



Characterization of different milk protein aggregates by Asymmetrical Flow Field-Flow Fractionation coupled with Multiangle Laser Light Scattering (A4F-MALLS)

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Characterization of different milk protein aggregates by Asymmetrical Flow Field-Flow Fractionation coupled with Multiangle Laser Light Scattering (A4F-MALLS)

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Journée Scientifique

du Groupe Francophone de Fractionnement Flux-Force

31 mars 2017



Contexte



Natural product
please!!!



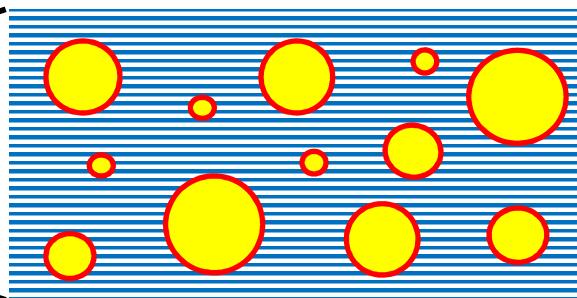
Milk Valley



11 Industriels



6 Scientifiques



Emulsion H/E

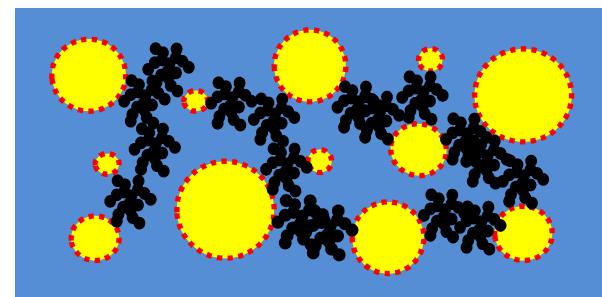
Gélification de la
phase **continue**

Objectifs

- ✓ Utiliser les **gouttes de matière grasse (MG)** comme des nœuds pour texturer le produit sans utiliser d'agents de texture



- ✓ Utiliser des **agrégats protéiques (Ag)** pour stabiliser l'interface et connecter les gouttes de MG



Objectifs

Éliminer les texturants de type polysaccharides

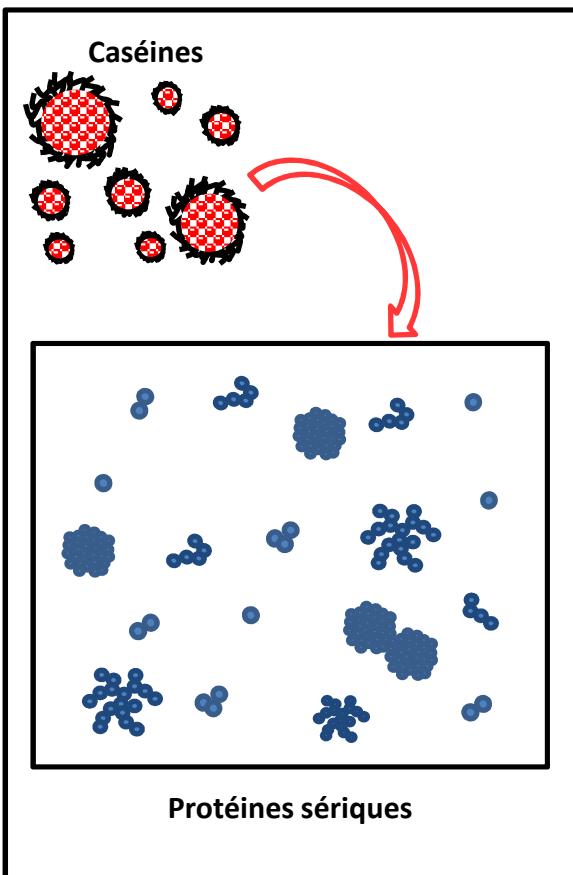
Texturer des produits à faibles taux de MG



LOW-FAT

Problématiques techniques

Agrégats de protéines laitières



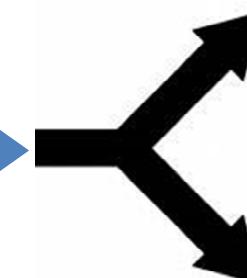
DLS

- ✓ Nous voyons uniquement les grosses particules
- ✓ Pas de différenciation entre les grosses particules

Mélange monomères/agrégats

Systèmes polydispers

Différente taille/forme/densité



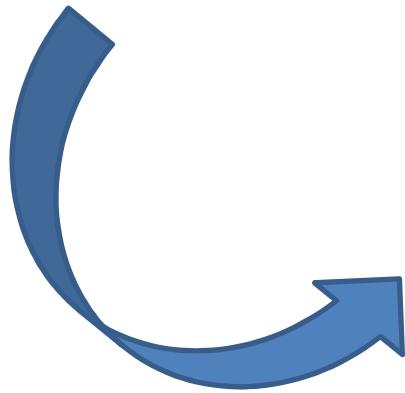
SEC

- ✓ Les caséines interagissent avec la colonne
- ✓ Nous voyons les monomères alors que les agrégats sortent dans le volume mort ou restent dans la colonne

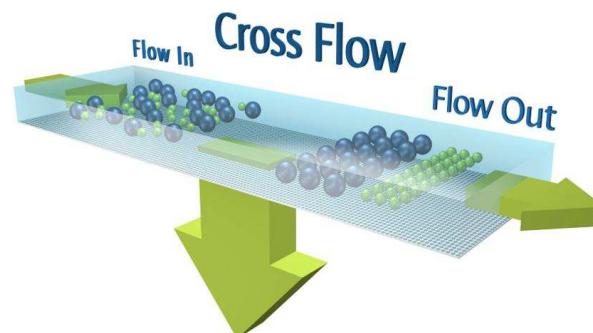
Problématiques techniques



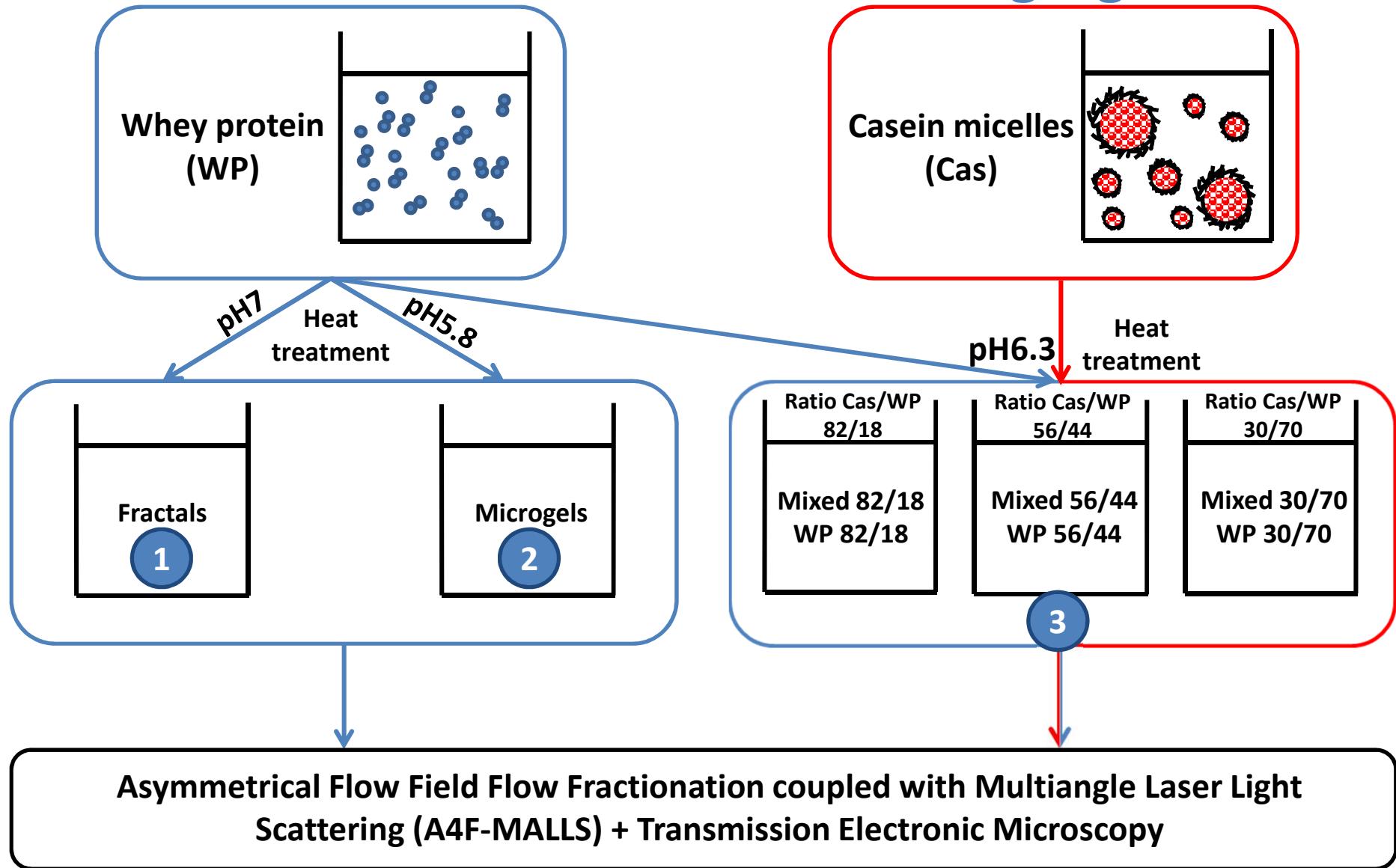
Trouver une technique séparative capable de fractionner les différentes entités y compris les assemblages avec des caséines (contrairement à la SEC)



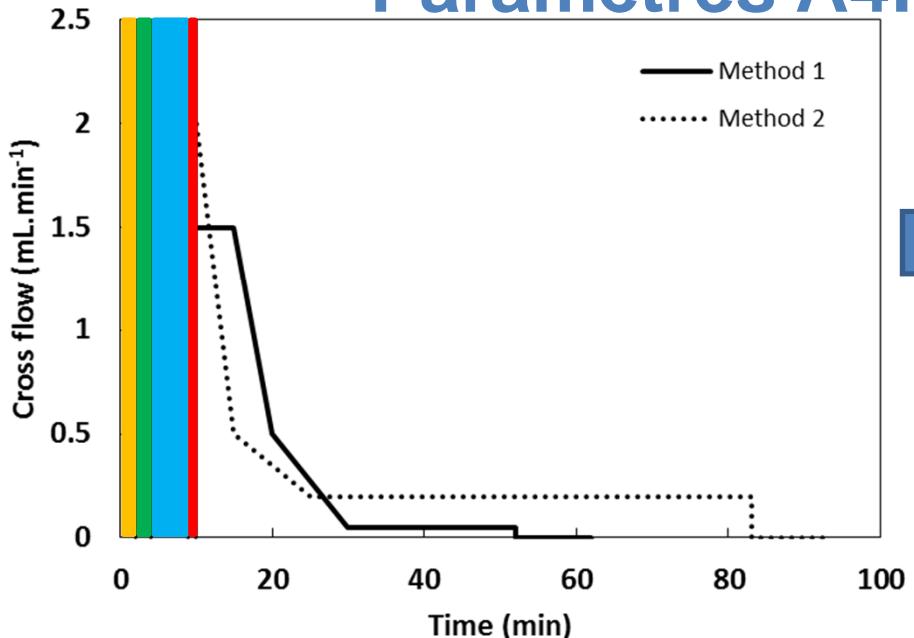
Asymmetrical Flow Field Flow Fractionation



Méthodes de fabrication des agrégats



Paramètres A4F et MET



Detectors

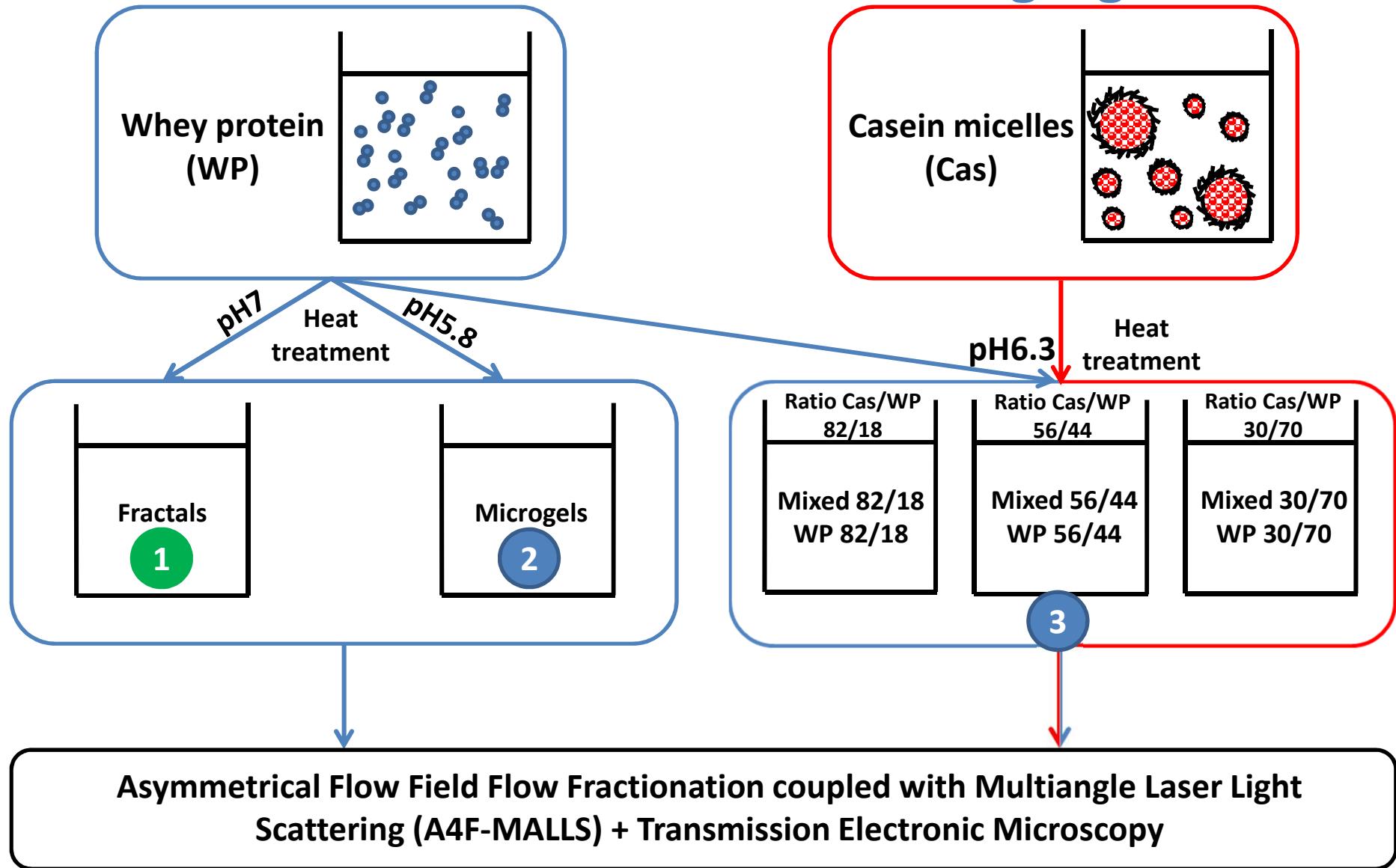
- ✓ MALLS
- ✓ UV Detector
- ✓ Differential Refractometer (DRI)

