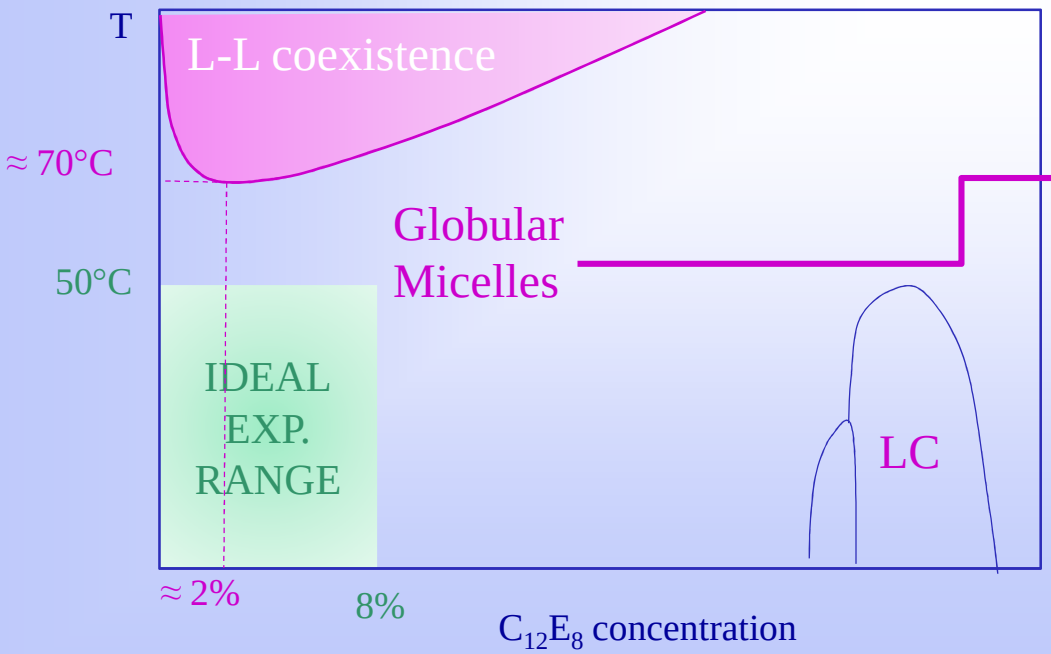
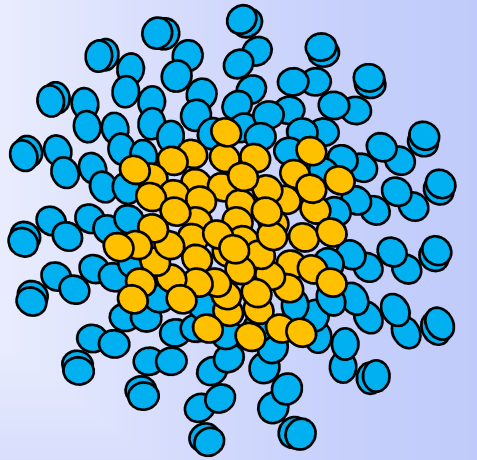
EXAMPLE: $C_{12}E_8$



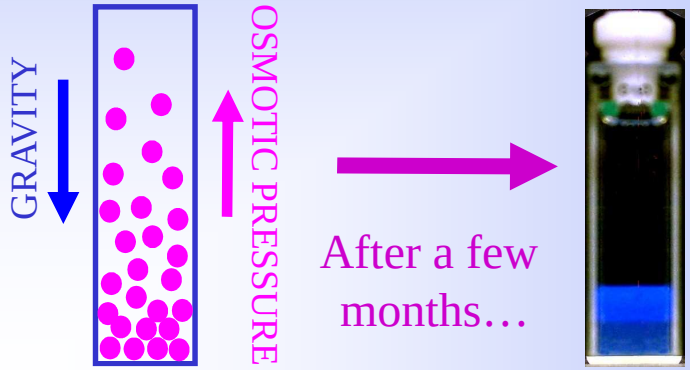
r ≈3.5 nm

Aggregation number
N ≈ 100

# STEP1: EXPLOITING EQUILIBRIUM SEDIMENTATION TO PROBE THE EQUATION OF STATE & PHASE DIAGRAM

Ref.: S. Buzzaccaro, R. Rusconi, and R. Piazza, *"Sticky" Hard Spheres: Equation of State, Phase Diagram, and Metastable Gels*, Phys. Rev. Lett. **99,** 098301 (2007)

Depolarized scattering intensity



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Depolarized scattering intensity

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Local particle concentration

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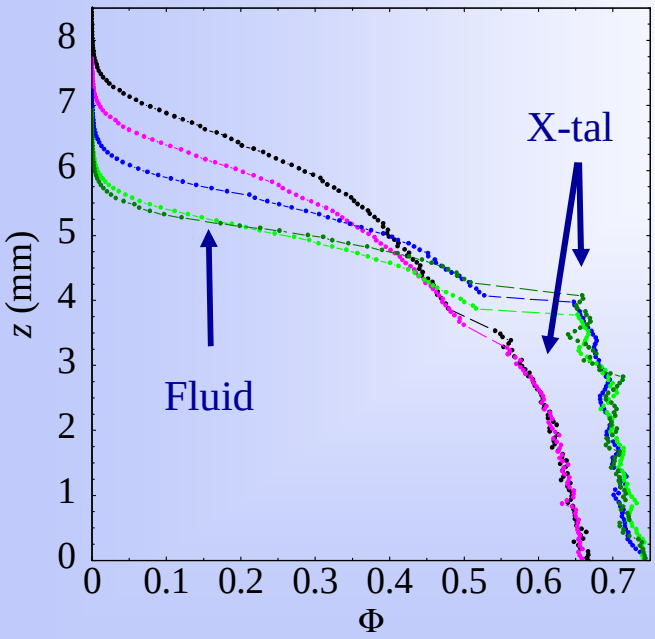
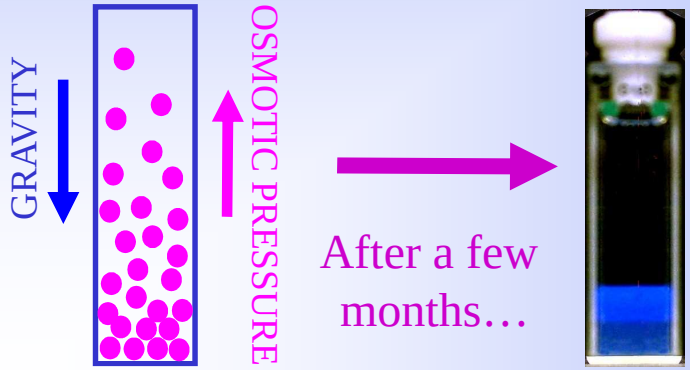
CONC. PROFILES



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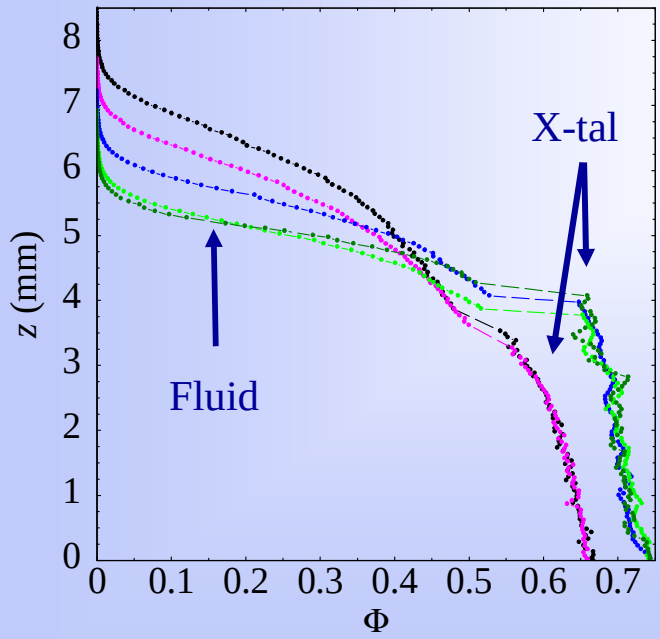
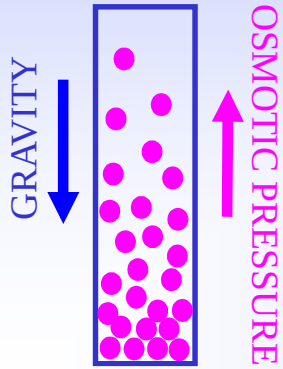


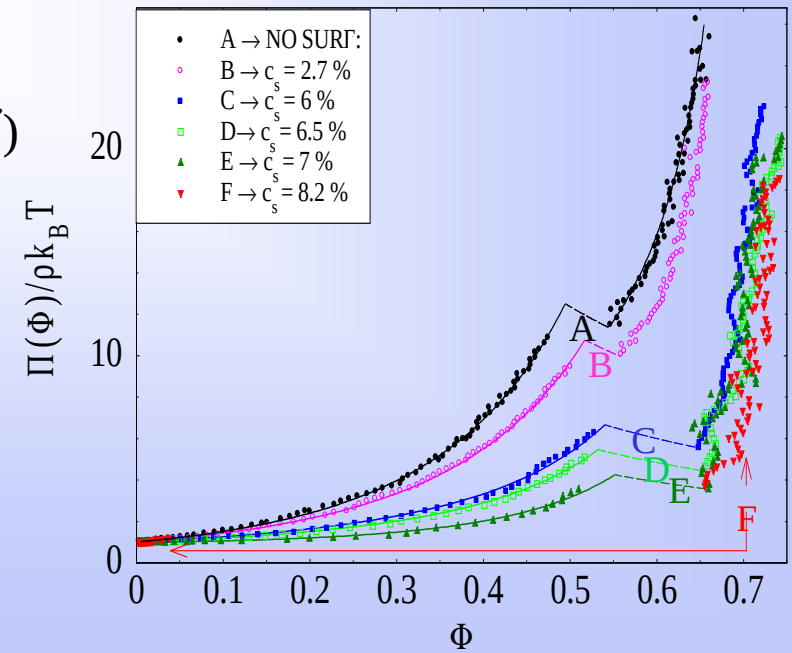




After a few months…

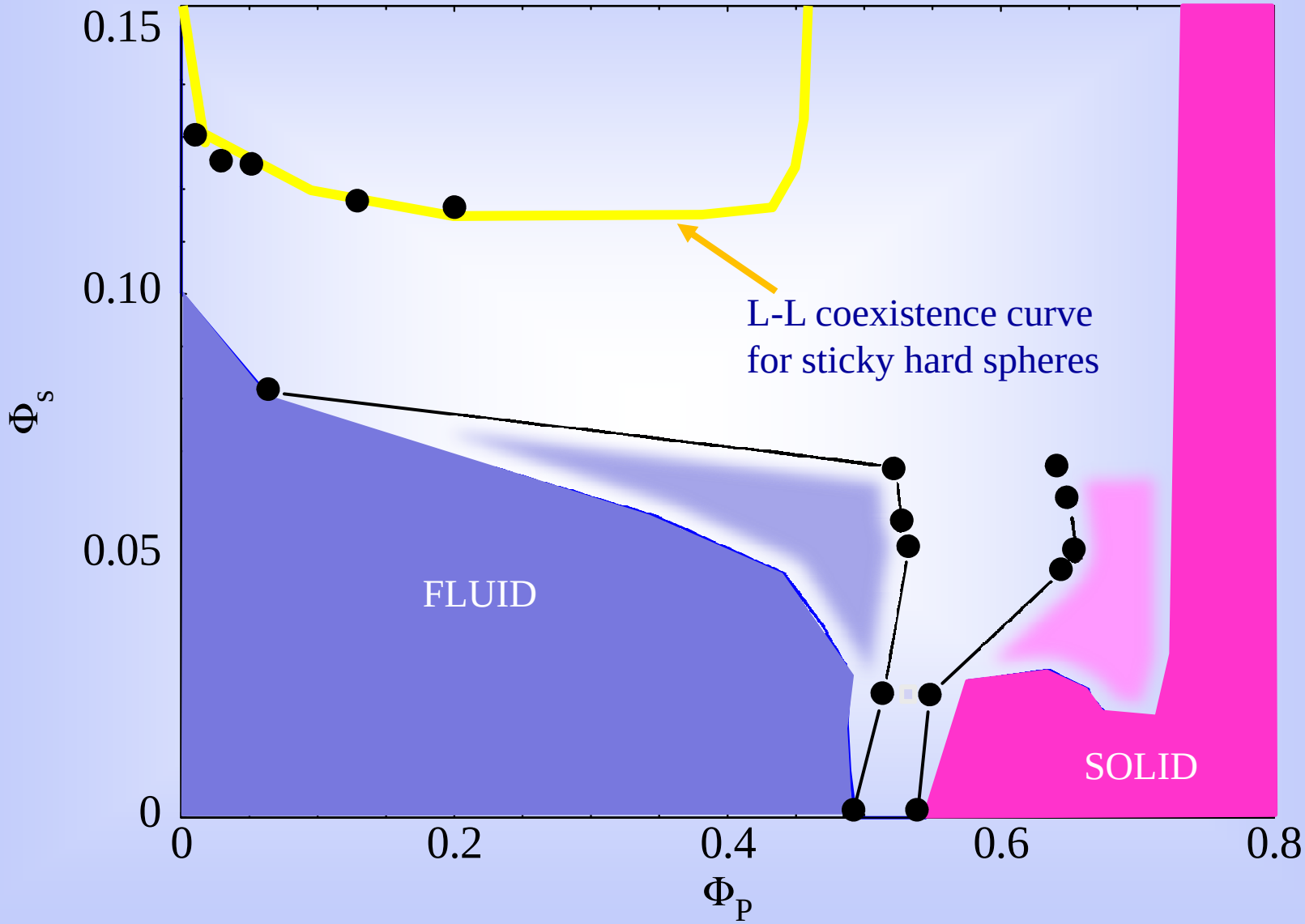
$$\Pi(z) = g\Delta\rho \int_z^h dz' \Phi(z')$$

