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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

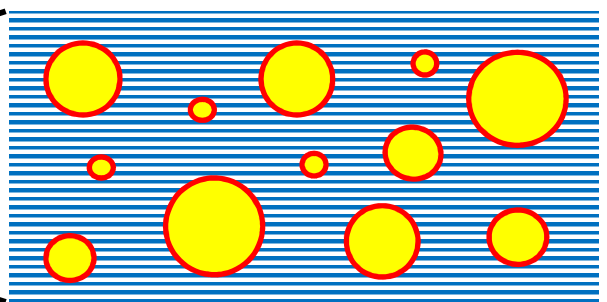
du Groupe Francophone de Fractionnement Flux-Force
31 mars 2017



Contexte



11 Industriels
6 Scientifiques



Emulsion H/E

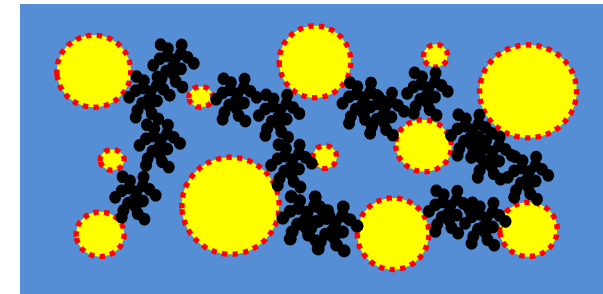
Gélification de la phase **continue**

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



Eliminer les texturants de type polysaccharides

Objectifs

Texturer des produits à faibles taux de MG

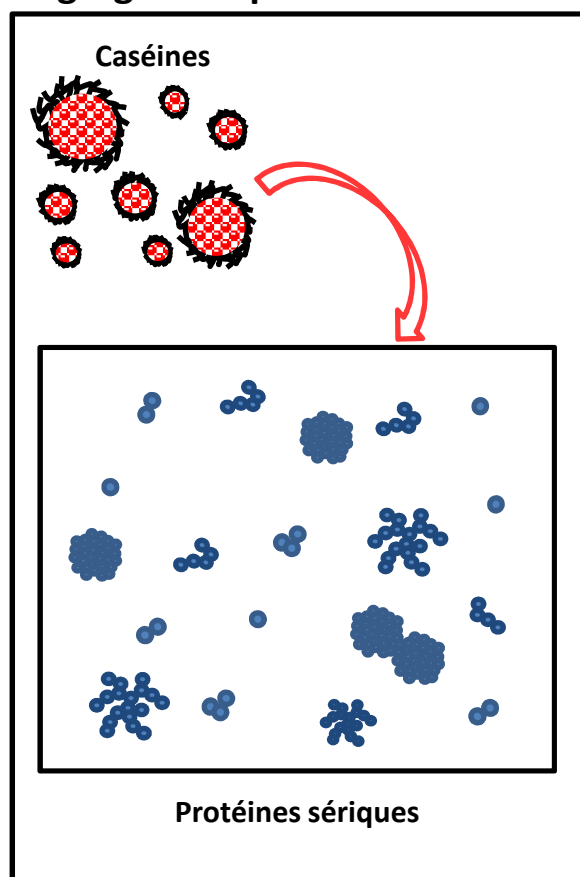


Problématiques techniques

DLS

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

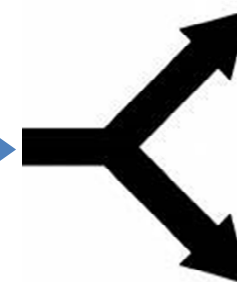
Agrégats de protéines laitières



Mélange monomères/agrégats

Systèmes polydispers

Différente taille/forme/densité



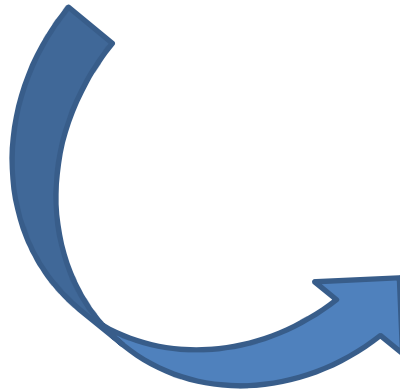
SEC

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

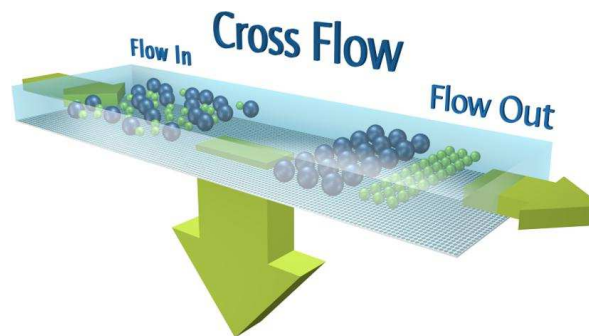
Problématiques techniques



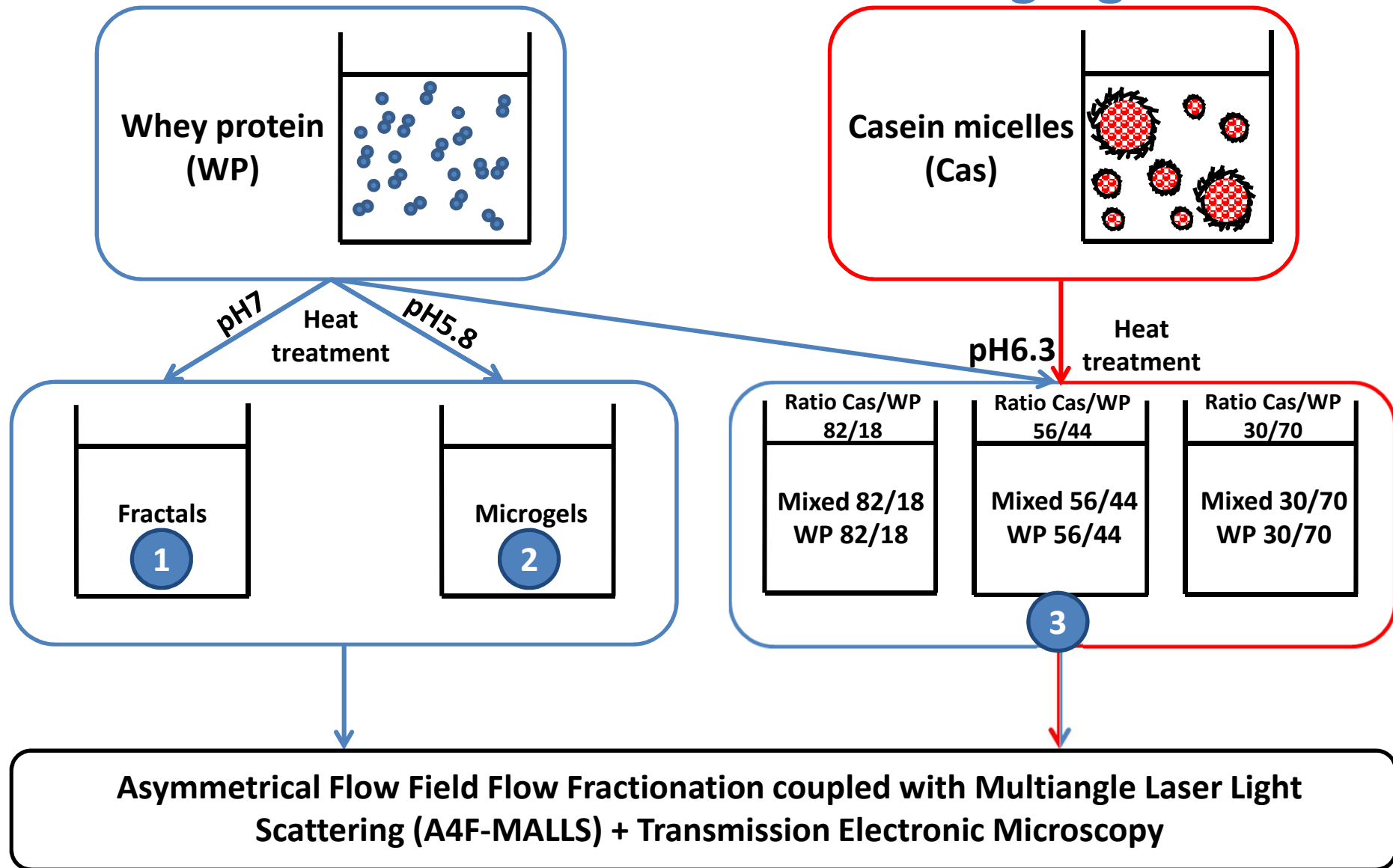
Trouver une technique séparative capable de fractionner les différentes entités y compris les assemblages avec des caséines (contrairement à la SEC)



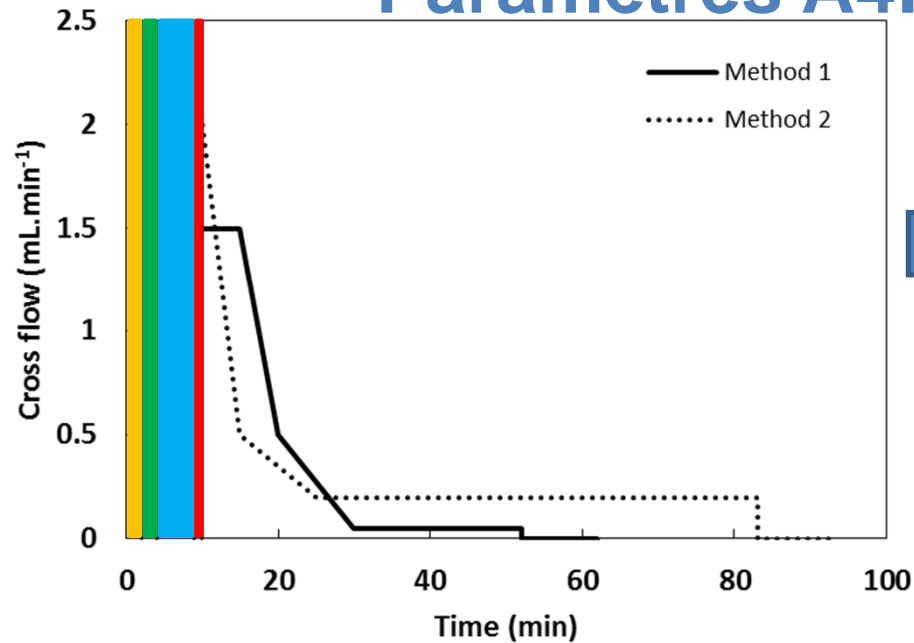
Asymmetrical Flow Field Flow Fractionation



Méthodes de fabrication des agrégats



Paramètres A4F et MET



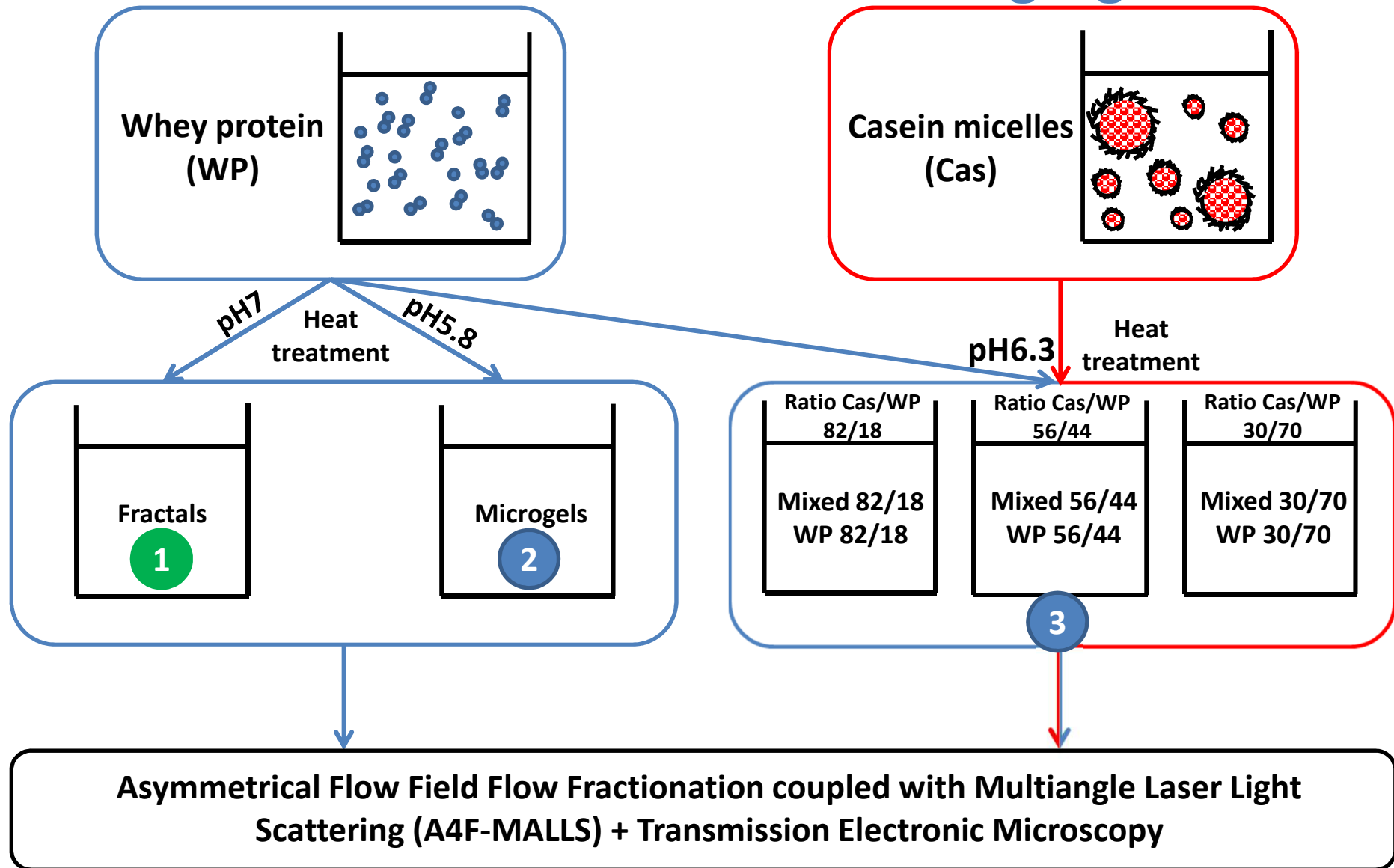
- ### Detectors
- ✓ MALLS
 - ✓ UV Detector
 - ✓ Differential Refractometer (DRi)