Elution : 2 min
Focus : 2 min
Focus + inject : 5 min
Relaxation : 1 min

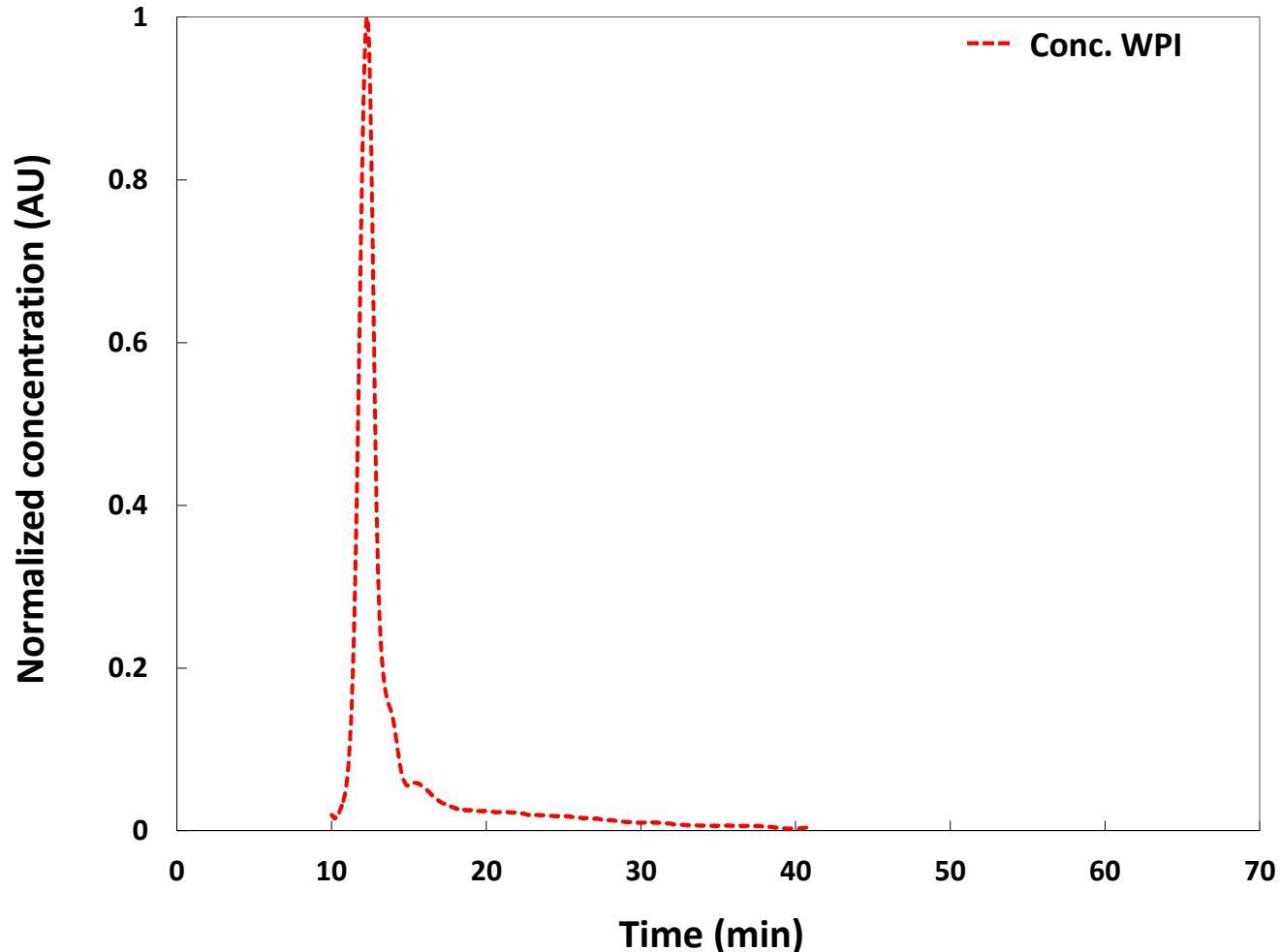
	Fractal aggregates	Microgel aggregates	Mixed aggregates
Eluent (pH)	Millipore water containing 0.02 w/v% NaN ₃ and 45 mM NaCl (pH 7)	Millipore water containing 0.02 w/v% NaN ₃ and 45 mM NaCl (pH 7)	Millipore water containing 0.02 w/v% NaN ₃ , 45 mM NaCl and 10mM CaCl ₂ (pH 6.3 or 7)
A4F method	2	1	2
Membrane	Regenerated cellulose		
Spacer (μm)	350		
Temperature (°C)	25		

MET: Coloration négative

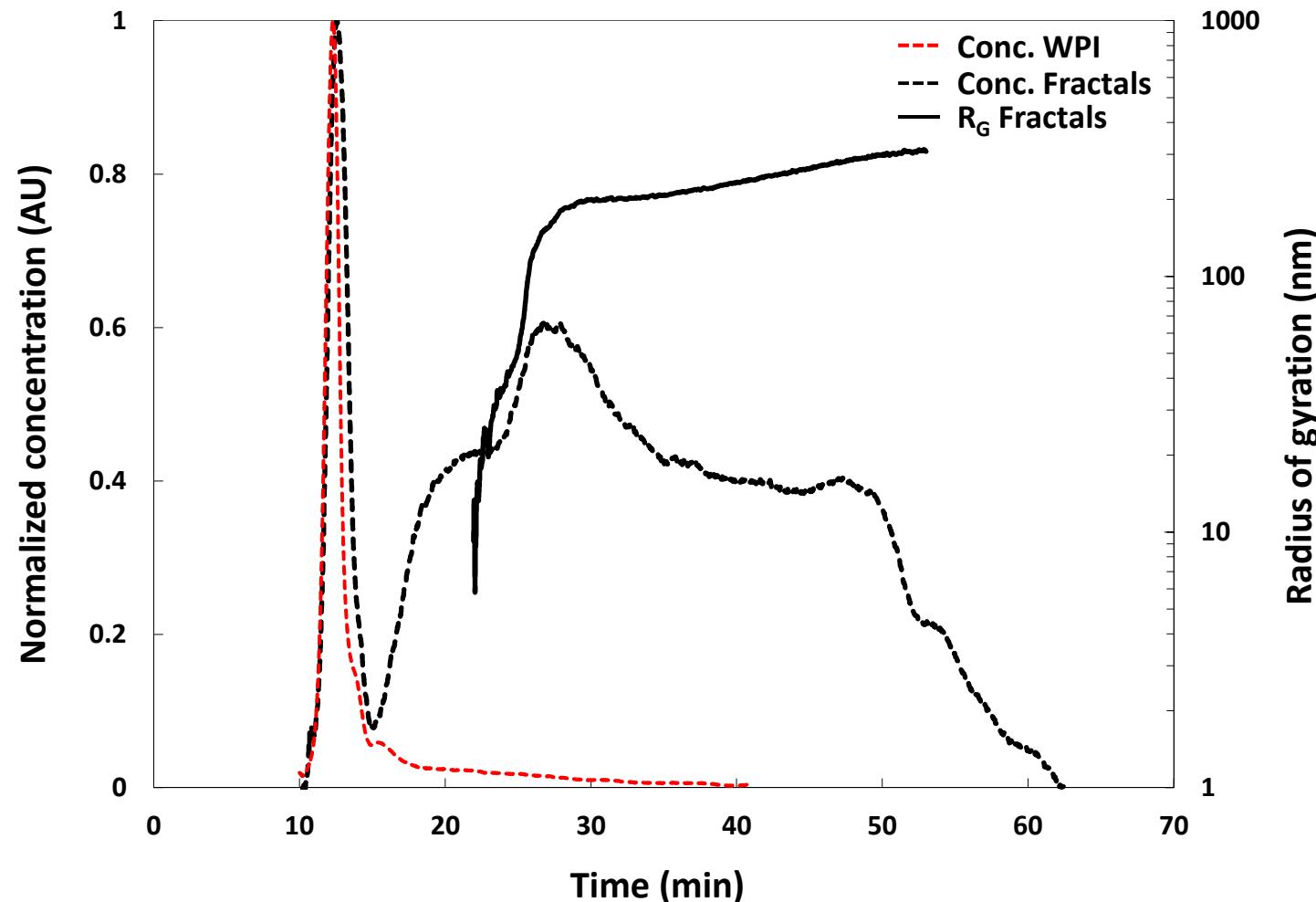
Méthodes de fabrication des agrégats



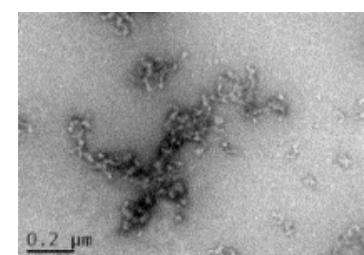
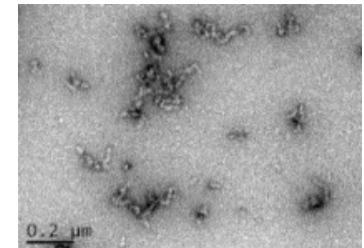
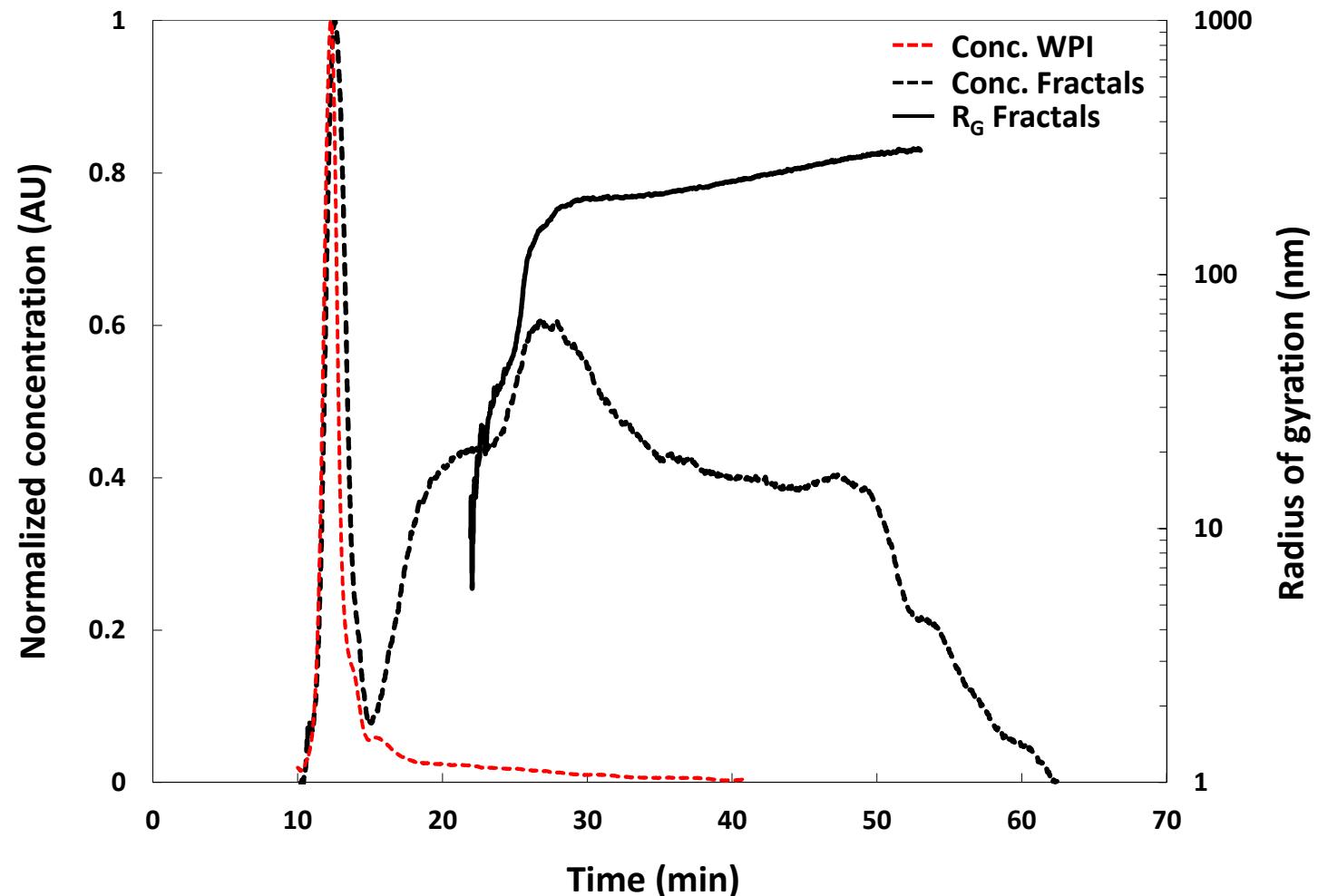
Fractals



