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# Understanding of casein micelles concentrated layer properties during cross flow ultrafiltration by *in-situ* Small Angle X-ray scattering (SAXS)

Floriane Doudières<sup>(1,2)</sup>, M. Loginov<sup>(1)</sup>, N. Hengl<sup>(2)</sup>, F. Garnier-Lambrouin<sup>(1)</sup>, N. Leconte<sup>(1)</sup>, J. Perez<sup>(3)</sup>, L. Sharpnack<sup>(4)</sup>, F. Pignon<sup>(2)</sup>, G. Gésan-Guiziou<sup>(1)</sup>

(1) UMR1253 STLO, Science and Technology of Milk and Egg, INRA, Agrocampus Ouest, Rennes, France; (2) Univ. Grenoble Alpes, CNRS, Grenoble INP, LRP, Laboratoire Rhéologie et procédés, Grenoble, France; (3) Beamline SWING, Synchrotron SOLEIL, Gif-sur-Yvette, France; (4) Beamline ID02, European Synchrotron Radiation Facility ESRF, Grenoble, France

## Context

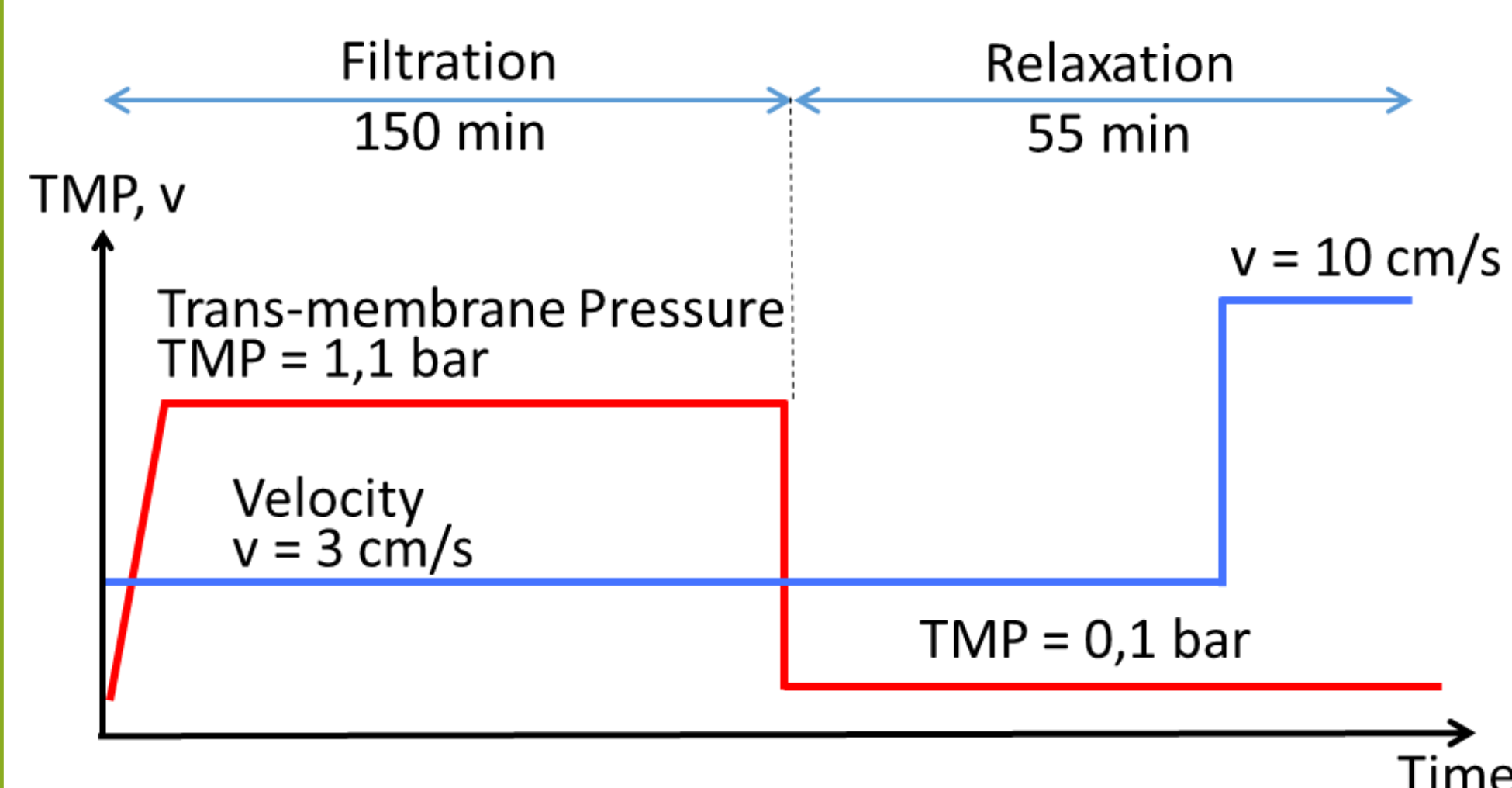
Micro- and ultrafiltration of skimmed milk are largely used in the dairy sector for protein fractionation and concentration. The performance of these operations (permeation flux, selectivity) are ruled by the formation of concentrated protein layers at membrane surface.