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

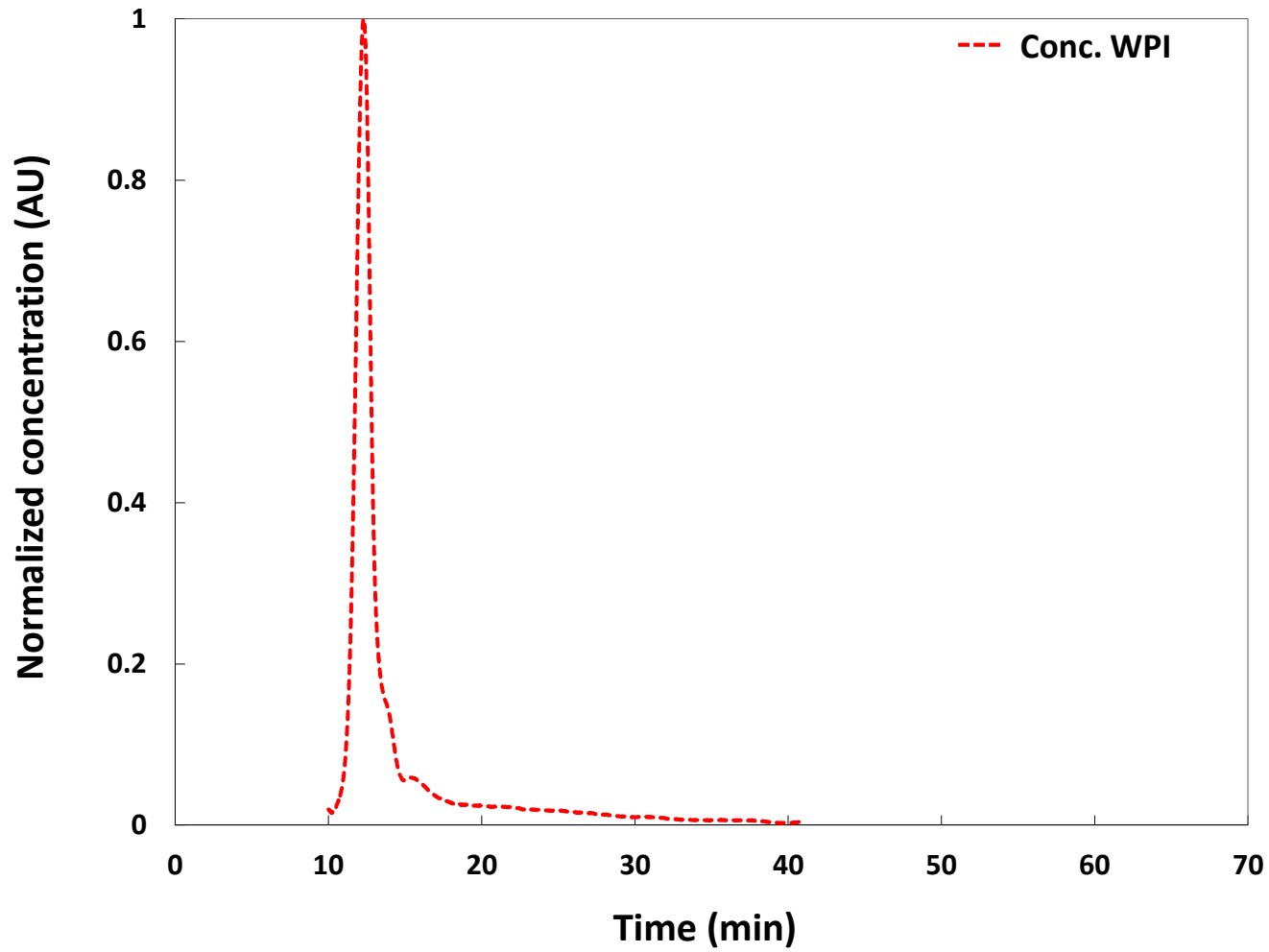
	Fractal aggregates	Microgel aggregates	Mixed aggregates
Eluent (pH)	Millipore water containing 0.02 w/v% NaN ₃ and 45 mM NaCl (pH 7)	Millipore water containing 0.02 w/v% NaN ₃ and 45 mM NaCl (pH 7)	Millipore water containing 0.02 w/v% NaN ₃ , 45 mM NaCl and 10mM CaCl ₂ (pH 6.3 or 7)
A4F method	2	1	2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

MET: Coloration négative

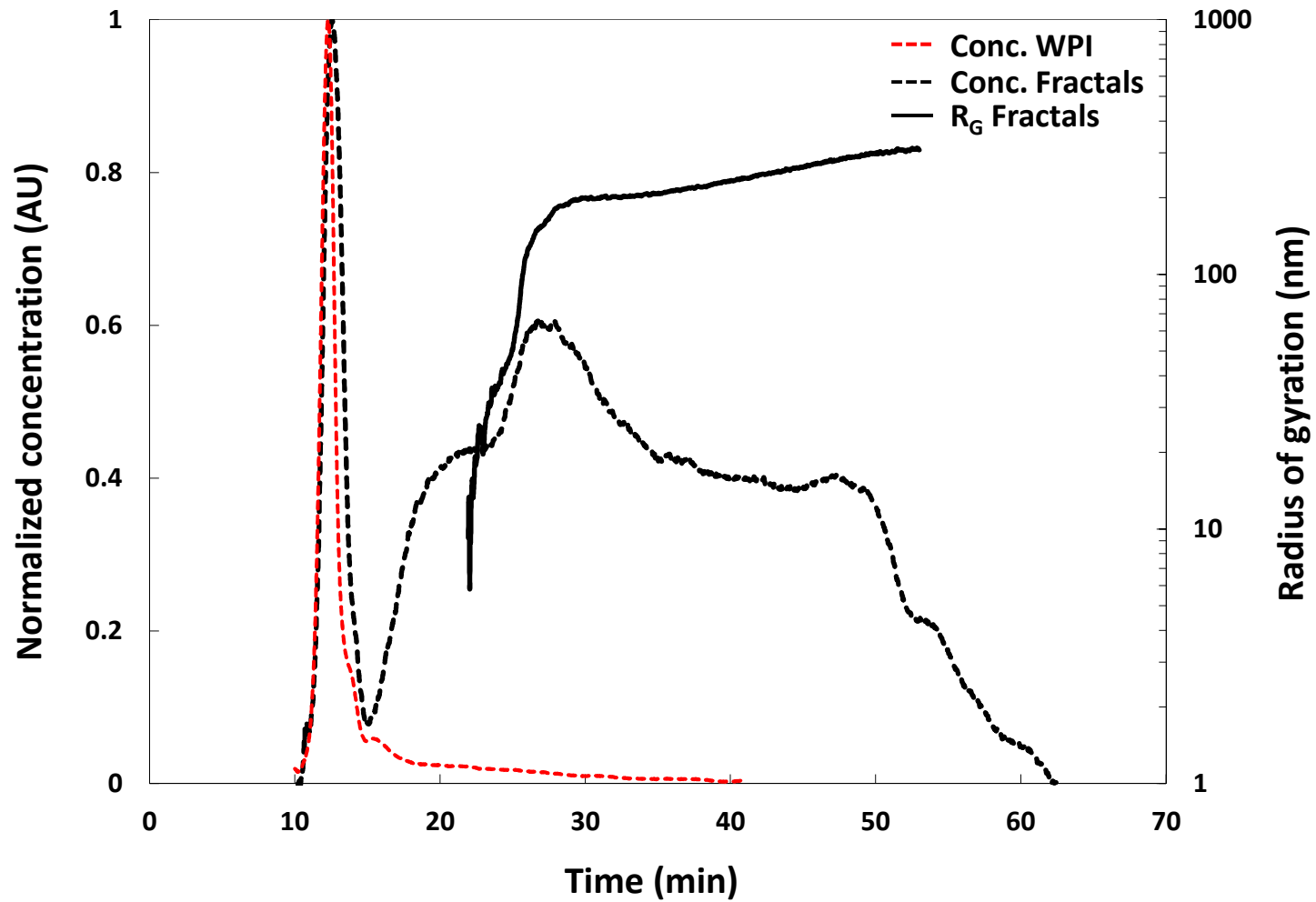
Méthodes de fabrication des agrégats



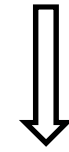
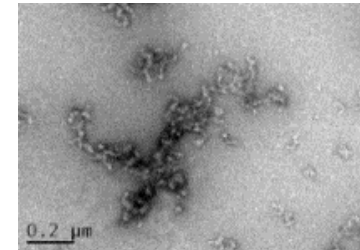
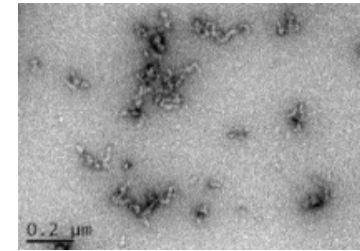
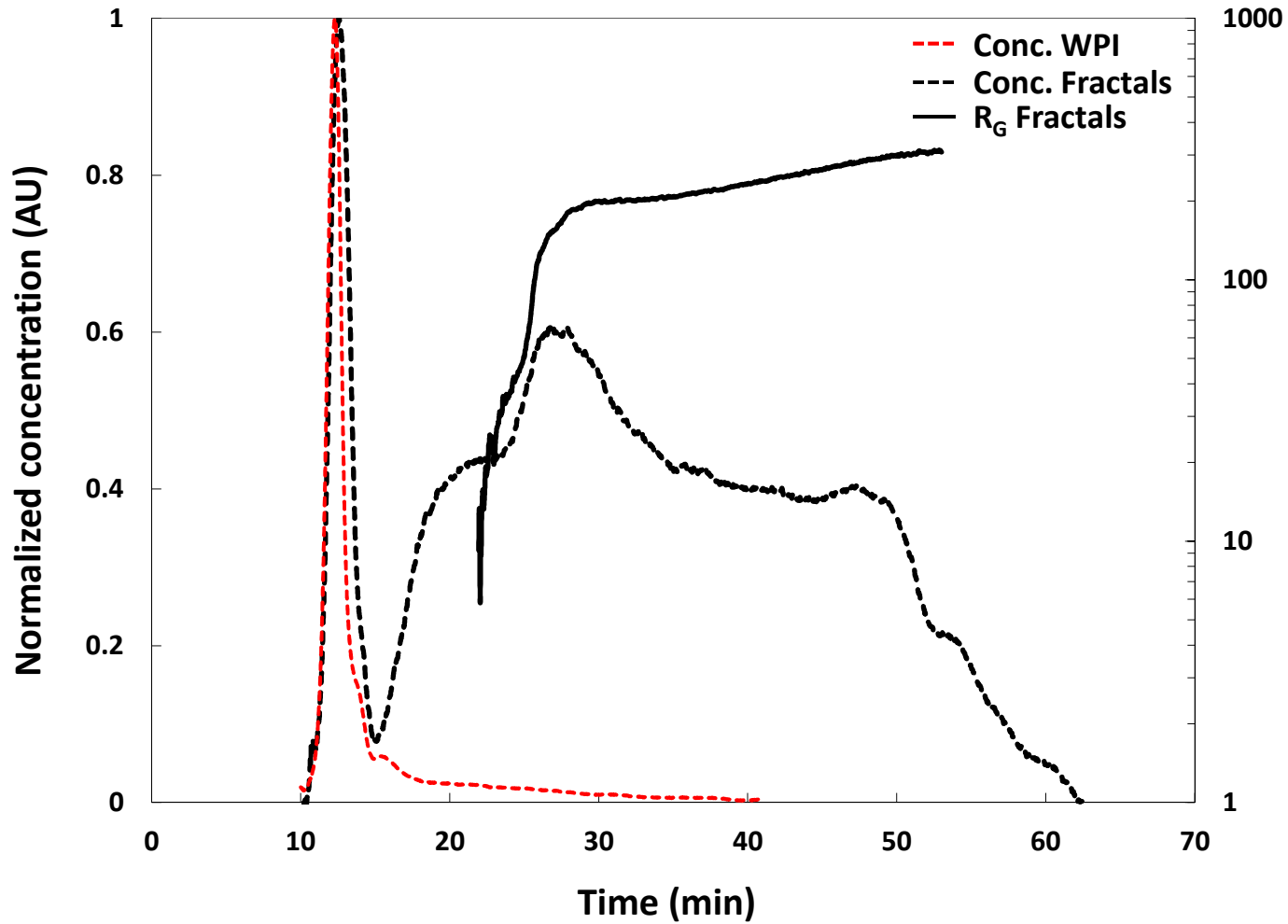
Fractals