Fractals



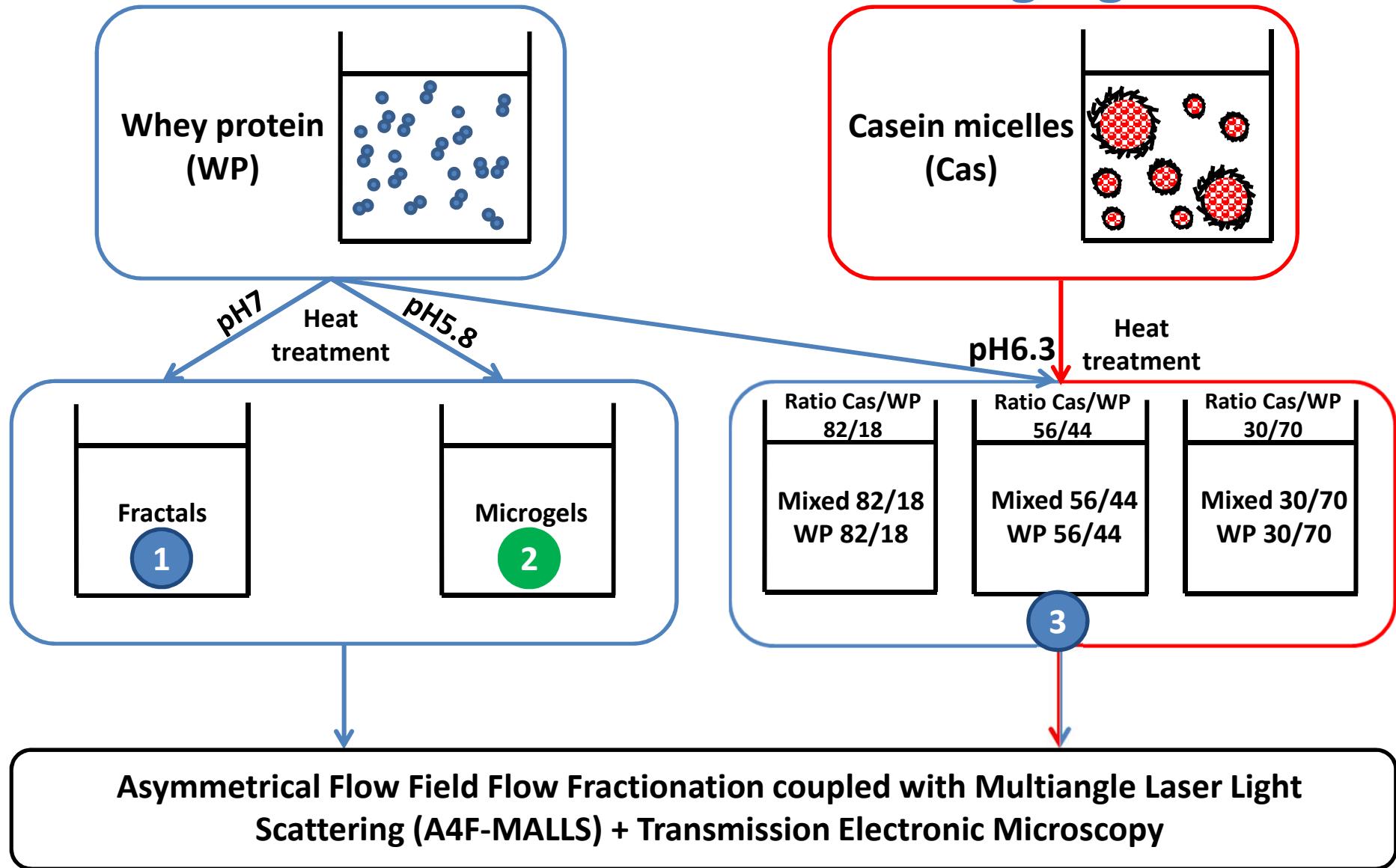
Fractals



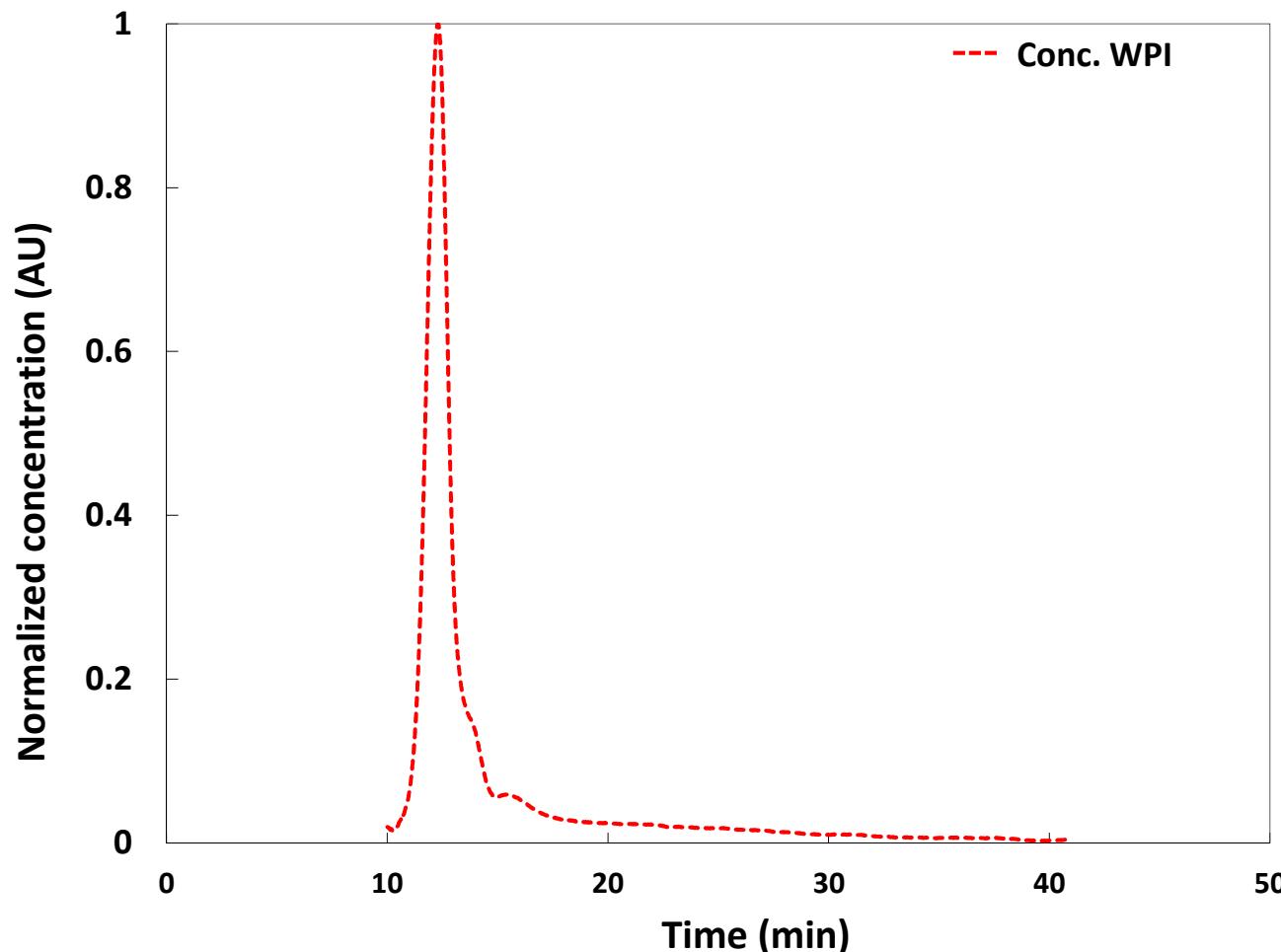
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Df : 2.3

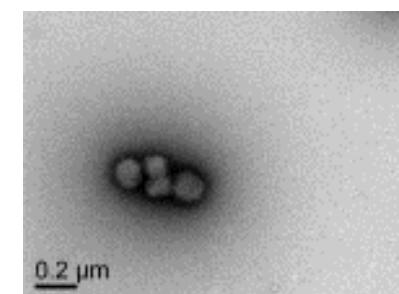
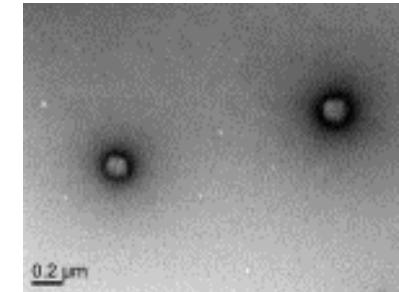
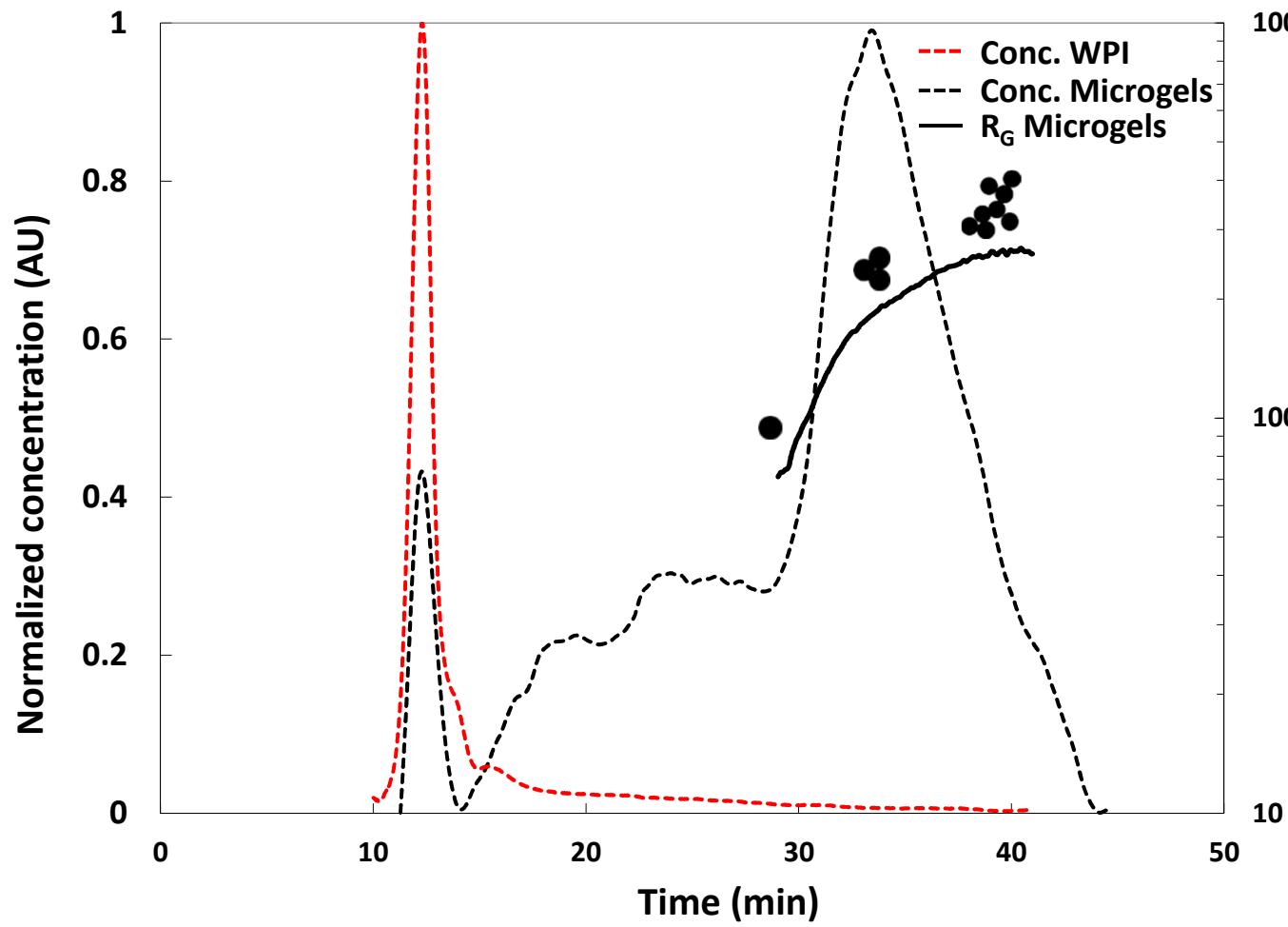
Méthodes de fabrication des agrégats



Microgels



Microgels

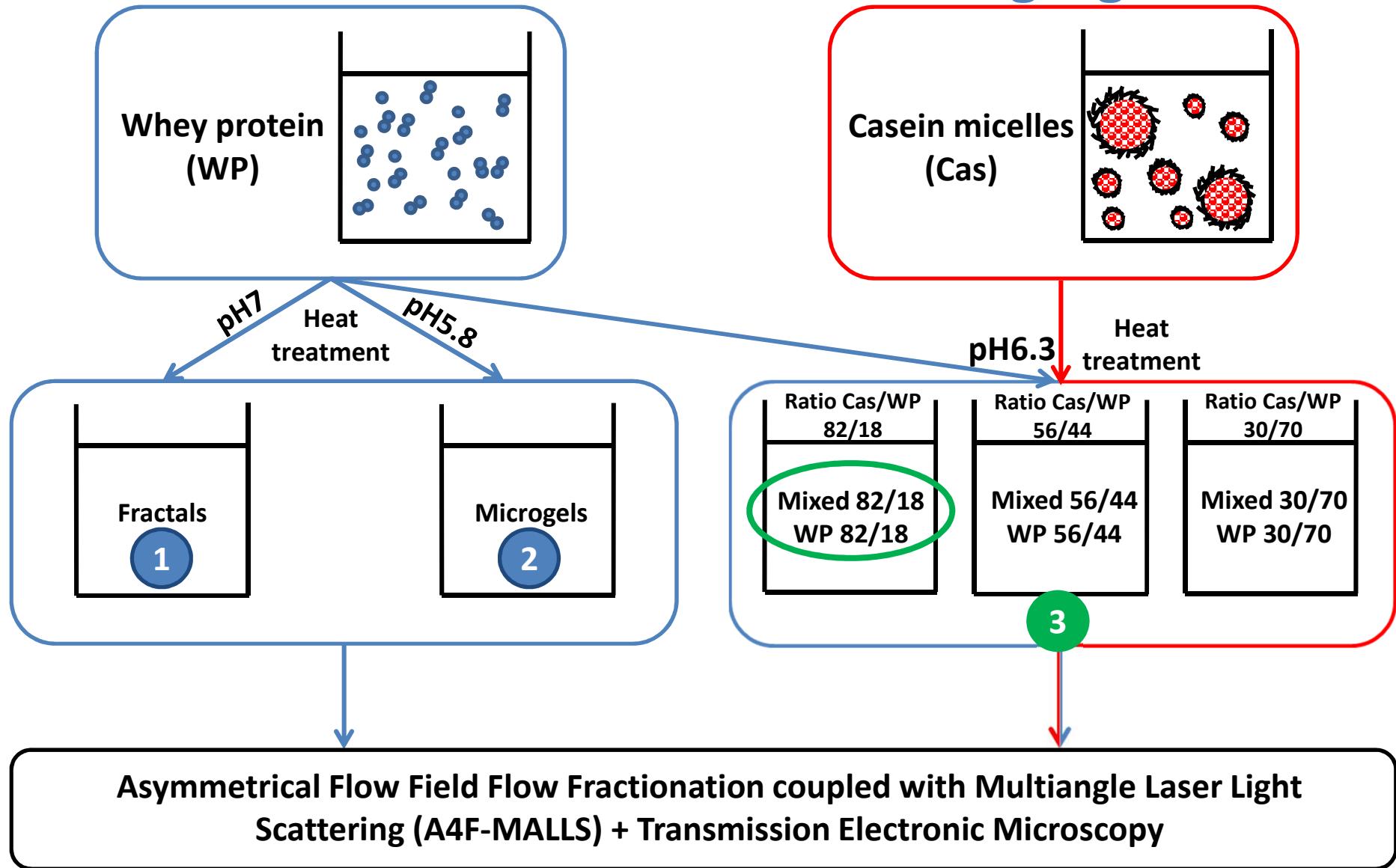


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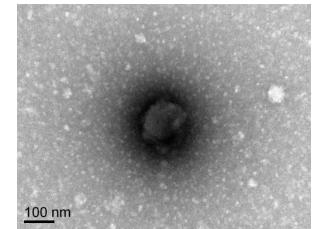
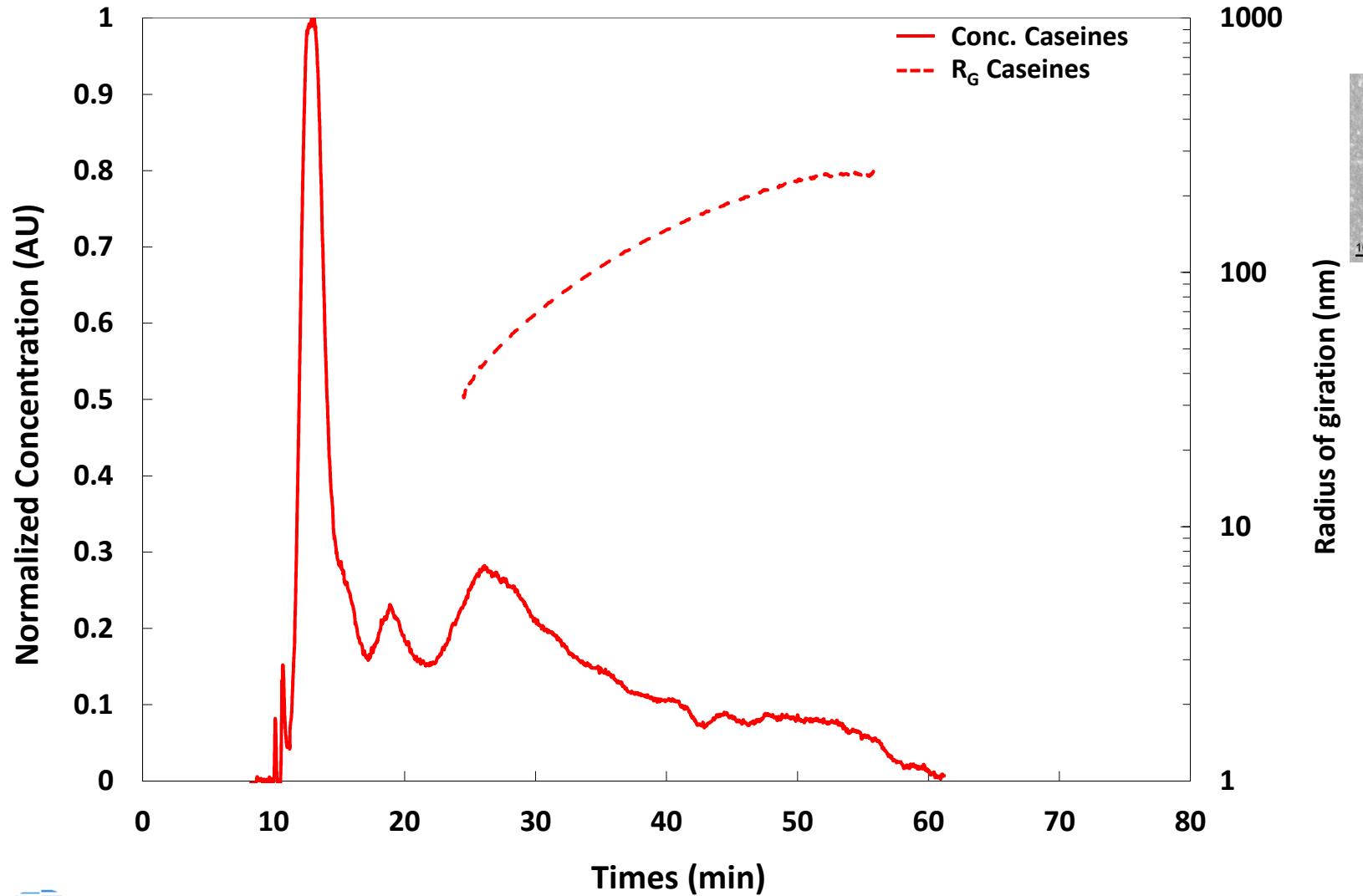
$$R_G = 100 \text{ nm}$$