- The objective of this study was to understand the structural organization and behaviour of concentrated casein micelles accumulated at the membrane surface during cross-flow ultrafiltration.
- We analysed fouling layer development during filtration step under several operating conditions and redispersion during pressure relaxation step. We focused on the effect of temperature (12, 25 & 42°C).

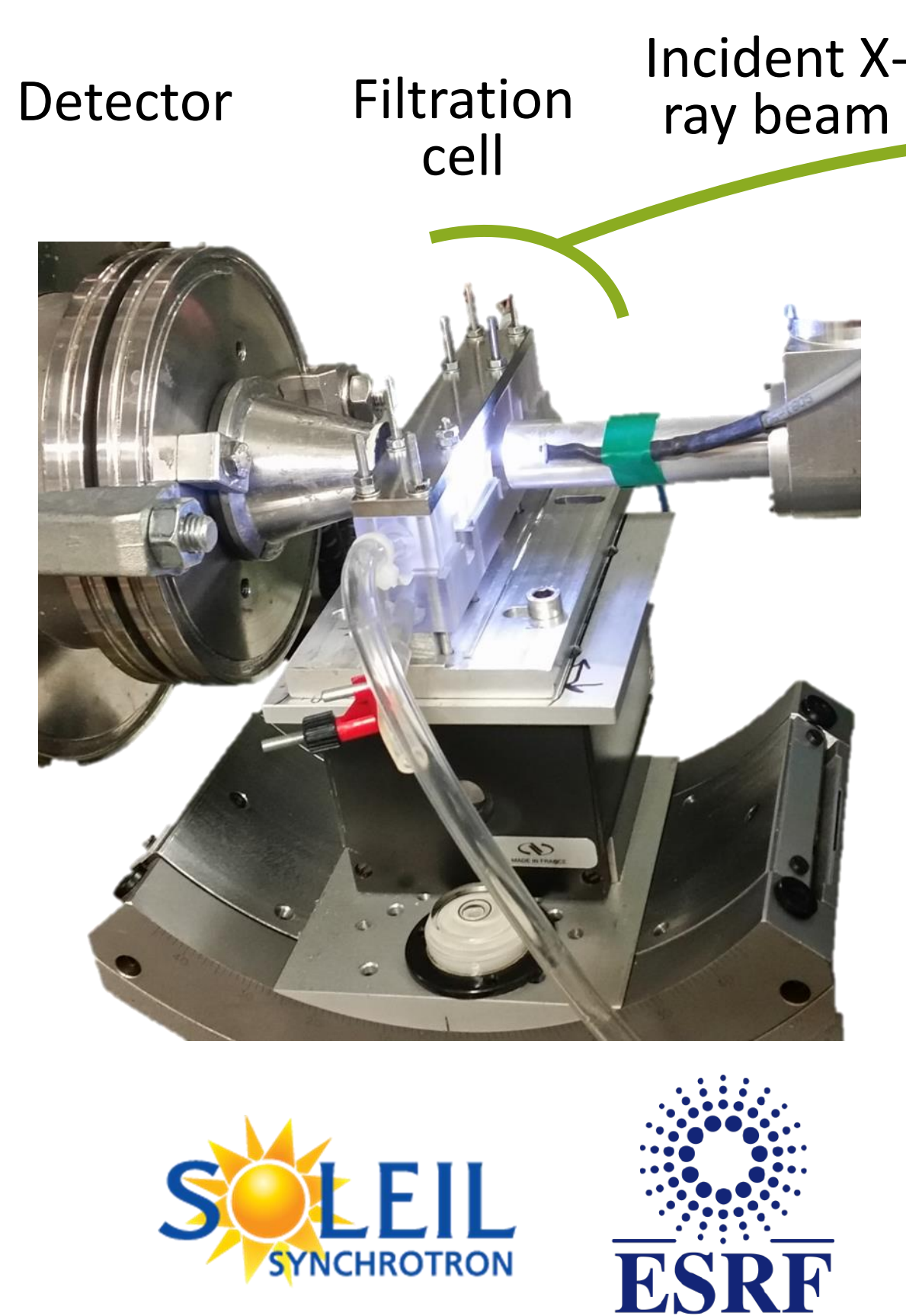
## Materials & Methods

### 1 Filtration and redispersion protocol

- Native casein micelles (native phosphocaseinate) powder dispersed in milk ultrafiltrate at ~50g/L
- Polyethersulfone membrane 100kD