Fractals

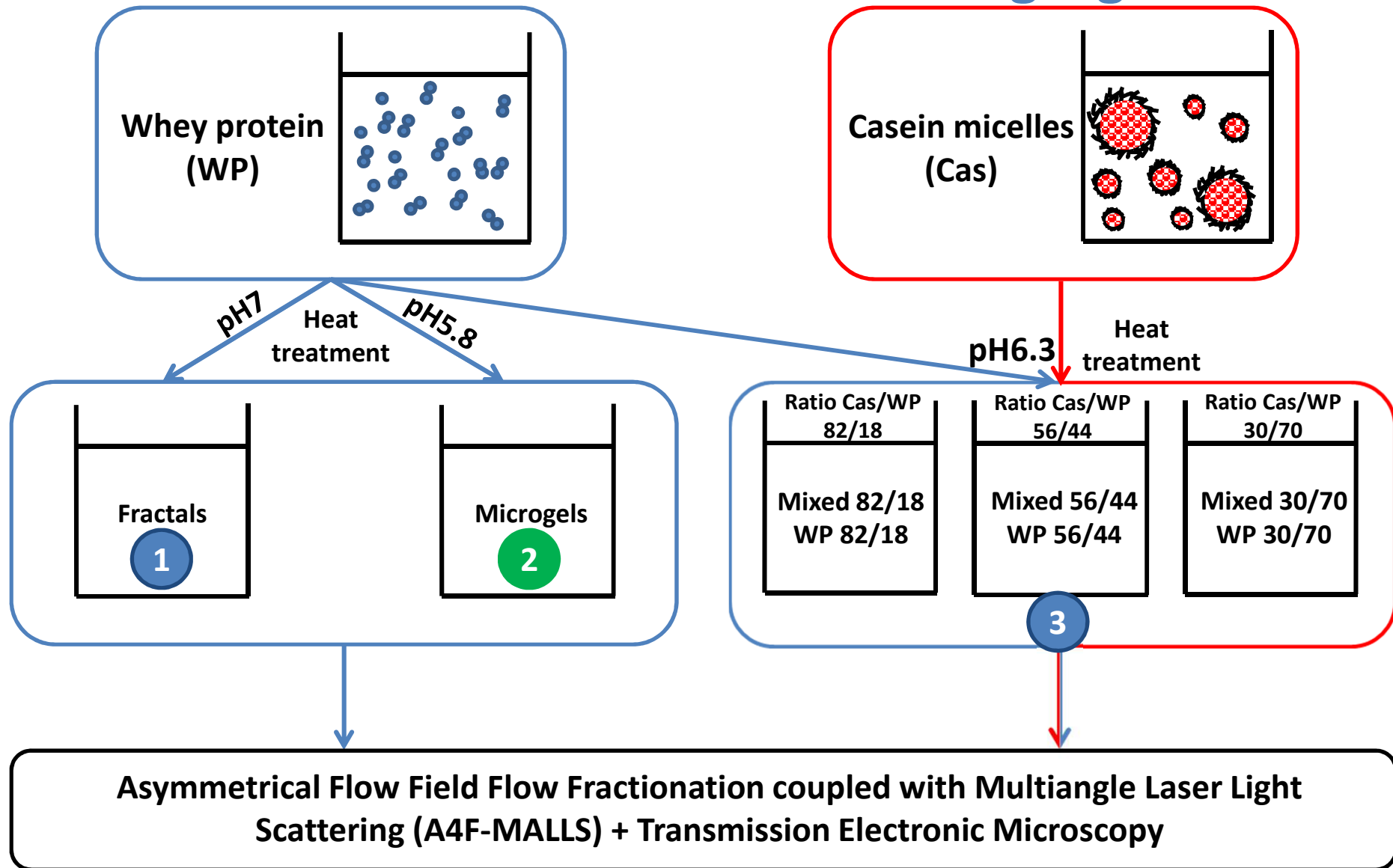


Fractals

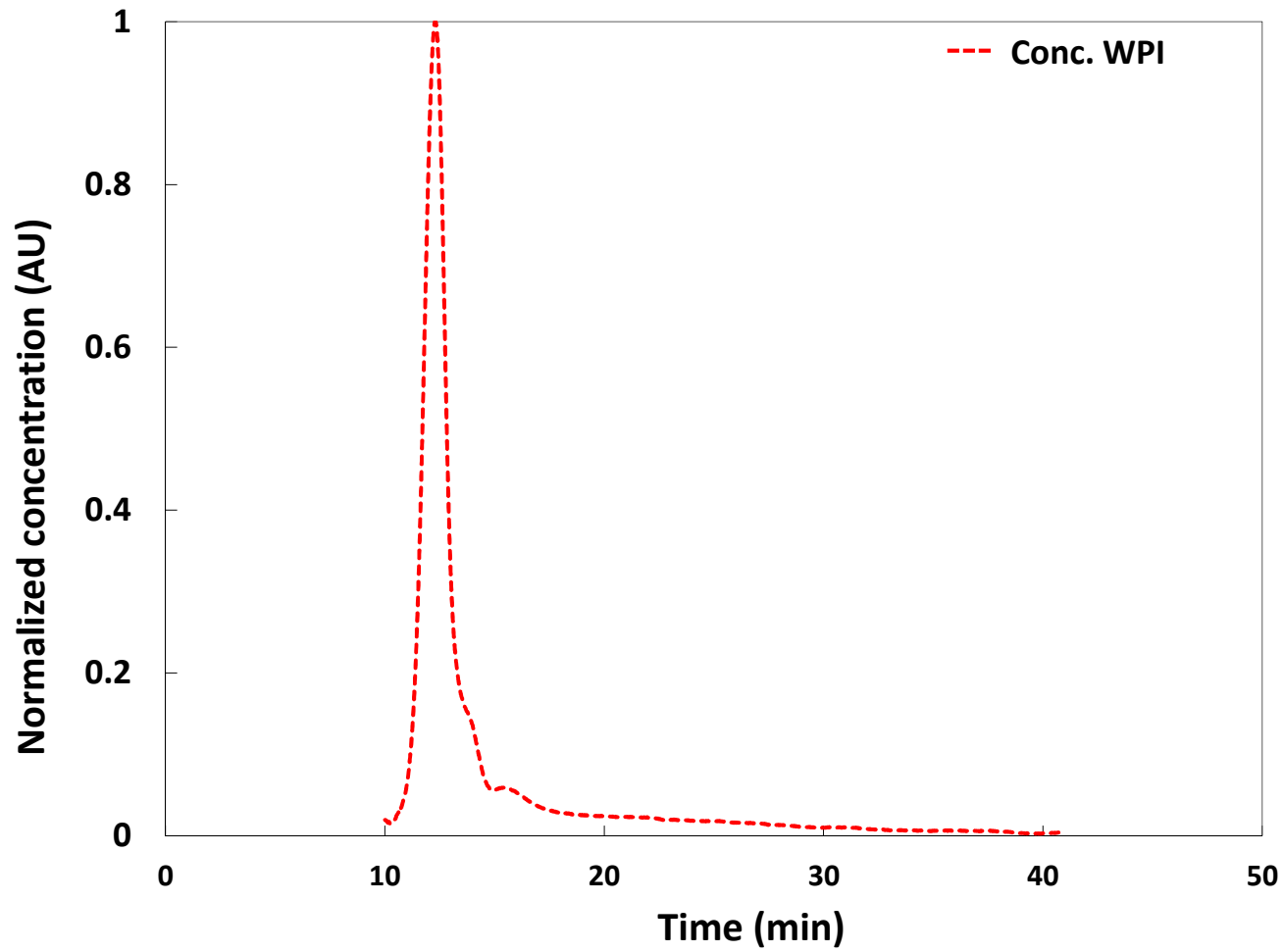


Df : 2.3

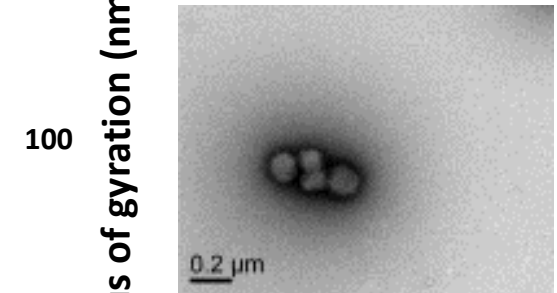
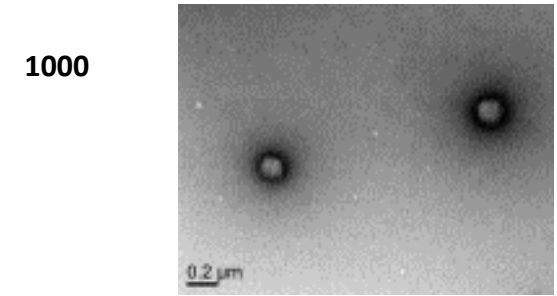
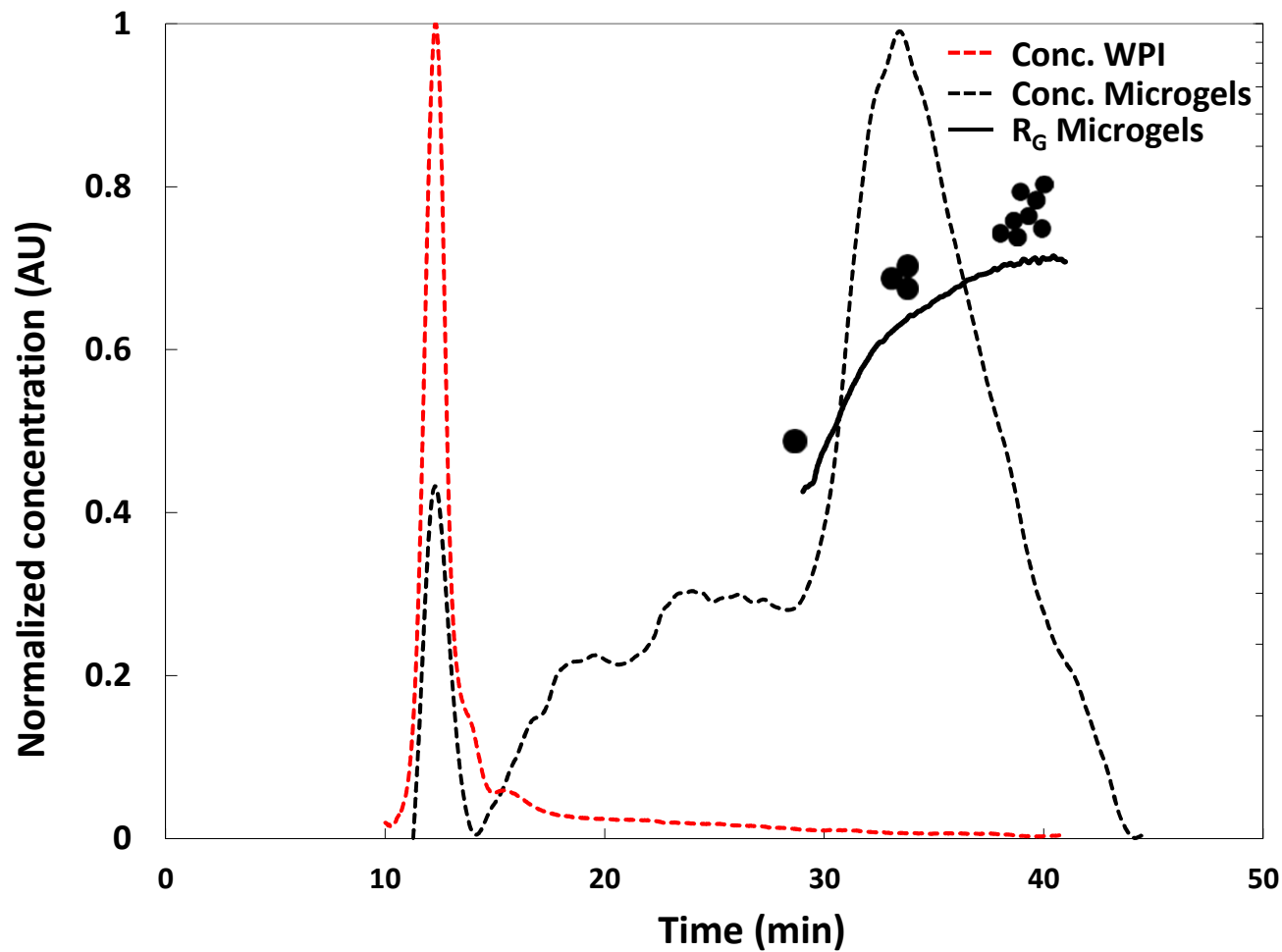
Méthodes de fabrication des agrégats



Microgels



Microgels



Radius of gyration (nm)

$R_G = 100\text{nm}$

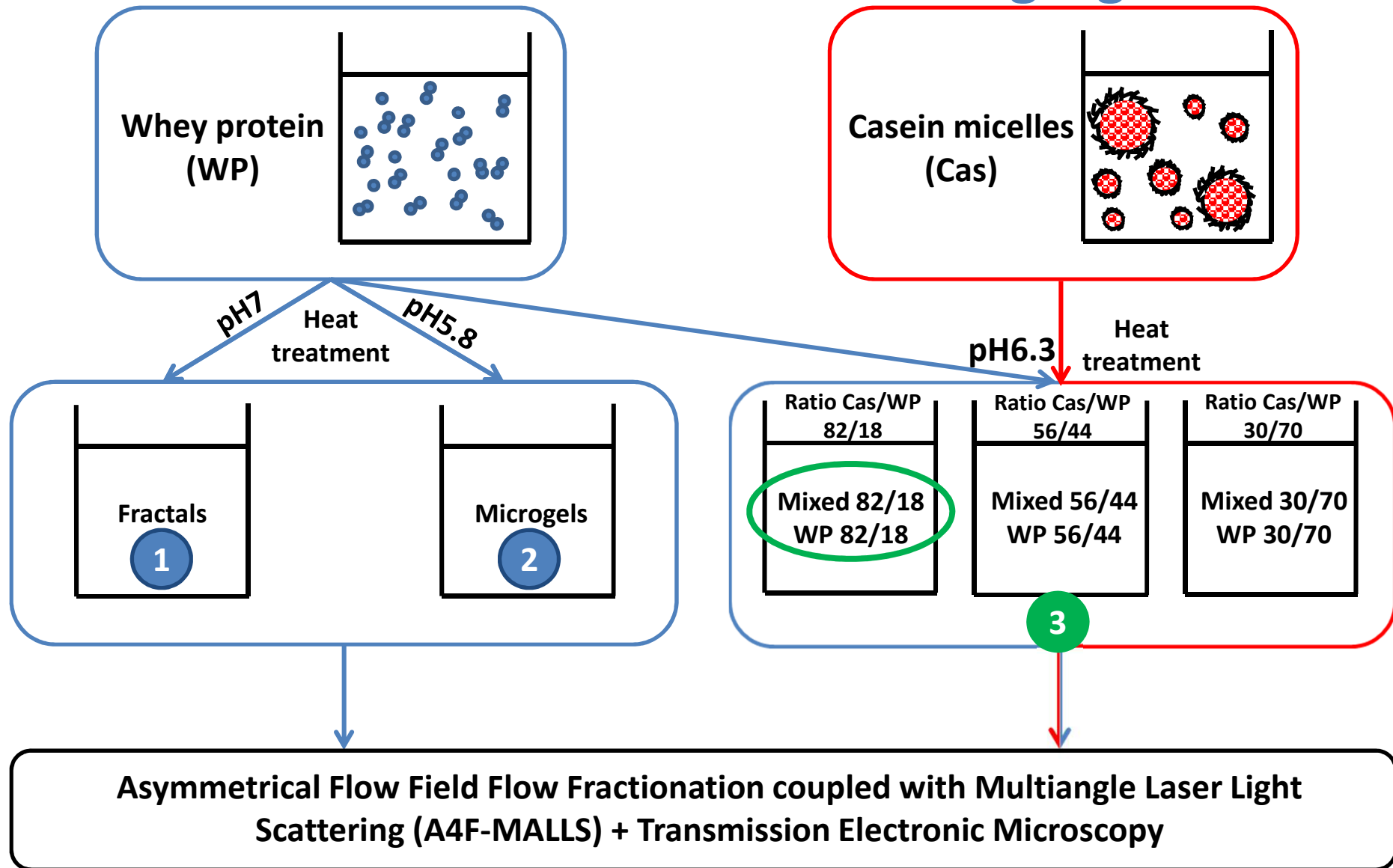


$M_W = 8.4 \cdot 10^7 \text{ g.mol}^{-1}$

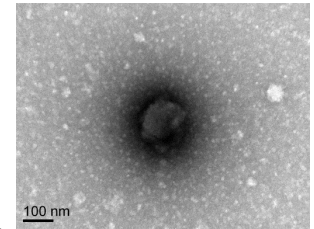
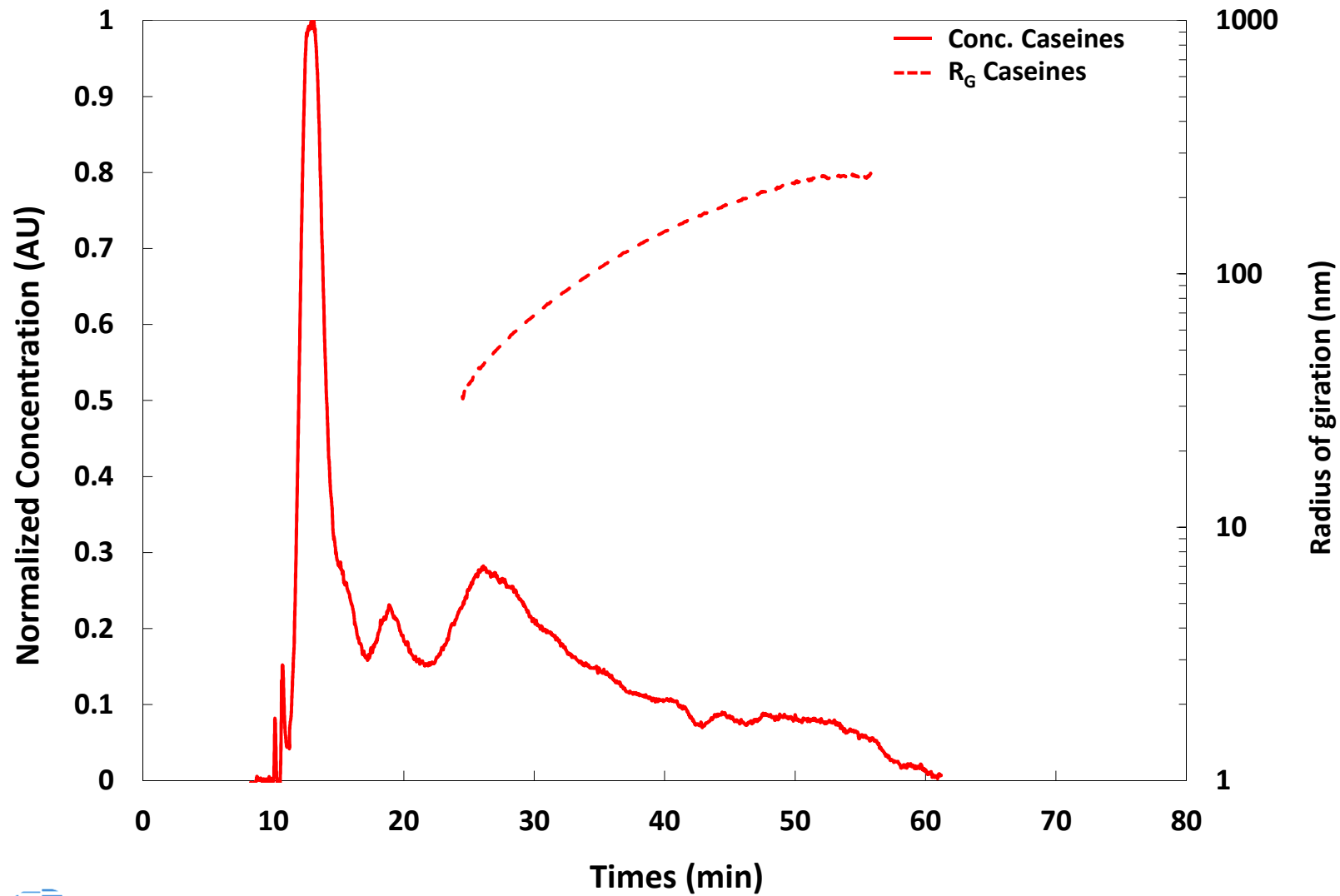