From Π(z) and Φ(z)

EQUATIONS OF STATE



# FULL PHASE DIAGRAM:
# TO THE ROOTS OF GELATION



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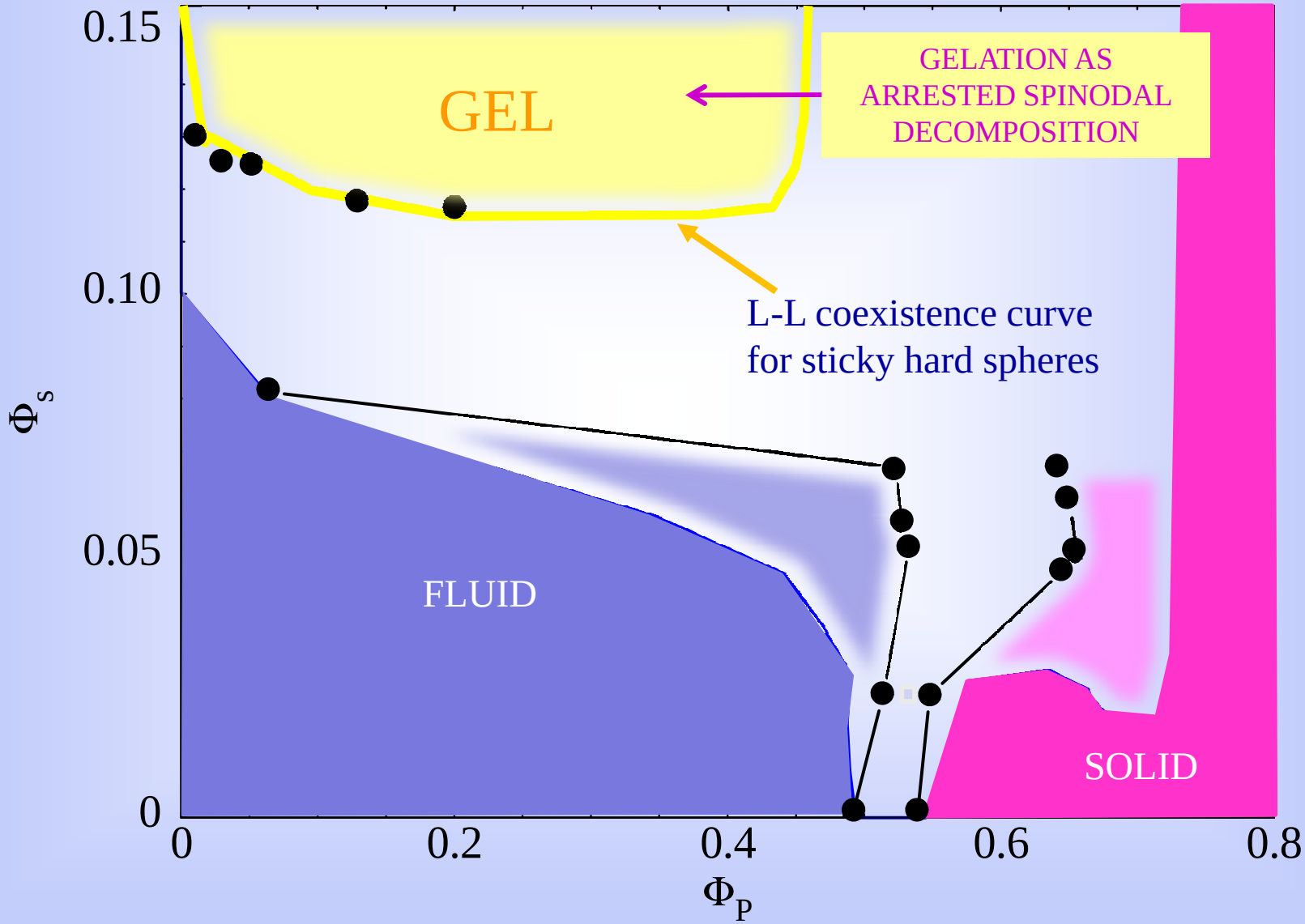

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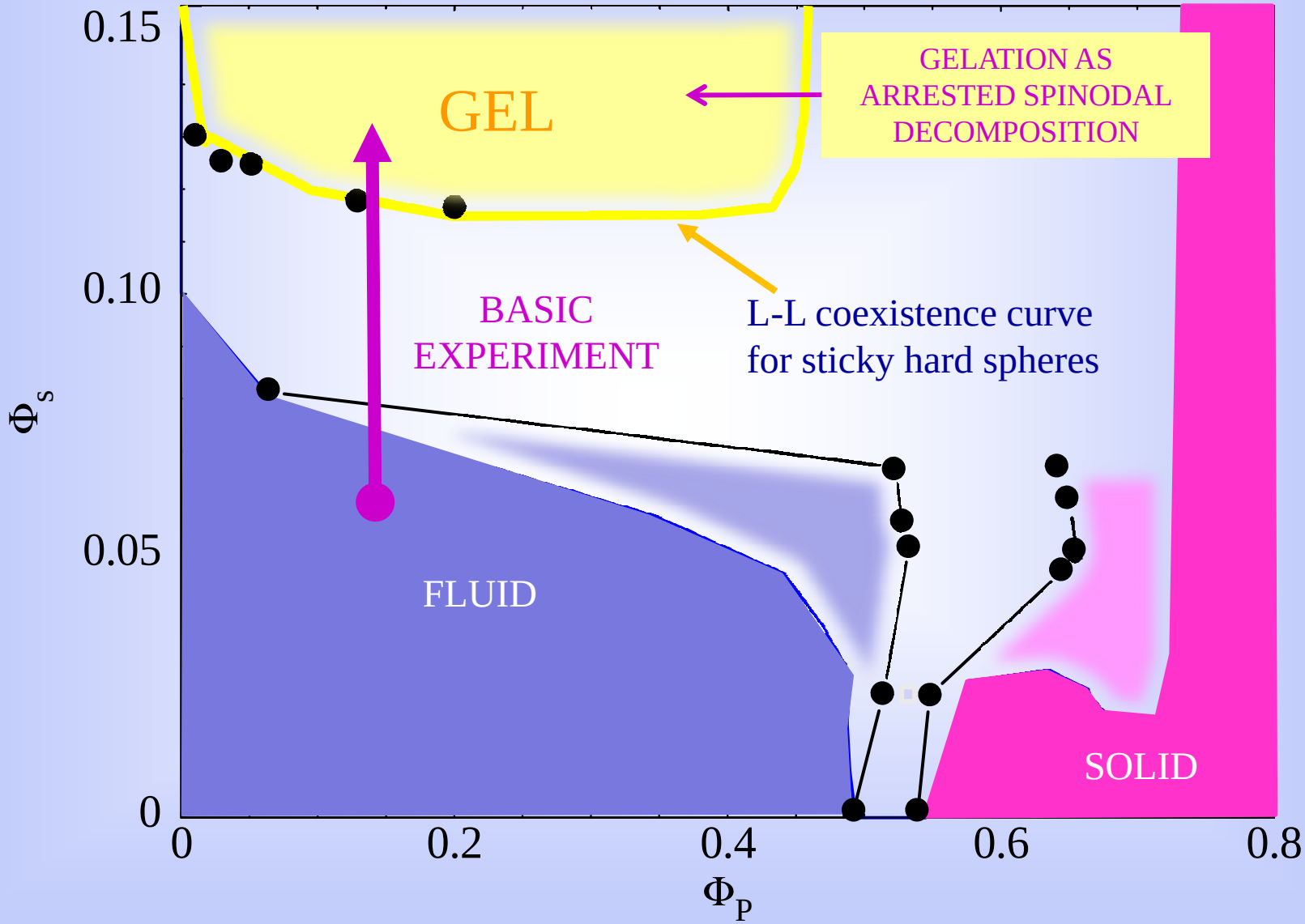

# BIRTH OF A GEL

*By quenching into L-L coexistence:*

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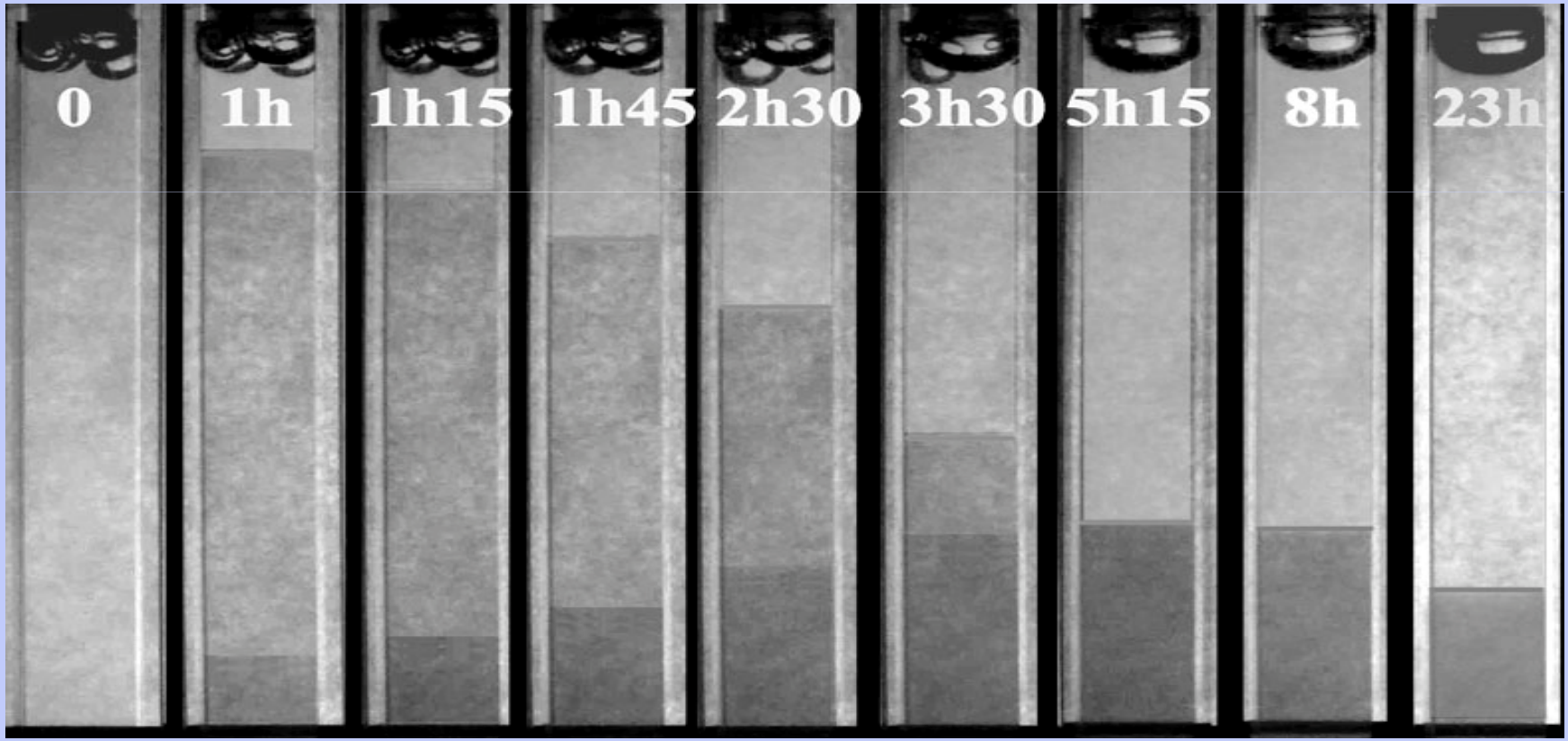
FAST SEDIMENTATION

(hours vs. months!)



