# Yielding properties of milk gels

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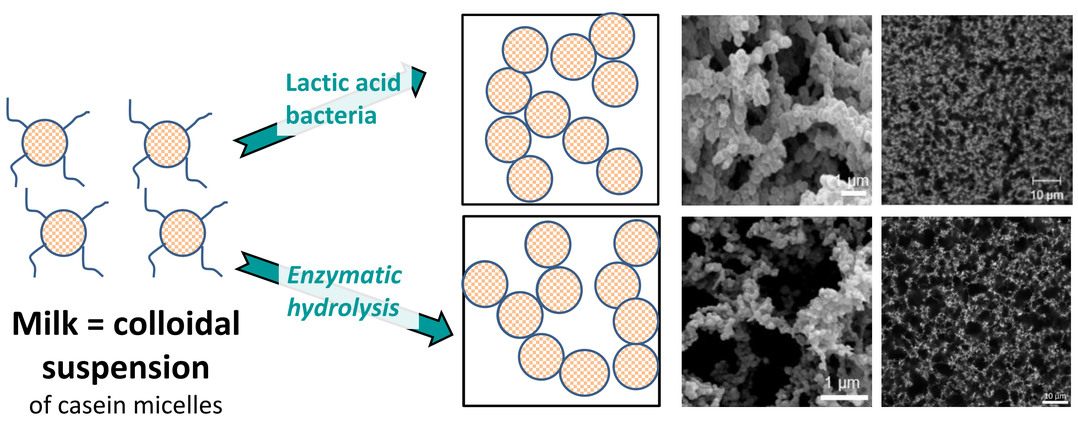
## Background




**Milk = colloidal suspension**
of casein micelles

**Colloidal gel**
with destabilized casein micelles

**Acid caseinate gel (ACG)** = Yogurt —Stirring→ Stirred yogurt

**Enzymatic milk gel (EMG)** —Cutting→ cheese

**Gel processing**
non-linear straining / breaking

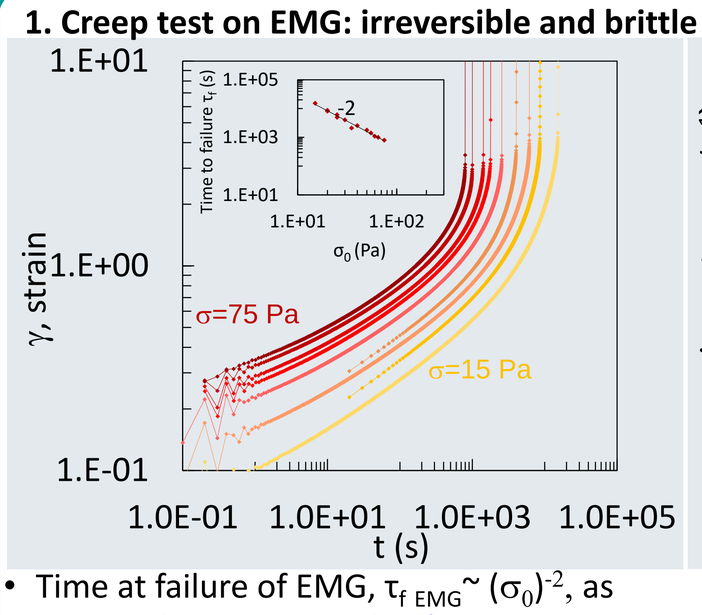
- **Enzymatic (or rennet) milk gel (EMG)** is a colloidal gel involved during cheese manufacture;
- Few studies on non-linear rheological properties of EMG (contrary to linear ones);
- Non-linear properties of materials can be studied by mean of creep, steps in shear rate or large angle oscillatory shear (LAOS) tests;
- The gel yielding is the shear-induced transition from solid to liquid state.