$M_W = 5.5 \cdot 10^8 \text{ g.mol}^{-1}$

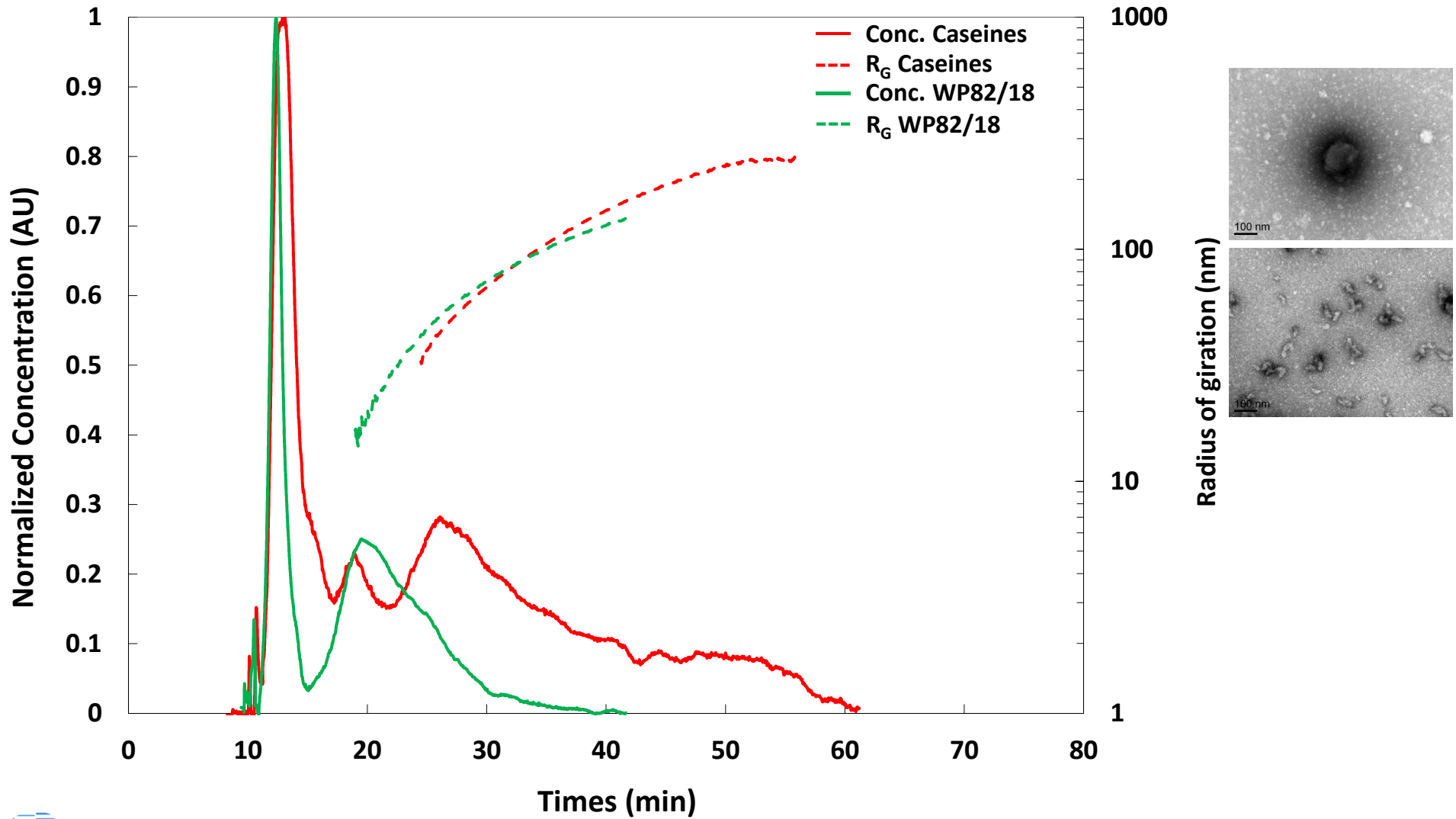
Méthodes de fabrication des agrégats



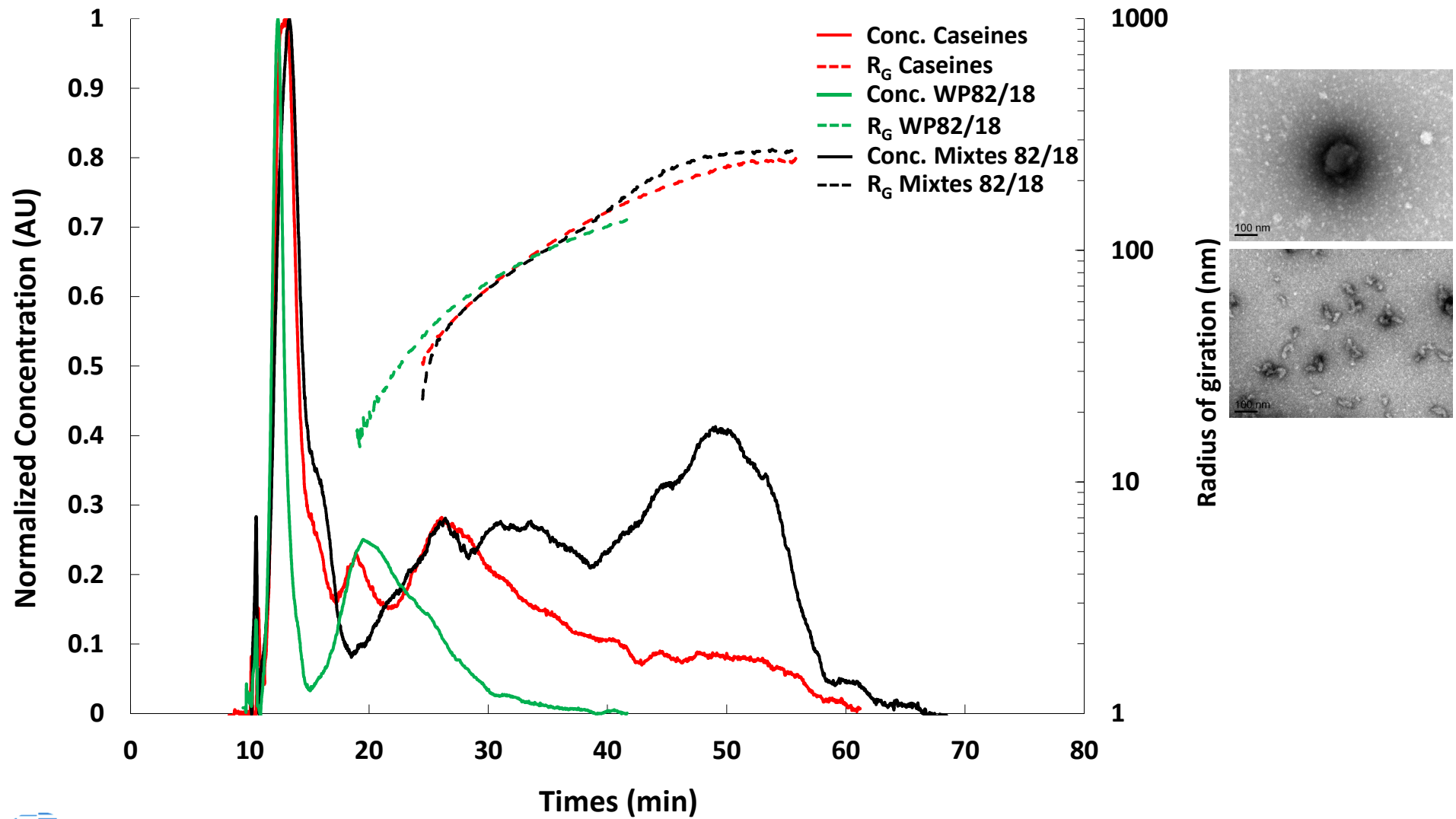
Mixtes 82/18



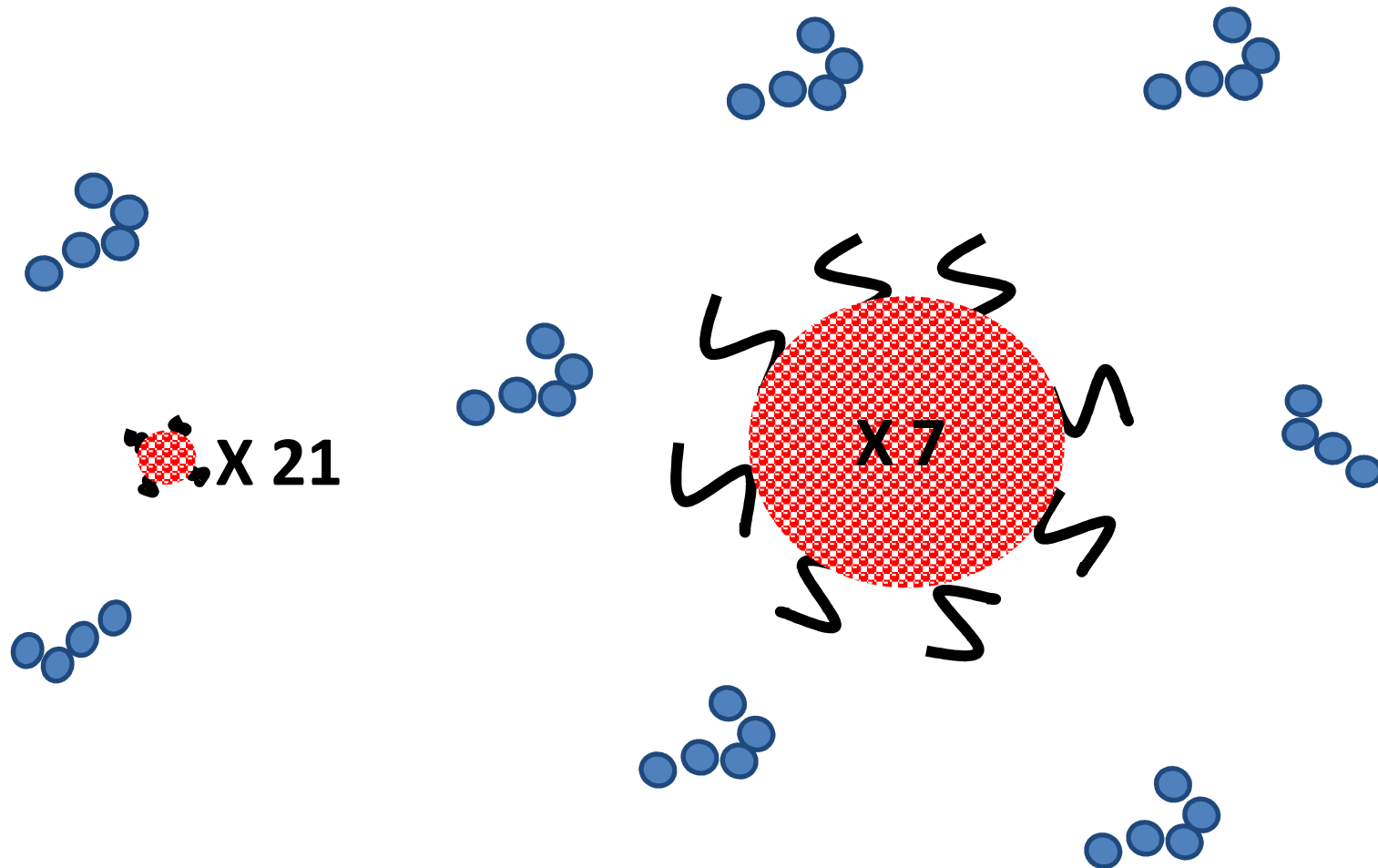
Mixtes 82/18



Mixtes 82/18



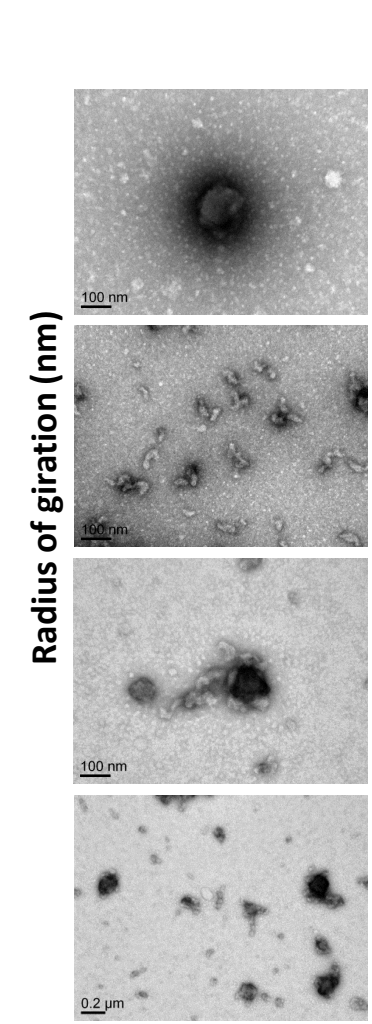
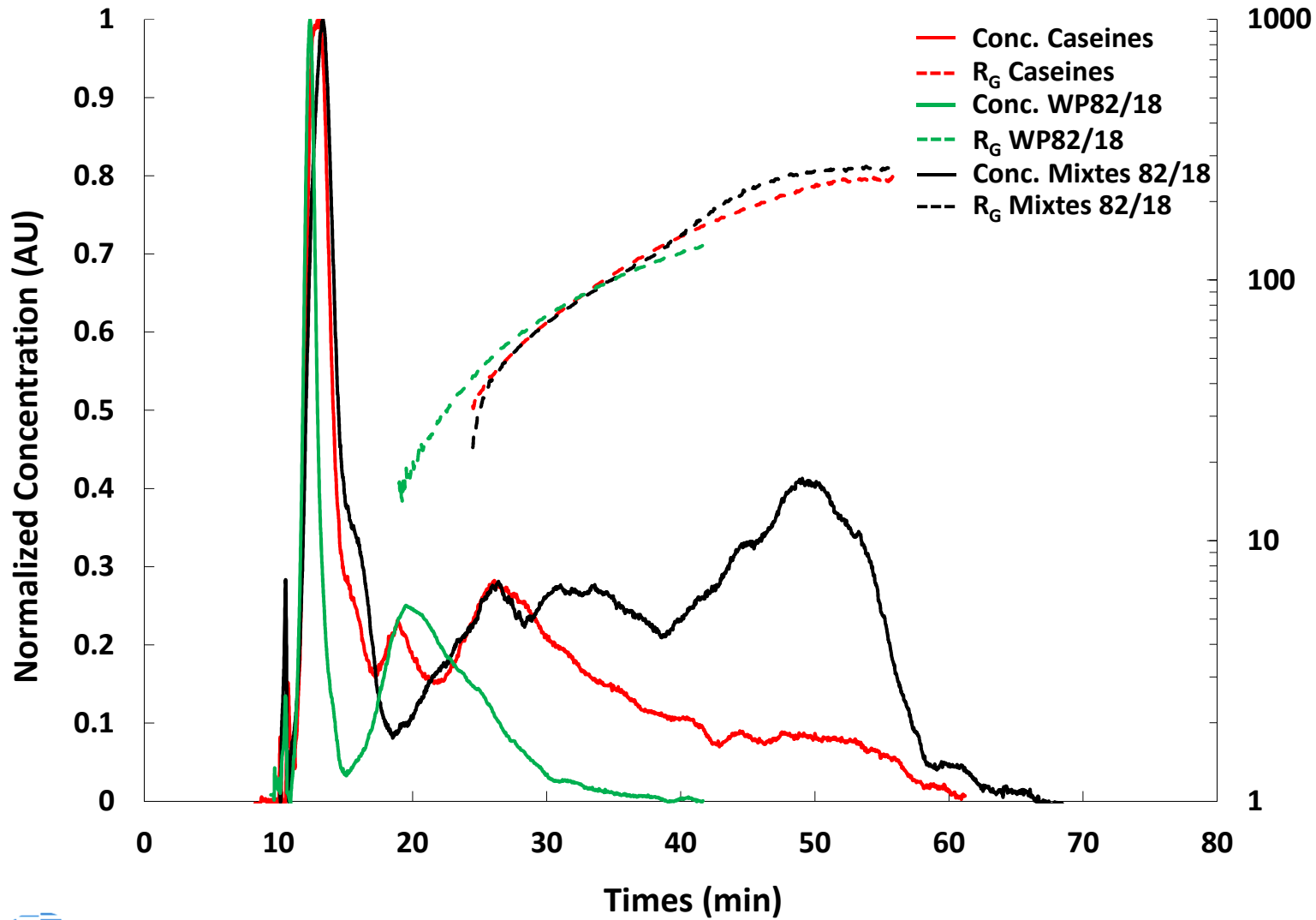