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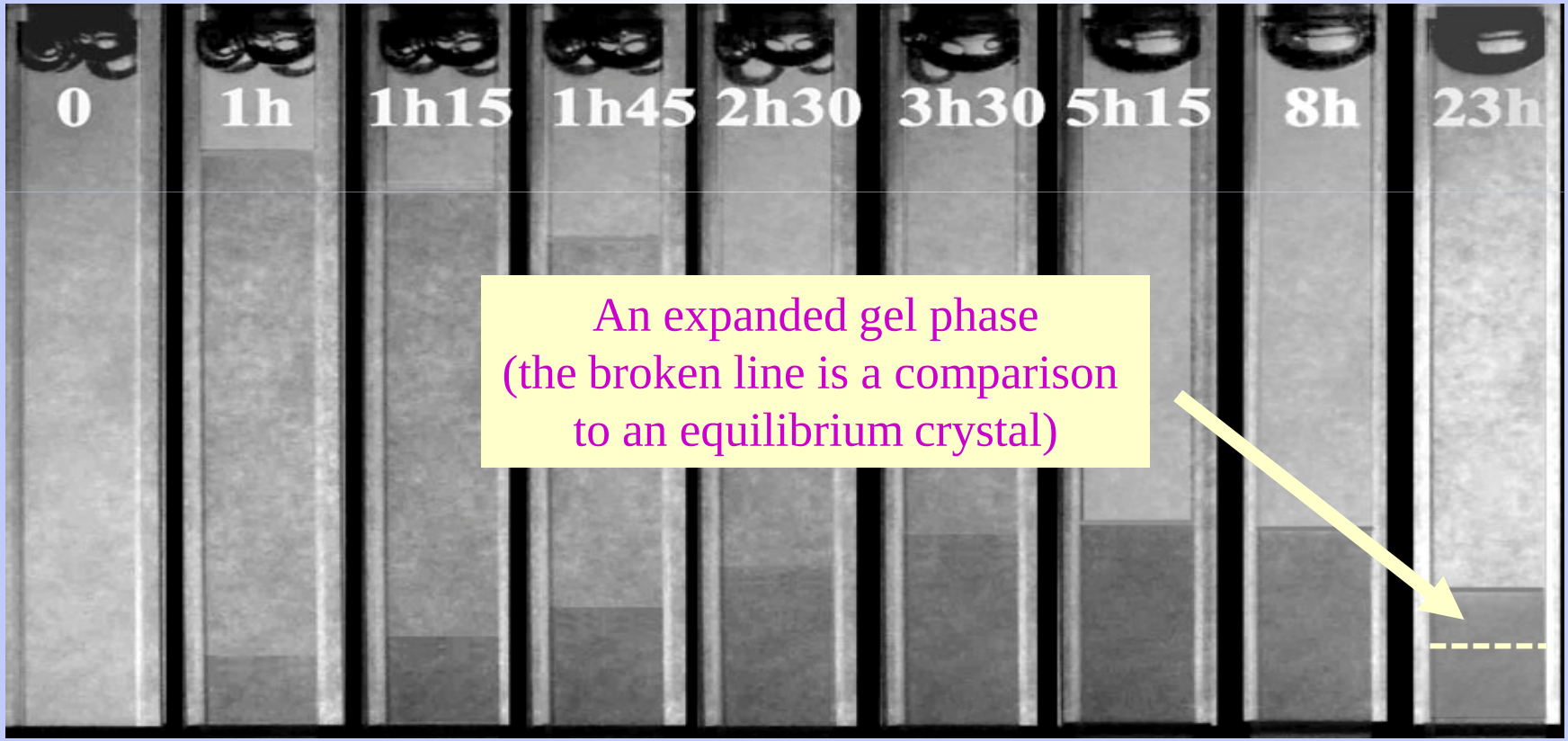
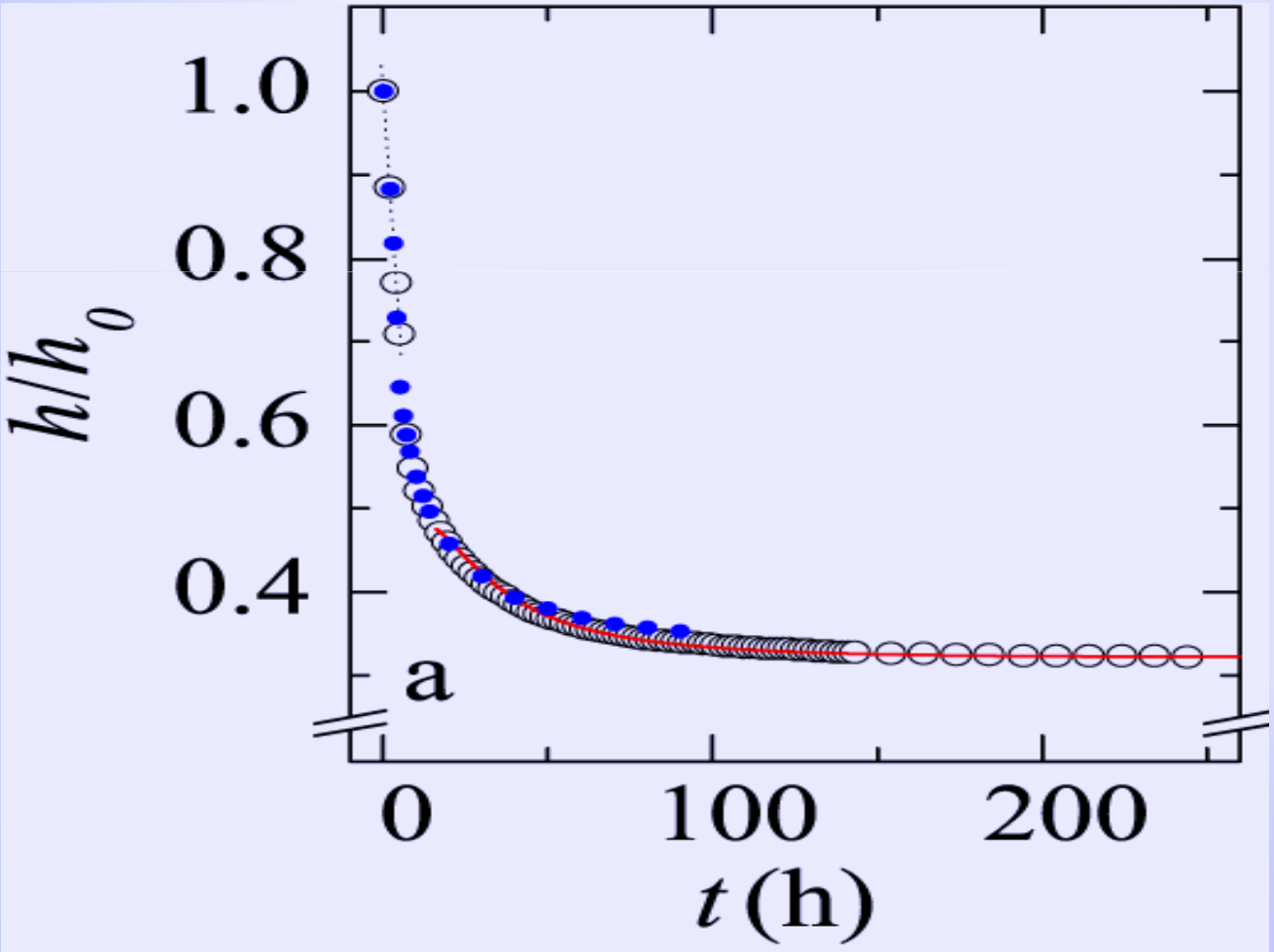



An expanded gel phase
(the broken line is a comparison
to an equilibrium crystal)

# STEP 2: GROUND EXPERIMENTS ON GEL COLLAPSE AND RESTRUCTURING DUE TO GRAVITATIONAL STRESS

Ref.: G. Brambilla, S. Buzzaccaro, R. Piazza, L. Berthier, and L. Cipelletti, *Highly nonlinear dynamics in a slowly sedimenting colloidal gel* , Phys. Rev. Lett. 106, 118302 (2011)

Time evolution of the gel height ($\Phi_P \approx 0.12, U_{att} \approx 4.5\ k_B T$ )



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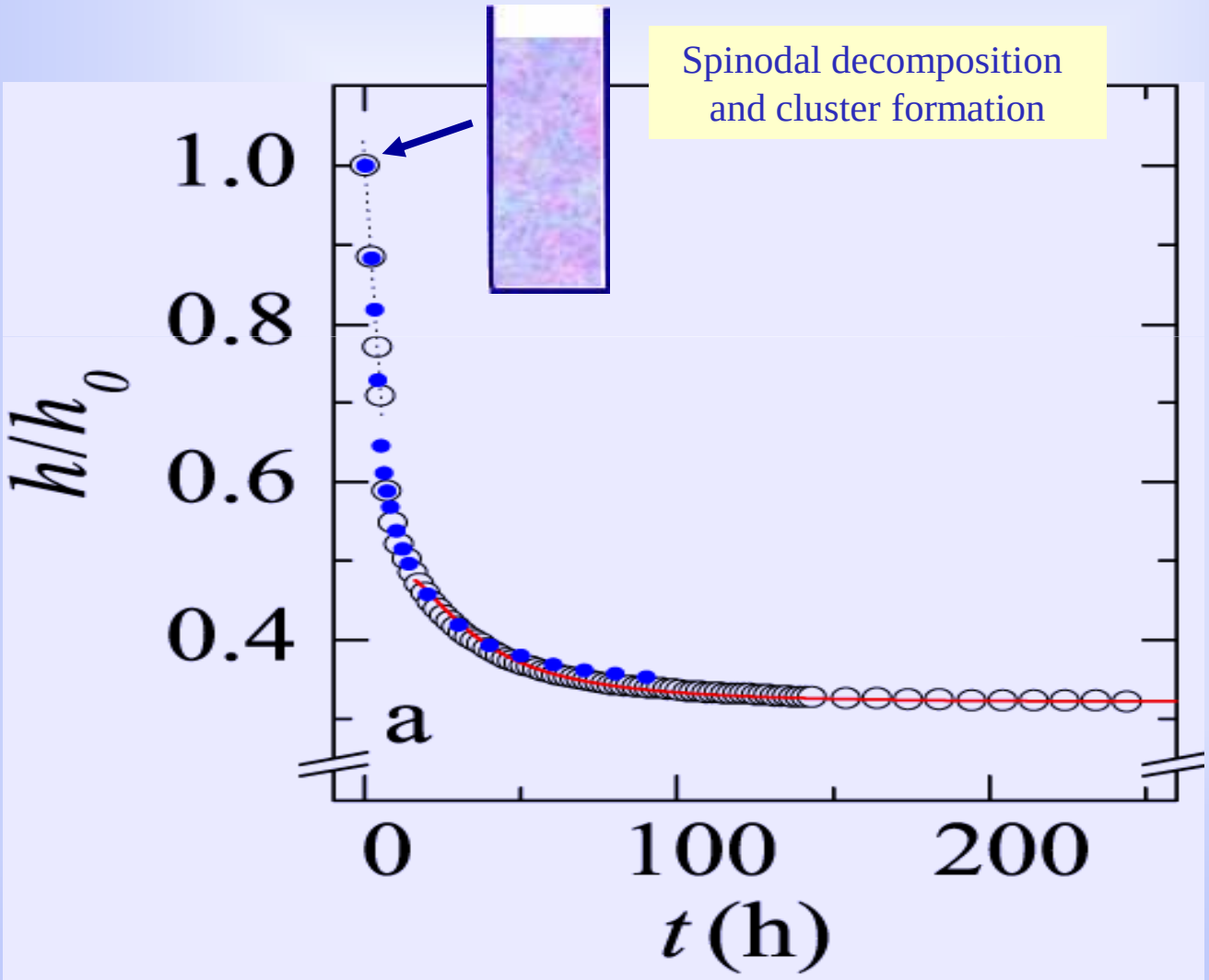

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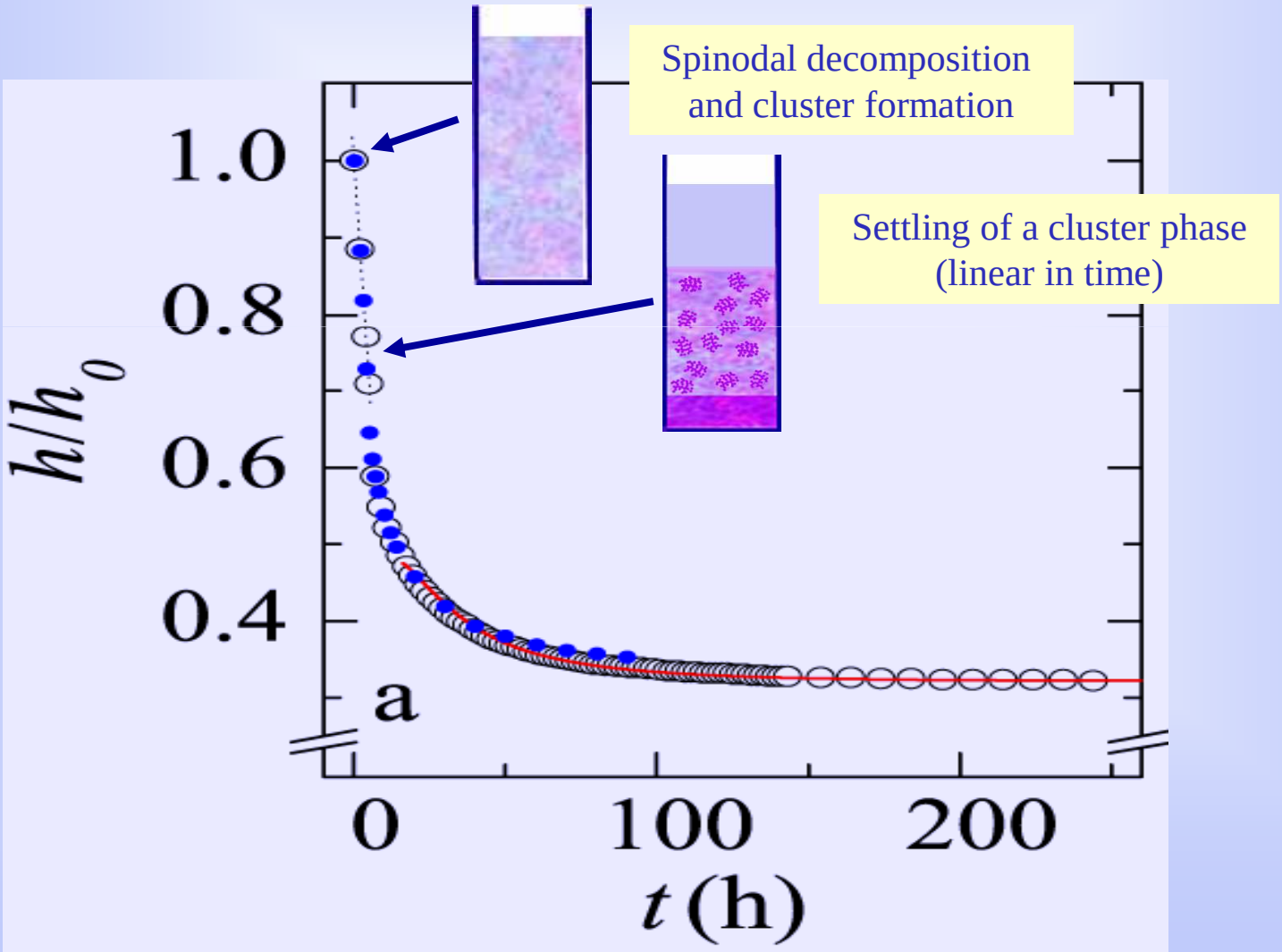