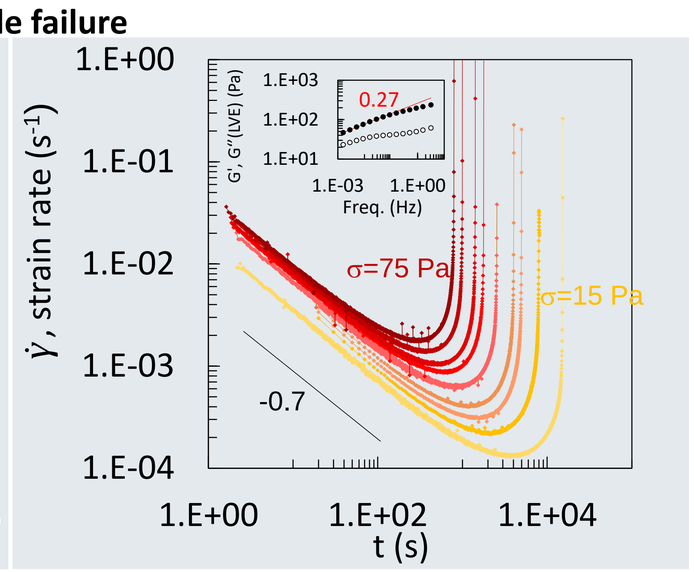
## Conclusions

- Dairy gels behave as hard solid during yielding with irreversible and brittle failure. This type of behaviour is predicted by fiber bundle-type models;
- Moreover, during stress sweep tests, EMG presents strain-softening/strain hardening before failure, that is typical of fibrous material;
- Non-linear behaviour of EMG stems from the network topology, i.e. number and size of strands and their bending/stretching rigidity.

## Results

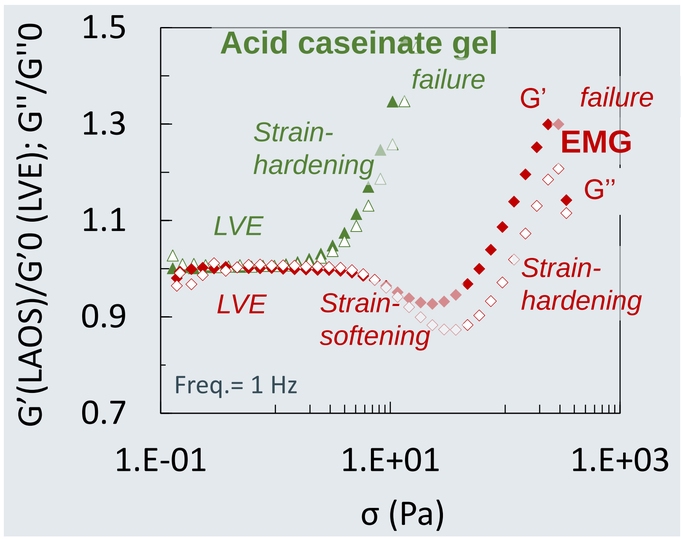
### 1. Creep test on EMG: irreversible and brittle failure




- Time at failure of EMG, $\tau_{f\ EMG} \sim (\sigma_0)^{-2}$, as obtained from Zoon's data[1]
- For acid caseinate gels, $\tau_{f\ ACG} \sim (\sigma_0)^{-5.50}$ [2,4]
- Lower damage accumulation rate in EMG

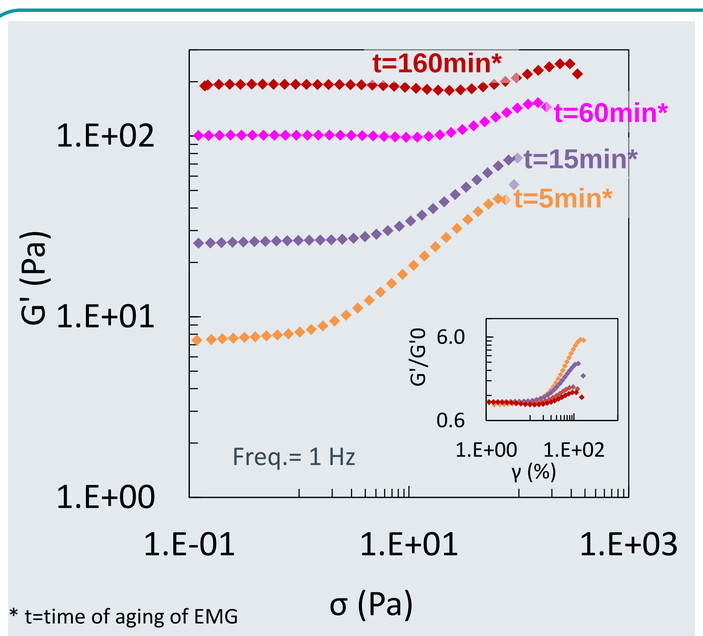
$\dot{\gamma}$ decrease ruled by LVE
$\dot{\gamma} \sim t^{\alpha}$, with $\alpha$ = - 0.7
$\gamma \sim t^{(1-\alpha)} \sim t^{(0.27)} \sim$ LVE




### 2. LAOS on EMG: an odd yielding behaviour

Looks like fibrous gels or as simulated by Bouzid et al.[3]:

1) Softening due to a low strand bending rigidity
2) Hardening when strands over-stretched