Mixtes 82/18



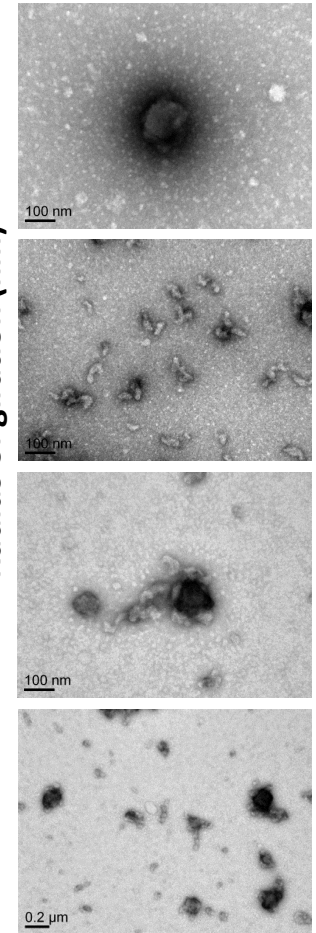
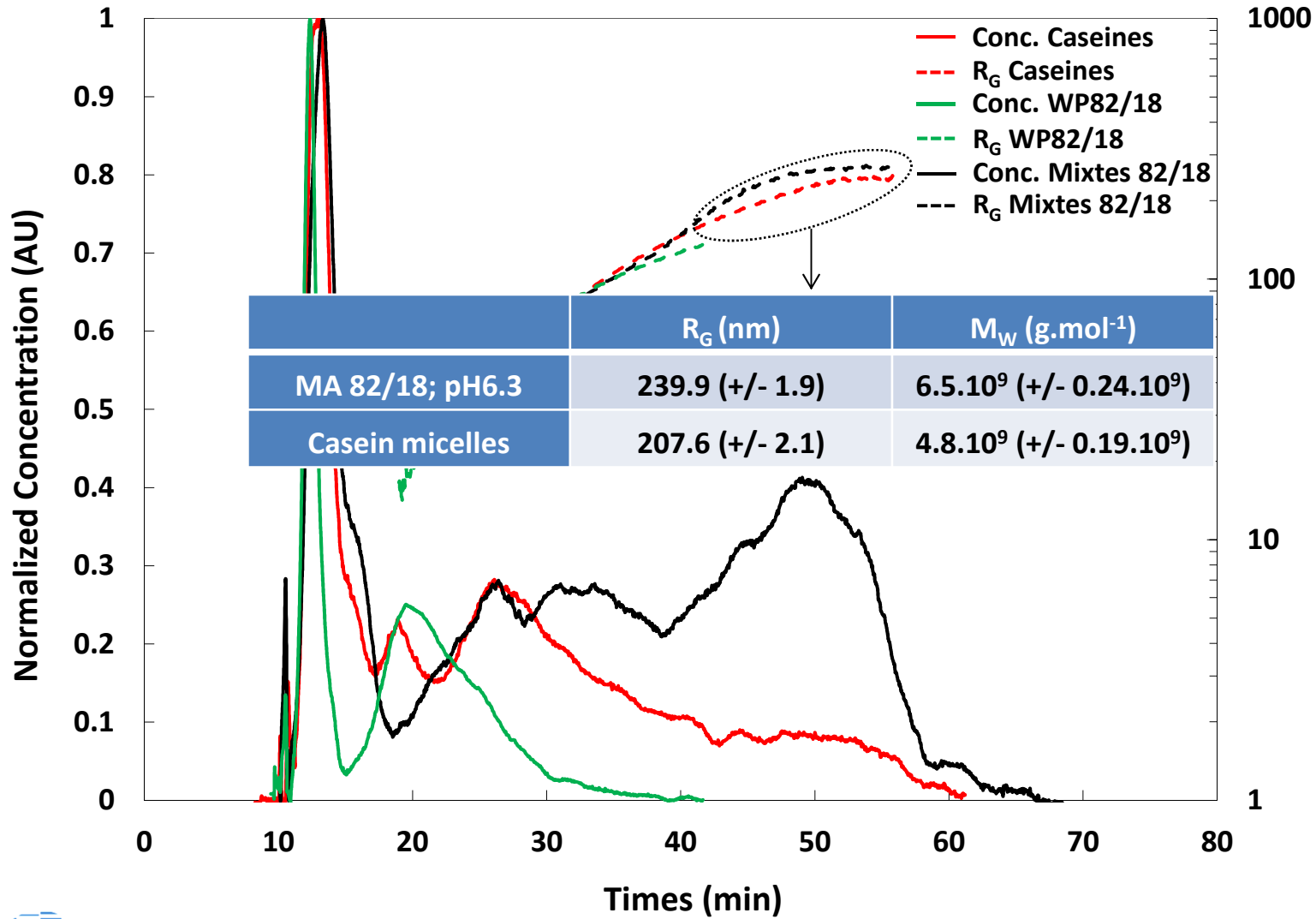
Pourquoi uniquement les micelles de grosses taille sont concernées?

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

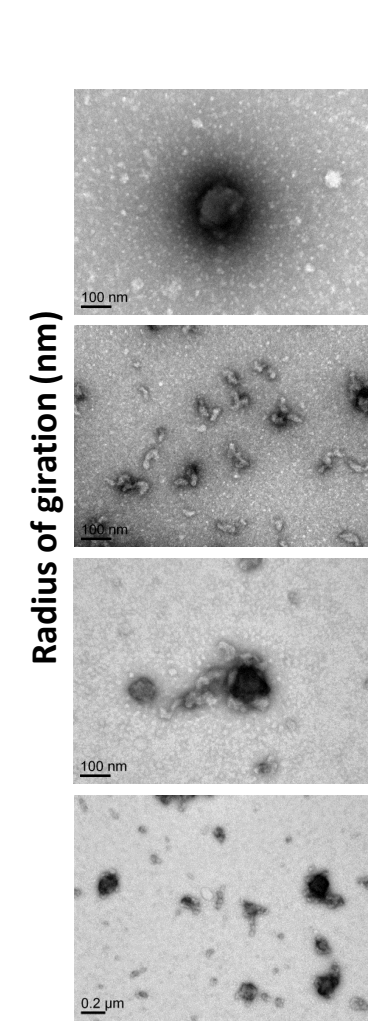
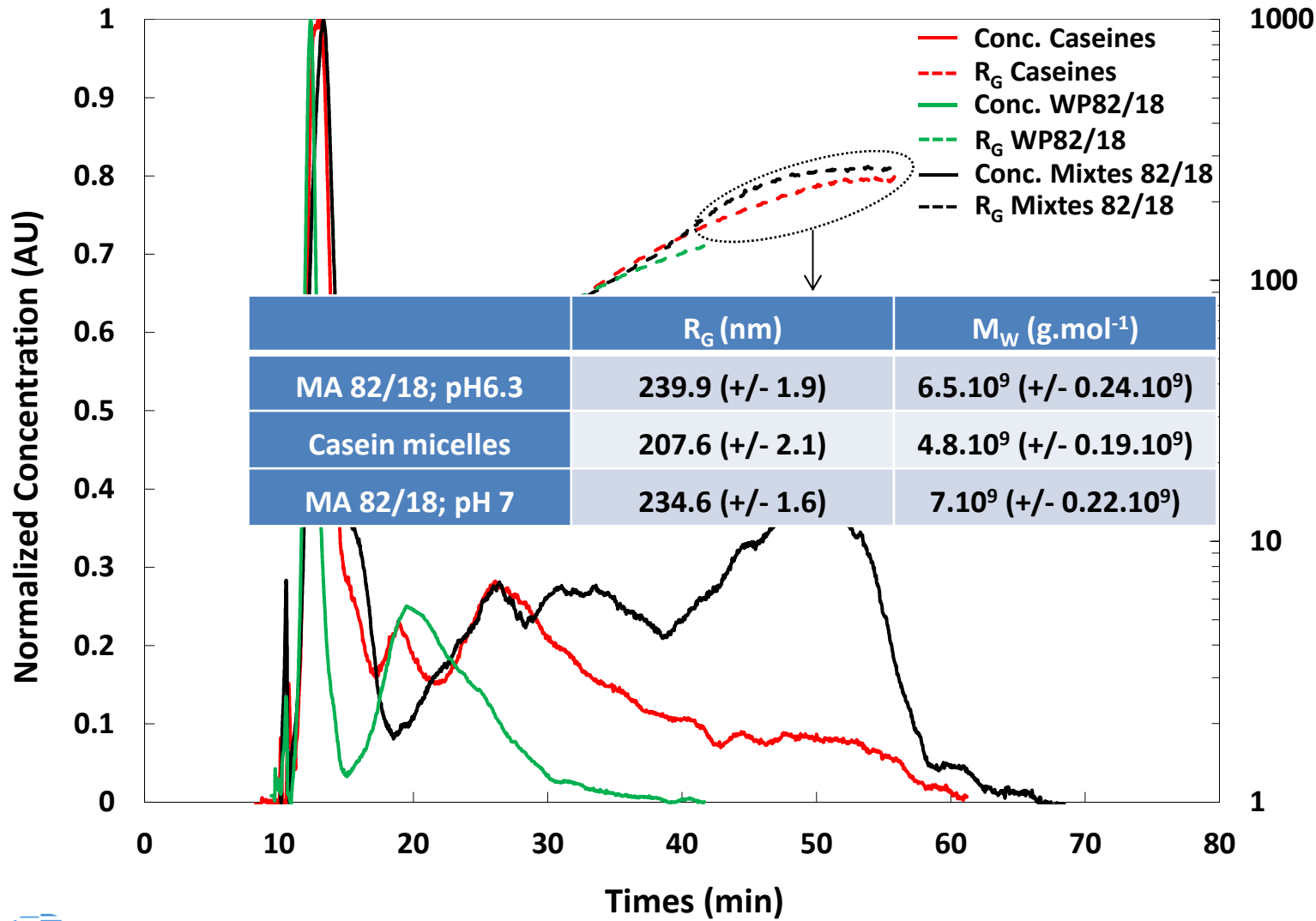
Mixtes 82/18



