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► **To cite this version:**

Y. Jin, S. Baup, F. Pignon, N. Gondrexon, M. Sztucki, et al.. Cross flow ultrafiltration of skim milk assisted by ultrasound: multi-scale characterization. 14th Meeting of the European Society of Sonochemistry, Jun 2014, Avignon, France. hal-01209774

**HAL Id: hal-01209774**

**<https://hal.science/hal-01209774v1>**

Submitted on 2 Jun 2020

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

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## 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<sup>[1-2]</sup>. Several strategies have been adopted to control fouling. Some proposed to increase the shear rate close to the membrane<sup>[3]</sup>, others suggested different procedures such as backpulsing<sup>[4]</sup>, pulsating or reversed feed flows<sup>[5]</sup>. 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<sup>[6-7]</sup>.

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<sup>-2</sup>), a significant increase of permeate flux was observed without damaging the membrane structure<sup>[8]</sup>. 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<sup>[9]</sup>.

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

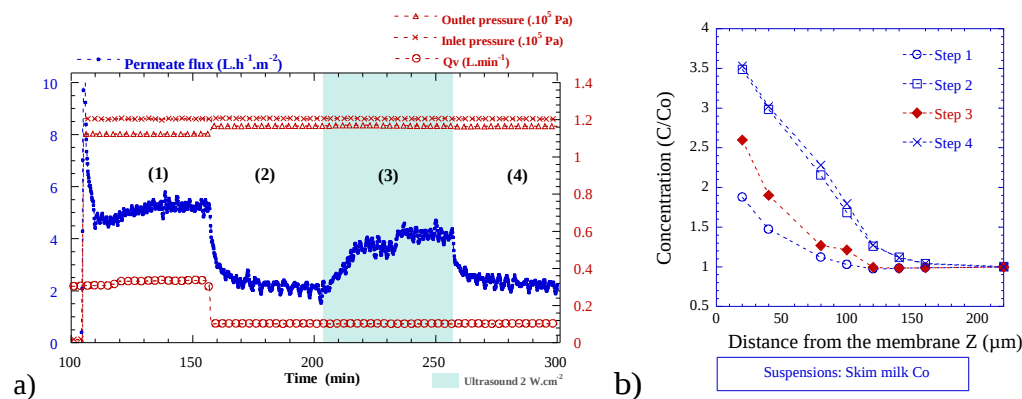
Skim milk suspensions were prepared from 'low heat' Bovine Skim Milk Powder, provided by 'INRA Agrocampus, STLO Science et technologie du lait et de l'œuf' (Rennes, France). A suspension of C<sub>0</sub>=29 g.L<sup>-1</sup> in casein micelle content has been prepared, corresponding to standard casein micelle concentration in skim milk suspension. To prevent the development of bacteria, 0.2 g.L<sup>-1</sup> sodium azide was added to the suspension. The SAXS measurements have been carried out at the ID02 High Brilliance Beamline<sup>[10]</sup>, at the European Synchrotron

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## Results

During filtration, two cross-flow fluxes,  $Q_v = 0.3 \text{ L}\cdot\text{min}^{-1}$  and  $0.1 \text{ L}\cdot\text{min}^{-1}$ , 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  $Q_v$  over time.



**Fig.1.** Combination of macro-scale results and nano-scale observations during milk ultrafiltration ( $C = C_o$ , pH 6.8).  $T = 25 \pm 1^\circ\text{C}$ . Ultrasound: 20 kHz,  $2 \text{ W}\cdot\text{cm}^{-2}$ . a) Cross-flow filtration curve: evolution of  $J_v$ , inlet/outlet pressures of filtration cell and cross-flow flux  $Q_v$  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  $0.1 \text{ L}\cdot\text{min}^{-1}$  between step 1 and step 2, inducing a growth of concentrated layer (Fig.1b). Thus the decline of permeate flux from  $5$  to  $2 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$  (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  $4 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ , 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  $2 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$  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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