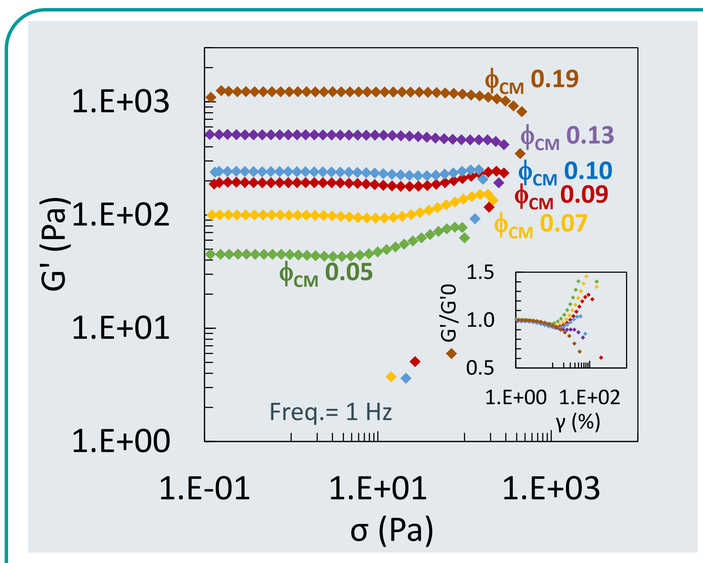



* t=time of aging of EMG

### 3. LAOS: Aging time effect on strain-hardening and strain-softening

- 5 min after gelation: strain-hardening then failure. Homogeneous gel, with small pores and short strands: low probability for bending strands, thus no softening
- 60 min after gelation: strain-softening + strain-hardening, then failure. Higher heterogeneity, larger pores and longer strands, more opportunity for bending and softening

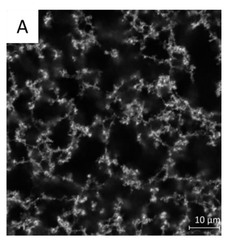
Changes in yielding behaviour due to local rearrangements or to local relaxation and stress localization on some strands of the network




### 4. LAOS: $\phi_{CM}$ (volume fraction of casein micelles) effect on hardening and softening

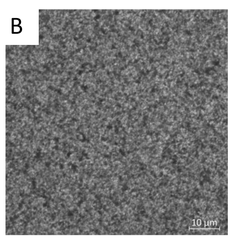
- Yielding begins at a higher σ (but at constant γ) as $\phi_{CM}$ increases
- Amplitude of hardening decreases with increasing $\phi_{CM}$
- At $\phi_{CM}$ = 0.19, only softening (contradictory to Bouzid et al.[3]).

Results due to network topology., i.e. number and size of strands and their bending/stretching rigidity

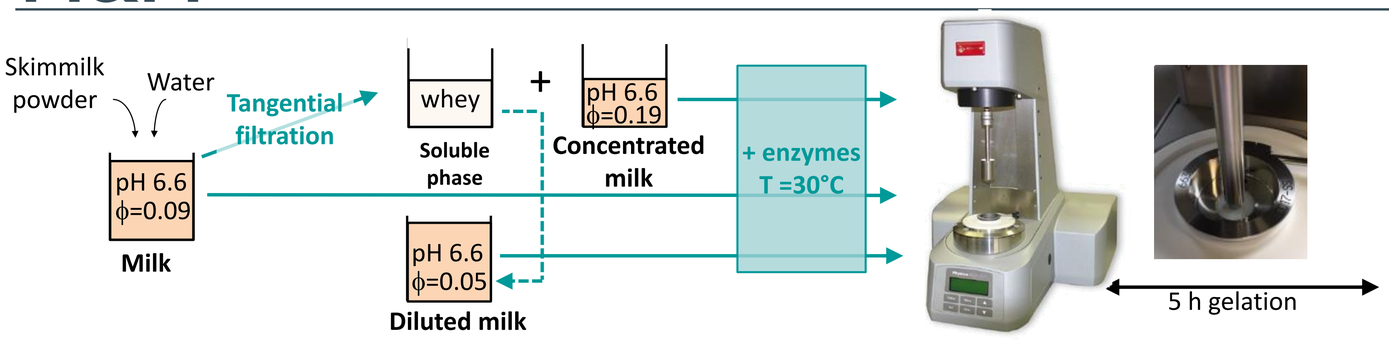



$\phi_{CM}$=0.052, very heterogeneous gel: large strands, large clusters and large pores

$\phi_{CM}$=0.19, more homogeneous, fine stranded and low pore size




## M&M




- Gels were formed in the rheometer and aged at 30°C during 5 h (MCR301 + coaxial cylinder CC1)
- Then, at 30°C:
  - Creep (strain at constant stress, $\sigma_0$)
  - LAOS ( increasing stress amplitude, σ)

1 test = 1 fresh gel aged 5h (or less when specified)

1 Zoon, PhD (1988) ;
2 Leocmach et al., Phys. Rev. Lett., 2014 ;
3 Bouzid & Del Gado, Langmuir, 2018 ;
4 Gibaud et al., Encycl. Complex. Syst. Sci., 2020