Food Science and Applied Biotechnology, 2022, **5**(2), 190-198

UFT Academic Publishing House, Plovdiv

Food Science and Applied Biotechnology
e-ISSN: 2603-3380

Journal home page: www.ijfsab.com
https://doi.org/10.30721/fsab2022.v5.i2

OPEN ACCESS

Research Article

Nanofiltration process monitoring and antioxidant activity analysis of skimmed milk samples

Iliyan Trayanov[1,2]✉

[1]*Department of Chemical Engineering, Faculty of Chemical and System Engineering, University of Chemical Technology and Metallurgy, Sofia, Bulgaria*
[2]*Institute of Chemical Engineering, Bulgarian Academy of Sciences, Sofia, Bulgaria*

Abstract

Dead-end stirred cell experiments were performed with reconstituted skimmed milk and model solutions of single components (lactic acid). When a low solids concentration is presented with the feed, dead-end filtration with a selected polymeric membrane with cut-off 300 Da is a viable option to assure a high rejection of the dissolved organic compounds (> 90%), combined with a good transport at moderate pressures (20 bar, selected from prior nano-milk-filtrations) with an average flux (J_P) increasing from 48 $L.m^{-2}.h^{-1}$ at 20°C and over 67 $L.m^{-2}.h^{-1}$ at 50°C until reaching a volume reduction of 5 . The DPPH antioxidant activity was effectively improved (~3 fold) with some insignificant loss due to fouling. Despite the high rejection, transmission of low-molecular weight components is detectable in the permeates, additionally the latter have astoundingly an antioxidant capacity, so each permeate can be divided into time interval fractions-aliquots each showing different DPPH antioxidant activity. This is further confirmed with statistical analysis where $p < 0.05$. As conclusion, because temperatures up to 50°C significantly increase the flux without much effect on the antioxidant activity, such conditions (temperature, pressure, dilution) are viable options for large-scale production applications.

Keywords: nanofiltration, flux, antioxidants, milk, fractionation

Abbreviations:

Da – daltons; DPPH – 2,2-diphenyl-1-picrylhydrazyl; %RSA – % radical scavenging activity; UF – ultrafiltration; NF – nanofiltration; J_P – permeate flux; MWCO – molecular weight cut-off; VCR – volume concentration reduction; VCF – volume concentration factor; VDPPH – volume of the reaction mixture in the DPPH assays; V_{sample} – volume of the reaction mixture in the DPPH assays; TMP – transmembrane pressure; V_F – feed volume; V_P – permeate volume; TDS – total deposited solids

✉*Corresponding author: chem. eng. Iliyan Trayanov, PhD Student, Department of Chemical Engineering, Faculty of Chemical and System Engineering, University of Chemical Technology and Metallurgy, Sofia, Bulgaria, 8 Kl. Ohridski blvd., 1756 Sofia, Bulgaria; E-mail: iliyantrayanov@gmail.com*

Article history:
Received 21 April 2022
Reviewed 30 July 2022
Accepted 25 August 2022
Available on-line 13 October 2022

https://doi.org/10.30721/fsab2022.v5.i2.193

Introduction

Membrane processes have been established as an effective option in the dairy industry. Ultrafiltration (UF) is perhaps the most used membrane technology for wide variety of applications thus prolonging shelf life of milk without heat exposure, processing of acid whey to obtain whey protein concentrate and lactose-rich permeate, and purification of the main bioactive compounds of milk for bringing new products to the market (Kumar et al. 2013). Besides having high nutritional value, milk formulations possess beneficial macro- and micronutrient composition, including high-quality proteins and high calcium content. Various liquid milk concentrates with antioxidant capacity, anti-angiotensin activity, antibacterial activity, and allergen-free were produced from skimmed milk by combination of membrane- and enzyme-based technologies. Milk naturally contains components that have antioxidant capacities (Fox et al. 2015), such as casein, lactoferrin, vitamin E, vitamin C, carotenoids and some flavonoids that play a role in radical scavenging. Many of the sulphur containing amino acids play that role. Milk processing into fermented products may produce bioactive peptides and other beneficial nutrients that increase antioxidant activity, inhibit oxidation and decrease lipid peroxidation (Khan et al. 2019b). Many synthetic antioxidants express toxic and carcinogenic effects, the reason why they are restricted or prohibited in a number of countries (El-Fattah et al. 2020)

The technical potential of nanofiltration (NF) lies between ultrafiltration and reverse osmosis with MWCO of membrane from 100 Da (tight) to 1000 Da (loose), and operating pressures much lower than those required for reverse osmosis. The small length scales determining separation can be advantageous to special applications such as partial demineralization of whey, production of low-lactose and even lactose-free dairy products or volume reduction of whey, as alternative to conventional thermal evaporation. The potential removal of salts is also important for preventing scaling and build-up on evaporators, thus facilitating the production of high-quality lactose and whey products (Zhang et al. 2020). The specific features of NF membranes are mainly the combination of very high rejection factors for multivalent ions (99%) with low to moderate rejection factors for monovalent ions (0-70%), and high rejection factors (90%) for organic compounds with molecular weights above that of the membrane MWCO (Meyer et al. 2017). Although there are several reported studies of milk concentration with the use of reverse osmosis and by coupling ultrafiltration, electrodialysis, and nanofiltration technologies, there are few works in the literature focused on the NF as a single process for milk concentration (Zacharof and Lovitt 2012a). Lipophilic antioxidants are durable by high temperatures, whilst being more important than hydrophilic antioxidants for organisms (Grazyna et al. 2017).