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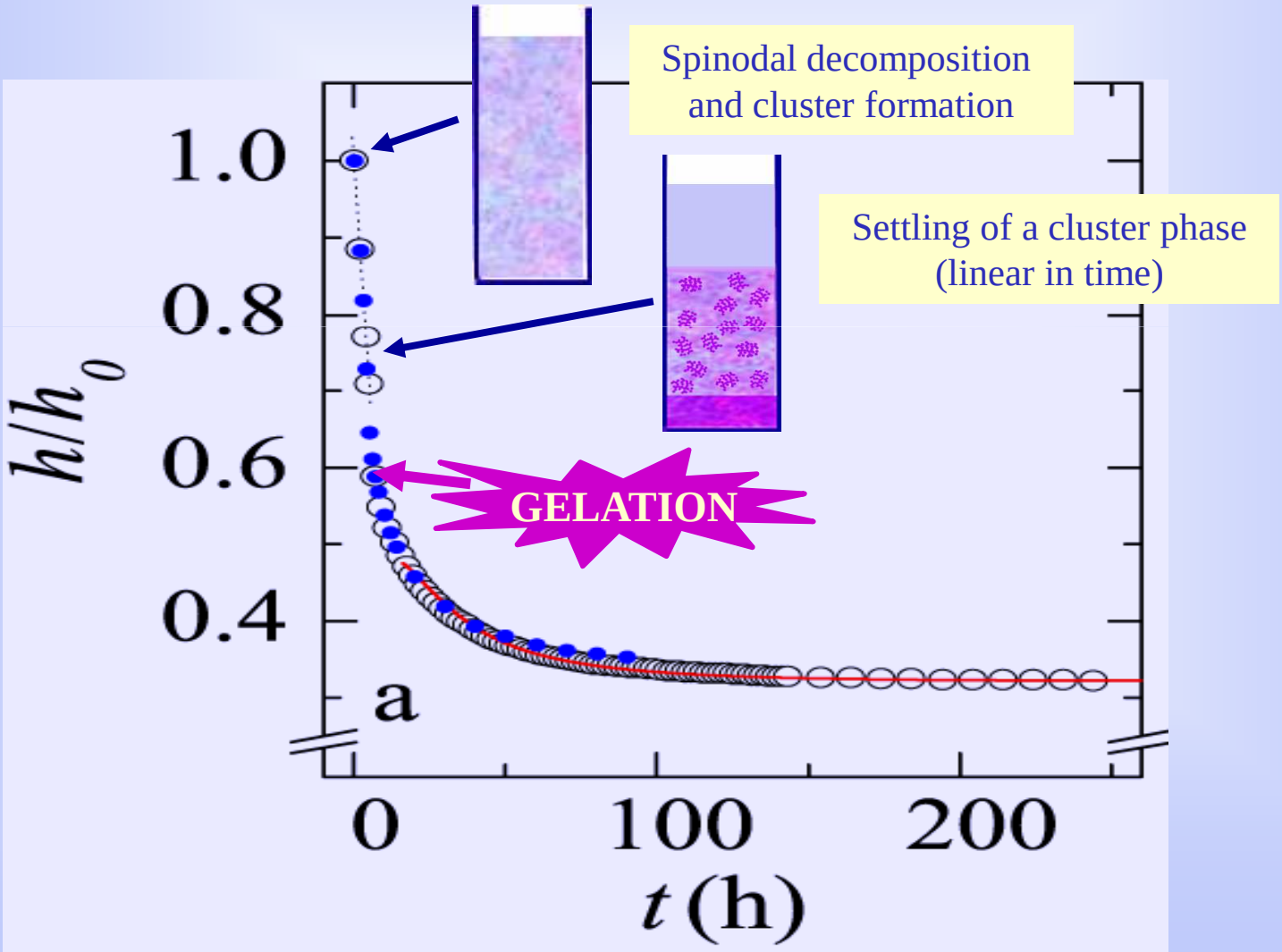

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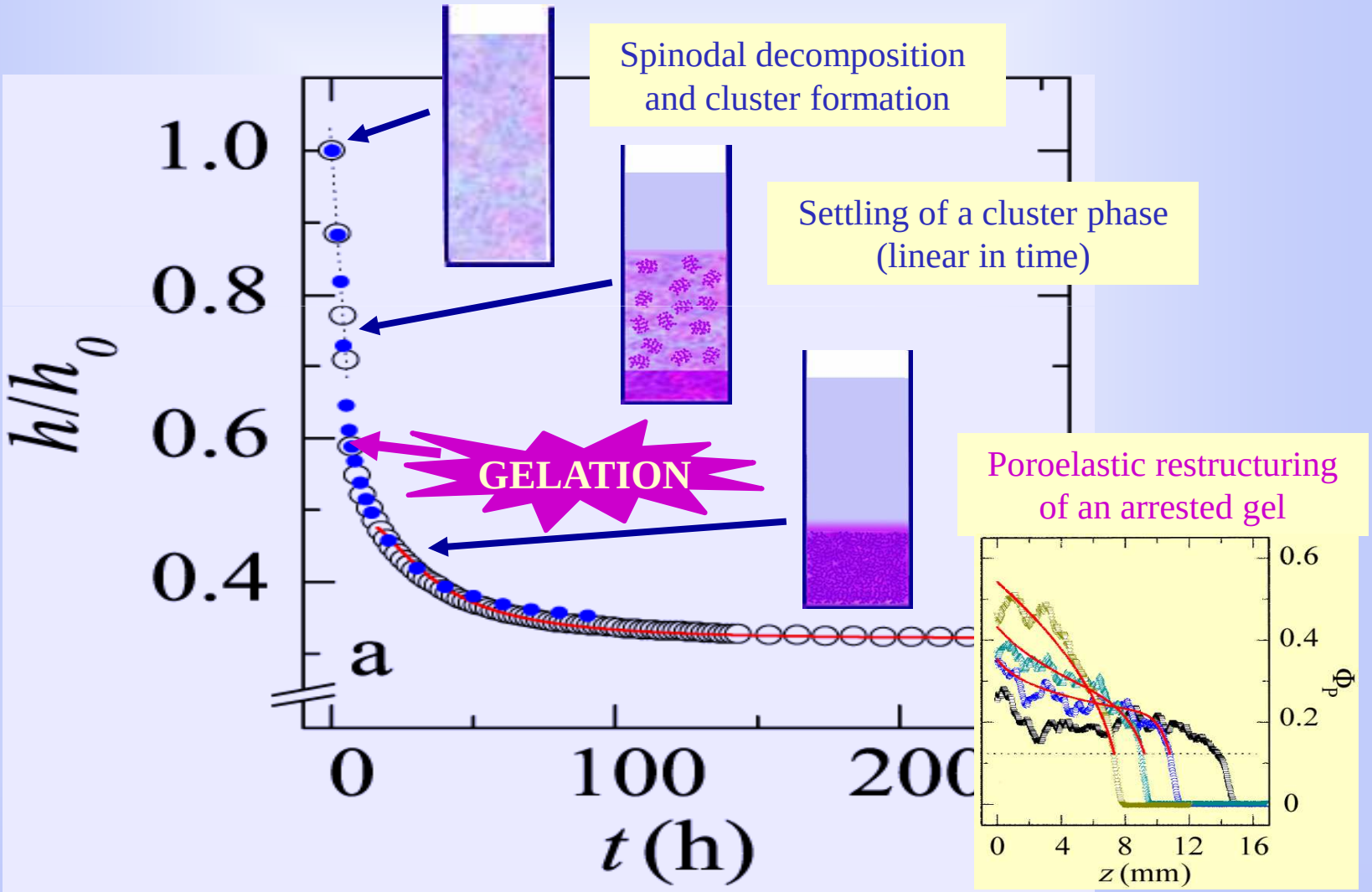

# THE POROELASTIC REGIME

## PICTURE: A FLUID (COUNTER)FLOWING THROUGH AN ELASTIC POROUS MEDIUM

- GENERAL EQUATION:

$$\frac{\partial \Phi}{\partial t} = \frac{\partial}{\partial z}\left[\frac{\Phi \kappa(\Phi)}{\eta}\left(\Delta \rho g \Phi + \frac{K(\Phi)}{\Phi}\frac{\partial \Phi}{\partial z}\right)\right]$$

PERMEABILITY

GRAVITATIONAL STRESS

ELASTIC RESPONSE

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PERMEABILITY

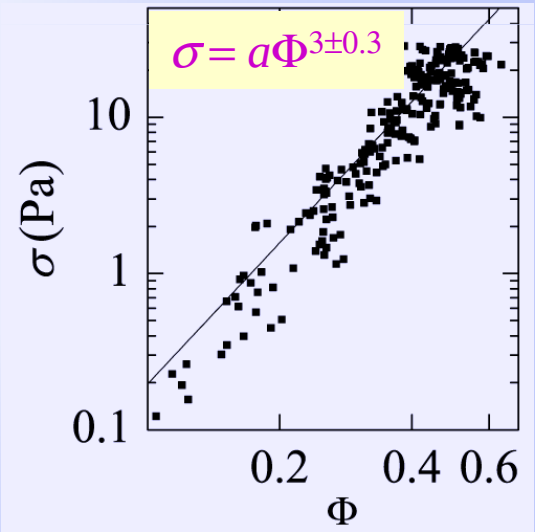
GRAVITATIONAL STRESS

ELASTIC RESPONSE

- INPUT FOR NUMERICAL SIMULATIONS:

$$i)\quad K(\Phi) = \Phi \frac{\partial \sigma}{\partial \Phi}$$

EFFECTIVE COMPRESSIONAL MODULUS IN RESPONSE TO AN APPLIED STRESS σ FROM STEADY-STATE PROFILE



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PERMEABILITY

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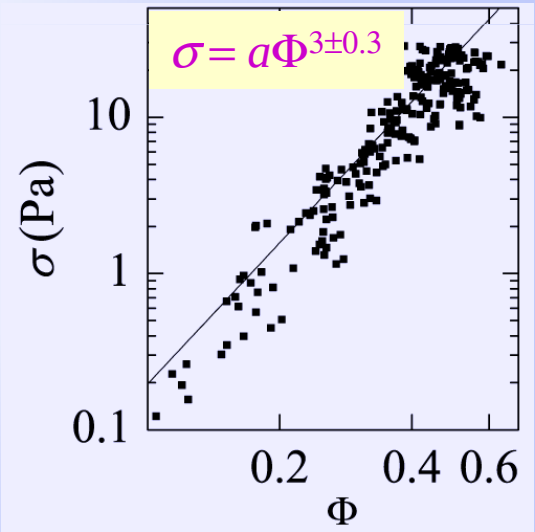


*ii)* $\kappa(\Phi) = \kappa_0 \dfrac{(1-\Phi)^m}{\Phi}$

WITH $\kappa_0$ AND $m$ CHOSEN TO FIT THE TIME-DEPENDENCE OF THE GEL HEIGHT

# VELOCIMETRY

NO HORIZONTAL COMPONENT OF THE DISPLACEMENT DURING GEL COMPRESSION

↓

NEGLIGIBLE STRESS ON THE CELL WALLS (CONFIRMED BY POLARIMETRY)

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POISSON RATIO ≈ 0 (LIKE CORK)

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NO HORIZONTAL COMPONENT OF THE DISPLACEMENT DURING GEL COMPRESSION

↓

NEGLIGIBLE STRESS ON THE CELL WALLS (CONFIRMED BY POLARIMETRY)

↓

POISSON RATIO ≈ 0 (LIKE CORK)

## LOCAL SETTLING VELOCITY v(t) (AT VARIOUS SETTLING TIMES)

- THE VELOCITY PROFILE IS ALMOST LINEAR FOR ANY SETTLING TIME, EXCEPT IN THE UPPERMOST LAYER OF THE GEL.

↓

- A *z*-INDEPENDENT (BUT *t*-DEPENDENT)

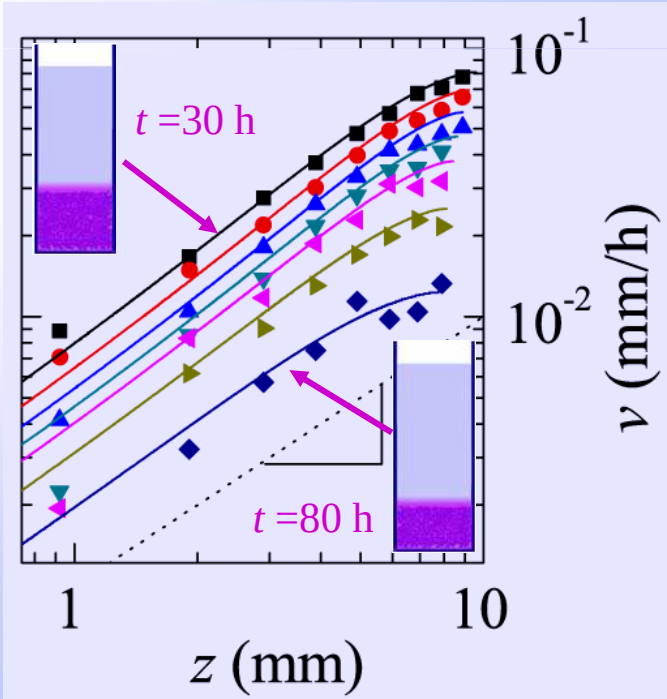
STRAIN RATE: $\dot{\varepsilon}(t) = dv/dz$

↓

- ON A PLANE AT COSTANT σ (SAME WEIGHT ON TOP)