- $M_W = 8.4 \cdot 10^7 \text{ g.mol}^{-1}$
- $M_W = 5.5 \cdot 10^8 \text{ g.mol}^{-1}$

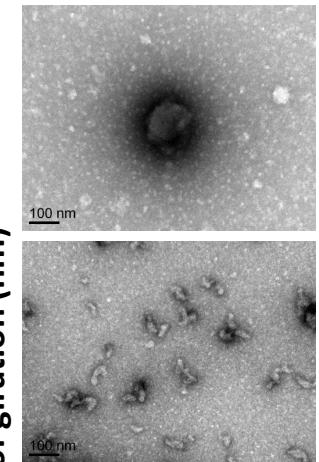
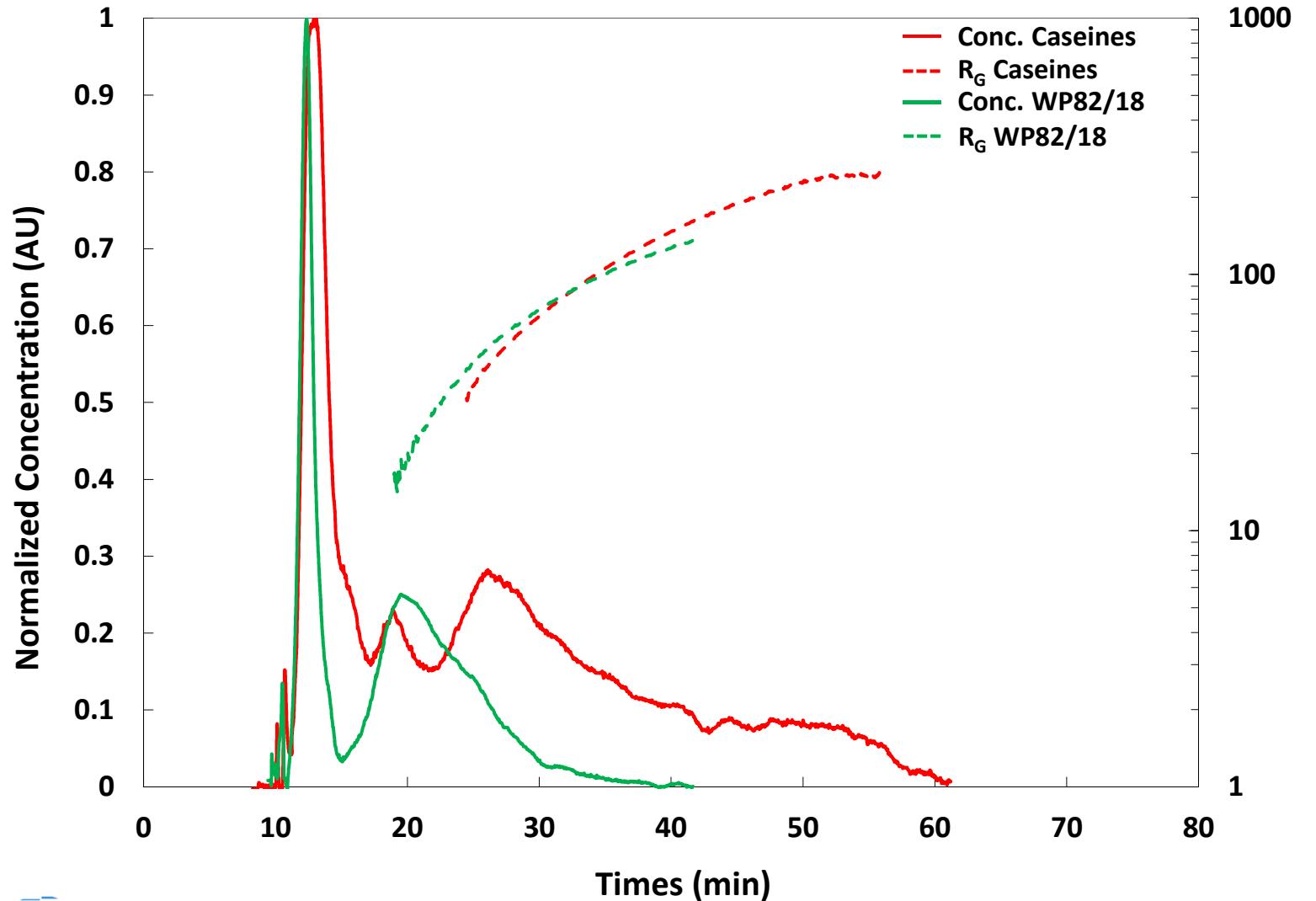
Méthodes de fabrication des agrégats



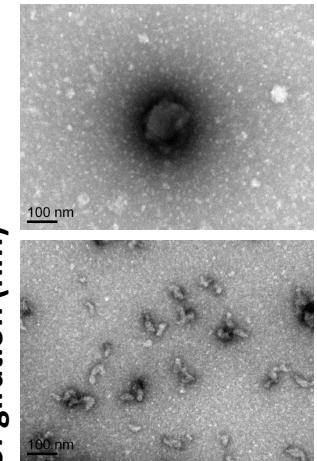
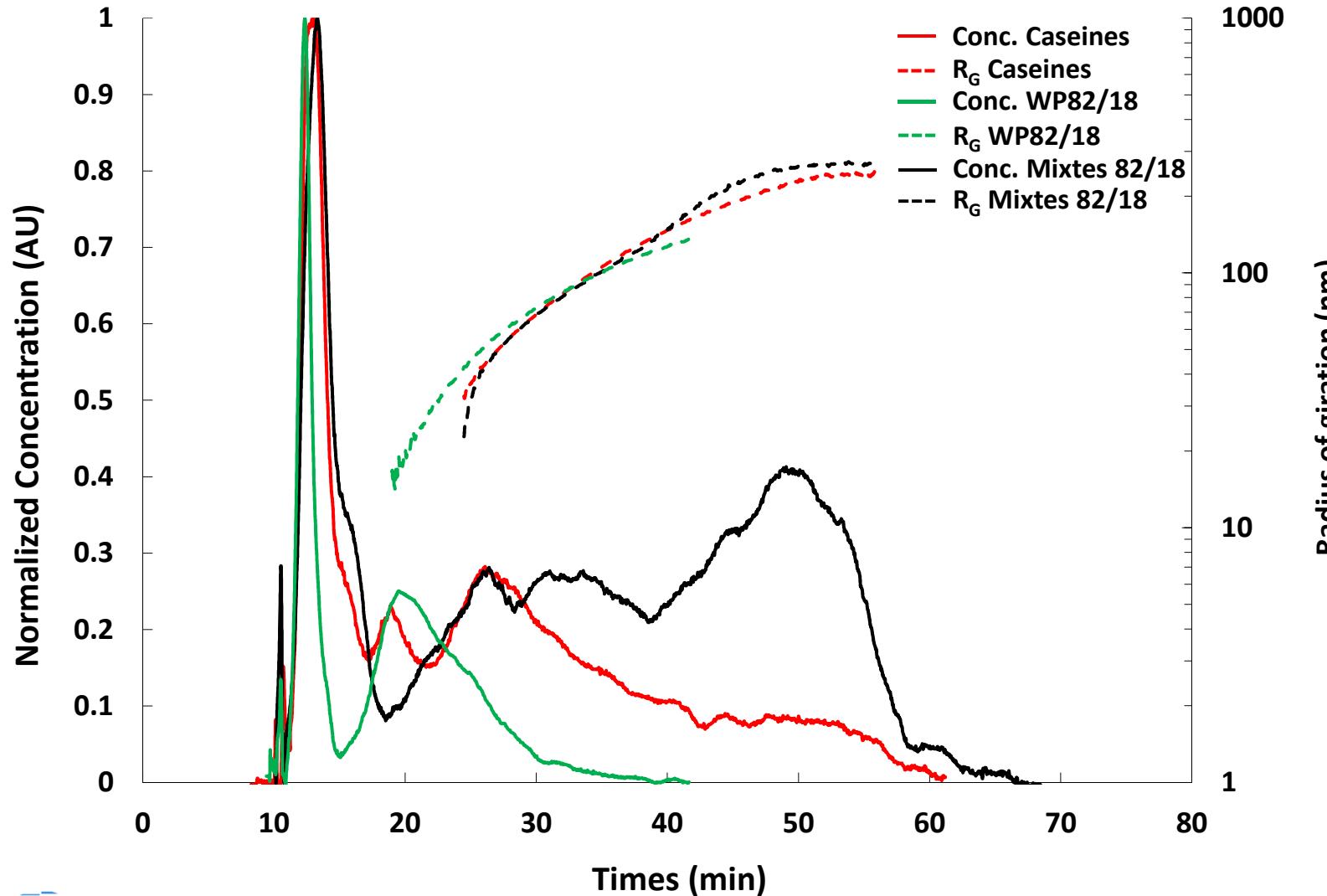
Mixtes 82/18



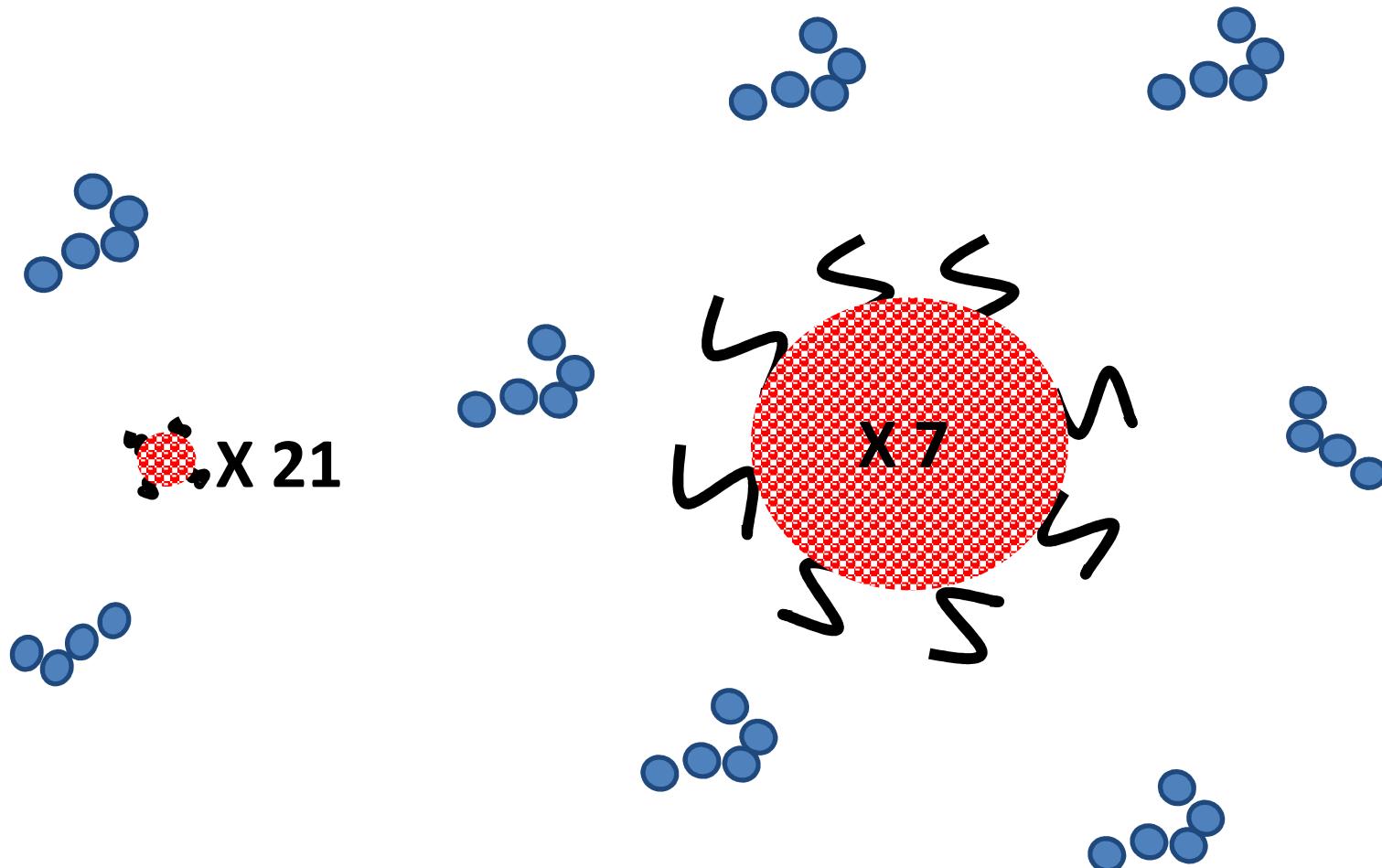
Mixtes 82/18



Mixtes 82/18



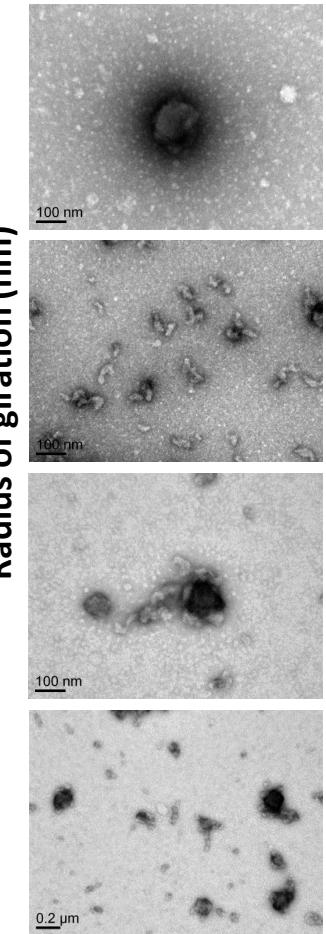
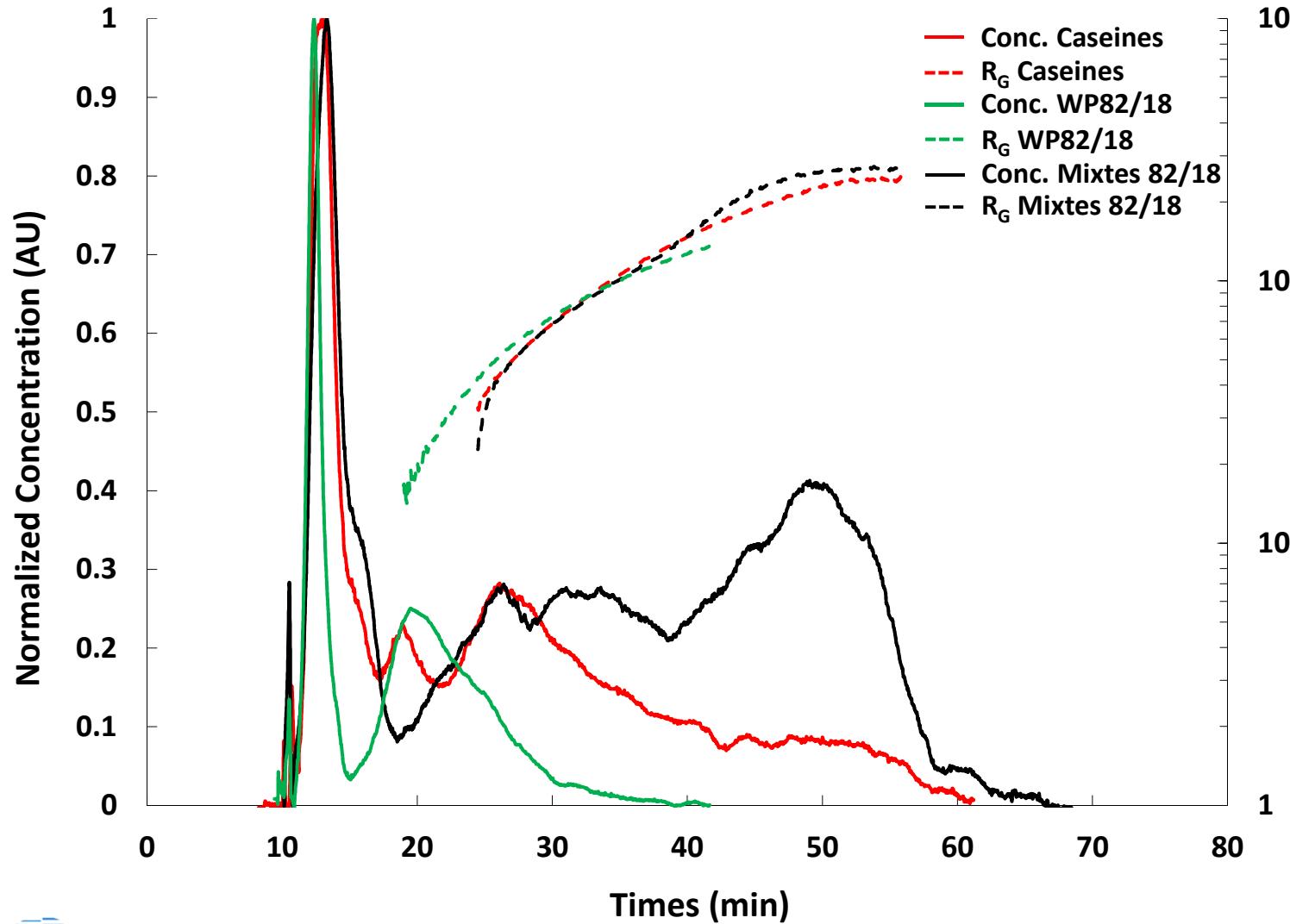
Mixtes 82/18