The objective of this work is to study the ability of a direct nanofiltration process to (pre)concentrate skimmed milk solutions, with a focus on antioxidant activity enhancement or potential loss in permeate side for some low molecular weight antioxidants.

Materials and Methods

Experimental set-up and membranes. Dead-end filtration experiments were performed in a laboratory high pressure equipment (METcell, Evonik Membrane Extraction Technology, UK) using a flat sheet thin film composite polyamide membrane (NF Alfa Laval) with the effective area of 0.054 m^2. Based on manufacturer's specification, the membrane selected (cut off 300 Da) is a high salt removal membrane, with rejection > 99% for $MgSO_4$, measured on 2000 ppm, 9 bar, and 25°C. Nitrogen gas is used for NF system pressurisation, at maximum operating pressure of 69 bar. Prior filtration experiments, special Initial Protective Layer Flush Procedure was applied according to the manufacturer recommendations, that took the following steps: one run 150 ml out of 175 ml distilled water at 10 bars, followed by four runs the same amounts of distilled water at 20 bars, 2 runs the same procedure at 50°C, 3 runs NaOH at pH 10, same amounts of fluid applied, and one last run 100 ml out of 125 ml distilled water at 20 bars (until neutral pH). Reconstituted skimmed milk (9.93% fat free dry residue, 3.51% protein, 5.43% lactose, pH 6.65 – 6.75, data from manufacturer) was prepared by adding the powder to hot water with gentle agitation. All trials consisted of a feed concentration of 2 mg/ml solutions. For NF concentration of the diluted solutions, feed solution of 75 – 150 ml was filled into the test cell. The cell was then pressurized at the operating pressure between 10 and 30 bar. The NF concentration was carried out until 70 – 80% of the feed volume which entered the module was recovered into permeate (dead volume of the cell 25 ml). The experiments were performed at two different temperatures of the feed, at room temperature 20±2°C and higher 50±2°C. The solutions were constantly agitated by a magnetic stirring plate at a stirring rate of 250 rpm to minimize concentration polarization and fouling layer formation near to or on the membrane surface. The system was cleaned between test runs, with distilled water being permeated through the membrane to restore membrane performance.

Characterization of separation performance.

The separation behaviour in dead-end mode was characterized in terms of volumetric permeate flux and rejection. During the NF experiments, the time taken to filter a given volume of feed solution was measured, and flux was calculated as:

$$J_P(t) = \frac{V_p(t)}{A \cdot \Delta t} \qquad (1)$$

where J_P is the permeate flux ($L.m^{-2}.h^{-1}$), Vp is the cumulated volume of permeate (L) at time t (h), A the effective membrane area (m^2) and Δt elapsed time from start (h). The volume concentration factor (VCF) was calculated as follows:

$$VCF = \frac{V_F}{V_R(t)} = \frac{V_F}{V_F - V_P(t)} \qquad (2)$$

where V_F is the feed volume, V_R and V_P - the retentate and permeate volume at time t. The permeate flux evolution during filtration can be described with a polynomial equation (second or higher degree) as a function of VCF:

$$J_p(VCF) = a + b\,(VCF) + c\,(VCF)^2 + d\,(VCF)^3 \qquad (3)$$

where a, b, c, and d denote the corresponding empirical polynomial coefficients. Then the average flux is obtained by integration of eq. 3:

$$J_{av} = \frac{\int_0^{VCF} J_p\, dVCF}{VCF} \qquad (4)$$

The separation capability of the membrane is quantified by the overall retention (R overall) with respect to the total dissolved solids (TDS), determined by comparing the concentration of the permeate to the feed:

$$R_{\text{overall}} = \left(1 - \frac{\overline{C}_P}{C_F} \cdot\right) x100\% \qquad (5)$$

where C_F and $\overline{C}_P$ are the concentrations of the feed (initial) and whole permeate (final averaged over the time of filtration, respectively. The content in TDS was obtained gravimetrically, after drying to constant weight. Feed and permeate samples were collected at different VCF and refrigerated for subsequent analysis. When the NF process was stopped, both retantate (R) and permeate (P) were collected and stored at 4°C.