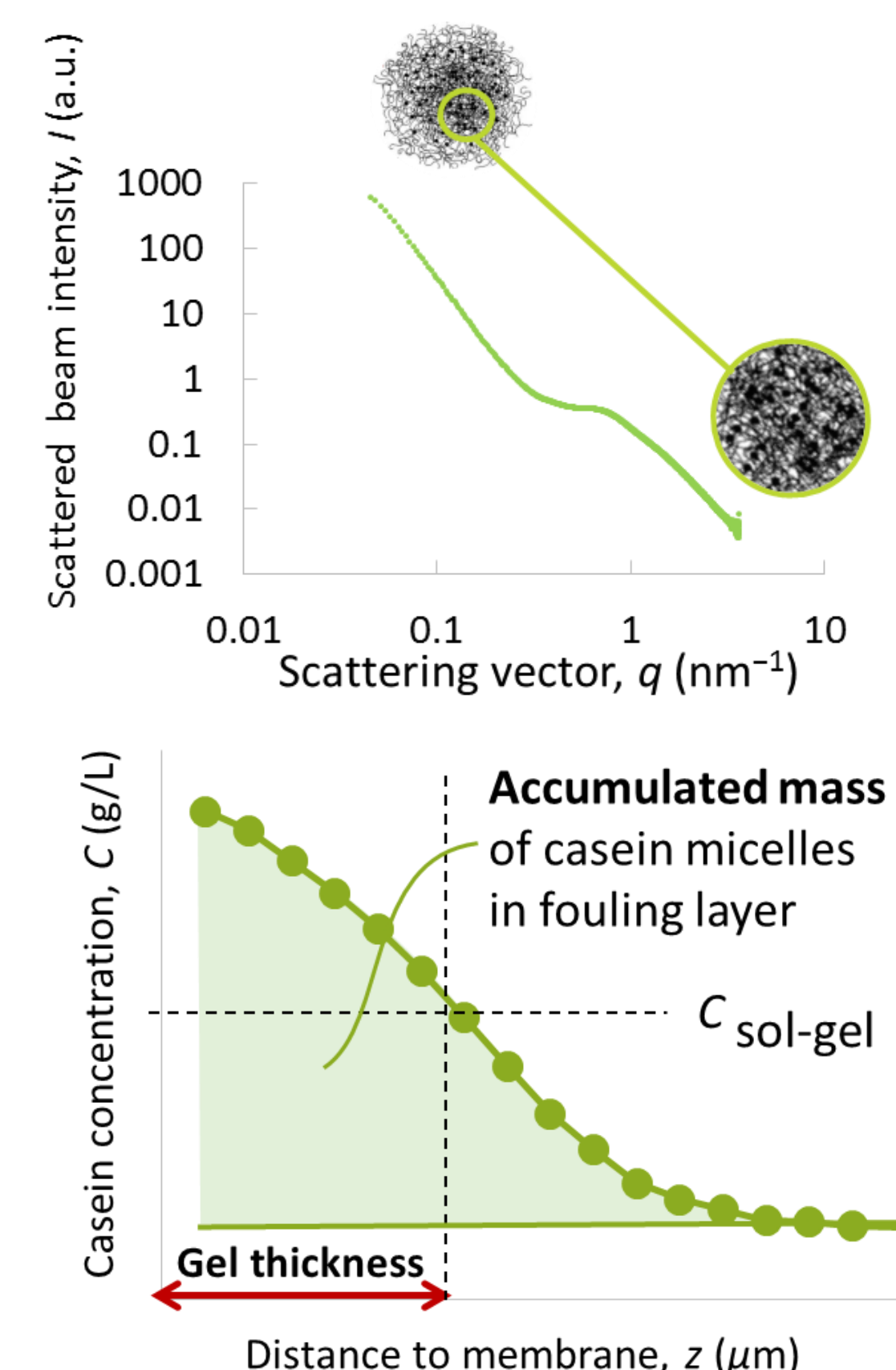


### 2 *In-situ* SAXS cross-flow filtration



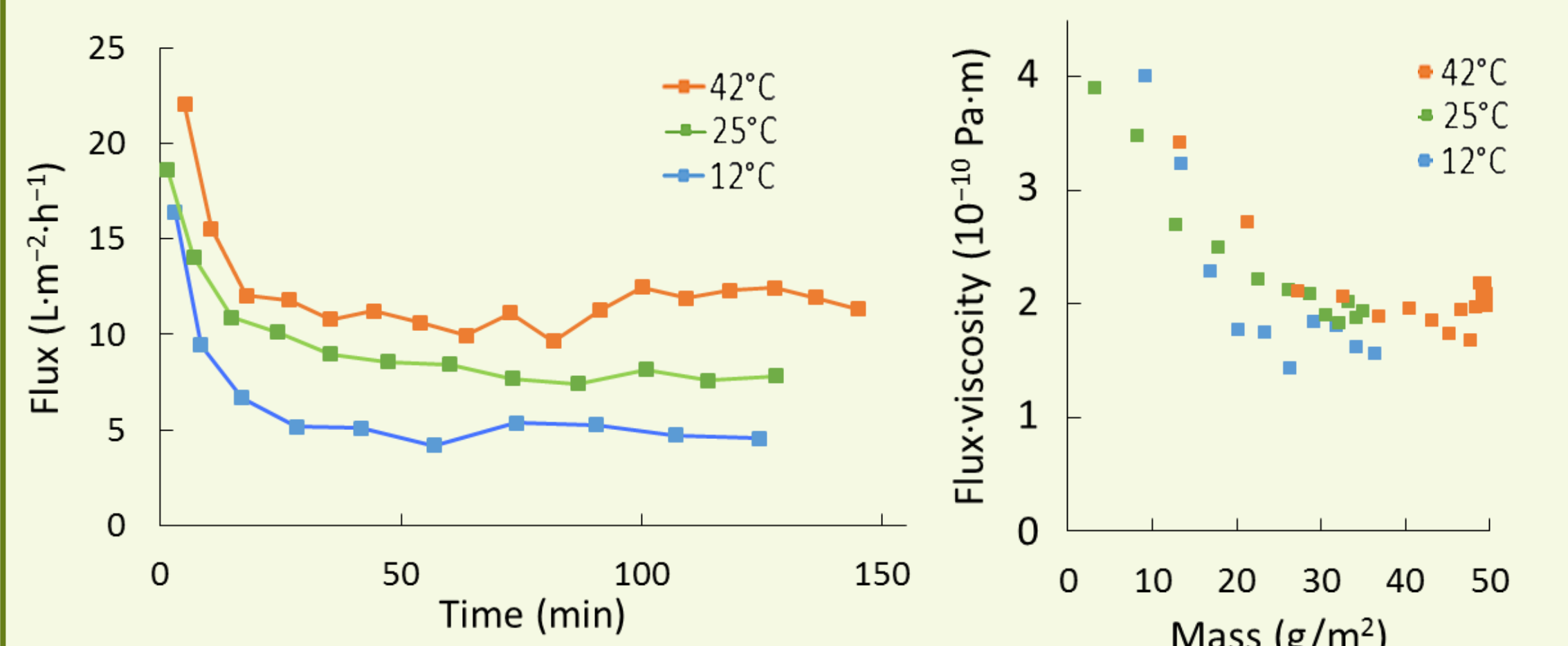