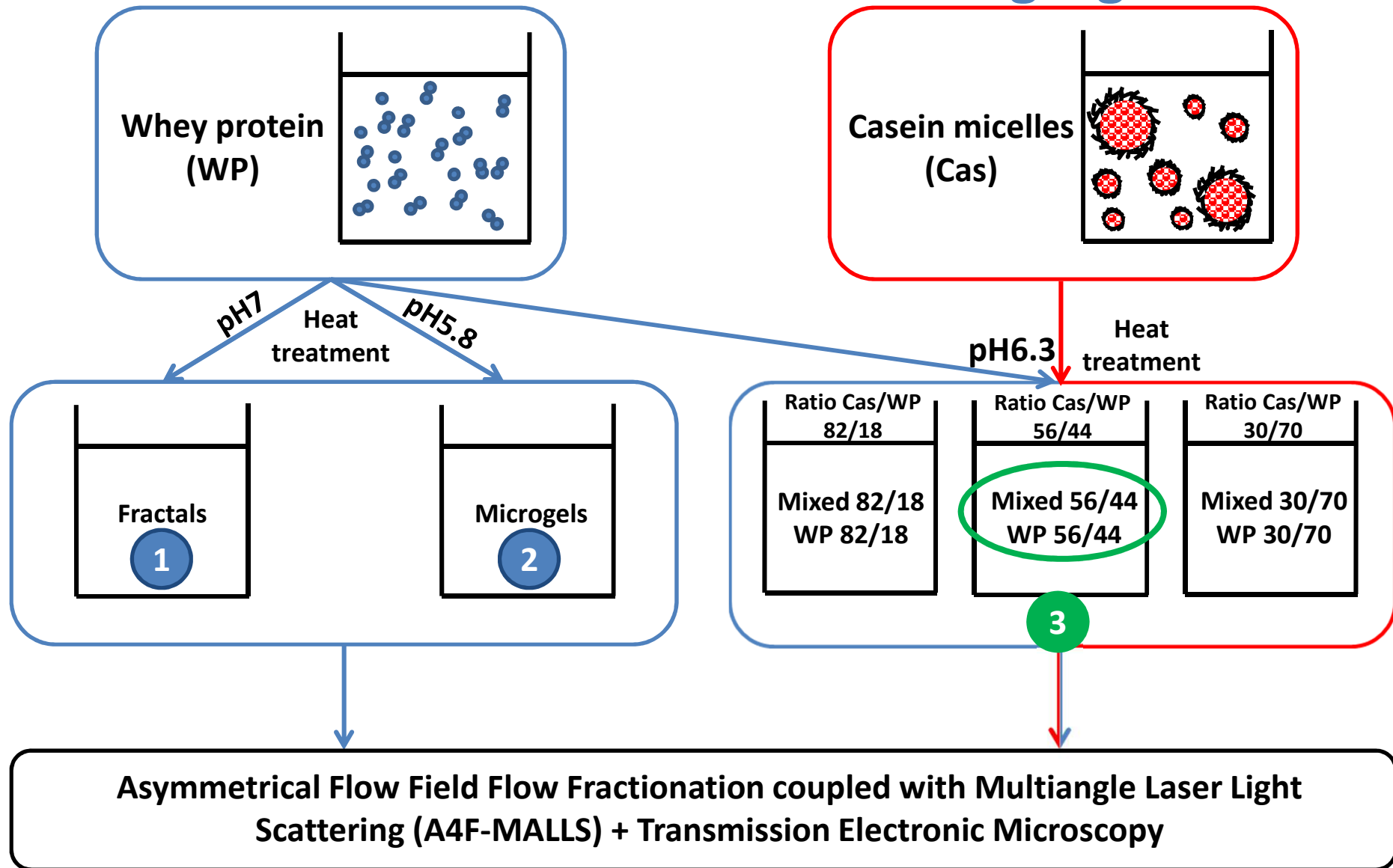
Mixtes 82/18



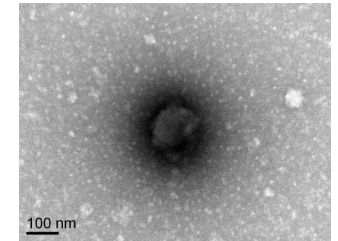
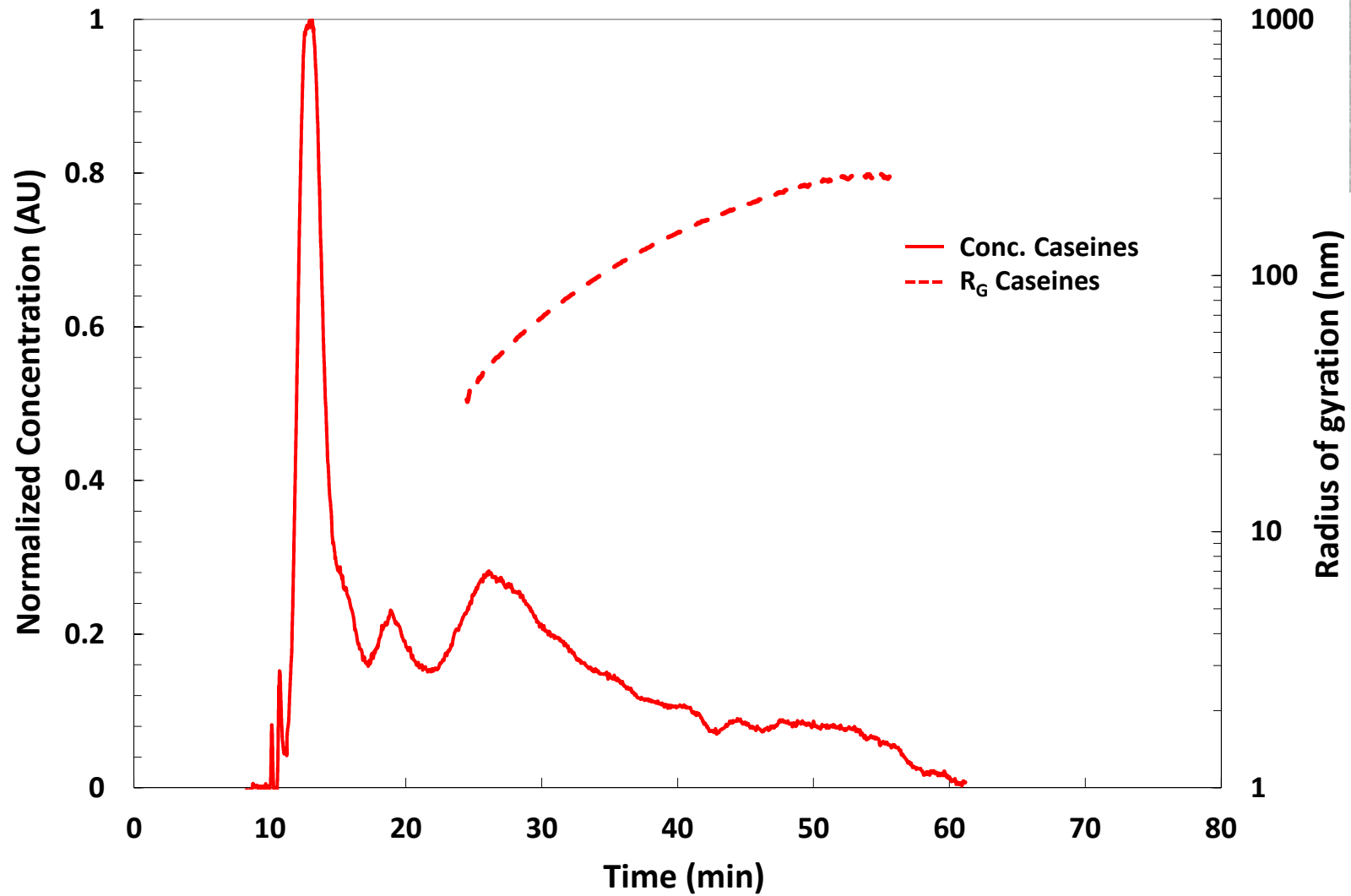
Mixtes 82/18



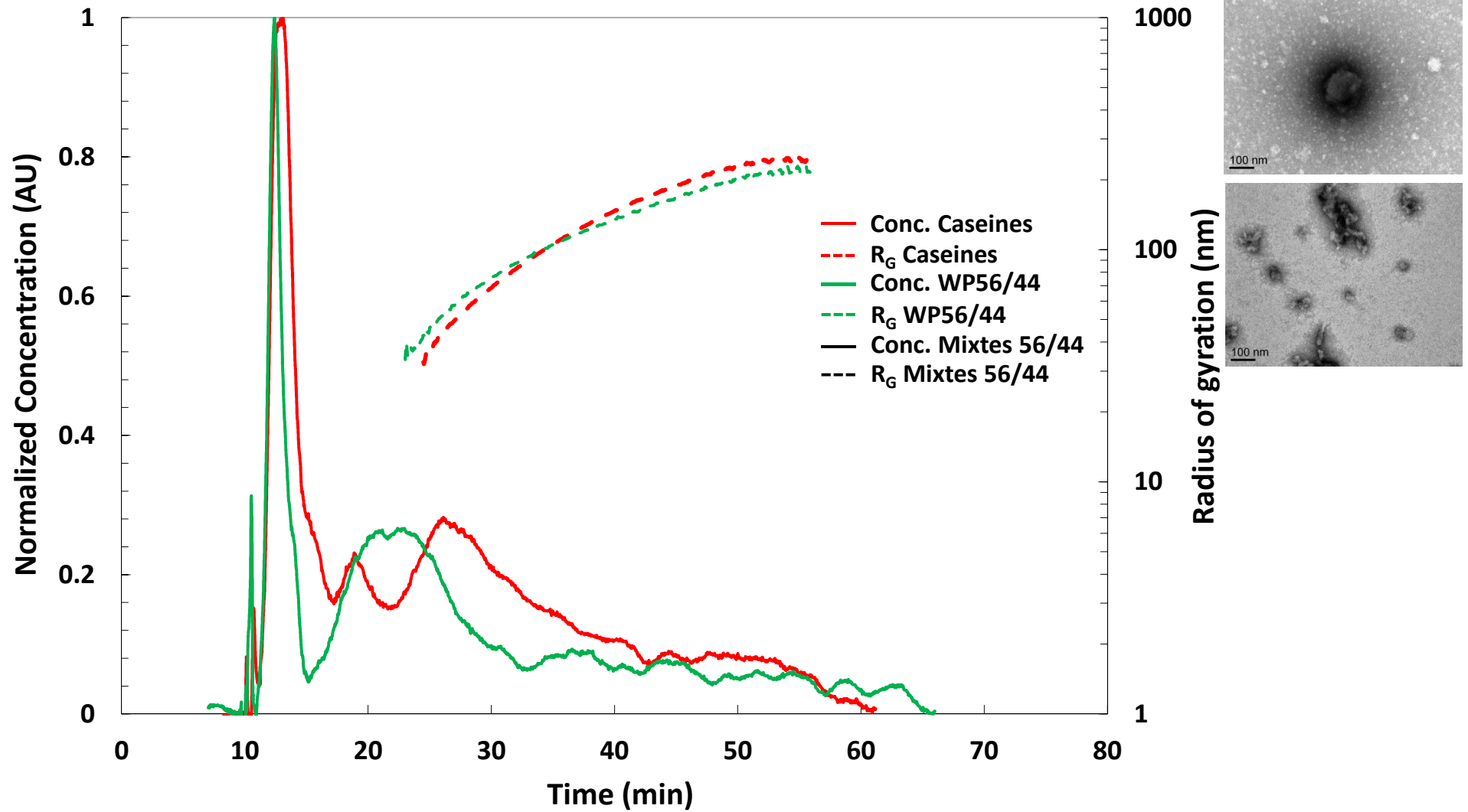
Méthodes de fabrication des agrégats



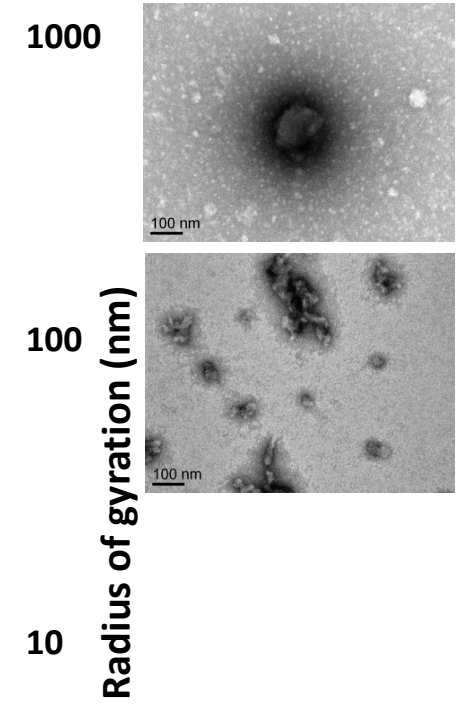
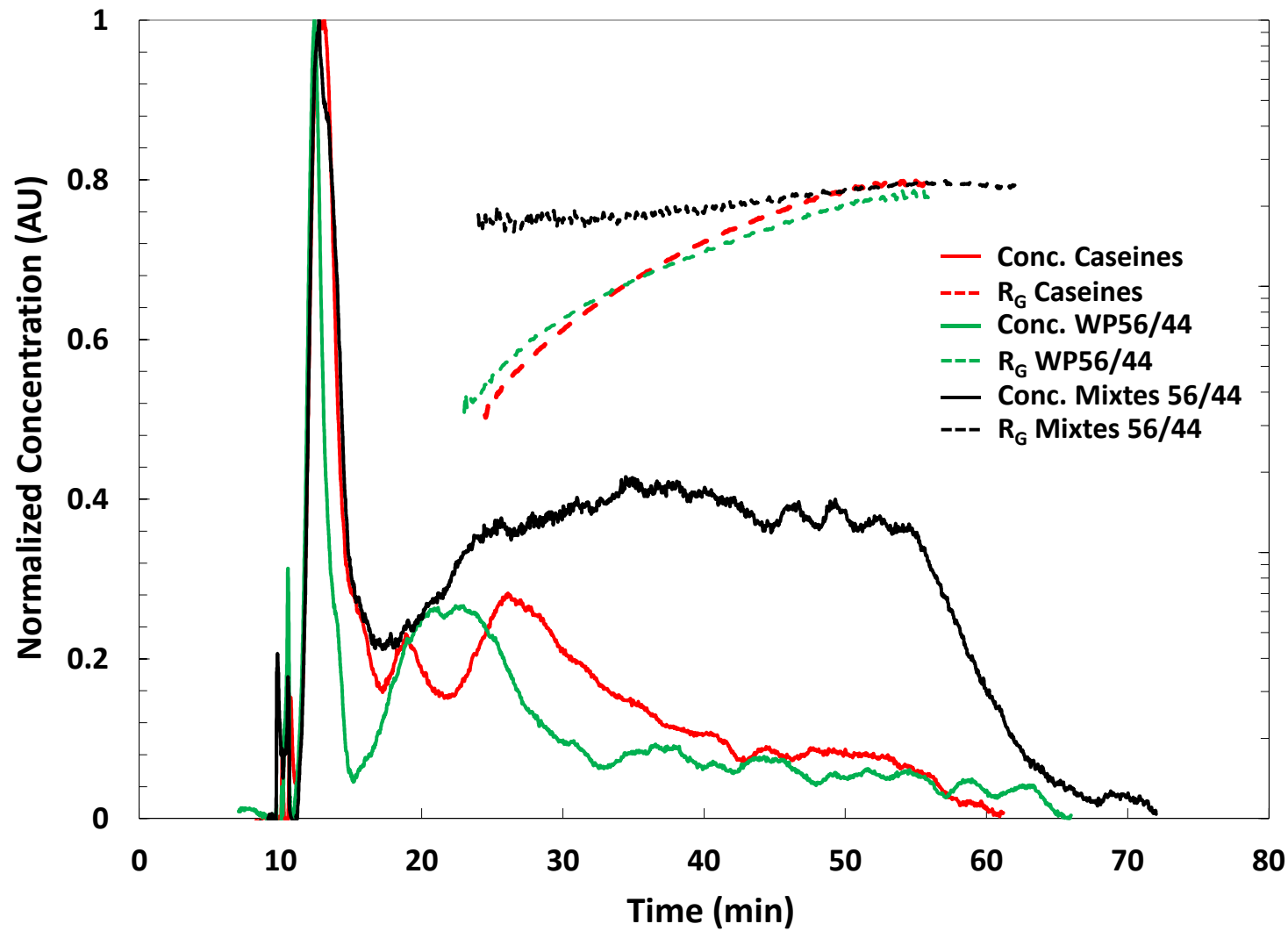
Mixtes 56/44