Determination of free radical scavenging activity. The activities of the feed (F), permeate (P) and retentate (R) were studied by using the 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) method, by the method of Brand Williams (Bondet et al. 1997). The reaction mixture consisted of 2 mL of tested solutions added to 2 mL of 200 μM solution of DPPH in 95% ethanol. The reaction takes place at dark in the spectrophotometer for maximum of 120 min, meanwhile the absorbance at 517 nm shows the concentration of remaining still-nonreacted DPPH in the UV-VIS cuvette. The radical scavenging activity (% RSA) of samples was calculated using the expression:

$$\%RSA = \frac{[A_s]_{t=0} - [A_s]_{t=final}}{[A_0]} x100 \qquad (6)$$

where A_s is the sample absorbance and A_0 is the control absorbance (DPPH and ethanol mixture) at 517 nm. Additionally, the remaining DPPH fraction during the reaction kinetics with the antioxidants was monitored and expressed according to:

$$DPPH_R = \frac{[Abs_{517}]_t}{[Abs_{517}]_{t=0}} \qquad (7)$$

Experiments were performed in triplicate (n = 3). The experimental error was represented by mean value with standard deviation (±values), which indicated very good reproducibility, the limits of percentage errors being lower than 4%.

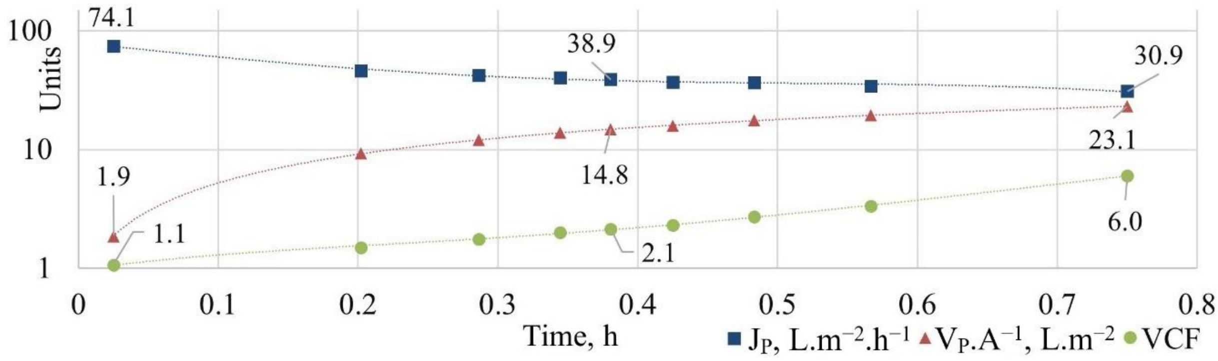
Results and Discussion

Effect of operating conditions on permeate flux and retention. Milk is a complex feed for membrane-based concentration and fractionation processes, because of the broad molecular weight distribution of multiple ingredients (< 1 nm to 20 µm) and high content of dissolved matter (10 – 13%). The major separation mechanisms of NF involve steric effect and the concentration and size of solutes are critical for their permeation and rejection. The separation efficiency for a particular application can also be influenced by the nature of the membrane material and surface properties (pore size distribution and shape, hydrophobicity, charge density) as well as processing parameters during dead-end and cross-flow mode of operation.

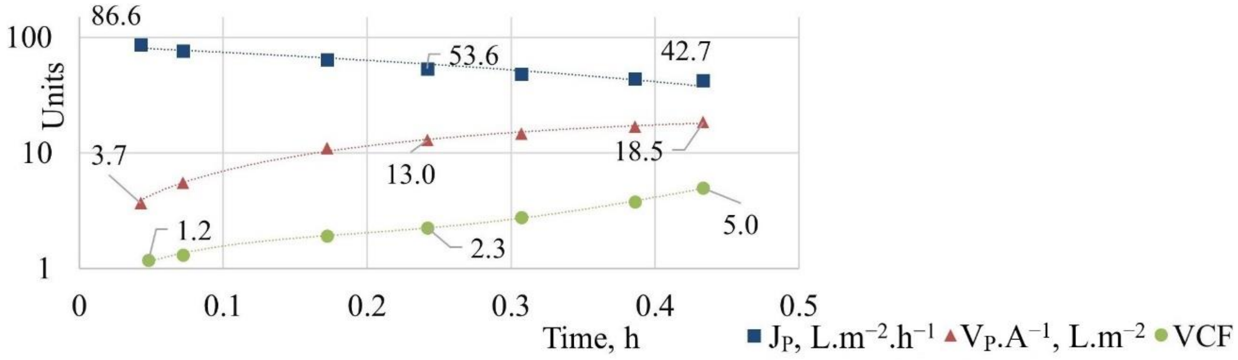
Preliminary experiments were carried out to evaluate the impact of operating conditions, as transmembrane pressure (TMP) and nature of product (feed concentration and pH) on the separation behaviour. The application of higher TMP enhanced the permeate flux though commonly not linearly up to 20 bar while with further increases in pressure no significant variation were observed and the flux becomes dependent rather on the mass transfer characteristics of the system (data non shown). From these results it was selected as optimal process parameters TMP of 20 bars.