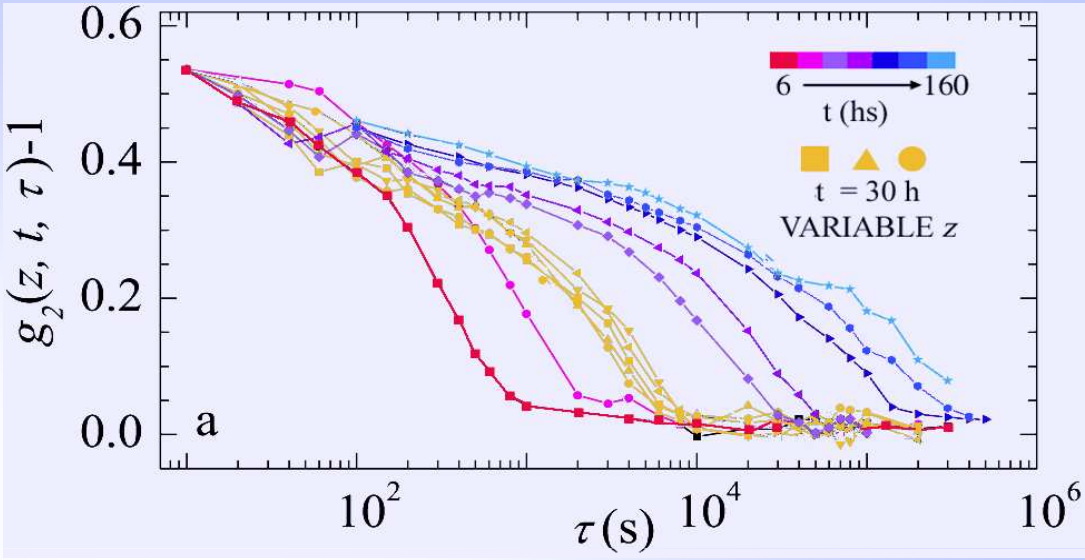
EFFECTIVE "COMPRESSIONAL" VISCOSITY:

$$\eta(t) = \frac{\sigma}{\dot{\varepsilon}(t)} \approx \dot{\varepsilon}(t)^{-1}$$



# MICROSCOPIC DYNAMICS

Local TRC correlation functions in the gel



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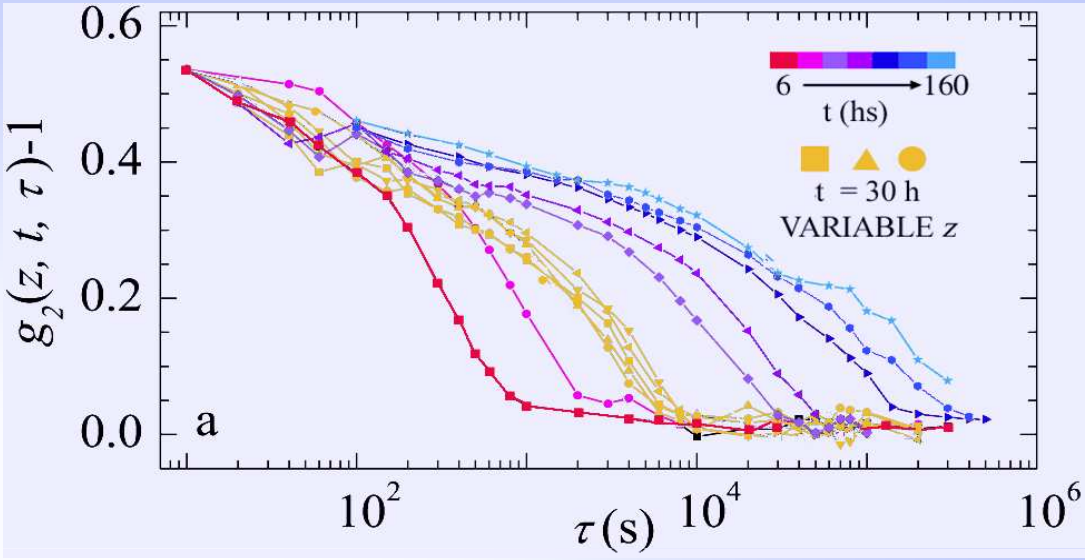
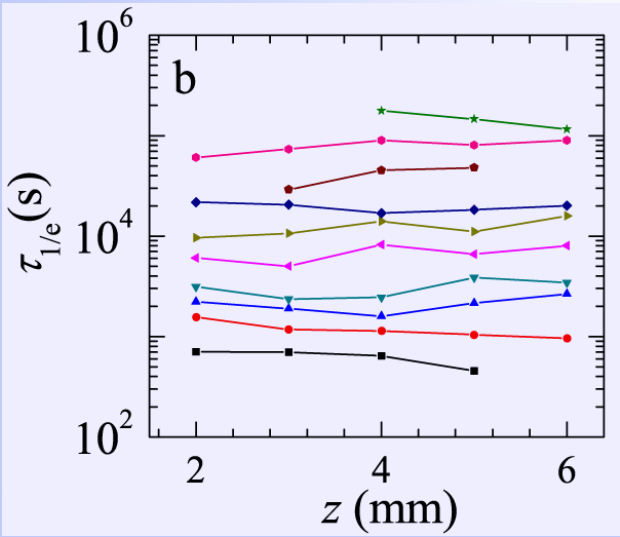



SAME decay time at all values of z
(as for strain rate!)



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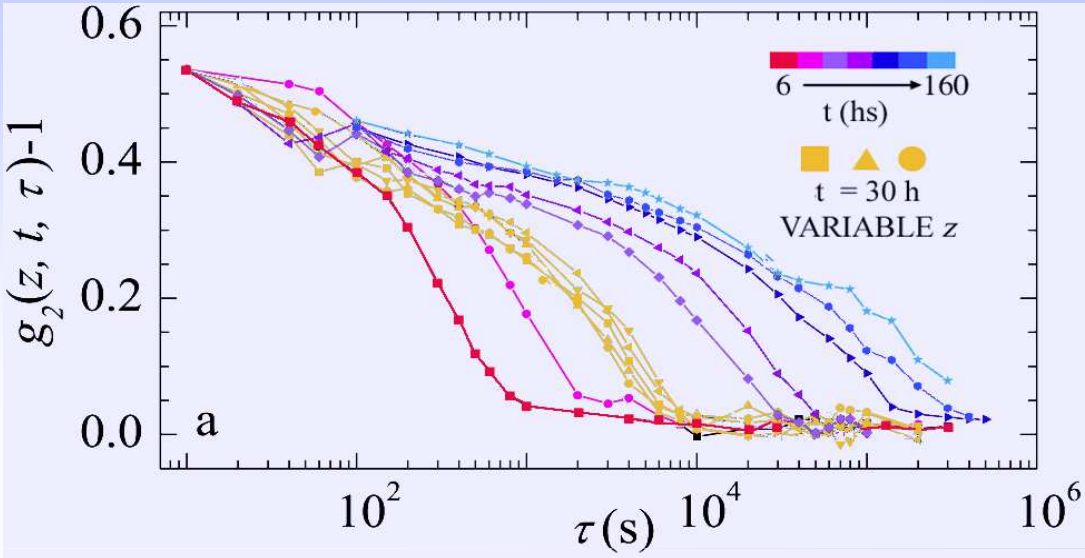


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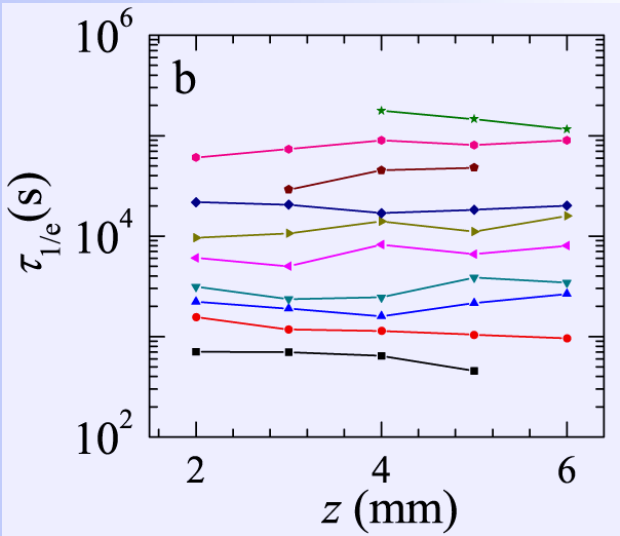
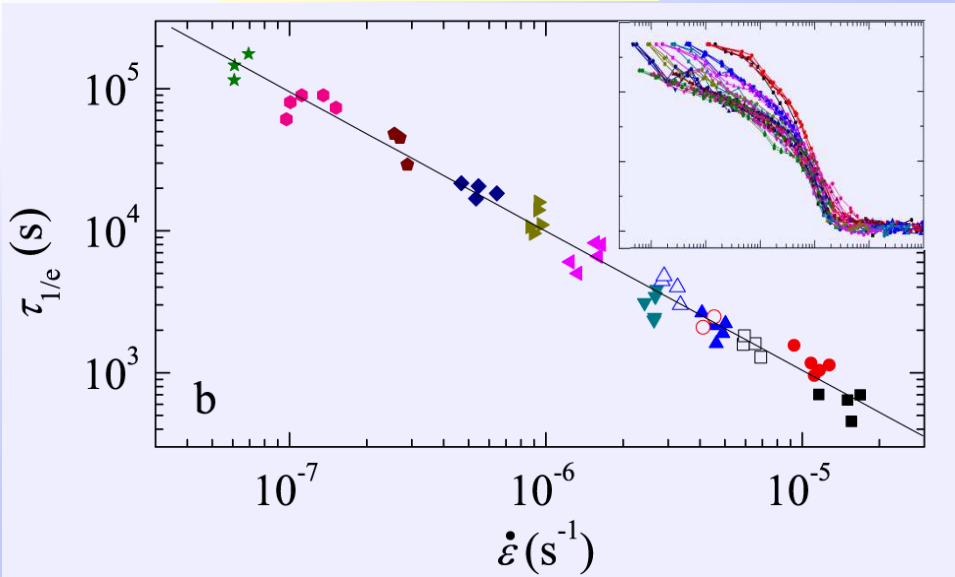



$\tau_{1/e}$ scales as $\varepsilon^{-1}$



## STEP3: EXPLOITING THE TEMPERATURE DEPENDENCE OF DEPLETION EFFECTS TO DRIVE GEL FORMATION

Ref.: S. Buzzaccaro, J. Colombo, A. Parola, and R. Piazza, *Critical Depletion*, Phys. Rev. Lett. **105**, 198301 (2010)

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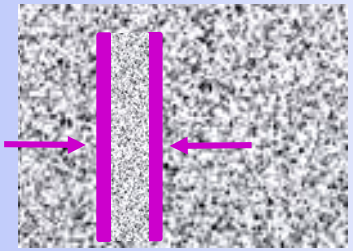

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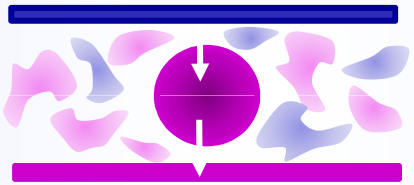

Universal scaling of the force between a colloidal particle immersed in a critical binary mixture and the container walls

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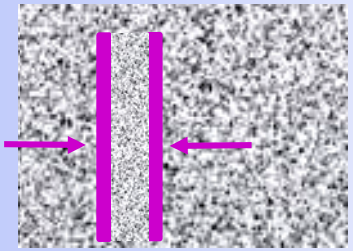

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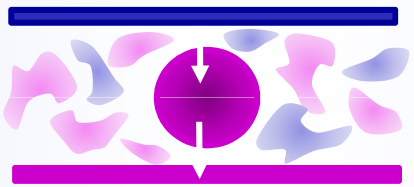

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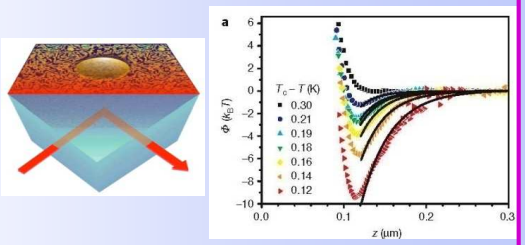


TIRM measurements of forces between a silica plate and a polystyrene sphere dispersed in a critical liquid mixture.

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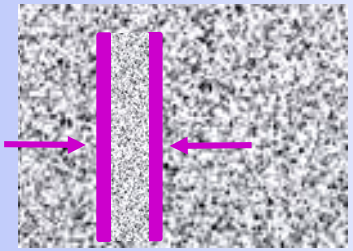

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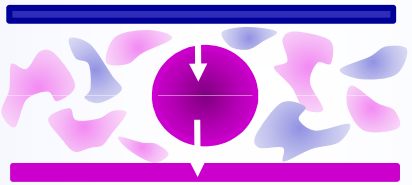