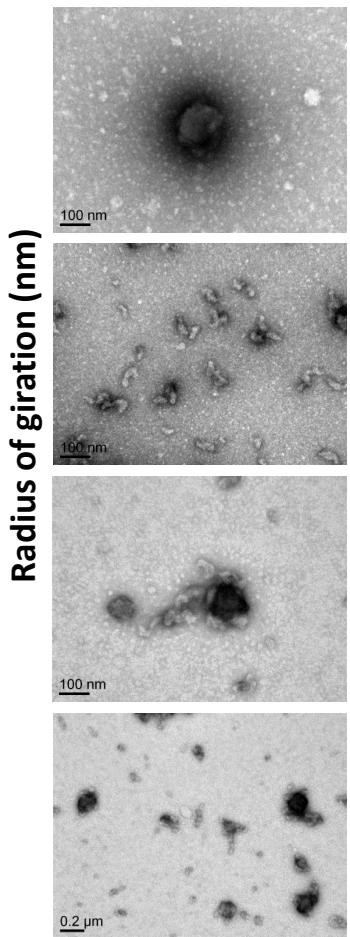
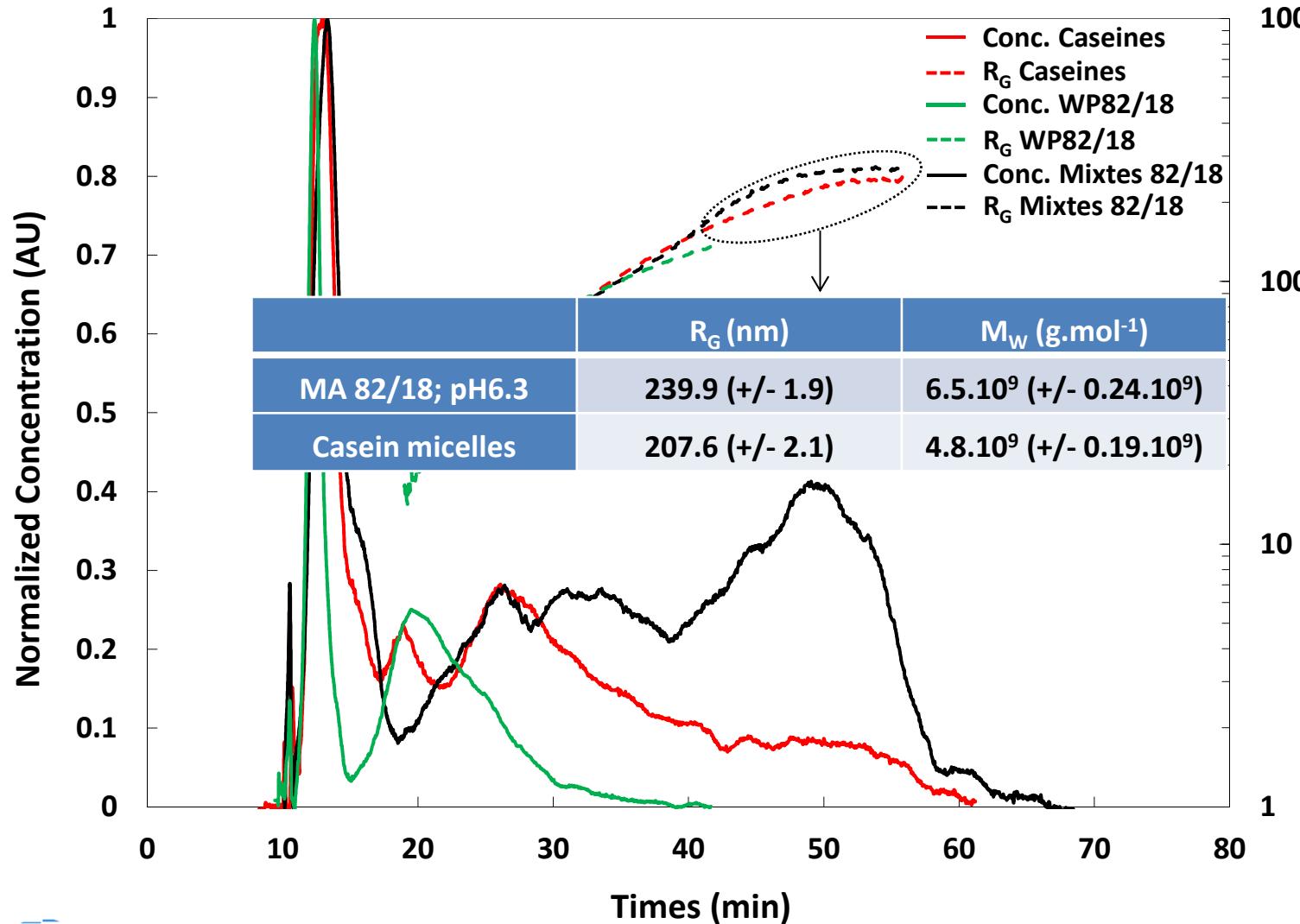
Pourquoi uniquement les micelles de grosses taille sont concernées?

- ✓ Plus grande quantité en nombre de caséines κ
- ✓ Encombrement stérique sur les petites micelles

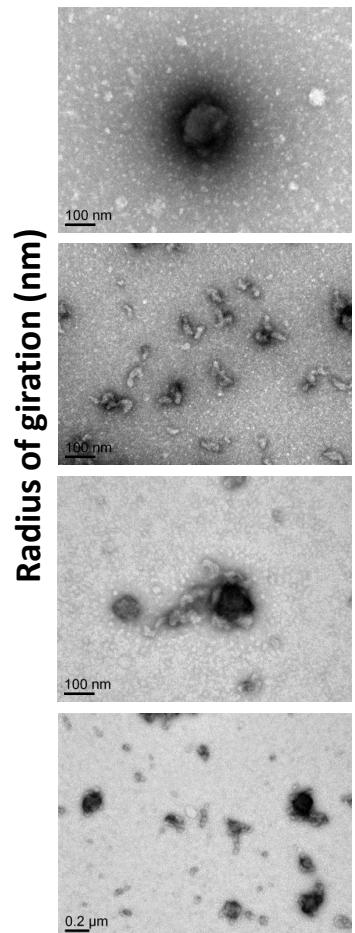
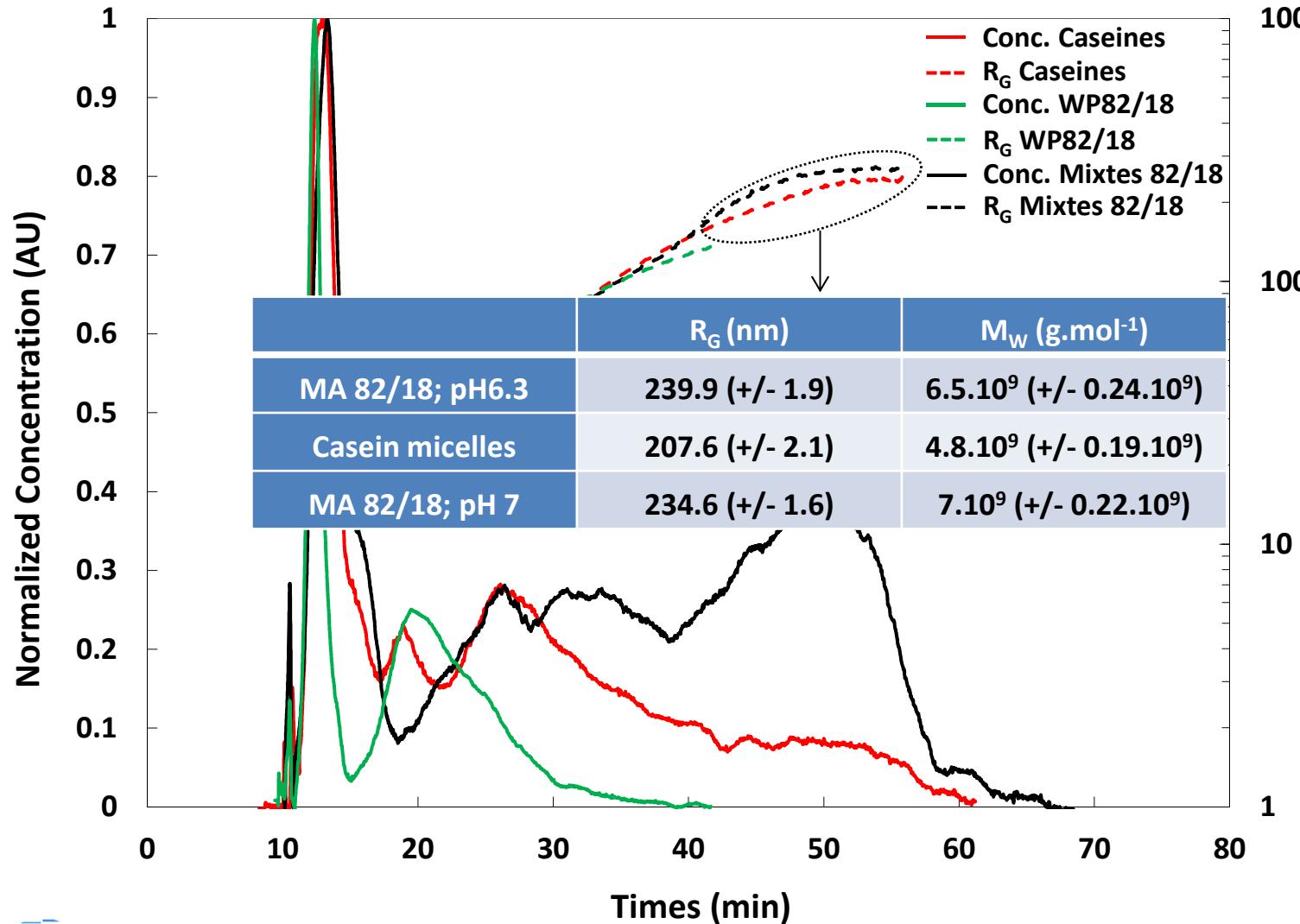
Mixtes 82/18



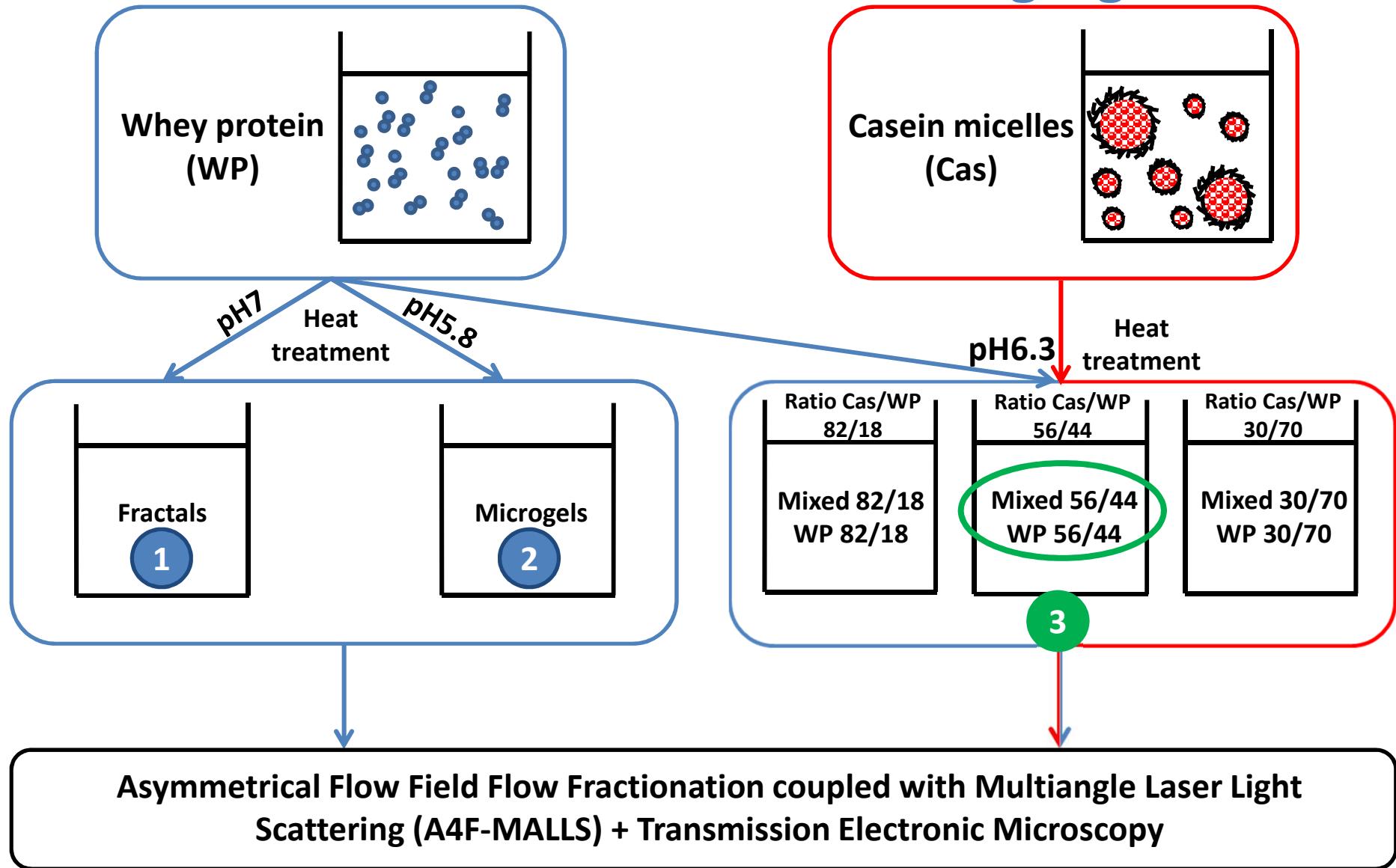
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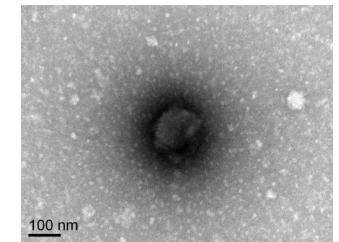
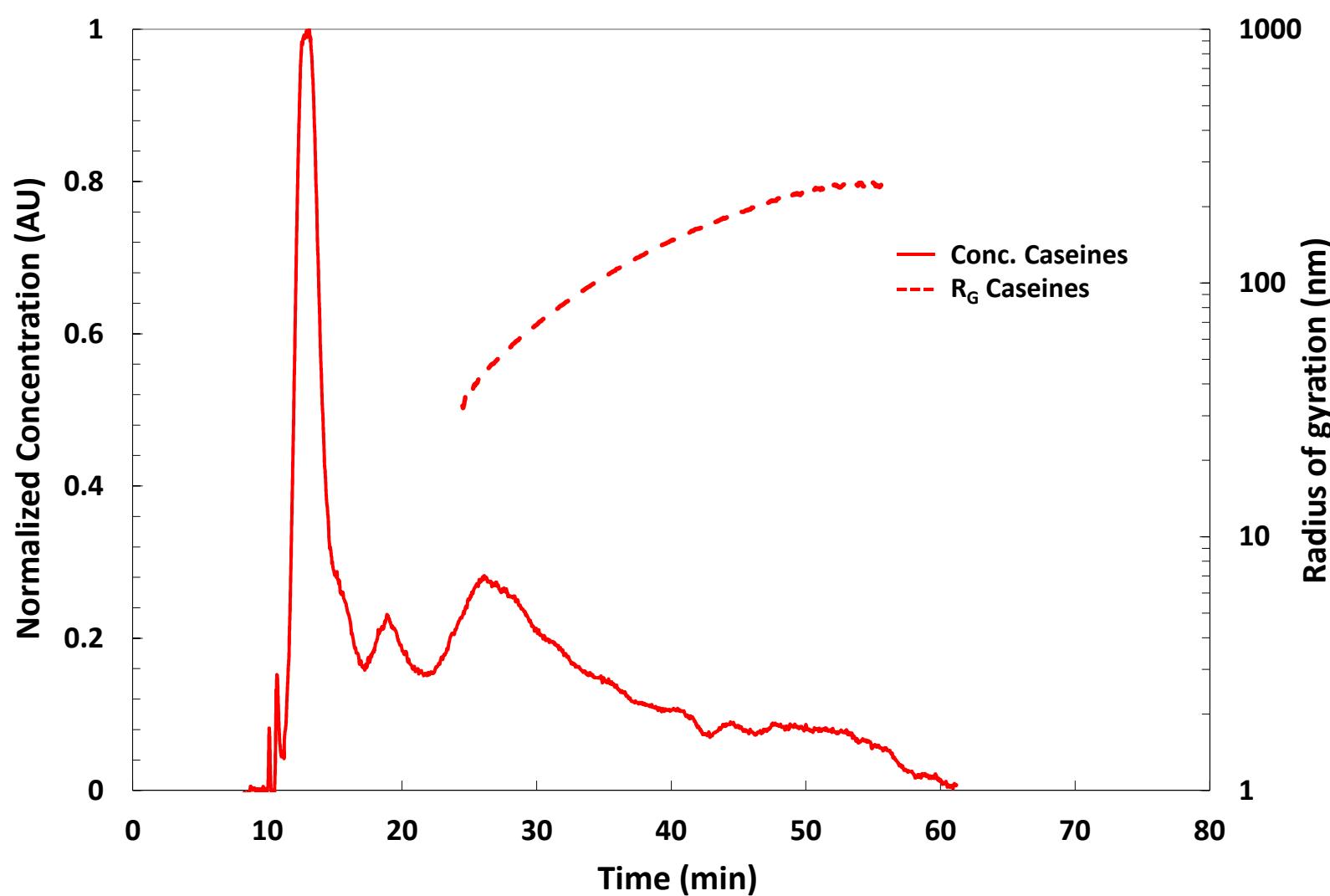
Mixtes 82/18