In agreement with the membrane's MWCO (300 Da), the rejections of the valuable compounds. of diluted skimmed milk solutions was over 90% ($R_{overall}$ > 90%). In which, macromolecules, as casein micelles ($10^7 – 10^9$ Da), α-lactalbumin and β-lactalbumin (~14500 – 36000 Da) are completely retained, whereas relatively small molecules of lactose (340 Da, 0.8 nm), a major carbohydrate present at about 51% in the skimmed milk powder, could be partially permeated, and finally small molecules as chloride ions (35 Da, 0.4 nm) and calcium ions (40 Da, 0,4 nm) found at trace levels, are supposed to pass through the membrane, driven by the difference in pressure on both sides of the membrane. The VCF attained at the end of run was 5 - 6 folds, whereas the concentration obtained for the TDS was only around 3.6 folds higher than that of the skim milk fed to the process. This behaviour may have been due to the establishment of fouling layer caused by adsorption of deposition of completely or partially rejected matter onto the membrane surface and in the membrane pores, thus reducing their concentration in the fraction retained. The susceptibility of membranes to fouling was further confirmed by the non-linear variation of volume concentration factor, cumulative permeate volume, and permeate flux with time, as presented in Fig.1 for each temperature condition.

In all filtration runs the permeate flux declined in a manner expected and in line with other studies (Zacharof and Lovitt 2012b). Overall, operating NF at 50°C gave better membrane permeability as evidenced by a better flux and shorter time required to reach a 5-folds volume concentration, as compared to that operated at 20°C. The comparison of permeate flux versus volume reduction further illustrate the difference in permeability obtained at 20 and 50°C (Fig. 2). The curves present relatively similar profile with an initial steep drop followed by a slowed down flux decline with time. The average permeation flux, calculated by eq.4, increased from 48 $L.m^{-2}.h^{-1}$ at room temperature to 67 $L.m^{-2}.h^{-1}$ at 50°C (about 40% increase). A relatively high permeability throughout the duration of the testing period to some extent suggests that the membrane is loose to enable complete rejection. Increasing the temperature favoured membrane filtration due to the coupled effect of lowering the liquid viscosity, increasing the solubility and mass transport across the membrane of permeated solutes, and possible changes in the membrane structural parameters (effective pore radius and membrane thickness). Results confirmed that the processing temperature of the feed is an important factor that can slow down the rate of membrane flux decline and retard fouling phenomena while non effect on the selectivity of the membrane and quality of the NF products occurred. By higher milk concentrations the casein micelle formation plays big role, as the micelles start to grow bigger (Li et al. 2016). Dairy milk effluents are responsible for 1-3% loss of milk components, which arises also as an environmental risk. (Luo et al. 2012) The changes in solvent viscosity and ion diffusivity (the so called mobilities) decrease and increase permeate concentration with increasing temperature respectively (22°C vs 50°C), meaning they partially cancel each other's effects (Roy and Lienhard 2019). Furthermore, membrane fouling by skim milk has been consistently caused primarily by proteins, with mineral fouling seen only on ceramic membranes (Ng et al. 2017) The concentration polarization was much stronger at 42°C than at 12°C (Doudiès et al. 2021) 1000kD PES membrane displayed improved flux and lower fouling at refrigeration temperatures than 0.1µm and 0.45µm membranes (Crowley et al. 2015).



a) T = 20±2°C; TMP = 20 bar; V_F = 150 ml; V_P = 125 ml



b) T = 50±2°C; TMP = 20 bars; V_F = 125 ml; V_P = 100 ml

Figure 1. Time course of cumulate volume per membrane area (V_P/A), volume concentration factor (VCF) and permeate flux (J_P) during nanofiltration of skimmed milk