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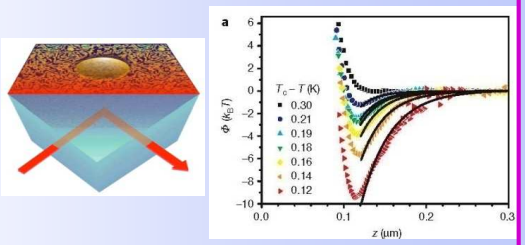
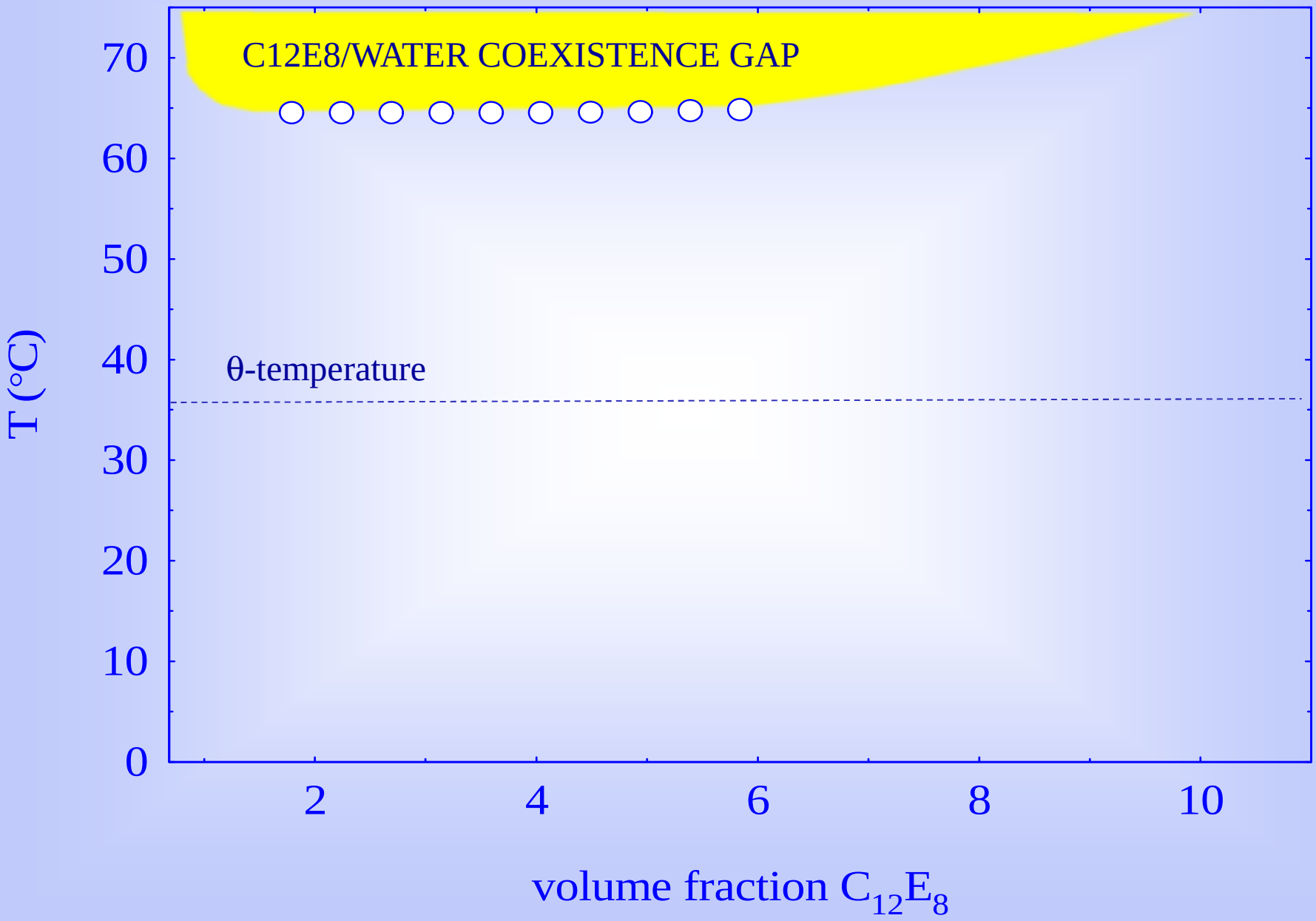



TIRM measurements of forces between a silica plate and a polystyrene sphere dispersed in a critical liquid mixture.

- NONIONIC SURFACTANT/WATER MIXTURES DEMIX AT HIGH T, thus CRITICAL EFFECTS YIELD CORRELATIONS IN THE DEPLETANT

ANY RELATION BETWEEN (CORRELATED) DEPLETION AND THE CRITICAL CASIMIR EFFECT?

# MINIMUM SURFACTANT AMOUNT TO INDUCE PHASE SEPARATION vs. T



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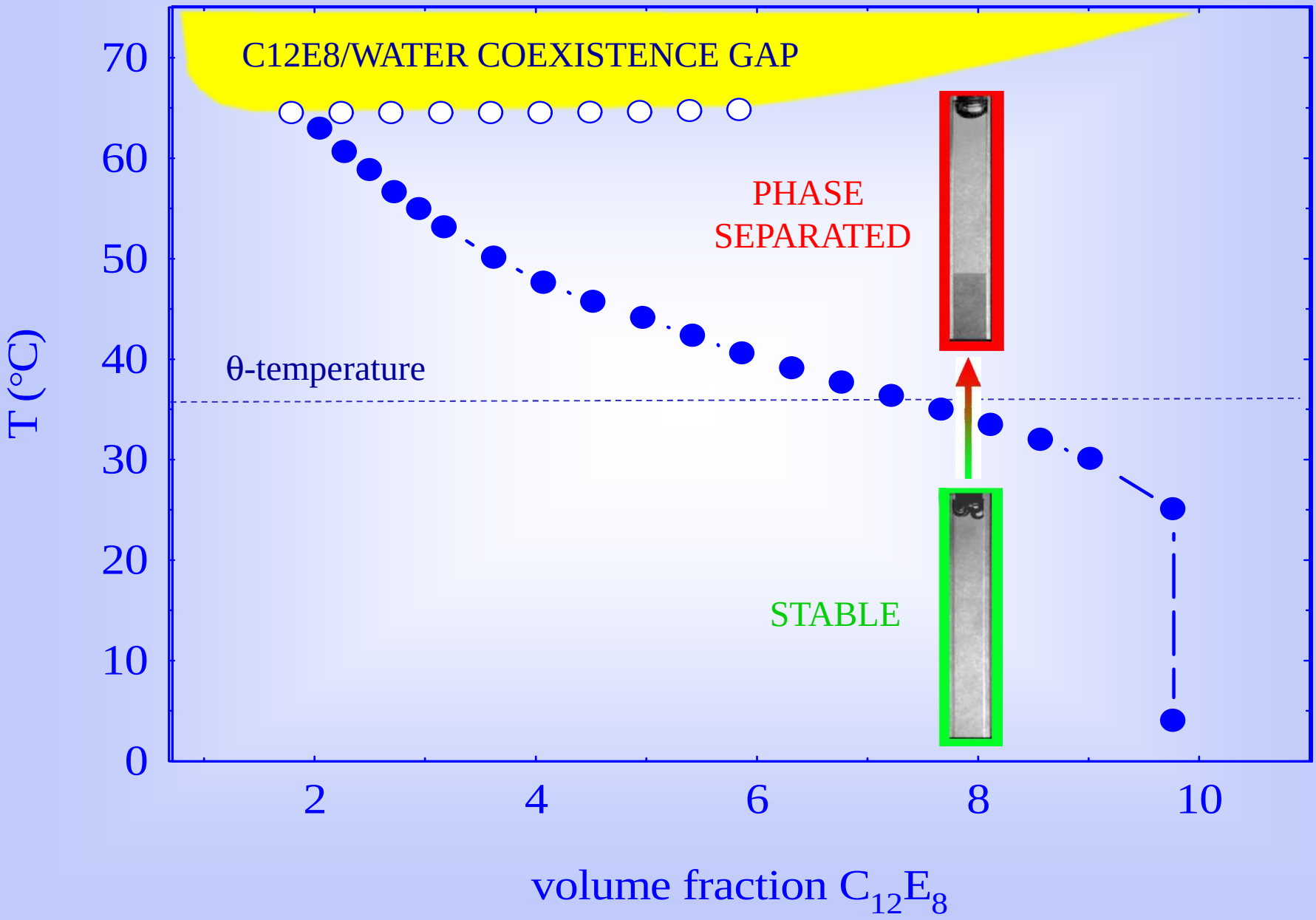

# DFT RESULTS: CONCEPTUAL

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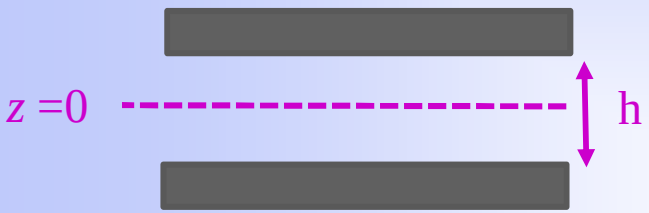
1) Force is still a pressure unbalance (as in simple depletion)




$$F(h) = p_n(z=0) - p_{bulk}$$

Force /surface at separation h

Normal pressure at midpoint

Bulk pressure

$$p_{bulk} = n_0 \frac{\partial f(n_0)}{\partial n_0} - f(n_0)$$

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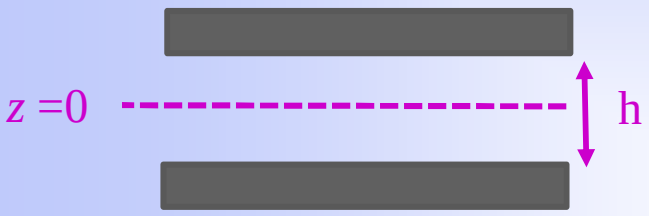


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Bulk pressure $p_{bulk} = n_0 \frac{\partial f(n_0)}{\partial n_0} - f(n_0)$

2) Continuity between depletion interactions an critical Casimir effect rigorously assessed.

- Far from the critical point ⟶ Asakura-Oosawa potential
- Approaching $T_c$ ⟶ Scaling form for the force:

$$F(h) = \frac{k_B T}{h^3} \vartheta(x; y^{1/\nu}) \qquad \begin{cases} x \propto \delta n \varepsilon^{\beta} \\ y \propto h \varepsilon^{\nu} \end{cases}$$

(exactly as in Dietrich model)

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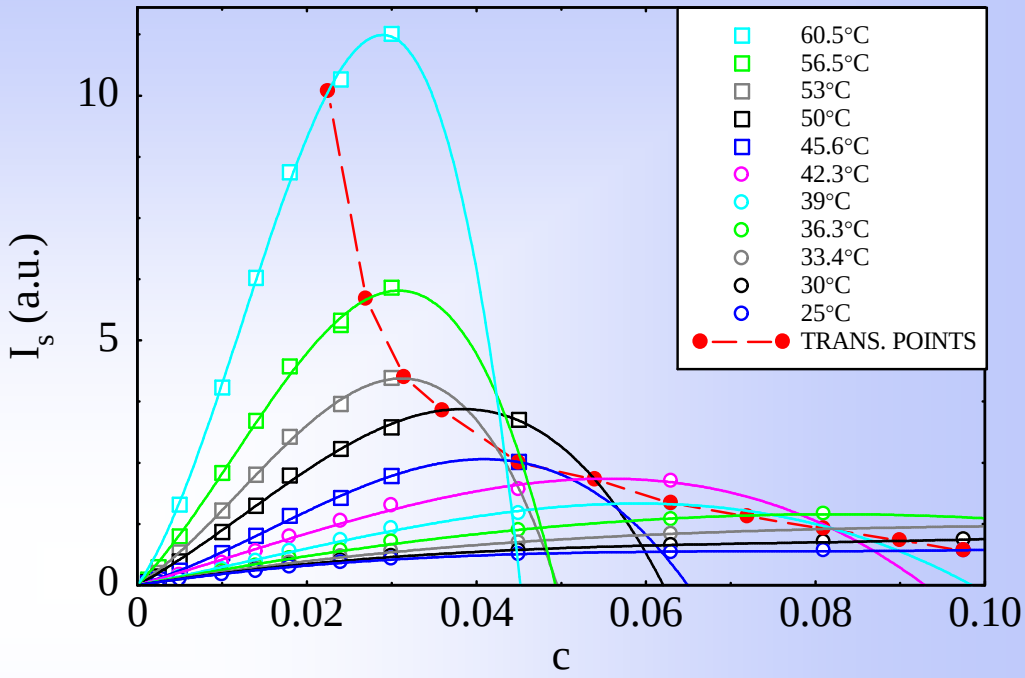

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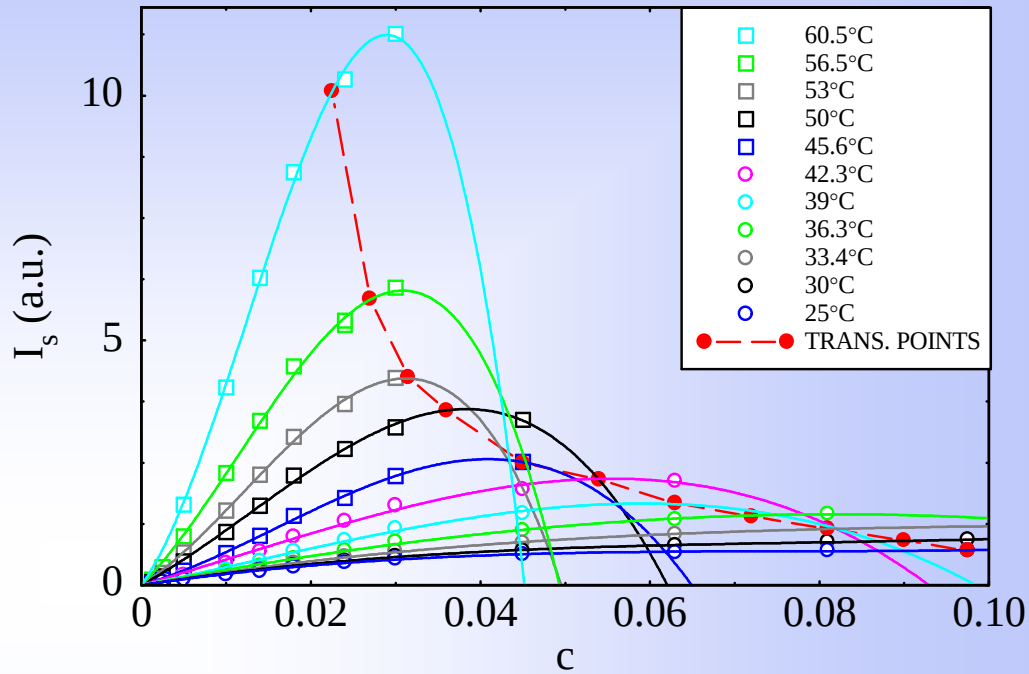


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$$\frac{\Delta p_{sing}\xi^3}{k_B T} \approx 0.1$$

