# Dead-end Filtration of Sponge-like Colloids: The Case of Casein Micelle

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**Dead-end filtration of sponge-like colloids: The case of casein micelle**

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In colloidal filtration, understanding the mechanisms responsible for deposit formation and determining the relations between filtration conditions and deposit characteristics are still major issues. Following the general - and now well admitted - description of colloidal filtration given by Bacchin *et al*. (2006, 2011), irreversible fouling occurs when a *critical concentration* in particles is reached at the membrane surface. At this concentration, the interactions between the particles get dominated by attractive forces and phase transition takes place from the liquid to the solid state (irreversible deposit – gel). In cross-flow filtration, this *critical concentration* is reached at a given flux (this is the well-known concept of *critical flux*) (Field *et al.,* 1995); while it is reached at a *critical filtered volume* in dead-end filtrations conducted at constant flux (Bessière *et al.,* 2005). Importantly, phase transition and deposit formation can also be interpreted in terms of a *critical osmotic pressure* $\Box_{crit}$, that can be viewed as the compressive pressure required to reach the critical concentration and go from the liquid to the solid state. The critical conditions for deposit formation, as well as the intrinsic characteristics of the deposit, are now well documented, especially for "hard" colloids like silica, latex, clays...; in opposition to "soft" colloids that can deform and even deswell when concentrated. As the "softness feature" might necessarily have an impact on the formation of a deposit and on its properties, it becomes essential to get new information about the behavior of soft colloids in filtration. It is all the more important that such soft objects (emulsions, microgels, protein aggregates...) are involved in an increasing number of industrial processes that use membrane operations.

In this work, we focus on the behavior of one particular soft and sponge-like natural colloid, the casein micelle that makes up to 80% of the protein content of cow milk. For that purpose, specific dead-end filtration experiments were designed and performed, involving successive filtrations interspersed with rinse periods at zero transmembrane pressure ($\Box P$ = 0). This methodology allowed us to precisely monitor the build-up -or not- of a deposit at the membrane surface, as well as to estimate the critical conditions at which the deposit is formed. A detailed analysis of the properties of the deposit is also proposed, based on the variation in its specific resistance (α) with the conditions of filtration.

From the results obtained, we first demonstrate that deposit formation is intimately linked to the colloidal nature of the micelle, as no irreversible deposit is formed when caseins are not organized into micelles. The deposit, or gel, forms through intermicellar bonds that are created when the micelles are forced to come in contact with each other.

We then discuss the critical conditions for deposit formation and we estimate the corresponding critical osmotic pressure at which colloidal phase transition occurs. Interestingly, the obtained value is a bit higher than the one measured in "static" osmotic stress experiments; which could be a direct effect of liquid flow between the particles.

From our filtration experiments, we find that *critical osmotic pressure* $\Box_{crit}$ Which corresponds to the compressive pressure that is required to reach the critical concentration and provokes phase transition, equals 31-35 kPa. Interestingly, this value is close to the one measured from "static" osmotic stress experiments. It is however sufficiently higher to postulate, among other assumptions, that the liquid flow between the particles and/or the kinetics of intermicellar bond formation have an effect on $\Box_{crit}$.


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We finally demonstrate that our results are consistent with a deposit made of closely-packed and connected casein micelles that are compressed during filtration, and that fully relax to their "native" initial dimensions when pressure is released (Figure 1). The deposit is found to be both compressible and relaxable; two properties that are related to the soft and elastic nature of the casein micelle alone. In most cases, the resistance to flow through the deposit is entirely governed by the internal porosity of the casein micelle; this porosity being directly associated to the degree of compression of the micelle body.

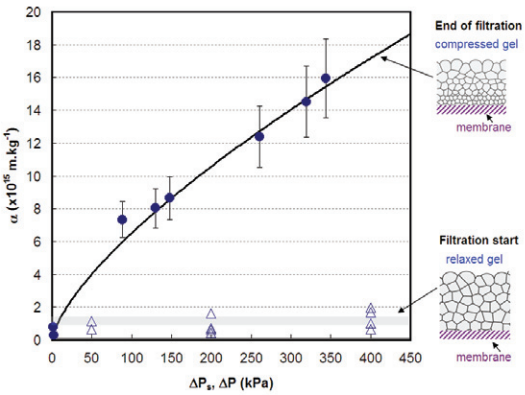


**Figure** : The average specific resistances of the casein micelle deposit ($\alpha$) as measured at the end of a filtration run (full circles) and at the beginning of a filtration run (open triangles). For resistances calculated at the end of a filtration, $\alpha$ is plotted as a function of the solid pressure drop $\Delta P_s$ across the deposit. For resistances calculated at the beginning of a filtration, $\alpha$ is plotted as a function of transmembrane pressure $\Delta P$. With this representation, one clearly sees that the deposit behaves as a compressible material during filtration and then relaxes to about the same resistance during the rinse steps.

Of course, much work still remains to be done to fully understand the behavior of casein micelles in filtration. In particular, we plan to better examine the quantitative relationship between the deposit resistance (and ultimately its selectivity) and the internal structure of the casein micelle. In future studies, another interesting point will be to investigate the filtration as it is performed in the industry, *i.e.*, in cross-flow mode, and to explore the effects of cross-flow velocity on the deposit formation ($\pi_{crit}$) and characteristics ($\alpha$). Finally, we hope that the approach described in this paper will contribute in understanding the general behavior of soft colloids in filtration and/or motivate other comparable studies on specific soft colloidal systems.

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