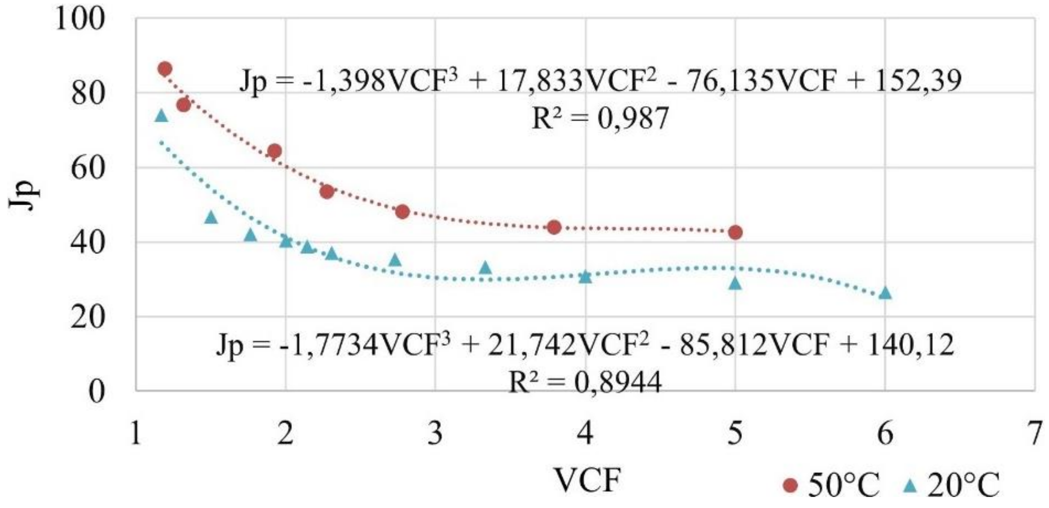


Figure 2. The influence of temperature on permeate flux (J_P) as a function of VCF

DPPH scavenging activity. Methods for fast evaluation the effectiveness of natural and synthetic antioxidants are usually based on their reactions with test radicals, e.g., DPPH radical. A literature review conducted showed that several protocols have been followed using different mixing (nature of solvents and polarity of the solution), incubation conditions (time, temperature), and compounds used as antioxidant standards (Mann et al. 2016), making the comparison between studies difficult, although as a whole the buffalo milk has more health benefits than cow milk including antioxidant properties (Khan et al. 2017). In Fig. 3 is exemplary shown how selected factors may affect the %RSA for two different sets of solutions, namely retentate obtained at 50°C and Lactic acid in solution. The experiments done at different values of the ratio of volume of sample to the volume of DPPH solution (V_{sample}: V_{DPPH}) reacted at very different rates and followed differing kinetics. As many of the compounds having antioxidant activity are weak or moderately strong organic acids, differing acidity may also affect the reaction time and final %RSA, as shown in the case of lactic acid at $pH < 2$ and pH 4, typical for fermented media. Also, the activity of the DPPH radical makes possible photochemical transformations caused by the light. Comparison of absorbance under irradiation in the spectrophotometer during reaction survey and in the dark for a fixed endpoint (usually 30 min) showed the impact of frequency / time of exposure to light (at 517 nm), and should also be taken into account in the interpretation of DPPH assay data.

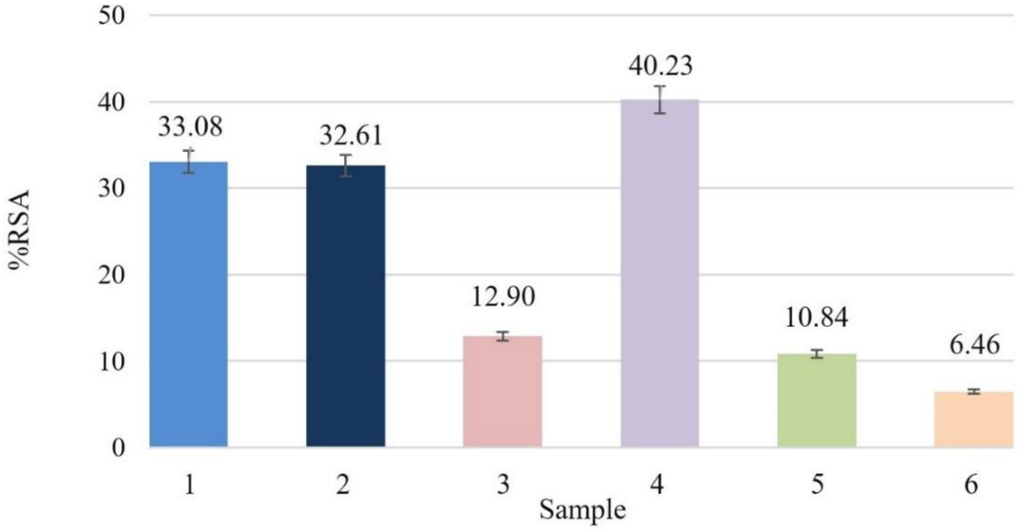


Figure 3. Sensitivity of DPPH assay of a skim milk concentrates from NF treatment compared to lactic acids solution at various conditions

1. Retantate 50°C, V_{sample}: VDPPH=1: 3; 2. Retantate 50°C V_{sample}: VDPPH = 1: 3; 3. Retantate 50°C, V_{sample}: VDPPH = 3:2; 4. Lactic acid (85%, $pH < 2$); 5. Lactic acid (diluted to pH 4), under VIS at 517 nm; 6. Lactic acid (diluted to pH 4), under dark

Fig. 4 shows the variation of %RSA of retentate and permeate, presumably as a result of the influence of fouling on the retention and the composition of the permeate and retentate. The desired effect of antioxidant activity increase in the NF concentrates was observed for each temperature condition. The %RSA of retentate solutions was more than 3 times higher than that of the initial solution, which is in line with the increase in total solids content at the end of the run.