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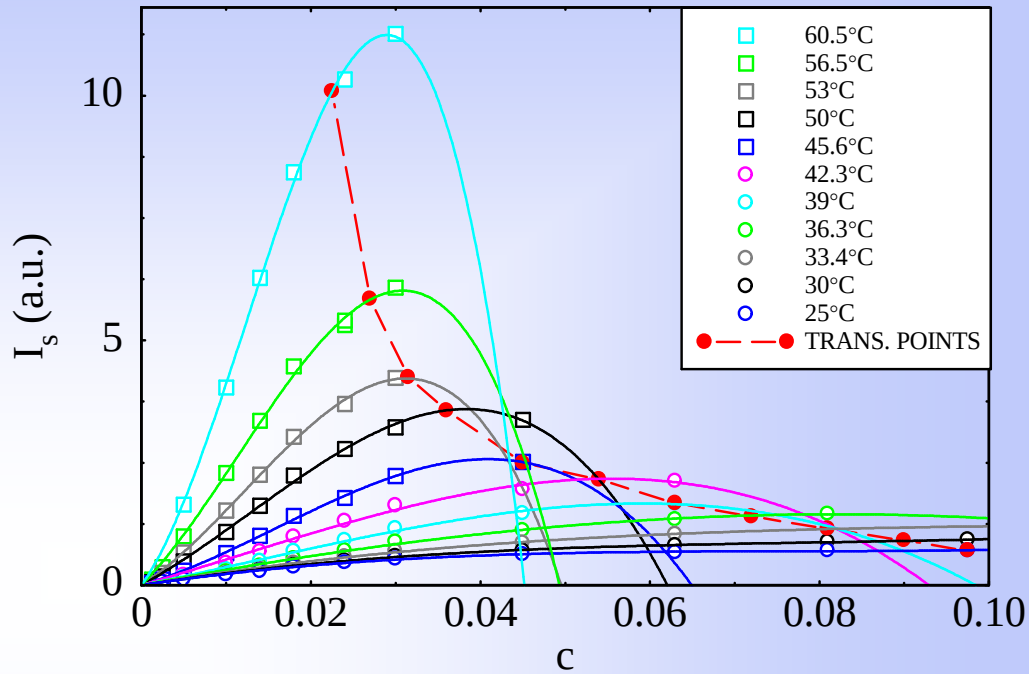


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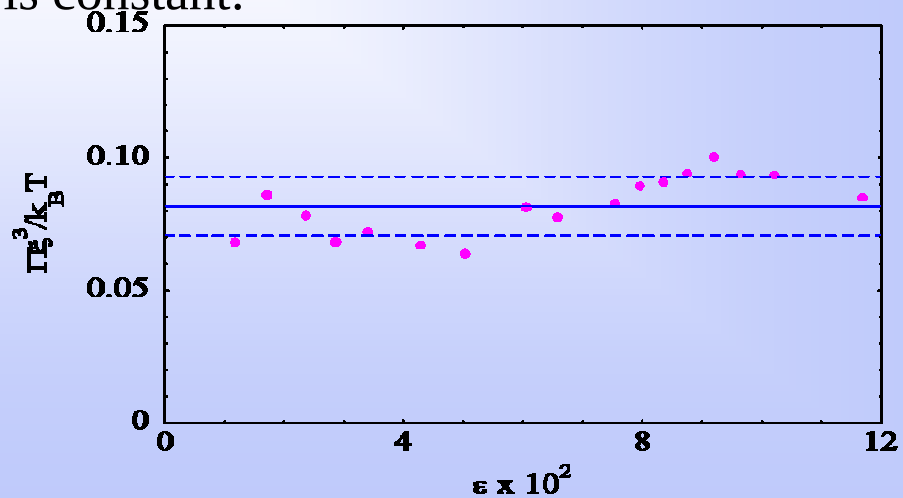

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- The microscope setup within ACE may allow for important and complementary studies.

# D) SPECIFIC EXPERIMENTS AND REQUIREMENTS

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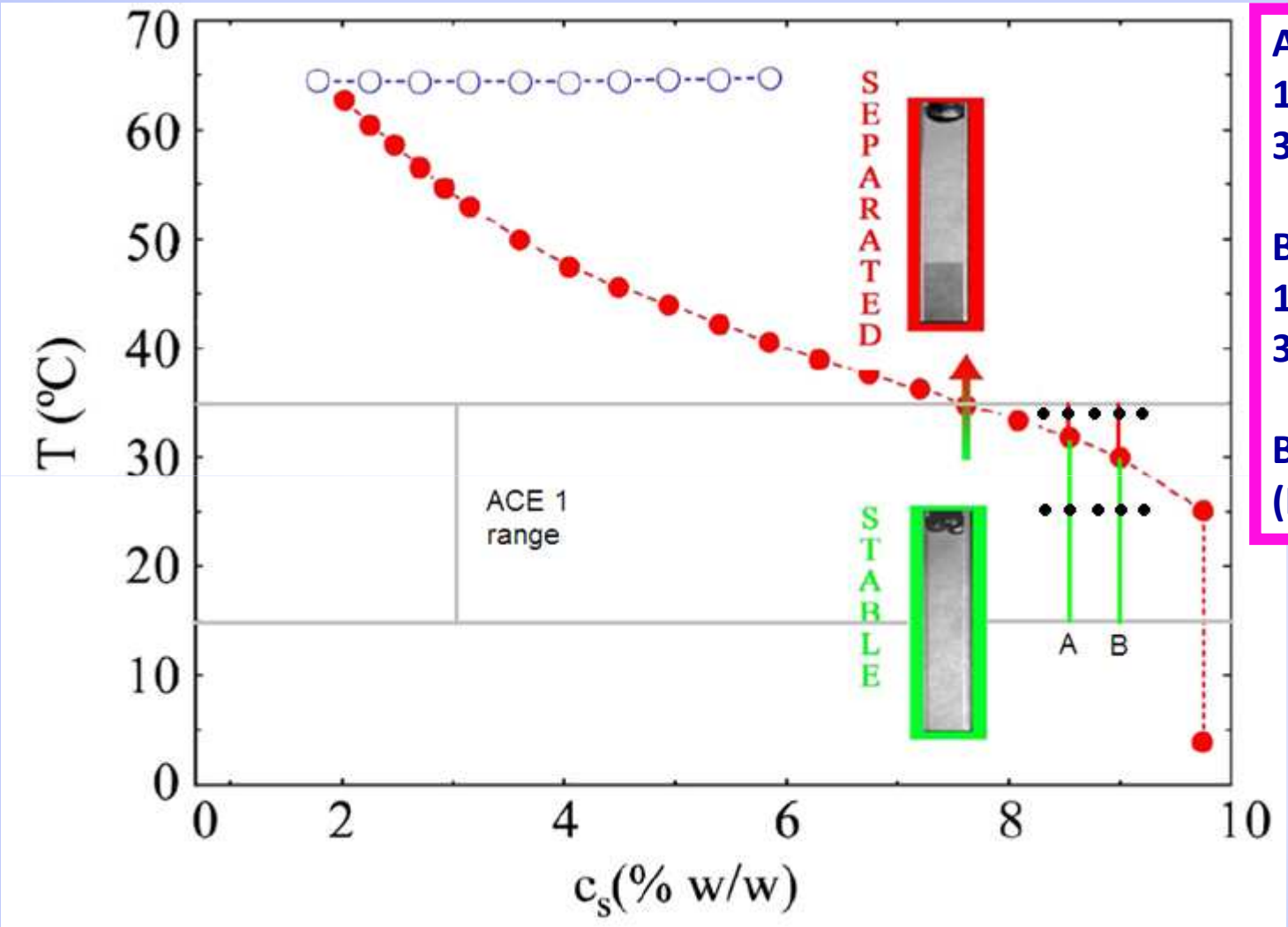


**A samples**
**15-32°C stable**
**32°-35°C gel**

**B samples**
**15°-30°C stable**
**30-35° gel**

**Black dots**
**(ISS experiment)**

## Samples

| | $\Phi$ $C_{12}E_8$ | $\Phi$ MFA | PRIORITY |
|---|---|---|---|
| B1 | 9% | 1% | (6) |
| B2 | 9% | 5% | (5) |
| B3 | 9% | 10% | (9) |
| B4 | 9% | 25% | (1) |
| B5 | 9% | 35% | (8) |
| A1 | 8.5% | 1% | (10) |
| A2 | 8.5% | 25% | (2) |
| C1 | 8.75% | 25% | (3) |
| D1 | 8.25% | 25% | (4) |
| E1 | 9.25% | 25% | (7) |

# REQUIREMENTS

| | |
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| • Samples (compositions) | • MFA particles (perfluoromethoxy resin obtained by copolymerization of tetrafluoroethylene and perfluoromethylvinylether)<br>• Water (solvent)<br>• Depletion Agent: Triton100 (surfactant) or $C_XE_Y$ (ethylene glycol ether non ionic surfactants)<br>• NaCl salt<br>• Urea |
| • Particle size | • Diameters between 0.160 and 0.180 µm |
| • Index of refraction | • Varies between 1.33 and 1.36 (most of the case 1.356) |
| • Volume fractions | • Between 0.05 and 0.60 |
| • Volume fraction verification | • The volume fraction shall be measured after the cells are filled to within ±0.003 |
| • Mixing | • Each sample should have to be mix before the use (not mandatory: continuous stirring after the launch). The stir bar or particles should be capable of being "parked" out of view or at the side when not being used. No mixing "cross-talk" between adjacent cells. On earth sample have to be mixed at least every 7 days (not mandatory: continuous stirring) |
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**Triton X100 or $C_{12}E_8$ depends on temperature control range**

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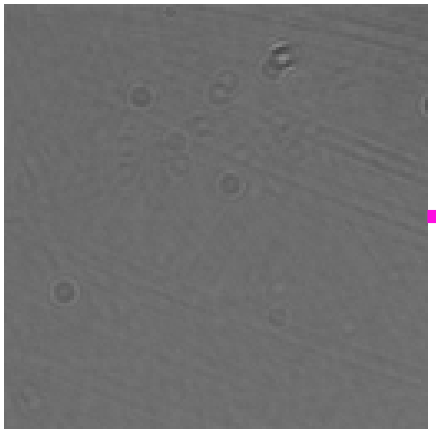
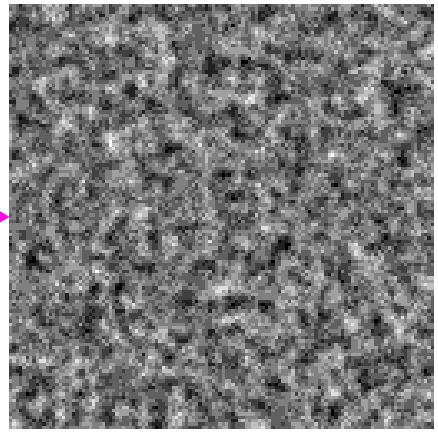
| • Volume fractions | • Between 0.05 and 0.60 → **0.35 sufficient!** |
| • Volume fraction verification | • The volume fraction shall be measured after the cells are filled to within ±0.003 |
| • Mixing | • Each sample should have to be mix before the use (not mandatory: continuous stirring after the launch). The stir bar or particles should be capable of being "parked" out of view or at the side when not being used. No mixing "cross-talk" between adjacent cells. On earth sample have to be mixed at least every 7 days (not mandatory: continuous stirring) |
| | |
| **Homogenization** | • Be able to homogenize samples one at a time.<br>• Simultaneous homogenization and video microscopy (via epi-illumination) to view the sample. |

| | |
|---|---|
| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
| • Dark field | • Desirable |
| • Imaging (camera) | • Need capability to periodically downlink data.<br>• B&W camera.<br>• 8-bit dynamic range is minimum requirement, at least 12-bit dynamic range strongly preferred.<br>• Resolution possible: at maximum field of view 1024 x 1024 min (signal to noise goes as the square root of the number of pixels).<br>• Region Of Interest selectable (512x512, 256x256, 128x128)<br>• Frame Rate: 30 Hz (highly desirable at least100 Hz at resolution of 128x128) for first 5 min after mixing, then lower and variable (usually 1 image/h for the last part of experiment)<br>• Selectable exposure time with a precision of 0.001 ms. (NOTE If is not possible to have an high frame rate, is it possible to infer sample dynamics by speckle visibility at different exposure time) |

**Signal is "noise" added to a static background.**
**Resolution cannot be controlled by exposure**





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|---|---|
| • Visual camera images | • High magnification, high-resolution visual images<br>d (to 0.001)<br>V (at least 12 bit<br><br>lesid rable 100 Hz at<br>e first 5 minutes<br><br>me downlink of<br>lignment<br>possible |
| | nochromatic light<br>ated ) |
| | ly downlink data.<br>• B&W camera.<br>8-bit dynamic range is minimum requirement, at lea 12-bit dynamic range strongly preferred.<br>• Resolution possible: at maximum field of view 1024 x 1024 min (signal to noise goes as the square root of the number of pixels).<br>• Region Of Interest selectable (512x512, 256x256, 128x128)<br>• Frame Rate: 30 Hz (highly desirable at least100 Hz at resolution of 128x128) for first 5 min after mixing, then lower and variable (usually 1 image/h for the last part of experiment)<br>• Selectable exposure time with a precision of 0.001 ms. (NOTE If is not possible to have an high frame rate, is it possible to infer sample dynamics by speckle visibility at different exposure time) |

| | |
|---|---|
| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
| • Dark field | • Desirable |
| • Imaging (camera) | • Need capability to periodically downlink data.<br>• B&W camera.<br>• 8-bit dynamic range is minimum requirement, at least 12-bit dynamic range strongly preferred.<br>• Resolution possible: at maximum field of view 1024 x 1024 min (signal to noise goes as the square root of the number of pixels).<br>• Region Of Interest selectable (512x512, 256x256, 128x128)<br>• Frame Rate: 30 Hz (highly desirable at least100 Hz at resolution of 128x128) for first 5 min after mixing, then lower and variable (usually 1 image/h for the last part of experiment)<br>• Selectable exposure time with a precision of 0.001 ms. (NOTE If is not possible to have an high frame rate, is it possible to infer sample dynamics by speckle visibility at different exposure time) |