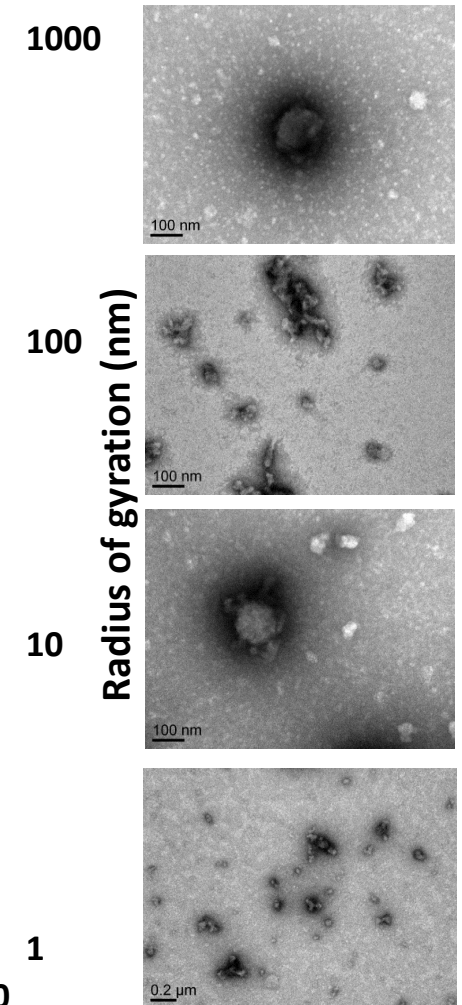
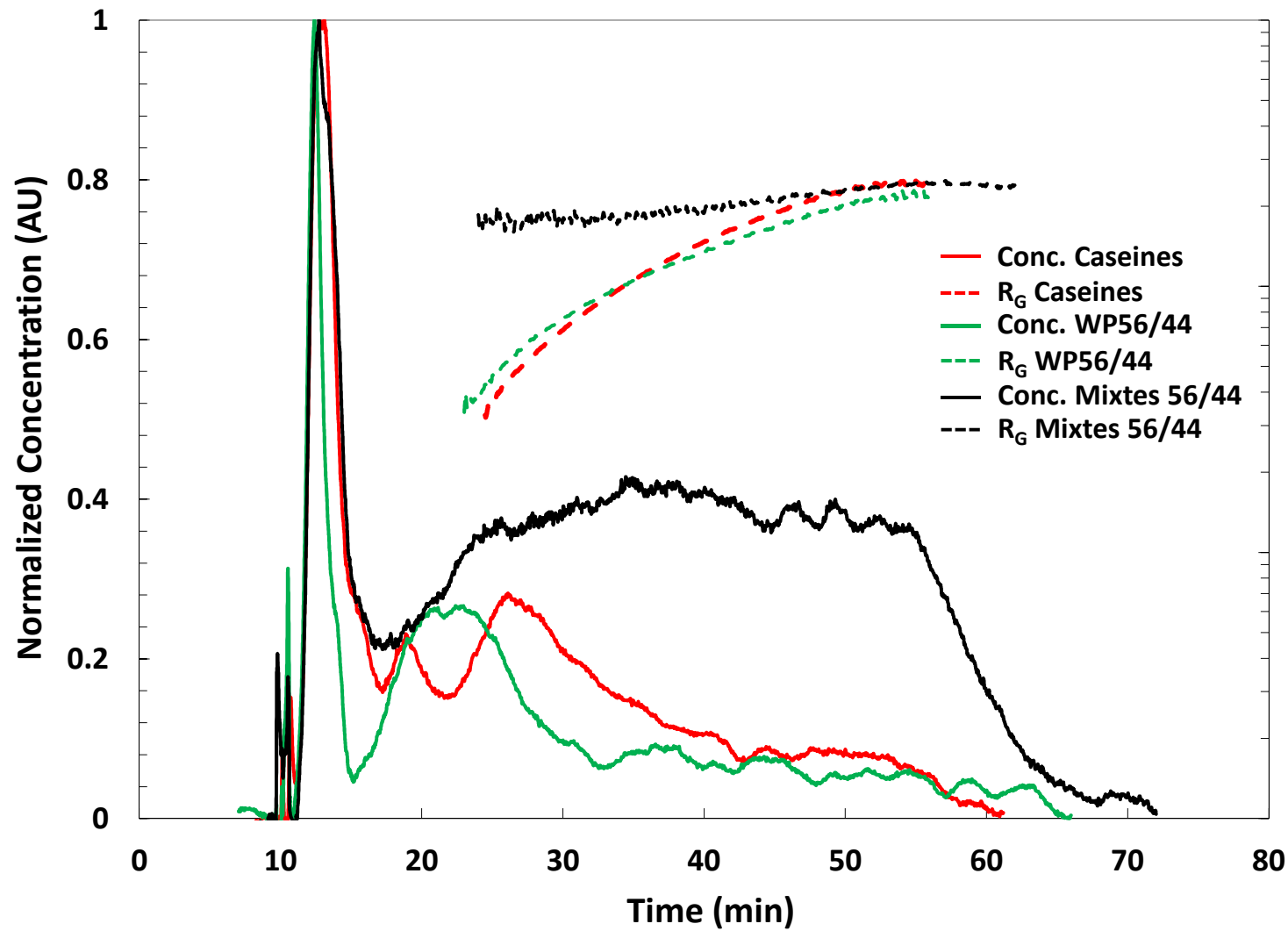
Mixtes 56/44