On the other hand, permeate solutions obtained over time presented also variable antioxidant activity, in the range between 3% to 20%, confirming the permeation of lactose, salt, and other antioxidants with increasing concentration over time. Fig. 5 depicts the different kinetic behavior of permeate samples collected at different VCFs, as compared to the whole permeate (P_{whole}).

.

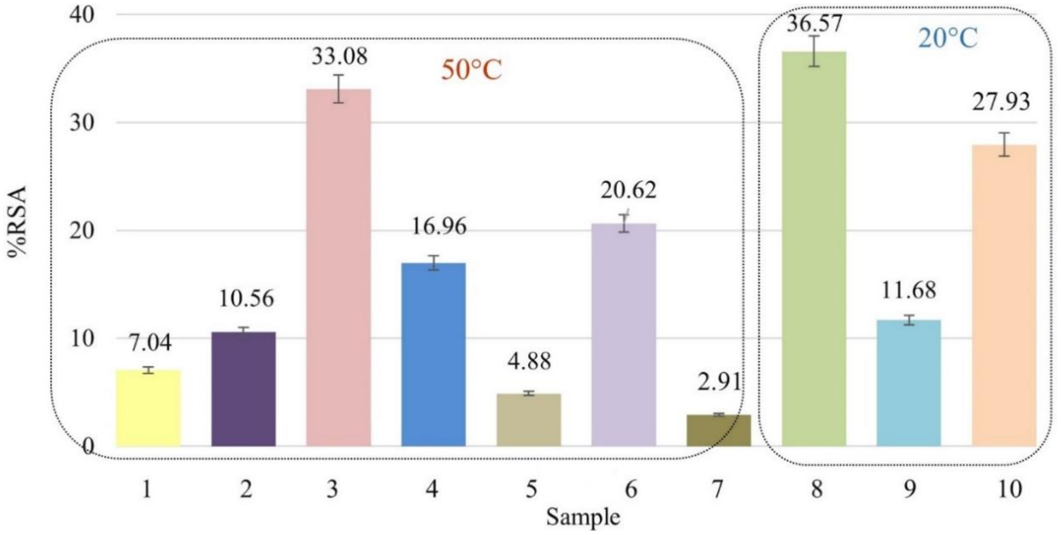


Figure 4. Estimated RSA % of feed, final skimmed milk concentrate and permeate samples taken at different VCF and temperatures

1. Permeate (whole; VCF = 5.00); 2. Skimmed milk (Feed); 3. Retentate; 4. Permeate (VCF = 1.07); 5. Permeate (VCF = 1.25); 6. Permeate (VCF = 2.12); 7. Permeate (VCF = 3.79); 8. Retentate; 9. Permeate (VCF = 1.16); 10. Skimmed milk (Feed)