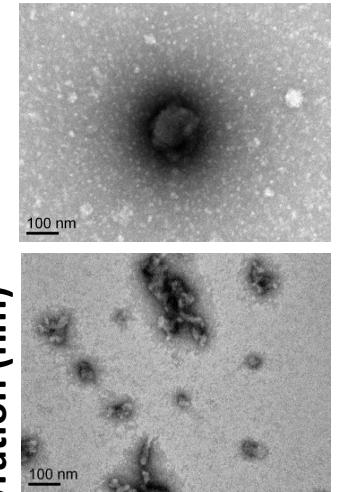
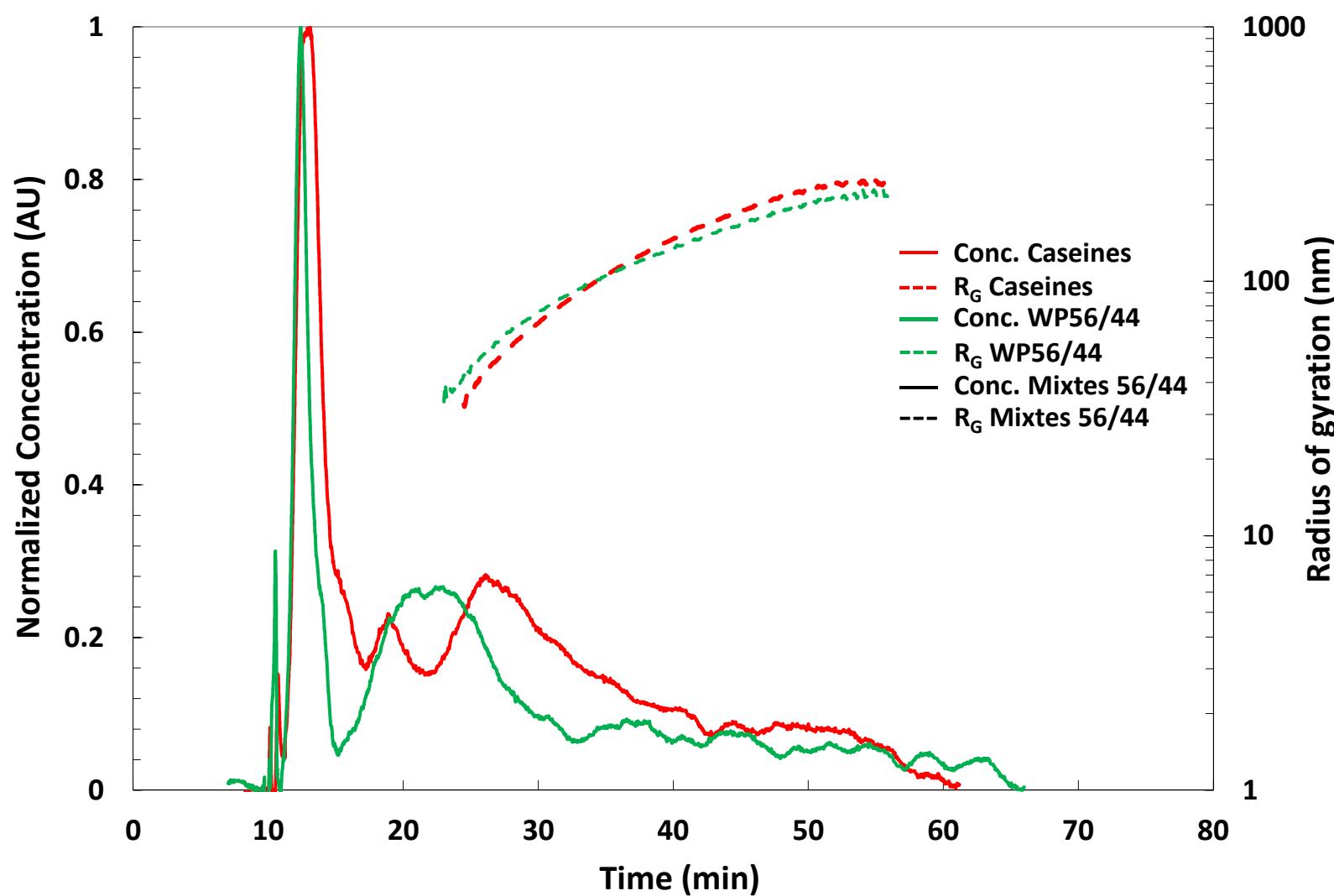
Méthodes de fabrication des agrégats



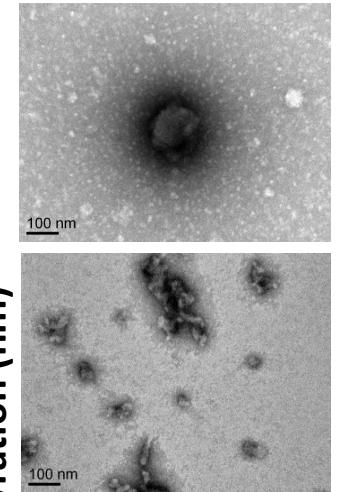
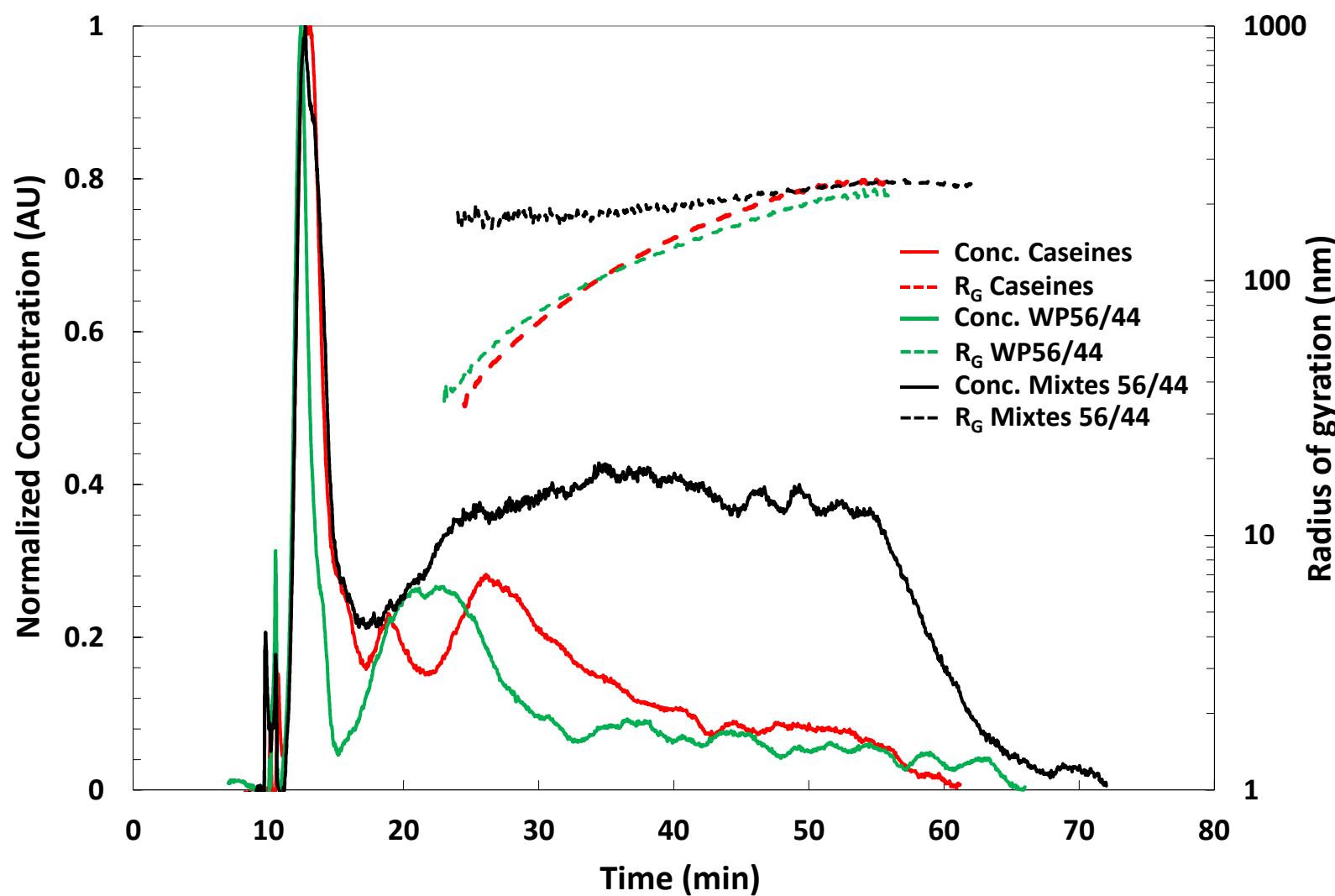
Mixtes 56/44