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| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
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**Allows us to perform DLS during first stage of spinodal decomposition**

| | |
|---|---|
| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
| • Dark field | • Desirable |
| • Imaging (camera) | • Need capability to periodically downlink data.<br>• B&W camera.<br>• 8-bit dynamic range is minimum requirement, at least 12-bit dynamic range strongly preferred.<br>• Resolution possible: at maximum field of view 1024 x 1024 min (signal to noise goes as the square root of the number of pixels).<br>• Region Of Interest selectable (512x512, 256x256, 128x128)<br>• Frame Rate: 30 Hz (highly desirable at least100 Hz at resolution of 128x128) for first 5 min after mixing, then lower and variable (usually 1 image/h for the last part of experiment)<br>• Selectable exposure time with a precision of 0.001 ms. (NOTE If is not possible to have an high frame rate, is it possible to infer sample dynamics by speckle visibility at different exposure time) |

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|---|---|
| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
| • Dark field | • Desirable |
| • Imaging (camera) | • Need capability to periodically downlink data.<br>• B&W camera.<br>• 8-bit dynamic range is minimum requirement, at least 12-bit dynamic range strongly preferred.<br>• Resolution possible: at maximum field of view 1024 x 1024 min (signal to noise goes as the square root of the number of pixels).<br>• Region Of Interest selectable (512x512, 256x256, 128x128)<br>• Frame Rate: 30 Hz (highly desirable at least100 Hz at resolution of 128x128) for first 5 min after mixing, then lower and variable (usually 1 image/h for the last part of experiment)<br>• Selectable exposure time with a precision of 0.001 ms. (NOTE If is not possible to have an high frame rate, is it possible to infer sample dynamics by speckle visibility at different exposure time) |

**Check if applicable to spinodal decomposition dynamics**

| | |
|---|---|
| • Visual camera images | • High magnification, high-resolution visual images of both highly index matched (to 0.001)<br>• Dynamic range of 8 bits B/W (at least 12 bit desiderable)<br>• Video recorded<br>• Video rates: at least 30 Hz (desiderable 100 Hz at resolution of 128x128) for the first 5 minutes<br>• Capability for some near real time downlink of video images to assist with alignment<br>• Trasmission configuration if possible |
| • DIC and phase contrast | • Desirable |
| • Bright field | • White light illumination (monochromatic light using a color filter is appreciated ) |
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| | |
|---|---|
| • Polarizer | • Polarizer filter for illumination and polarizer filter for imaging (axis angle selectable, at least parallel/perpendicular) are strongly desiderated |
| • Objective lenses | • Chosen to allow range of field of views, from about 100 μm to 2 mm.<br>• Recommended choice: 5x (or 10x), 20x, 40x, 60x 100x.<br>• High numerical aperture (60x 0.7 < N.A. < 0.9).<br>• NOTE: Choice of lenses should be done to optimize all requirements, and will depend highly on manufacturer of the microscope. |
| • White light source for imaging | • Imaging of colloidal particles using Köhler illumination for highest resolution.<br>• N.A. variable (at least the possibility of close the condenser to have a "point-like" source). Easier solution is to have a pinhole (diameter <1mm )<br>• May also consider monochromatic source if necessary. |

**Depolarized LS depends only on particles concentration.**

| | |
|---|---|
| | • Polarizer filter for illumination and polarizer filter for imaging (axis angle selectable, at least parallel/perpendicular) are strongly desiderated |
| • Objective lenses | • Chosen to allow range of field of views, from about 100 µm to 2 mm.<br>• Recommended choice: 5x (or 10x), 20x, 40x, 60x 100x.<br>• High numerical aperture (60x 0.7 < N.A. < 0.9).<br>• NOTE: Choice of lenses should be done to optimize all requirements, and will depend highly on manufacturer of the microscope. |
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| • White light source for imaging | • Imaging of colloidal particles using Köhler illumination for highest resolution.<br>• N.A. variable (at least the possibility of close the condenser to have a "point-like" source). Easier solution is to have a pinhole (diameter <1mm )<br>• May also consider monochromatic source if necessary. |

**In trasmission configuration, the control of condenser N.A. allows to change the spatial coherence of the source. LS measurements can be made WITHOUT LASER if not available.**

| | |
|---|---|
| • Polarizer | • Polarizer filter for illumination and polarizer filter for imaging (axis angle selectable, at least parallel/perpendicular) are strongly desiderated |
| • Objective lenses | • Chosen to allow range of field of views, from about 100 μm to 2 mm.<br>• Recommended choice: 5x (or 10x), 20x, 40x, 60x 100x.<br>• High numerical aperture (60x 0.7 < N.A. < 0.9).<br>• NOTE: Choice of lenses should be done to optimize all requirements, and will depend highly on manufacturer of the microscope. |
| • White light source for imaging | • Imaging of colloidal particles using Köhler illumination for highest resolution.<br>• N.A. variable (at least the possibility of close the condenser to have a "point-like" source). Easier solution is to have a pinhole (diameter <1mm )<br>• May also consider monochromatic source if necessary. |

| **Temperature control** | |
|---|---|
| • Large scale | • 20–60°C variable temperature range (20-30° as minimum/ 20-40°C highly desirable ). Temperature controls particles aggregation. Before measurement samples have to be maintained at T<25°C to prevent coagulation. For T>25°C gel forms. Interaction strength are controlled by temperature: wider temperature range implies more "effective" sample with the same cell<br>• Temperature stability 0.05°C during measurement (1/2weeks), 1°C but T< 25°C during storage<br>• simultaneously, be capable of performing high magnification imaging |
| | |
| **Sample Cell Requirements** | |
| Cell | • ACE1 samples |
| Number | • At least 2 for ACE1 |

| **Temperature control** | |
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| • Large scale | • 20–60°C variable temperature range (20-30° as minimum, 20-40°C highly desirable ). Temperature controls particles aggregation. Before measurement samples have to be maintained at T<25°C to prevent coagulation. For T>25°C gel forms. Interaction strength are controlled by temperature: wider temperature range implies more "effective" sample with the same cell<br>• Temperature stability 0.05°C during measurement (1/2weeks), 1°C but T< 25°C during storage<br>• simultaneously, be capable of performing high magnification imaging |
| | |
| **Sample Cell Requirements** | |
| Cell | • ACE1 samples |
| Number | • At least 2 for ACE1 |

# E) PRELIMINARY TEST MATRIX

| Sample no. | Sample type | Day no. | General description of test | Manipulation, diagnostics, and acquisition rate | Test procedure: Sequence and approx. time taken | Acquisition frequency |
|---|---|---|---|---|---|---|
| All | ACE1b Workhorse cell<br><br>0.180 micron MFA spheres in water-surfactant mixtures<br>NaCl 250mM<br>Urea<br><br>volume fraction 0.01-0.35 | 1 | Homogenize samples.<br><br>Record | Each sample is individually stirred and observed with 10x lens to assure homogeneous melting at T=25°C | 1) Each sample is individually stirred (T=25°C) and observed with 10x lens to assure homogeneous mixing.[2min/sample]<br><br>2) For each sample 1000 photos at maximum frame rate (at least 30Hz) with 50x lens. Laser illumination if present or with low N.A. condenser in transmission configuration [30 sec ca/sample]<br><br>3)For each sample 400 photos at 1 frame/s with 50x lens. Laser illumination if present or with low N.A. condenser in transmission configuration [ 7 min ca/sample ]<br><br>Total time 60 minutes (for 8 samples) ~ 1 hour | once<br><br>1000 photos at maximum frame rate (at least 30Hz)<br><br>400 photos at 1 frame/s |

| Sample no. | Sample type | Day no. | General description of test | Manipulation, diagnostics, and acquisition rate | Test procedure: Sequence and approx. time taken | Acquisition frequency |
|---|---|---|---|---|---|---|
| All | ACE1b Workhorse cell<br><br>0.180 micron MFA spheres in water-surfactant mixtures<br>NaCl 250mM<br>Urea<br><br>volume fraction 0.01-0.35 | 1 | Record<br><br>Record | Temperature is raised at 34°C (all samples) at maximum heating rate. Temperature of 34°C ± 0.5°C will be maintained for 7 days<br>. | 1) Temperature is raised at 34°C (all samples) at maximum heating rate.<br><br>1) During heating sample B4 is imaged with 50x lens. Photos at maximum 1 frame/s rate until final temperature will be reached. Laser illumination if present or with low N.A. condenser in transmission configuration. [heating time 5 min ca]<br><br>2)At 34°C sample B4 is imaged with 50X lens. 1000 photos at maximum frame rate [at least 30Hz]. Laser illumination if present or with low N.A. condenser in transmission configuration. [40 sec ca]<br><br>Total time 6 minutes ca | once<br><br>1 frame/s<br><br>1000 images at maximum frame rate |

| Sample no. | Sample type | Day no. | General description of test | Manipulation, diagnostics, and acquisition rate | Test procedure: Sequence and approx. time taken | Acquisition frequency |
|---|---|---|---|---|---|---|
| All | ACE1b Workhorse cell<br><br>0.180 micron MFA spheres in water-surfactant mixtures<br>NaCl 250mM<br>Urea<br><br>volume fraction 0.01-0.35 | 1 | Record<br><br>Record | Temperature of 34°C ± 0.5°C will be maintained for 7 days.<br>. | 1) Each samples is imaged with with 50x lens and 10x lens. 600 photos for each lens and samples at frame = 1 photo/min. Laser illumination if present or with low N.A. condenser in transmission configuration. [600 min ca] | 1 frame/min<br>600 frames each samples and lens |
| | | | | | 2) Each samples is imaged with with 50x lens and 10x lens. 600 photos for each lens and samples at frame = 1 photo/5min. Laser illumination if present or with low N.A. condenser in transmission configuration. [3000 min ca] | 1 frame/5 min<br>600 frames each samples and lens |
| | | | | | 3) Each samples is imaged with with 50x lens and 10x lens. 240 photos for each lens and samples at frame = 1 photo/30min. Laser illumination if present or with low N.A. condenser in transmission configuration. [7200 min ca] | 1 frame/30 min<br>2400 frames each samples and lens |
| | | | | | Total time 10800 min ca - 7.5 days ca | |

| Sample no. | Sample type | Day no. | General description of test | Manipulation, diagnostics, and acquisition rate | Test procedure: Sequence and approx. time taken | Acquisition frequency |
|---|---|---|---|---|---|---|
| B4 | ACE1b Workhorse cell<br><br>0.180 micron MFA spheres in water-surfactant mixtures NaCl 250mM Urea | 1 | Record<br><br>Record | Temperature is lowered at 25°C ± 0.5°C at maximum cooling rate. | 1) During cooling Sample B4 is imaged with 50x lens. Photos at 1frame/s until final T is reached. Laser illumination if present or with low N.A. condenser in transmission configuration. [supposed 5 min] | 1 frame/sec Until final T is reached |
| | volume fraction 0.01-0.35 | | | | 2) Sample B4 is imaged with 50x lens. Photos at 5 frame/s until clear remix is reached (test for sufficient remix will be defined). [supposed 5 min] | 5frames/1 min |
| | | | Homogenize samples. | Sample B4 is stirred and observed with 10x lens to assure homogeneous melting. | 3) Sample B4 is stirred and observed with 10x lens to assure homogeneous melting.[2min] | once |
| | | | | | Total time 12 min ca | |

| Sample no. | Sample type | Day no. | General description of test | Manipulation, diagnostics, and acquisition rate | Test procedure: Sequence and approx. time taken | Acquisition frequency |
|---|---|---|---|---|---|---|
| B4 | ACE1b Workhorse cell<br><br>0.180 micron MFA spheres in water-surfactant mixtures NaCl 250mM Urea<br><br>volume fraction 0.01-0.35 | 1 | Record | Temperature is raised at 34°C ± 0.5°C with well defined cooling rate [depends on hardware] and maintained for 30 min ca | 1) Temperature is raised at 34°C with the defined cooling rate. | once |
| | | | | | 2) During heating sample B4 is imaged with 50x lens. Photos at 5 frame/s until final temperature will be reached [heating time5 min] | 1 frame/sec Until final T is reached |
| | | | | | 3) At 34°C Sample B4 is imaged with 50X lens. 1000 photos at maximum frame rate (at least 30Hz) [40 sec ca] | 1000 images at maximum frame rate |
| | | | | | 4) Sample B4 is imaged with 50xlens. 1000 photos at 1 frame/s. [1000 s] | 1000 images at 1 frame/s |
| | | | | | 5)Sample B4 is imaged with 50x lens. 120 photos at 1 frame/min [120 min] | 120 images at 1 frame/min |
| | | | | Temperature is lowered at 25°C ± 0.5°C. | 1) Temperature is raised at 34°C with the defined cooling rate. | Once |
| | | | | Sample B4 is stirred and observed with 10x lens to assure homogeneous melting | 1)Sample B4 is stirred and observed with 10x lens to assure homogeneous melting [2min] | once |
| | | | | | Total time 140 min ca | |

**N.B. This procedure will be repeated at different heating rates**

| | |
|---|---|
| **Atmosphere Requirements** | • Normal cabin pressures and temperatures environment is sufficient. |
| **Vibration Control and Measurement** | • Averaged over an hour, needs no better than $10^{-3}$ g of dc g-level. During measurement image misaligned less than 1/10 of field of view |
| **Imaging requirements (Image viewability requirements)** | • Near real time Downlinked CCD images of the samples are needed just before and after mixing |
| **Astronaut Involvement** | • minimal– depends on the extent of hardware automation. |
| **Data (Storage) Requirements** | • Images should be stored with a record of when the time measurement was made, exposition time, temperature<br>• Temperature is to be recorded whenever the rack power is on, and with each image data;<br>• Accelerations in excess of $10^{-3}$ g should be recorded and time-tagged for comparison with data from the experiment.<br>• Collected data should be time-tagged to estimate precisely the real frame rate.<br>• Need capability to periodically downlink any of the above data. |