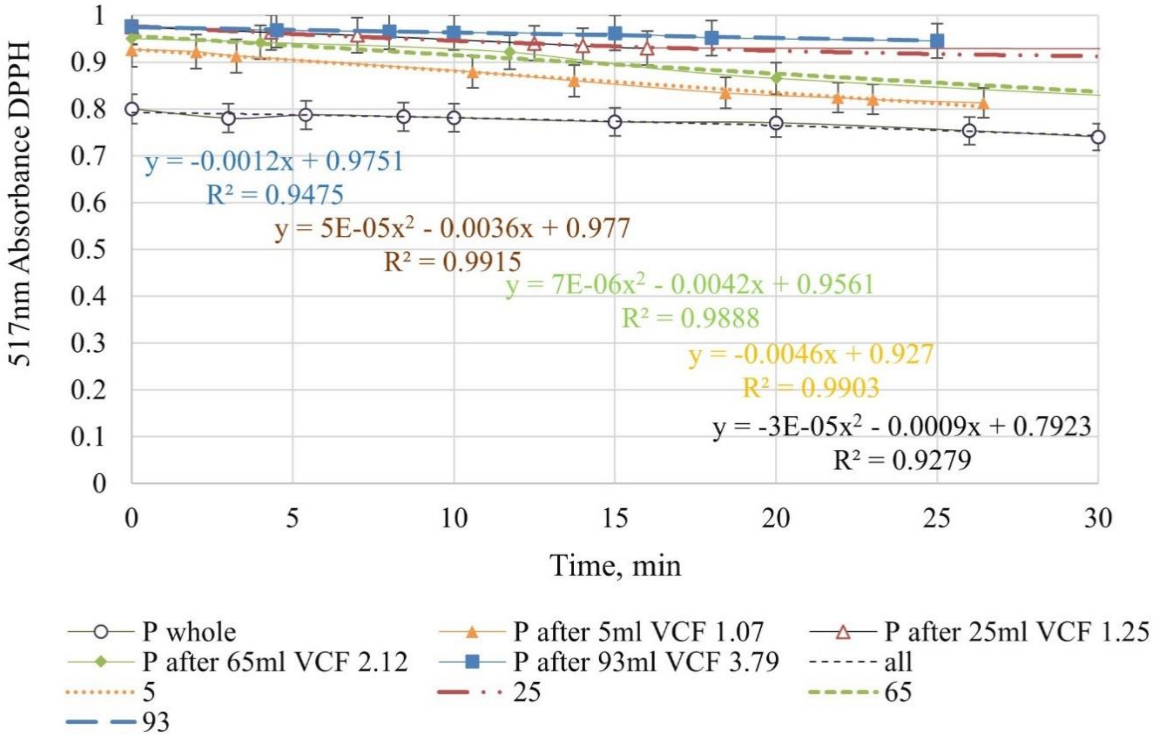


Figure 5. Antioxidant activity during nanofiltration at 50°C. Kinetics of reaction between DPPH radicals and permeate solutions recovered at different VCFs. At statistical standpoint with sample “P whole” the Variance at the beginning 0-1 min is 0.005948, without “P whole” the Variance is 0.0005385, which is more than one magnitude less

These fluctuations in the antioxidant action could be attributed to possible changes in membrane selectivity over time. Pore blocking and an enhanced sieving effect due to the increased fouling layer probably modify the instant retention of permeated antioxidant compounds. This reflects the very complex nature of fouling mechanisms in NF membranes caused by solute-solute and solute-membrane interactions which cannot be easily identified and predicted. Statistical analysis for level of significance $p < 0.05$ where the F value is 13.76, which is greater than F critical at 2.66 so rejecting the null hypothesis that the aliquots are the same in our experiments – they are different at their DPPH antioxidant capacity and at their DPPH antioxidant activity kinetics. Our results are comparable to the results by other researchers that UHT milk reaches higher values ($p < 0.05$) in all assays than MF and HQ milk, whereas the values are $p > 0.05$ (Manzi and Durazzo 2017). Protein content, added probiotic microflora and type and quality of flavouring preparations are main contributors for antioxidant properties of fermented milks (Najgebauer-Lejko and Sady 2015), which shows good prospects for future investigation. Genetically manipulated milk products are also a topic in the scientific world (Khan et al. 2019a).

Conclusions

NF membrane with MWCO of 300 Da can be considered as a slightly open structure for a complete retention of skimmed milk components, but may be suitable for simultaneous concentration and partial demineralization at moderate operating conditions. Appropriate sequencing of UF/NF process may enhance the separation performance (decreased flux decline and rejection) able to produce an UF permeate with a reduced organic load, thus minimizing the complexity of the fouling layer. Increasing the temperature from 20±2°C to 50±2°C improves the flux with 40%, meanwhile the process is 40% more time efficient with no significant DPPH antioxidant capacity losses. Supporting our proposal for 50±2°C processes, is the statement that lipophilic antioxidants are durable at high temperatures, while being more important than hydrophilic antioxidants for organisms, pointing at our decision as a healthy production choice.

References

Adebayo-Tayo B., Fashogbon R. In vitro antioxidant, antibacterial, in vivo immunomodulatory, antitumor and haematological potential of exopolysaccharide produced by wild type and mutant Lactobacillus delbrueckii subsp. Bulgaricus. *Heliyon*, 2020, 6(2): E03268. https://doi.org/10.1016/j.heliyon.2020.e03268

Bondet V., Brand-Williams W., Berset C. Kinetics and mechanisms of antioxidant activity using the DPPH• free radical method. *LWT - Food Science and Technology*, 1997, 30(6): 609-615. https://doi.org/10.1006/fstl.1997.0240

Crowley S.V., Caldeo V., McCarthy N.A., Fenelon M.A., Kelly A.L., O'Mahony J.A. Processing and protein-fractionation characteristics of different polymeric membranes during filtration of skim milk at refrigeration temperatures. *International Dairy Journal*, 2015, 48(9):23-30. https://doi.org/10.1016/j.idairyj.2015.01.005

Doudiès F., Loginov M., Hengl N., Karrouch M., Leconte N., Garnier-Lambrouin F., Pérez J., Pignon F., Gésan-Guiziou G. Build-up and relaxation of membrane fouling deposits produced during crossflow ultrafiltration of casein micelle dispersions at 12°C and 42°C probed by in situ SAXS. *Journal of Membrane Science*, 2021, 618(1): 118700. https://doi.org/10.1016/j.memsci.2020.118700

El-Fattah A. A., Azzam M., Elkashef H., Elhadydy A. Antioxidant properties of milk: Effect of milk Species, milk fractions and heat treatments. *International Journal of Dairy Science*, 2020, 15(1): 1-9. https://doi.org/10.3923/ijds.2020.1.9

