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Skim milk ultrafiltration with a PES membrane: Effect of milk thermal pretreatment and concentration on the irreversible fouling

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The skim milk ultrafiltration (UF) is widely used industrially to standardize milk protein concentration before cheese making. These UF membranes are made of polyethersulfone (70% of the market for HFK 131, Koch) and are used on several consecutive stages. During production cycles the membrane is fouled with skim milk components. It leads to flux and selectivity decrease. To restore membrane performances, a chemical cleaning is performed at least once a day (2-3 h of cleaning every 6-8h of production). The irreversible fouling that remains after the water rinsing is the target of the cleaning. Cleaning procedures actually performed cannot avoid a decrease of about 50% of the production flux over several months nor sometimes an alteration of the membrane selectivity. This justifies the fundamental studies on cleaning optimization and the need of information on the nature of the overall and irreversible fouling.

Rabiller-Baudry *et al.* (2002) showed previously by FTIR-ATR and SEM-EDX analysis that the irreversible fouling after skim milk UF, with the HFK 131 membrane in batch mode circulation (VRR=1), is exclusively made of proteins. In this latest study the skim milk used is UHT type whereas at industrial scale the milk is only pasteurized before ultrafiltration process (lower temperature treatment). These sterilization modes induce a different denaturation state for the whey proteins (alpha-lactalbumin and beta-lactoglobulin) suspected to be the main responsible of the irreversible fouling. In this study the effect of the soluble proteins' denaturation on the irreversible fouling of the membrane (skim milk UF at 2 bar, 50°C, 60 min) is shown. The influence of milk concentration is also highlighted as, industrially, it increases from the first to the last stage. Either skim milk and water fluxes, minerals (analyzed by SEM-EDX) and protein (determined by FTIR-ATR) amount on the membrane and cleanability by a sodium hydroxide solution (pH 11.5, 2bar, 50°C, 60 min) are compared.

Results show that the permeability of the membrane during skim milk UF or in water after rinsing is quite the same regardless the mode of milk sterilization (UHT or pasteurization): no significant impact of soluble protein thermal denaturation is evidenced on the flux. Nevertheless the irreversible protein amount is quite different on the membrane: 30 and 116 $\mu\text{g}\cdot\text{cm}^{-2}$ with UHT and pasteurized skim milk, respectively. There is no mineral irreversible fouling on both fouled membranes. The fact that the same water permeability is obtained with so different protein amount allows thinking that the denaturation state of proteins induced by the thermal treatment leads to deposits of different structures. It would be less open and/or less hydrophilic with UHT denatured proteins.

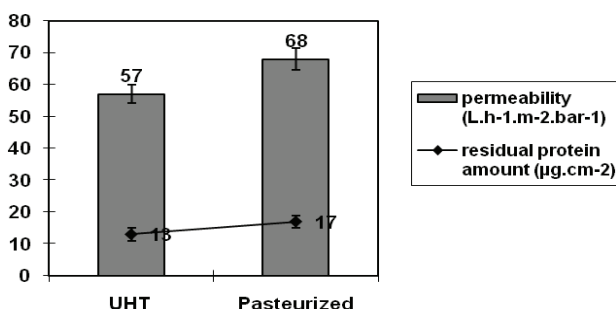


Fig 1: Residual protein amount and permeability of membrane fouled during UF (HFK 131) of UHT or pasteurized skim milk.

The amount of residual proteins remaining after NaOH cleaning is a bit lower for the membrane previously fouled with UHT skim milk than with the pasteurized one - 13 vs 17 $\mu\text{g}\cdot\text{cm}^{-2}$ (Figure 1). Nevertheless the water permeability after NaOH treatment is higher with membrane previously fouled with pasteurized milk (Figure 2). This is in good agreement with previous hypothesis on opened or hydrophilic fouling structure induced by thermal denaturation of proteins.

Figure 2 shows the influence of the UHT skim milk concentration on the irreversible fouling, obtained by UF at various volume reduction ratios (VRR). Membrane permeability decreases regularly with VRR traducing an increase of the overall fouling. After rinsing, the water permeability is close to 30 $\text{L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}\cdot\text{bar}^{-1}$ at both VRR= 1 and 2 and close to 25 at higher VRR (4 and 6). There are here two levels of irreversible fouling. But regardless the concentration ratio, the residual protein amount (after rinsing) is constant and the SEM-EDX analysis doesn't show any mineral fouling. So at higher concentrations the water permeability is lower whereas the protein amount remains the same: the concentration ratio seems to also have an impact on protein deposit structure. The irreversible fouling would be less open and/or less hydrophilic at high concentration rate.

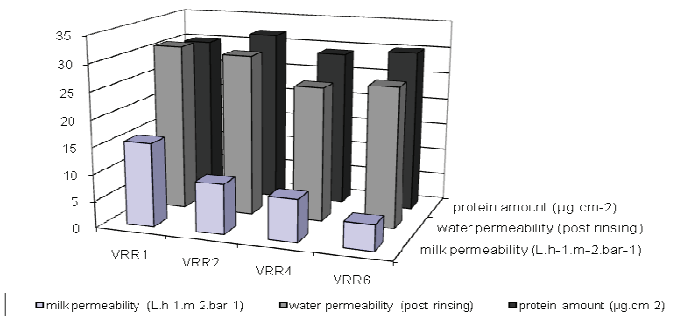


Fig 2: Residual protein amount, water and milk permeability after the fouling of HFK 131 at VRR=1,2,4,6.

The comparison of permeability and protein amount after NaOH cleaning for membranes fouled at VRR=1 and up to VRR=6 shows clearly a difference of cleanability (Figure 3). The more concentrated the milk was the less cleanable the membrane was. The residual protein amount is also significantly different. Once again, the irreversible deposit would be more cohesive at high concentration ratio than at low one.

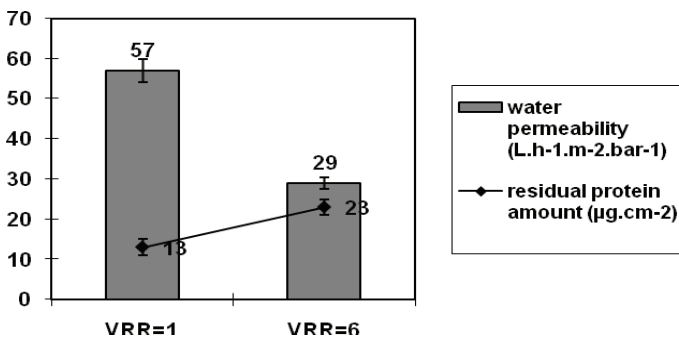


Fig 3: Residual protein amount and permeability of membrane fouled during UF (HFK 131) at VRR=1 and 6.

This work highlights the role of protein structure and concentration on the irreversible fouling characteristics in terms of permeability, irreversible protein amount before and after cleaning and thus of membrane cleanability. The fouling by UHT skim milk is probably more cohesive than that induced by pasteurized skim milk. Moreover, the cohesion increases with concentration rate.

References

M. Rabiller-Baudry, M. Le Maux, B. Chaufer, L. Begoin, Desalination, 146 (2002) 123-128.

Keywords: PES membrane fouling, skim milk, thermal treatment, protein