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Geneviève Gésan-Guiziou

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Cross flow ultrafiltration of skim milk assisted by ultrasound: multi-scale characterization

Jin Y.^a, Hengl N.^{a*}, Baup S.^a, Pignon F.^a, Gondrexon N.^a, Sztucki M.^b, Narayanan T.^b, Gésan-Guiziou G.^c

^a Laboratoire Rhéologie et Procédés, UJF, G-INP, CNRS, BP 53, 38041 Grenoble Cedex 9

^b European Synchrotron Radiation Facility, BP220 38043 Grenoble Cedex 9

^c INRA UMR1253 STLO Science et technologie du lait et de l'œuf, 65 rue de Saint-Brieuc -35042 Rennes Cedex

^d Agrocampus Ouest, UMR1253 STLO 65 rue de Saint-Brieuc - 35042 Rennes Cedex

Introduction

The bottleneck of skim milk ultrafiltration (UF) is the fouling phenomenon that affects the process performance thus limits the productivity. Therefore, a cleaning step is often integrated in the production ^[1-2]. Several strategies have been adopted to control fouling. Some proposed to increase the shear rate close to the membrane ^[3], others suggested different procedures such as backpulsing^[4], pulsating or reversed feed flows^[5]. The ultrasonic-assisted filtration processes have been also proposed to enhance the cleaning process or the permeate flux. Several successful examples can already be found in literature ^[6-7].

Our recent work has shown a great interest to apply *in-situ* ultrasound (US) in colloidal suspensions filtration: with a designed 'SAXS Cross-Flow US-coupled Filtration Cell' at low US power (2 W.cm⁻²), a significant increase of permeate flux was observed without damaging the membrane structure^[8]. Moreover, structural organization at nanometer length scale during filtration was revealed thanks to the *in-situ* SAXS measurements, suggesting that the permeate flux increase resulted from the break-up of the concentrated layer^[9].

In using the designed 'SAXS Cross-Flow US-coupled Filtration Cell', this study is devoted to enhance skim milk ultrafiltration by applying *in-situ* ultrasonication and to characterize effects of ultrasound at multi-scales: the macroscopic results, presented by the permeate flux, will be combined with simultaneous observations at nanometer length scale, revealed by *in-situ* synchrotron SAXS measurements.

Materials and Methods

Skim milk suspensions were prepared from 'low heat' Bovine Skim Milk Powder, provided by 'INRA Agrocampus, STLO Science et technologie du lait et de l'œuf' (Rennes, France). A suspension of $C_0=29$ g.L⁻¹ in casein micelle content has been prepared, corresponding to standard casein micelle concentration in skim milk suspension. To prevent the development of bacteria, 0.2 g.L⁻¹ sodium azide was added to the suspension. The SAXS measurements have been carried out at the ID02 High Brilliance Beamline^[10], at the European Synchrotron Radiation Facility (ESRF) (Grenoble, France). Results

During filtration, two cross-flow fluxes, $Qv = 0.3 \text{ L.min}^{-1}$ and 0.1 L.min⁻¹, were applied and ultrasonication was added for a given duration. These different conditions divided the filtration run into 4 steps, as presented in Fig.1a, displaying the evolution of permeate flux J_v , inlet/outlet pressures of filtration cell and cross-flow flux Qv over time.



Fig.1. Combination of macro-scale results and nano-scale observations during milk ultrafiltration (C = Co, pH 6.8). T = $25\pm1^{\circ}$ C. Ultrasound: 20 kHz, 2 W.cm⁻². a) Cross-flow filtration curve: evolution of Jv, inlet/outlet pressures of filtration cell and cross-flow flux Qv over time. b) Related concentration profiles at steady state from the membrane surface to the bulk, deduced from in-situ SAXS measurements. Permeate flux value corresponds to the concentrated layer; a partial disruption of concentration profile by US is observed.

The related concentration profiles at steady state of each step are presented in Fig.1b. Applied cross-flow flux was changed from 0.3 to 0.1 L.min⁻¹ between step 1 and step 2, inducing a growth of concentrated layer (Fig.1b). Thus the decline of permeate flux from 5 to 2 L.h⁻¹.m⁻² (Fig.1a) is observed due to an increase of the mass transfer resistance. US was applied at step 3, a progressive increase of permeate flux followed, from 2 to 4 L.h⁻¹.m⁻², which can be explained by a partial removal of concentrated layer (Fig.1b). When US was switched off at step 4, the permeate flux got down back to 2 L.h⁻¹.m⁻² and exactly the same concentrated layer as step 2 is obtained at steady state, indicating that the US effect on filtration is almost instantaneous: no further change took place during filtration when US is switched off.

Conclusion

A simultaneous ultrasonic irradiation has been showed an efficient way to improve cross-flow ultrafiltration of skim milk with a factor of 2: it has led to a significant increase of permeate flux, which can be explained by a partial disruption of the concentrated layer, as evidenced from *in-situ* SAXS measurements These results emphasis the promising potential in applying in-situ ultrasonication to membrane separation processes in dairy industry.

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