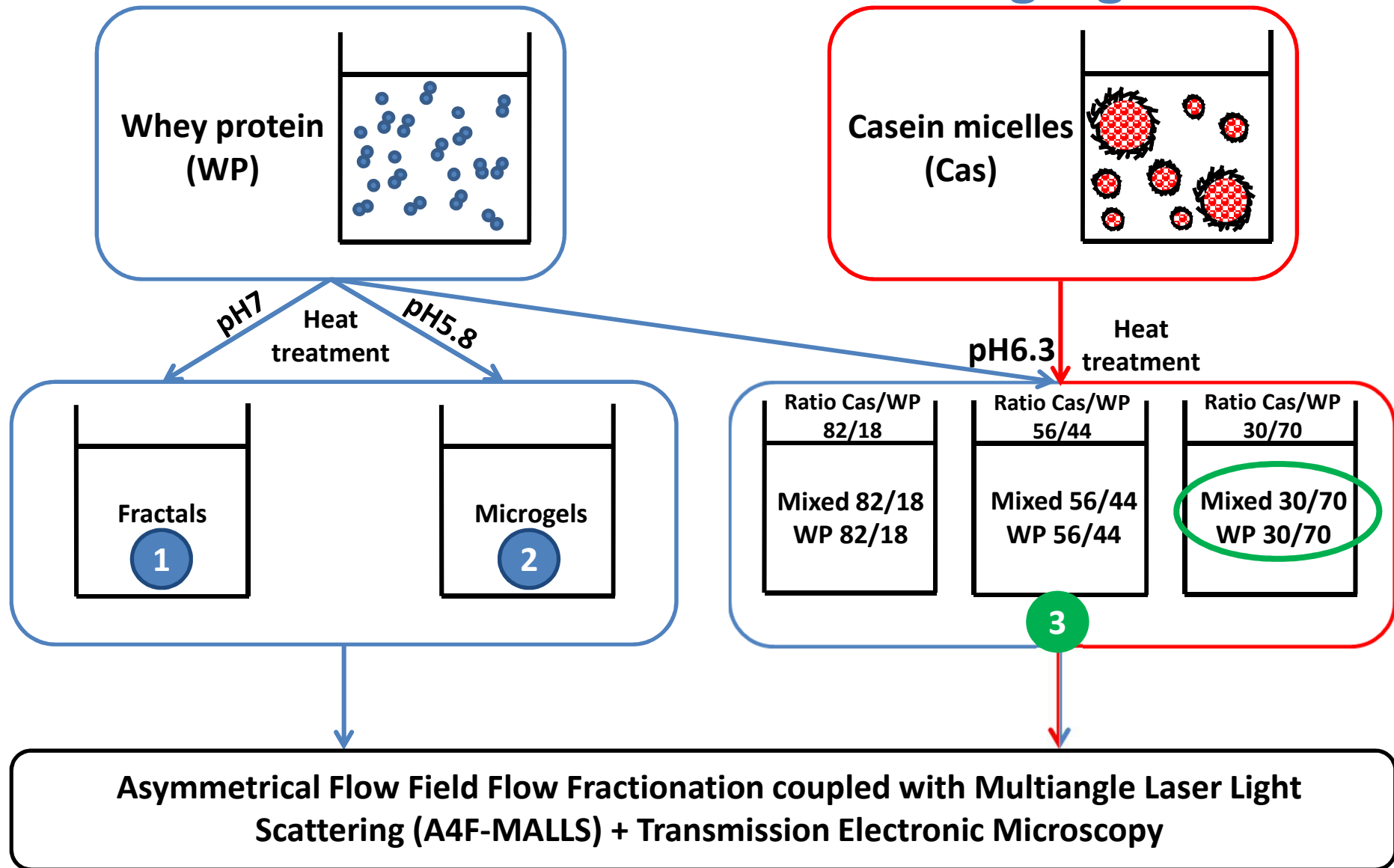
Mixtes 56/44



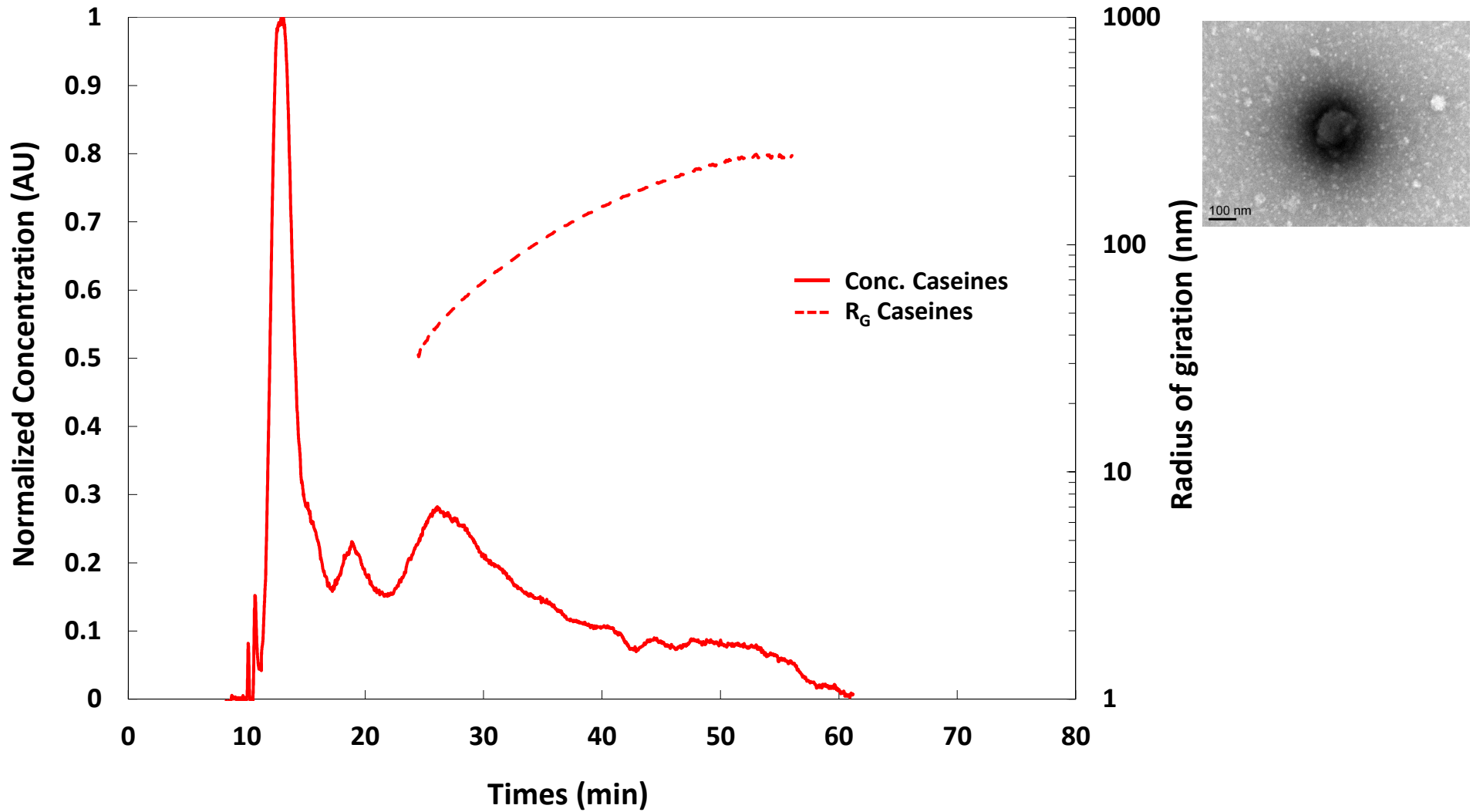
Mixtes 56/44



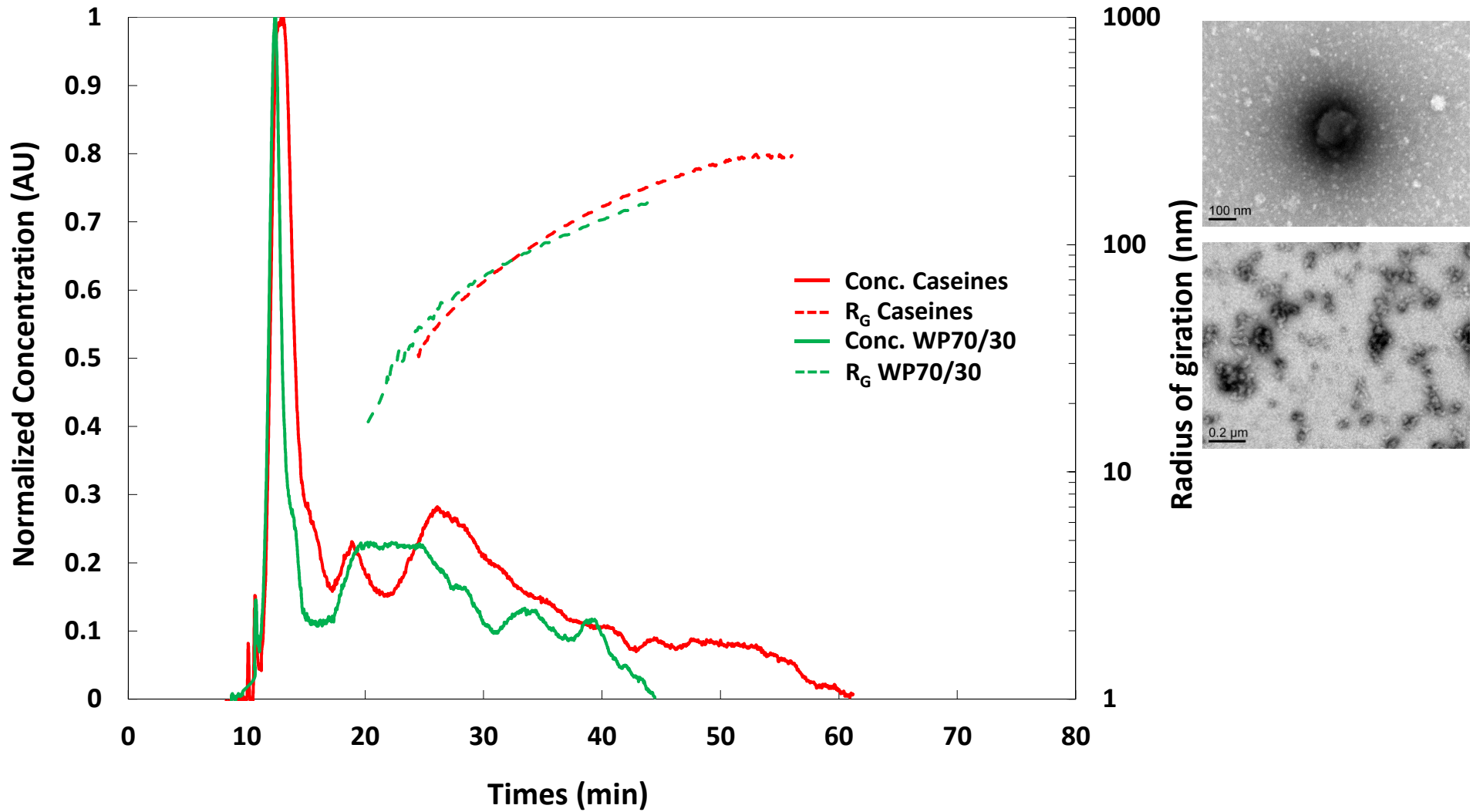
Méthodes de fabrication des agrégats



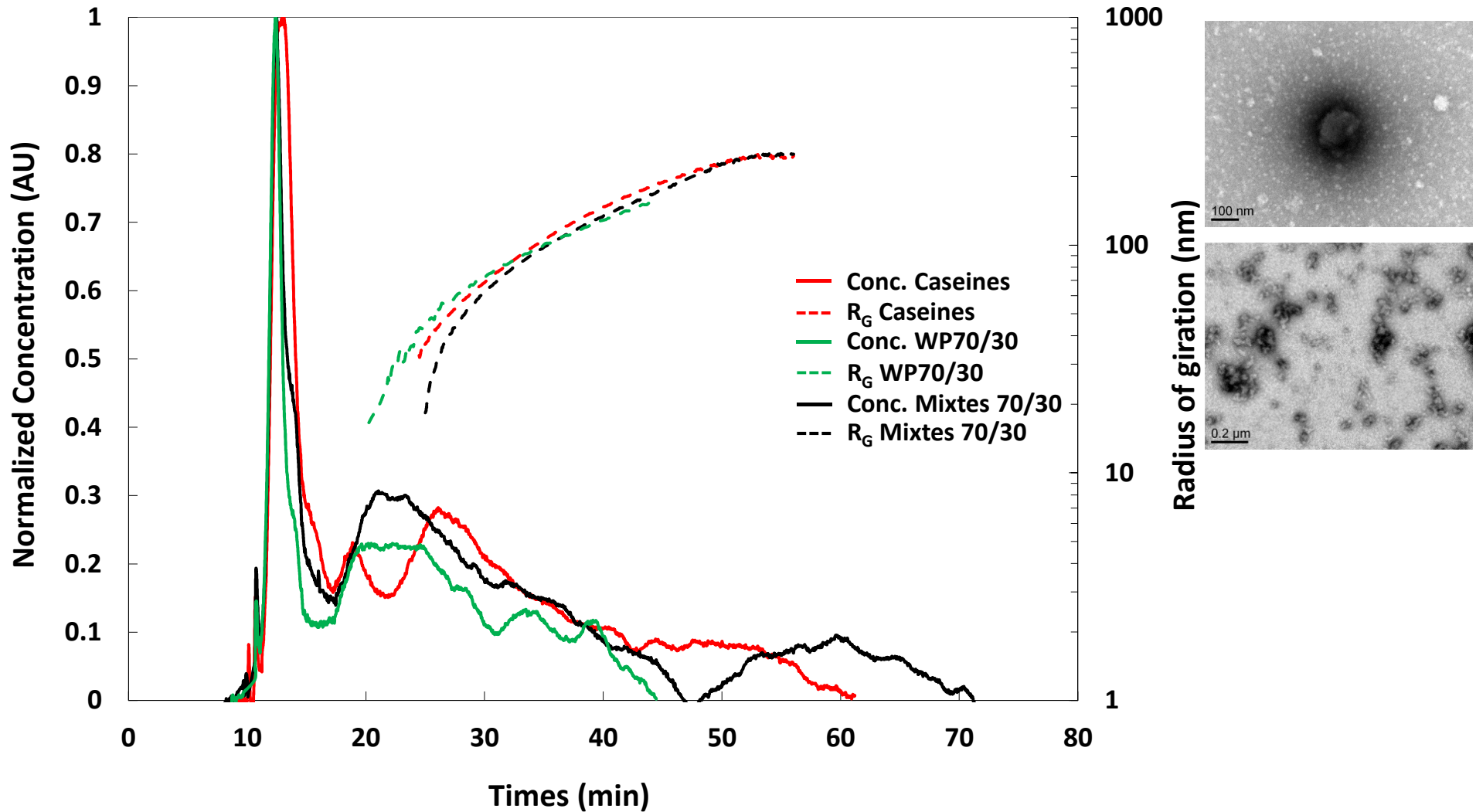
Mixtes 30/70



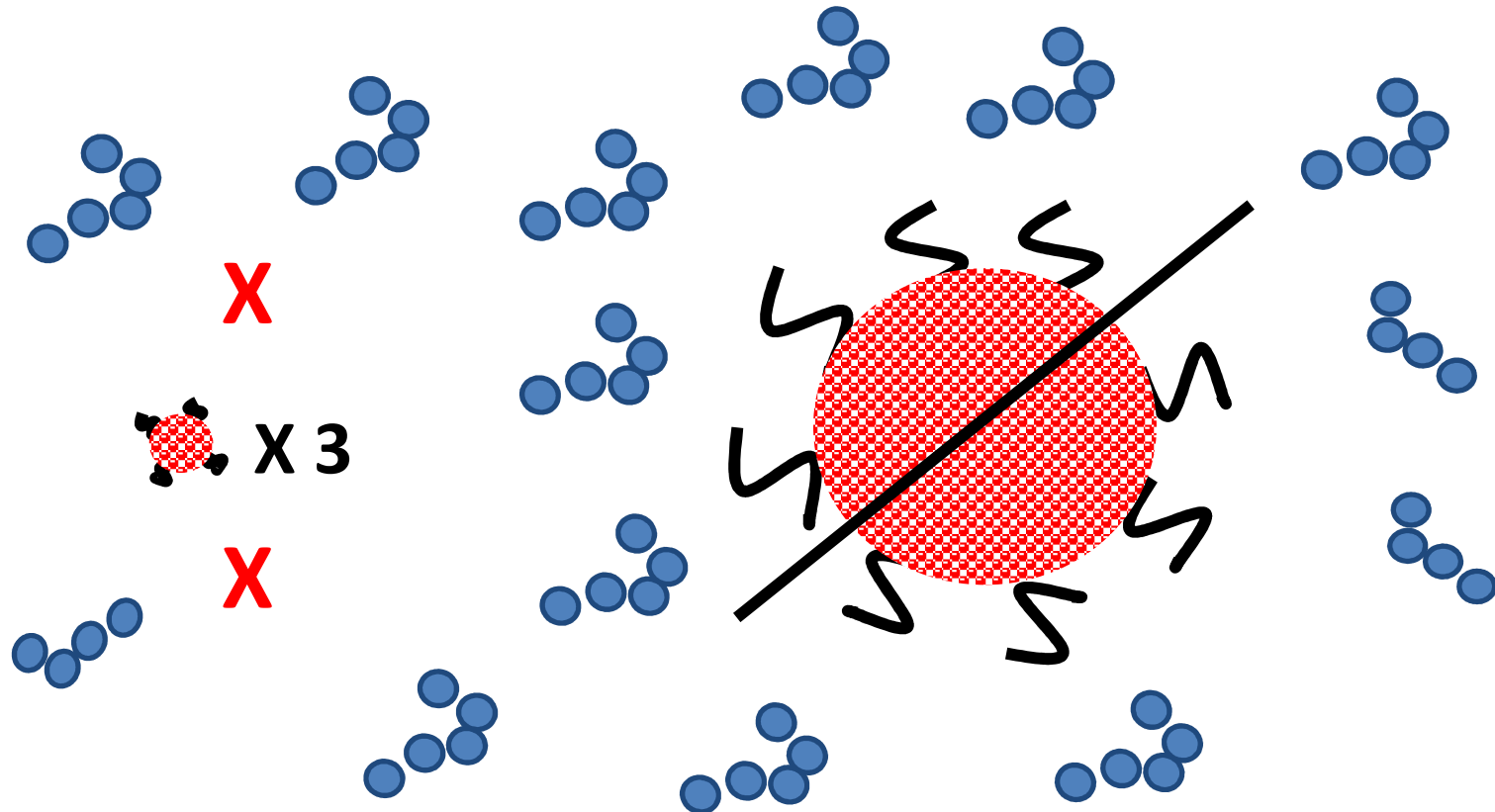
Mixtes 30/70



Mixtes 30/70



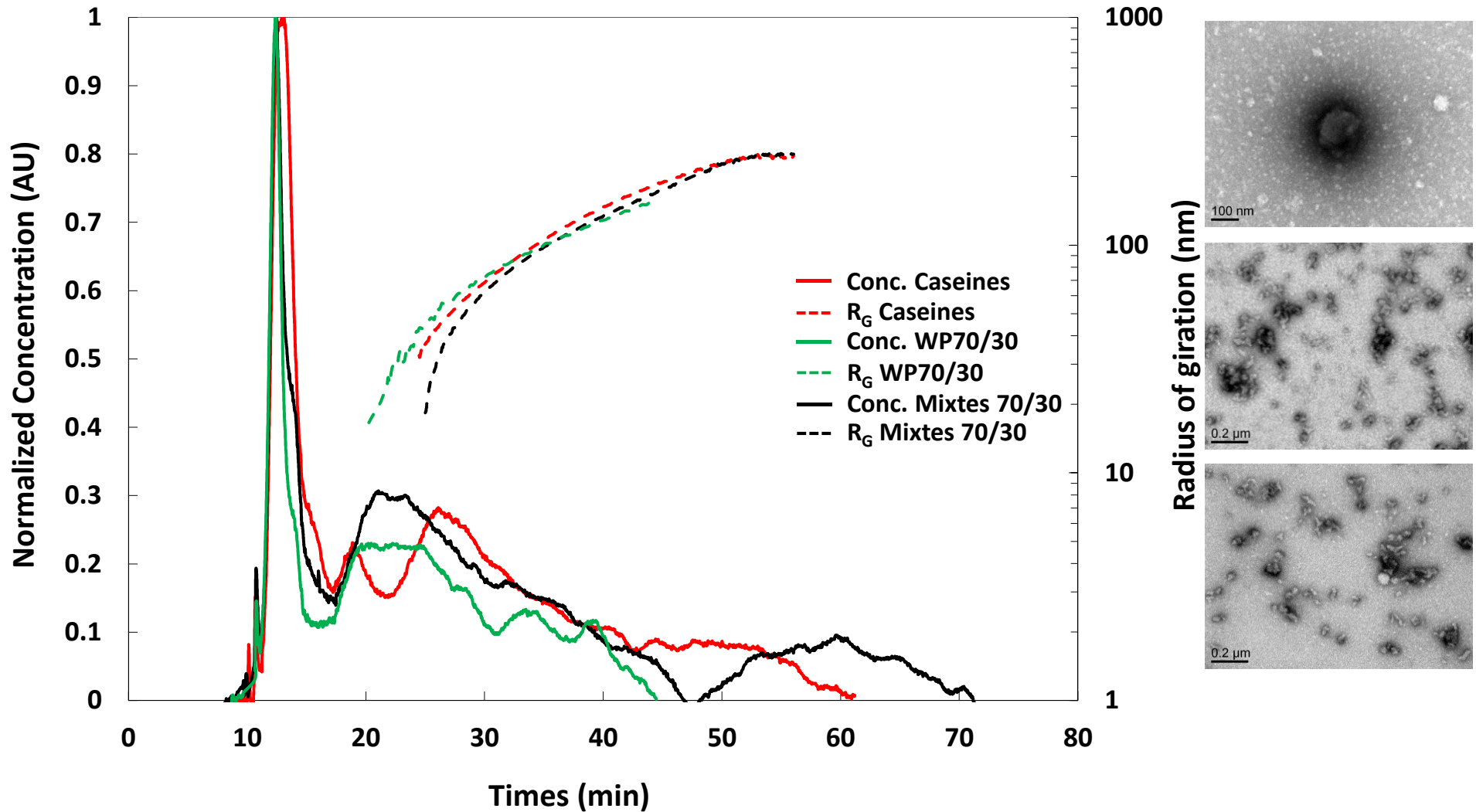
Mixtes 30/70



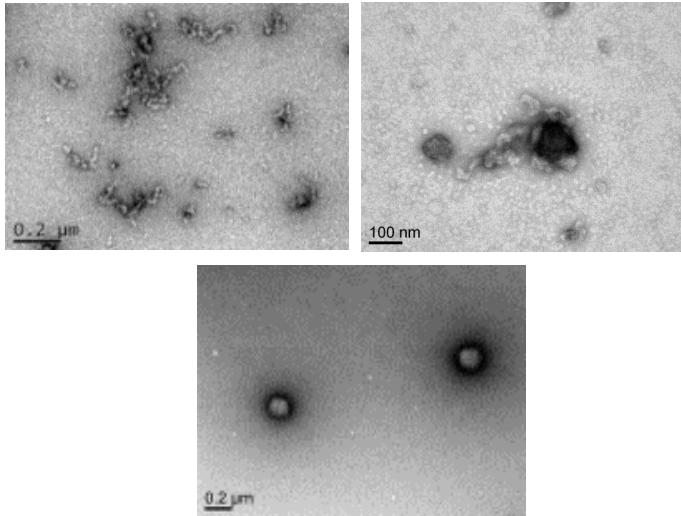
Pourquoi le profil des mixtes 30/70 et des fractals sont similaires?

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

Mixtes 30/70



Conclusion



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



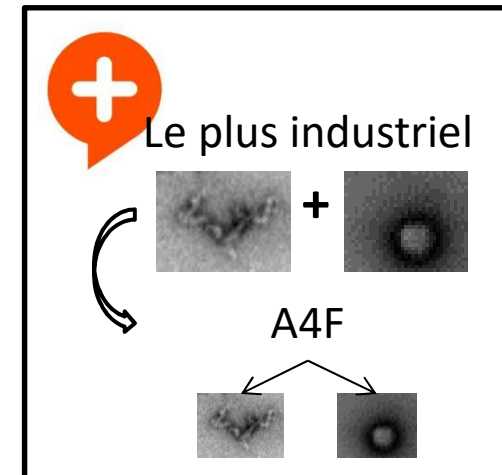
**Asymmetrical
Flow Field
Flow
Fractionation**

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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