Fox P. F., Uniacke-Lowe T., McSweeney P. L. H., O'Mahony. J. A. *Dairy chemistry and biochemistry* (Second Edition). Springer Cham, Springer International Publishing Switzerland 2015, 584 pages, Hardcover ISBN: 978-3-319-14891-5, eBook ISBN: 978-3-319-14892-2, https://doi.org/10.1007/978-3-319-14892-2

Grazyna C., Hanna C., Adam A., Magdalena B.M. Natural antioxidants in milk and dairy products. *International Journal of Dairy Technology*, 2017, 70(2): 165-178 https://doi.org/10.1111/1471-0307.12359

Khan I.T., Nadeem M., Imran M., Ayaz M., Ajmal M., Ellahi M.Y., Khalique A. Antioxidant capacity and fatty acids characterization of heat-treated cow and buffalo milk. *Lipids in Health and Disease*, 2017, 16(8): 163. https://doi.org/10.1186/s12944-017-0553-z

Khan I.T., Bule M., Ullah R., Nadeem M., Asif S., Niaz K. The antioxidant components of milk and their role in processing, ripening, and storage: Functional food. *Veterinary World*, 2019a, 12(1): 12-33.

https://doi.org/10.14202/vetworld.2019.12-33

Khan I.T, Nadeem M., Imran M., Ullah R., Ajmal M., Hayat Jaspal M.H. Antioxidant properties of milk and dairy products: a comprehensive review of the current knowledge. *Lipids in Health Disease*, 2019b, 18(2): 41. https://doi.org/10.1186/s12944-019-0969-8

Kumar, P., Sharma, N., Ranjan R., Kumar S., Bhat Z.F., Jeong, D.K. Perspective of membrane technology in dairy industry: A review. *Asian-Australasian Journal of Animal Sciences*, 2013, 26(9): 1347-1358. https://doi.org/10.5713/ajas.2013.13082

Li H., Hsu Y., Zhang Z., Dharsana N., Ye Y., Chen V. The influence of milk components on the performance of ultrafiltration/diafiltration of concentrated skim milk, *Separation Science and Technology*, 2016, 52(2): 381-391. https://doi.org/10.1080/01496395.2016.1217243

Luo J., Cao W., Ding L., Zhu Z., Wan Y., Jaffrin M. Y., Treatment of dairy effluent by shear-enhanced membrane filtration: The role of foulants, Separation and Purification Technology, 2012, 96(8): 194-203. https://doi.org/10.1016/j.seppur.2012.06.009

Mann S., Shandilya U.K., Sodhi M., Kumar P., Bharti V.K., Verma P., Sharma A., Mohanty A., Mukesh M. Determination of antioxidant capacity and free radical scavenging activity of milk from native cows (*Bos indicus*), exotic cows (*Bos taurus*), and riverine buffaloes (*Bubalus bubalis*) across different lactation stages. *International Journal of Dairy Processing & Research*, 2016, 3(4): 66-70. http://doi.org/10.19070/2379-1578-1600013

Manzi P., and Durazzo A. Antioxidant properties of industrial heat-treated milk. *Journal of Food Measurement and Characterization*, 2017, 11(5): 1690-1698. https://doi.org/10.1007/s11694-017-9549-7

Meyer P., Petermeier J., Hartinger M., Kulozik U. Concentration of skim milk by a cascade comprised of ultrafiltration and nanofiltration: investigation of the nanofiltration of skim milk ultrafiltration permeate. *Food Bioprocess Technology*, 2017, 10(11): 469-478. https://doi.org/10.1007/s11947-016-1836-5

Najgebauer-Lejko D., Sady M. Estimation of the antioxidant activity of the commercially available fermented milks. *Acta Scientiarum Polonorum Technologia Alimentaria*. 2015, 14(4): 387–96. https://doi.org/10.17306/J.AFS.2015.4.38

Ng S.Y., Haribabu M. , Harvie D.J.E., Dunstan D.E., Martin G.J.O. Mechanisms of flux decline in skim milk ultrafiltration: A review. *Journal of Membrane Science*, 2017, 523(2): 144–162. https://doi.org/10.1016/j.memsci.2016.09.036

Roy Y., Lienhard J.H., 2019. Factors contributing to the change in permeate quality upon temperature variation in nanofiltration. Desalination, 2019, 455(4): 58–70, http://doi.org/10.1016/j.desal.2018.12.017

Zacharof M., Lovitt, R. Bacteriocins produced by lactic acid bacteria a review article. *APCBEE Procedia*, 2012ab, 2(4): 50–56. https://doi.org/10.1016/j.apcbee.2012.06.010

Zhang H., Yanyao T., Yubin H., Jiefeng P., Preparation of low-lactose milk powder by coupling membrane technology. *ACS Omega*, 2020, 5(15): 8543–8550. https://doi.org/10.1021/acsomega.9b04252