### 3 Side view of filtration channel

### 4 At each filtration time *t*, SAXS spectra & concentration profile



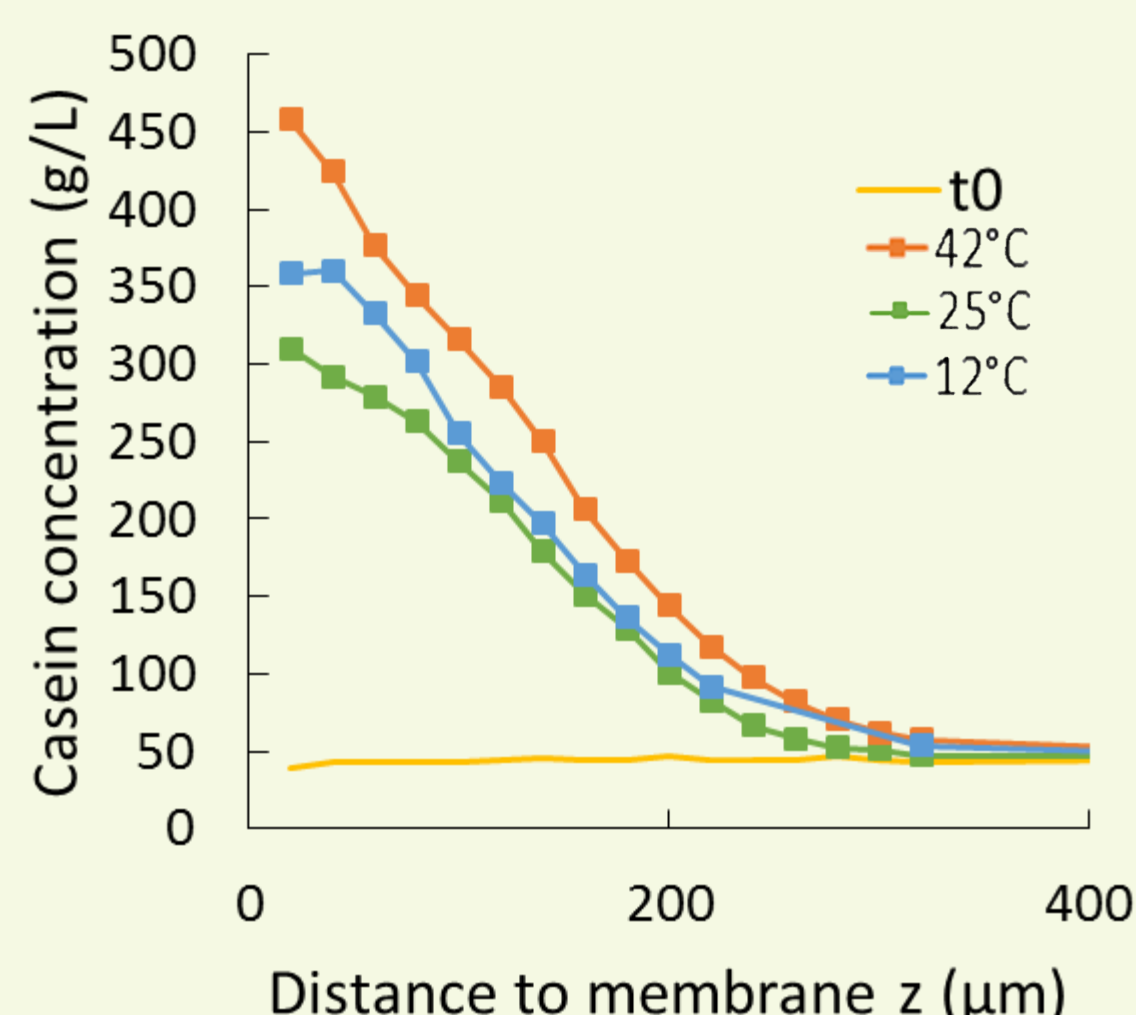
## Results

### Filtration step: Mass