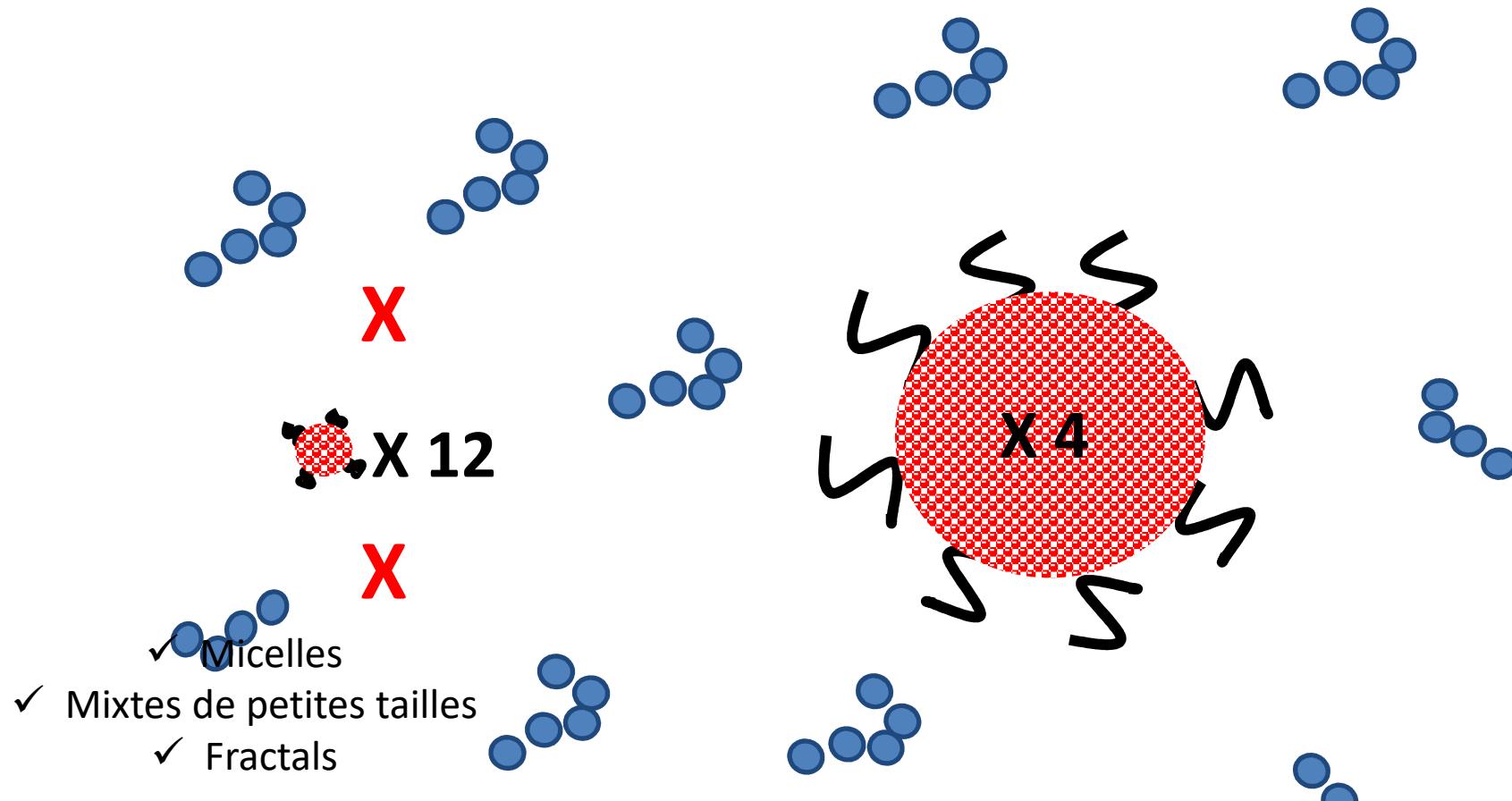
Mixtes 56/44



Mixtes 56/44



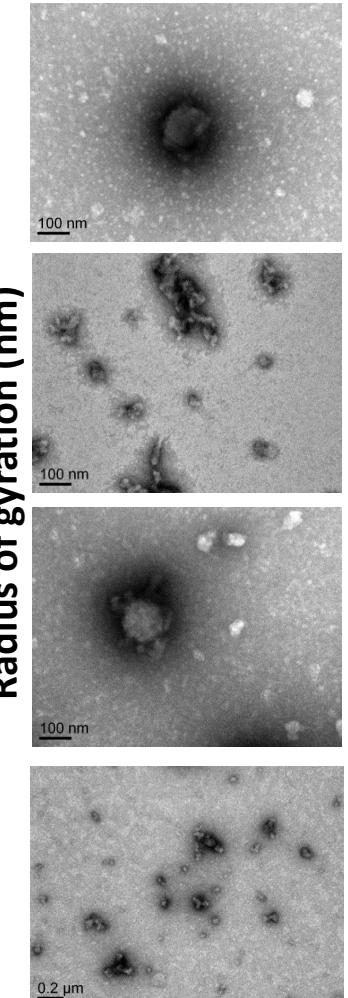
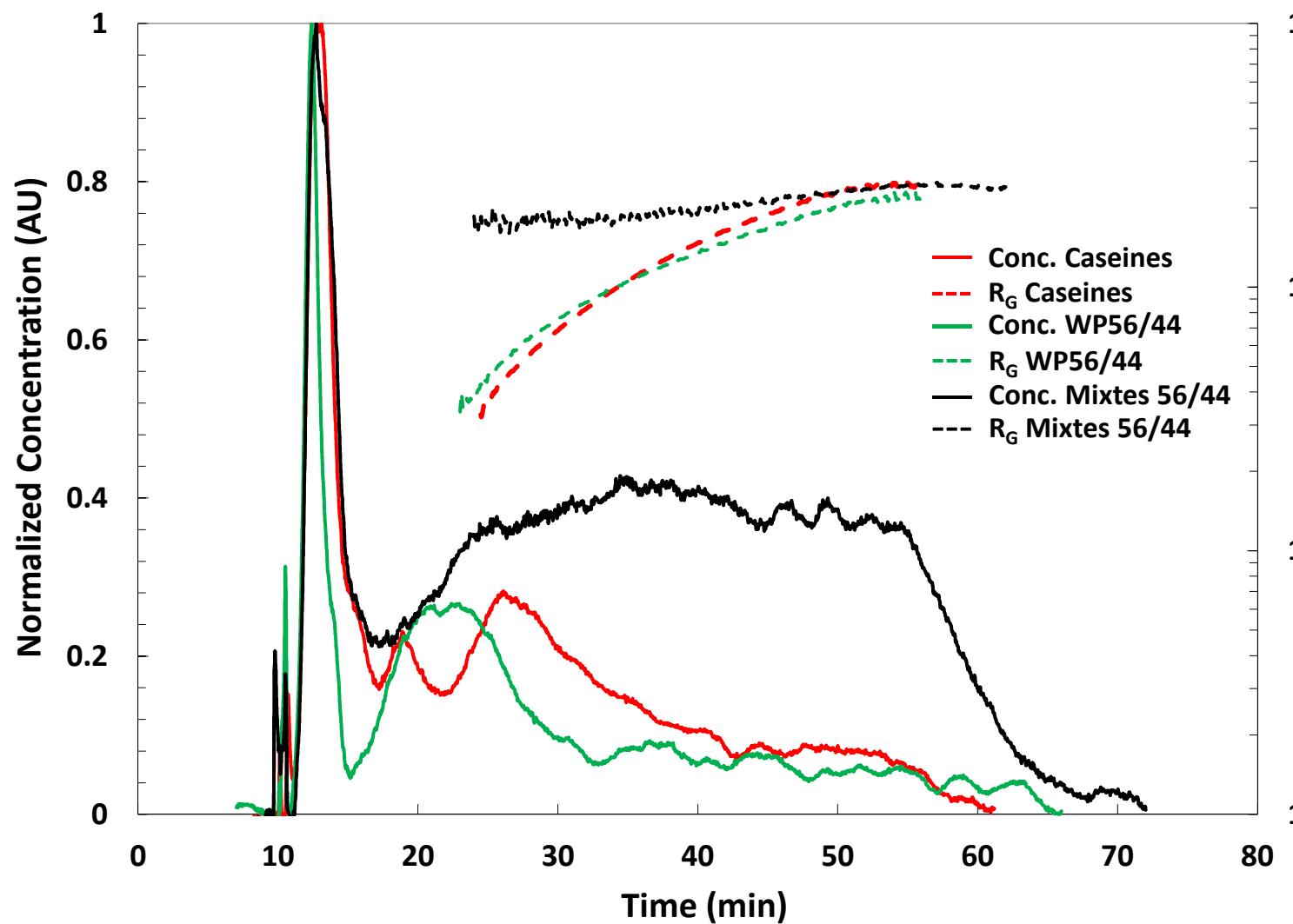
Mixtes 56/44



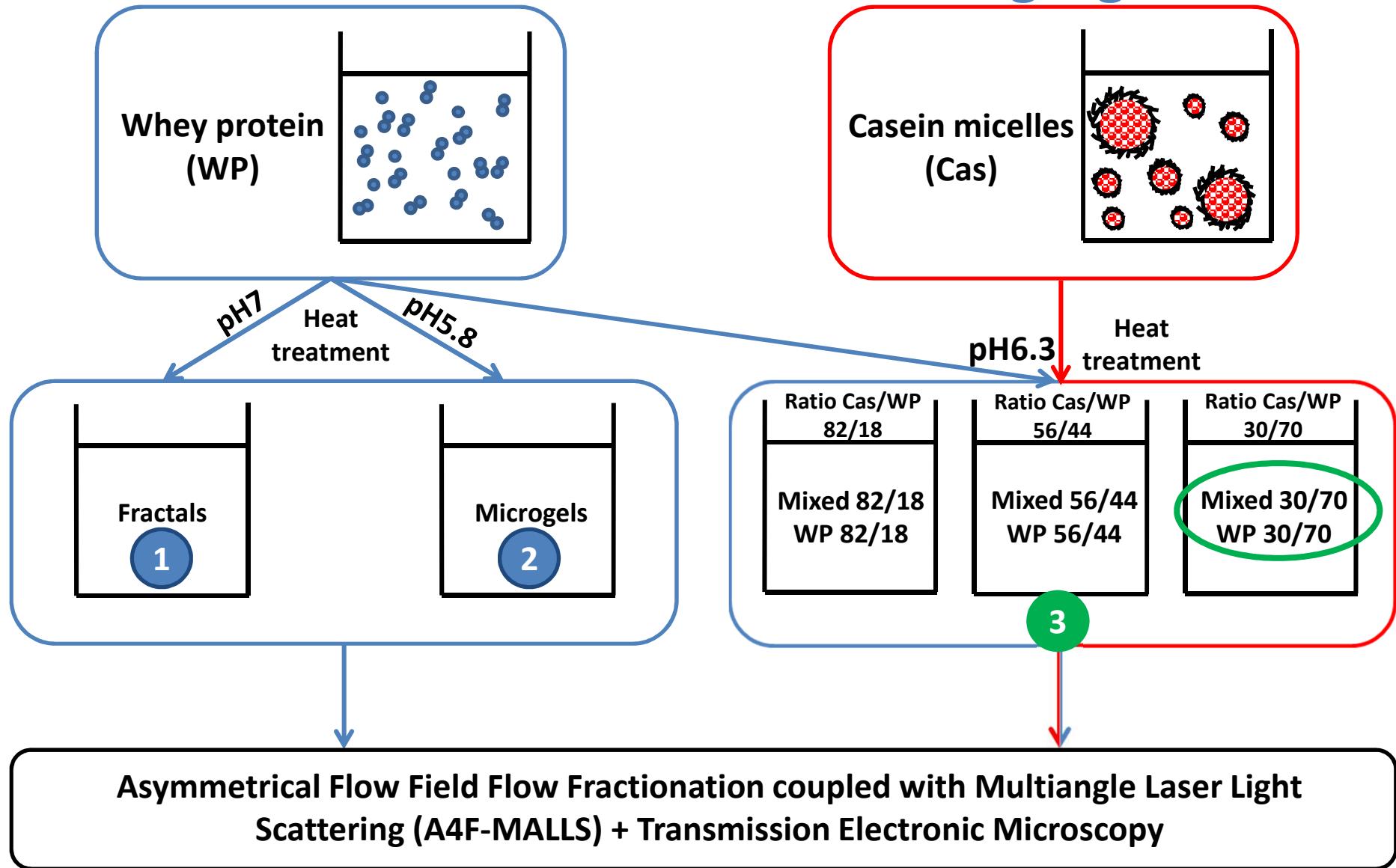
Pourquoi nous avons un mélange d’éléments de différentes tailles et formes?

- ✓ Formation essentiellement de mixtes de petites tailles car peu de grosses micelles
- ✓ Présence d’agrégats fractals en solution car problème d’encombrement stérique sur les petites micelles (cinétique d’adsorption plus lente)

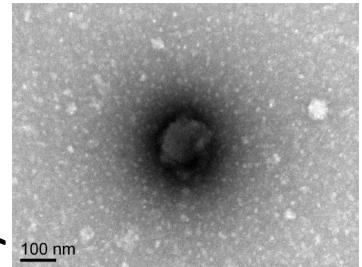
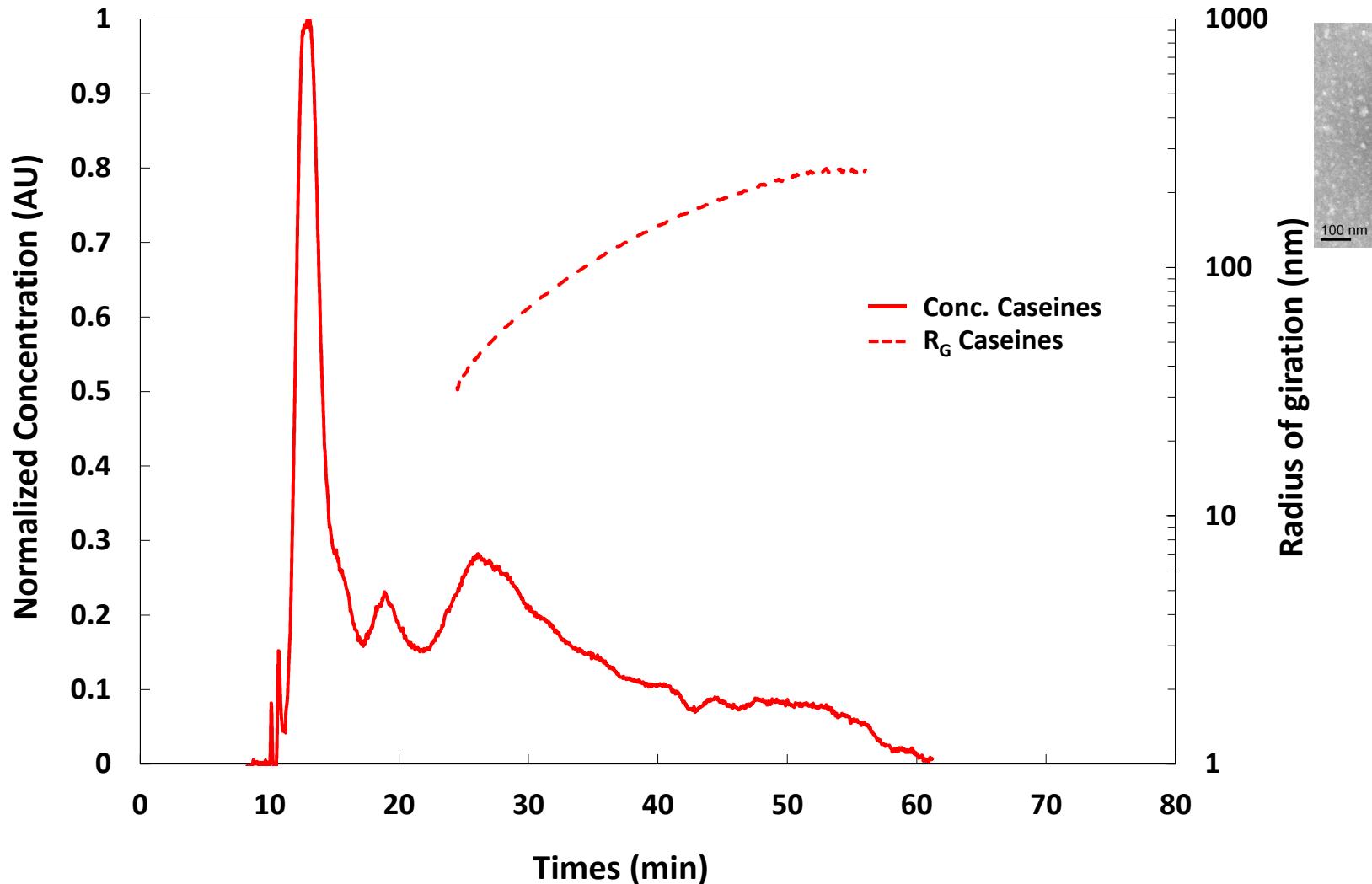
Mixtes 56/44



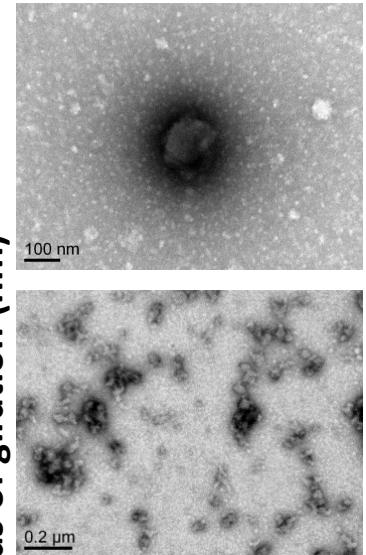
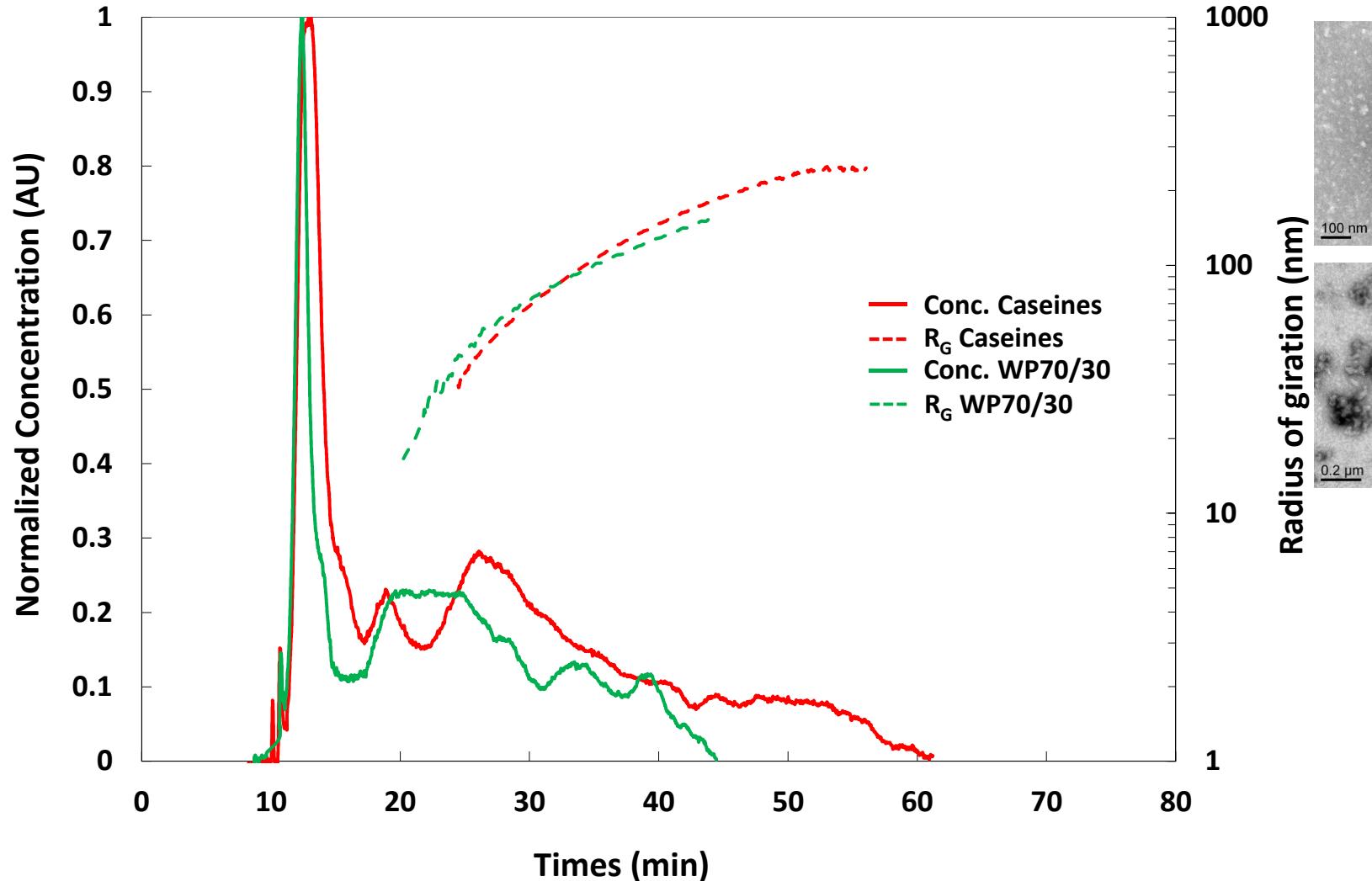
Méthodes de fabrication des agrégats