accumulation and gel formation

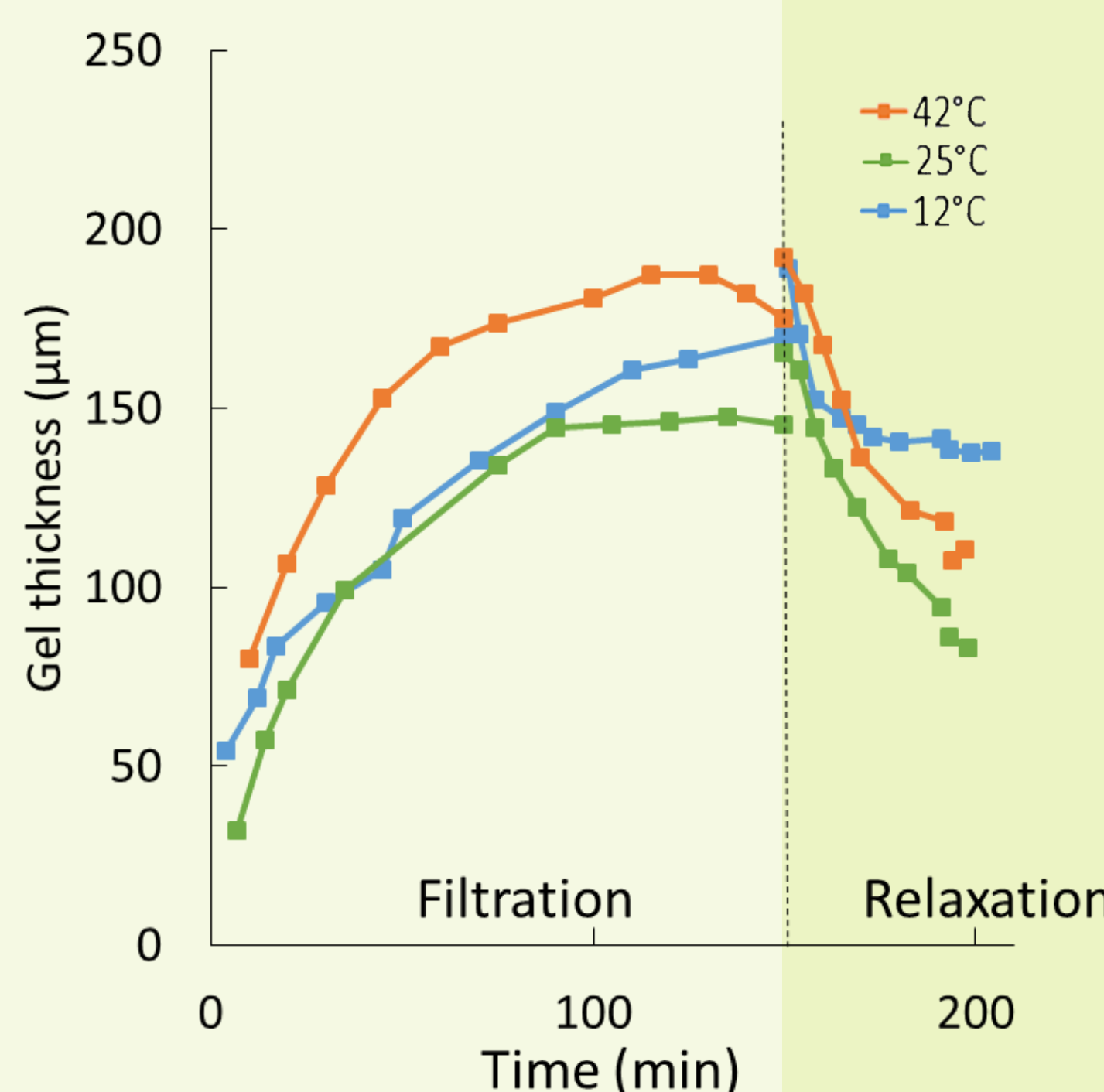
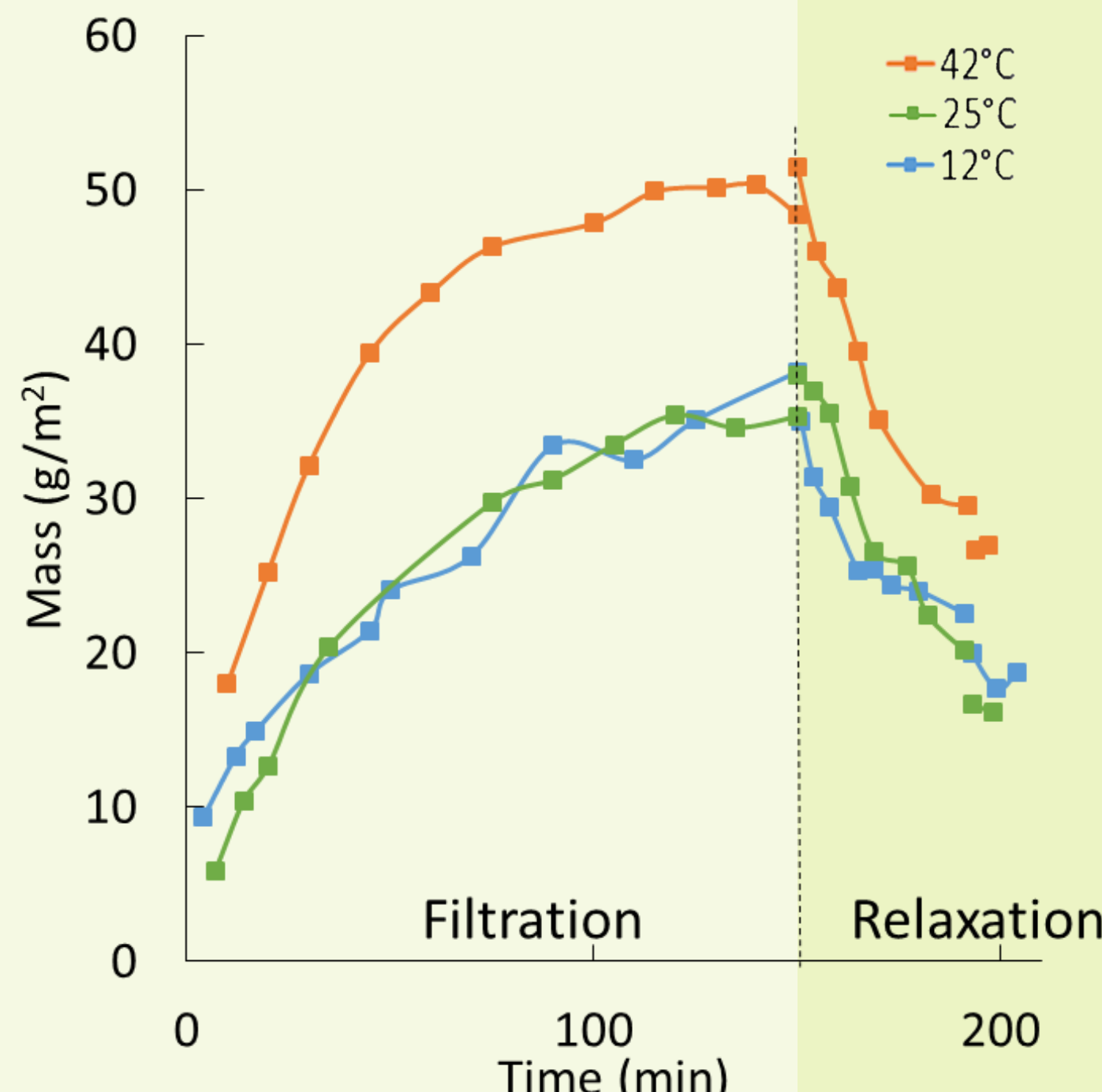


Permeate flux decreases over time for each temperature; mass accumulation impacts permeate flux (for each temperature and so for each filtrate viscosity).

At the end of filtration step,  $t = 150$  min

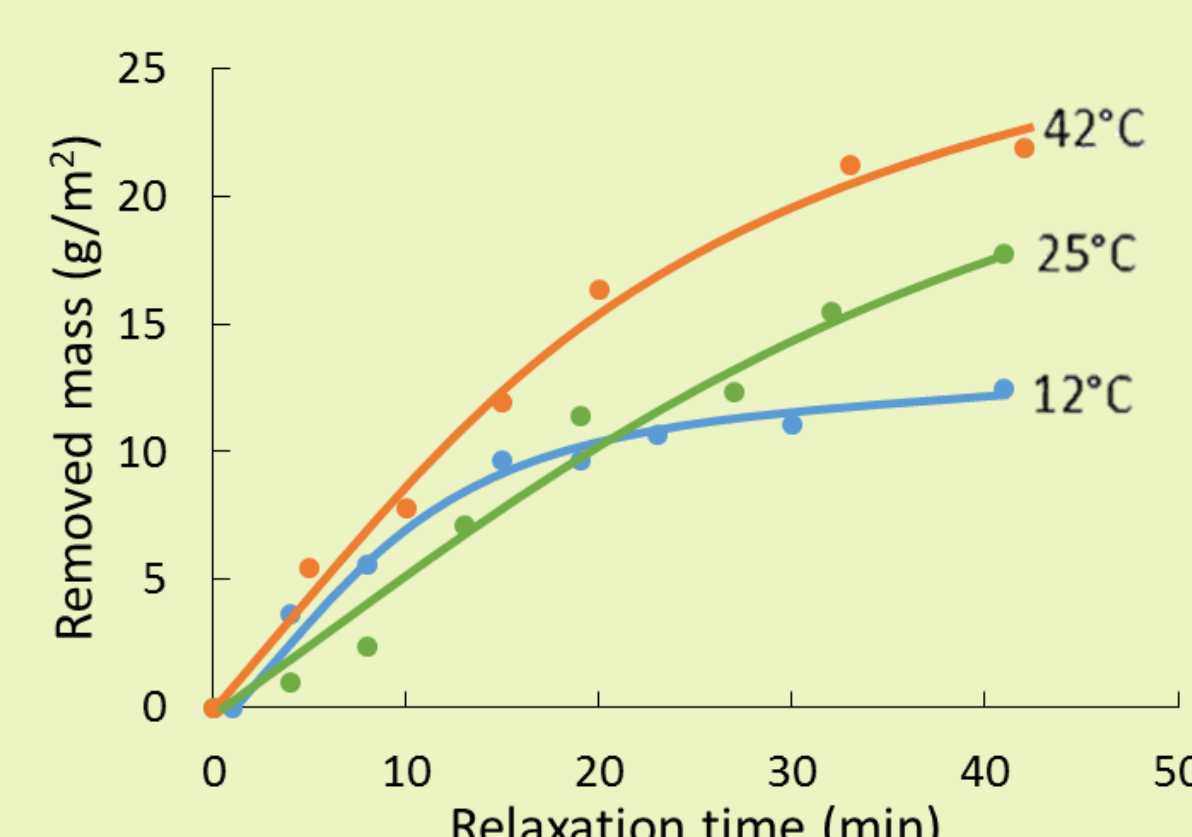


Accumulated mass and gel thickness for each temperature rise over time. At 42°C, accumulation is faster and higher than at 12 or 25°C. Gel is thicker and more concentrated than at 12 or 25°C.