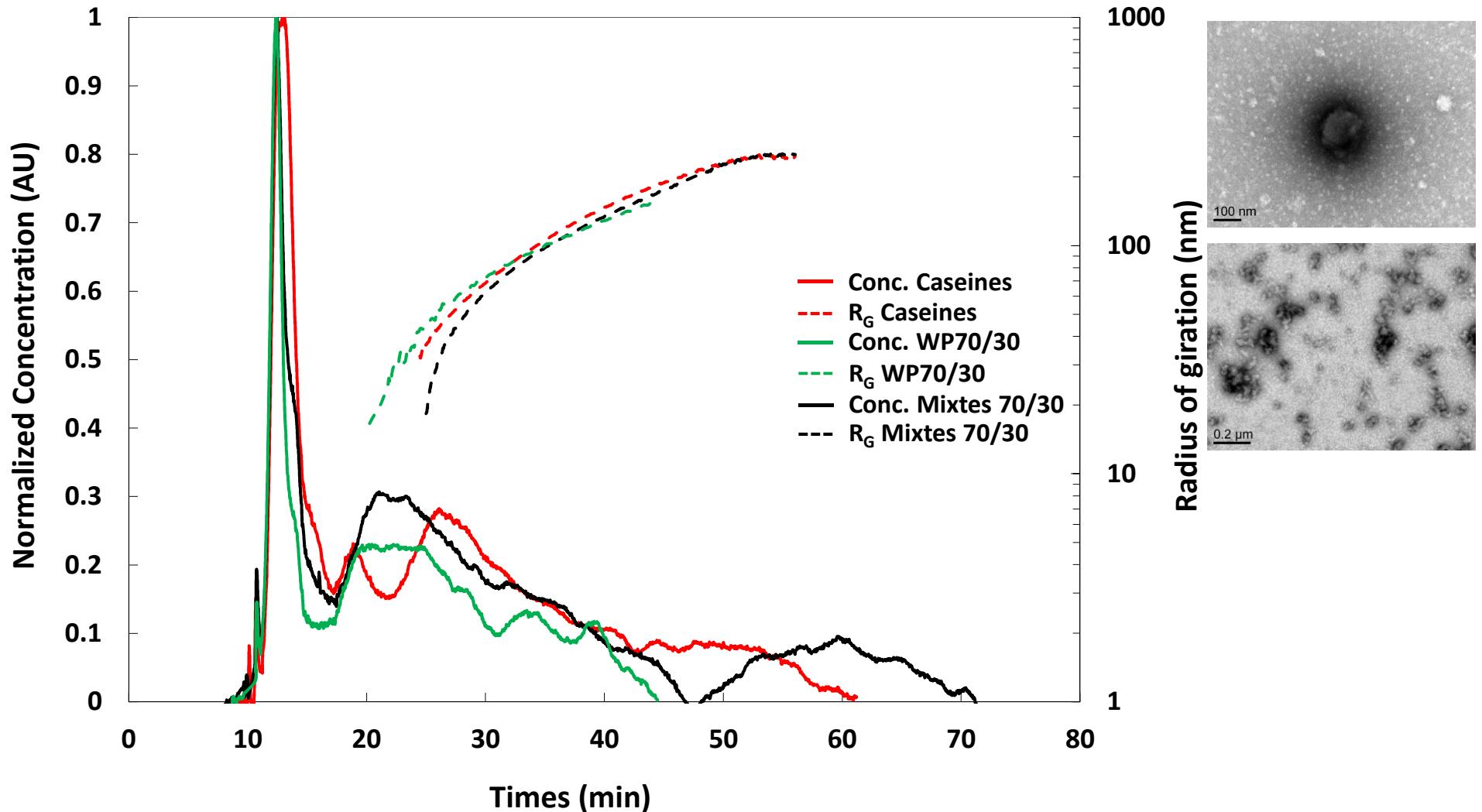
Mixtes 30/70



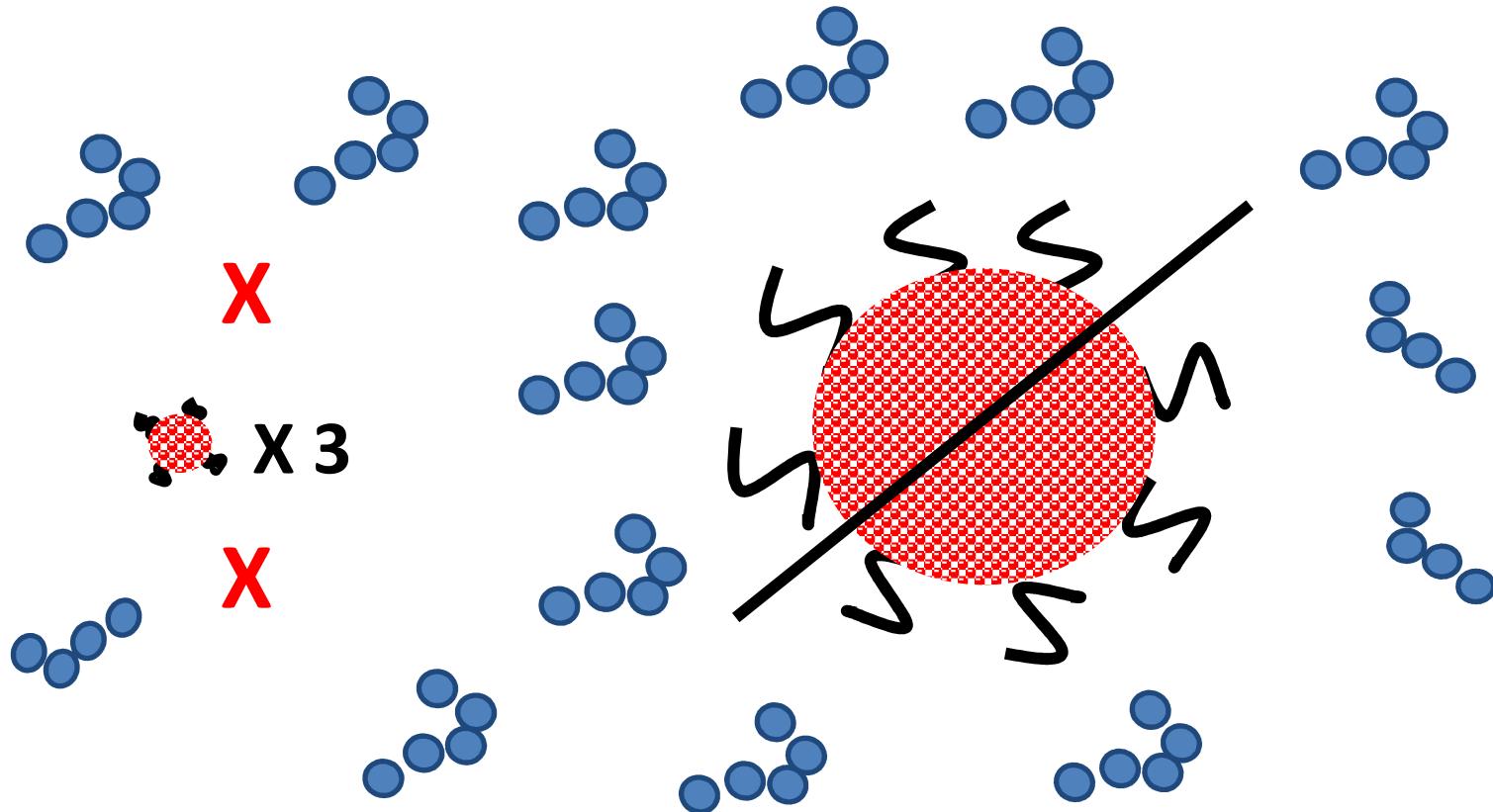
Mixtes 30/70



Mixtes 30/70



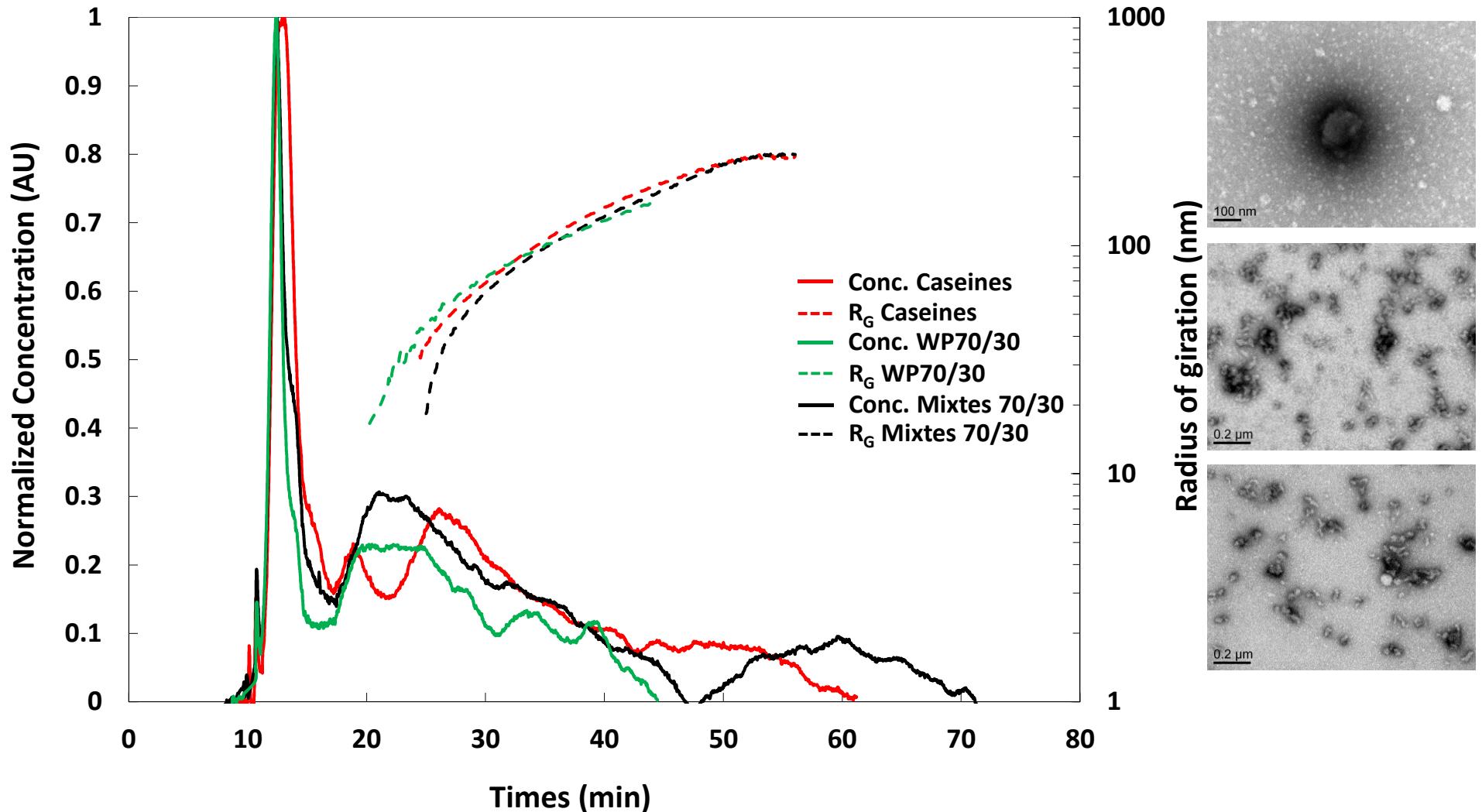
Mixtes 30/70



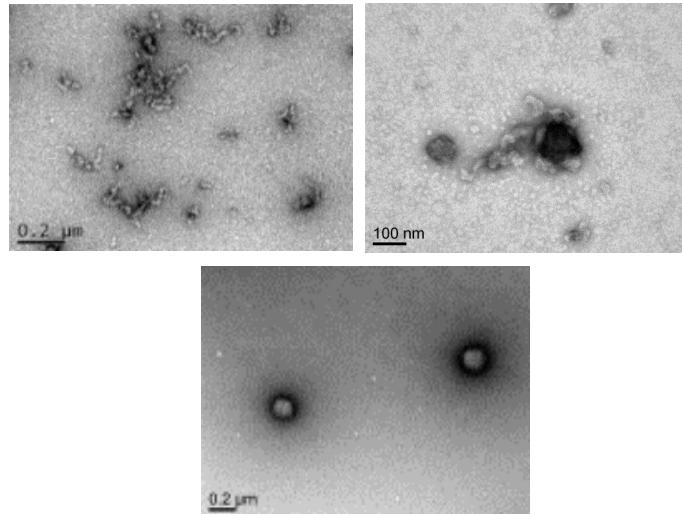
Pourquoi le profil des mixtes 30/70 et des fractals sont similaires?

- ✓ Très peu de petites caséines présentent dans la solution et quasiment aucune grosse micelle

Mixtes 30/70



Conclusion



- ✓ Polydisperses
- ✓ Mélange monomères/agrégats
- ✓ Différente taille et forme
- ✓ Présence de caséines



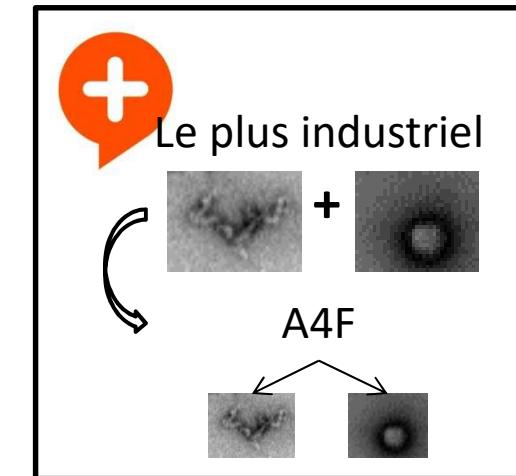
Asymmetrical Flow Field Flow Fractionation

Fractionner les monomère et les agrégats

Fractionner les agrégats

Travailler avec des agrégats contenant des micelles

Idée des mécanismes de formation des agrégats mixtes





Merci de votre attention

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Journée Scientifique

du Groupe Francophone de Fractionnement Flux-Force

31 mars 2017