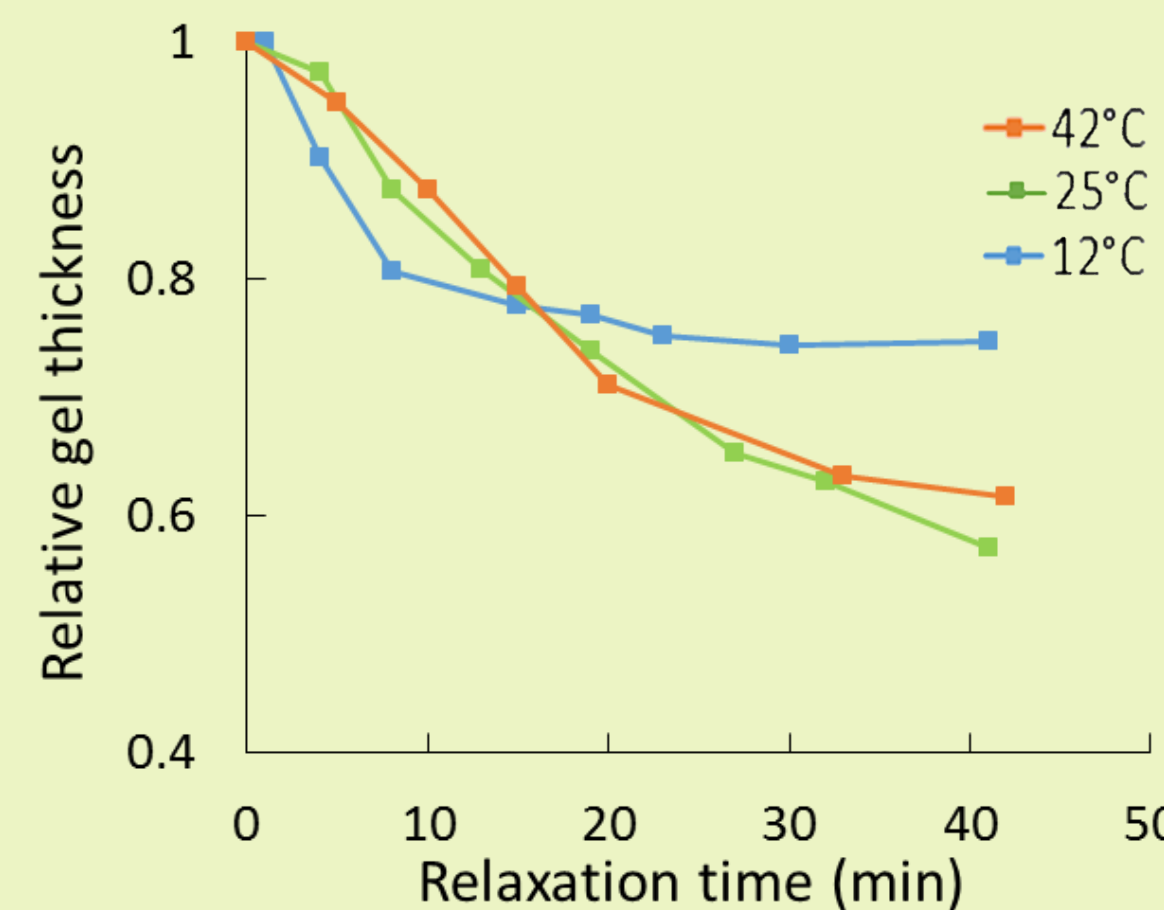


### Pressure relaxation step : Removal of a part of accumulated layer

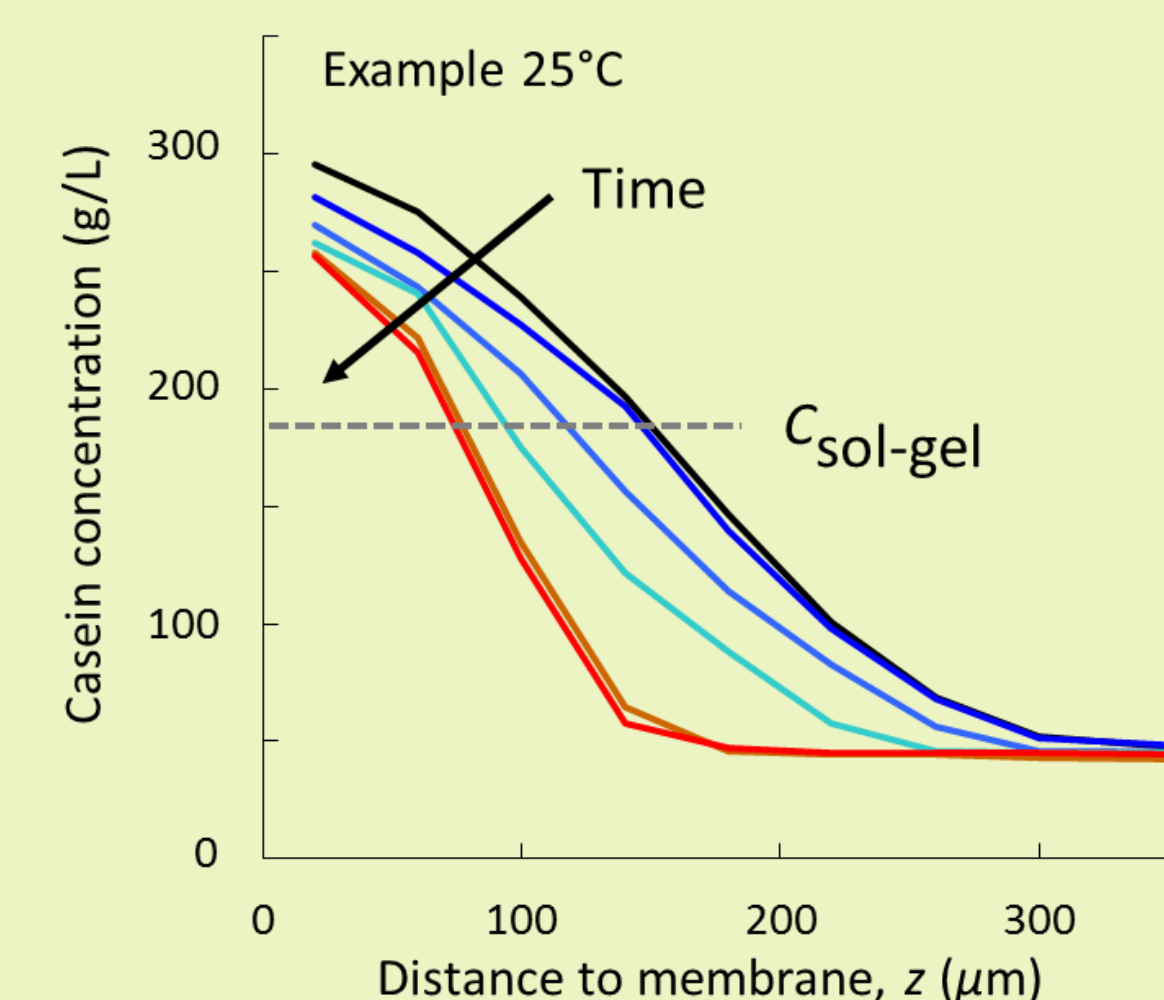
Relaxation speed is relatively low (similar to accumulation speed).



Removal of accumulated micelles and gel layer starts simultaneously with pressure decrease and rises with temperature.



Gel removal is limited at 12°C. Layer is more cohesive at 12°C than at 25 or 42°C.



Fouling removal occurs via gel swelling and redispersion of external swelled part. The nature of fouling (repulsive gel/attractive gel) could depend on temperature.

## Summary

*In-situ* SAXS cross-flow filtration allowed analysis of casein micelles fouling layer with an unique resolution of 20 µm. Quantity of accumulated micelles and gel thickness increase when temperature increases due to the lower filtrate viscosity and higher permeation flux.

An important part of fouling can be reduced by simple pressure release. The fouling by casein micelles is removed via the swelling-redispersion mechanism. A limited efficiency of pressure release at 12°C can be explained by transformation of repulsive gel (swells and redisperses) into attractive gel (swells but doesn't redisperse).