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Industrial dairy wastewater purification by shear-enhanced membrane filtration: The effects of vibration

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Abstract. Membrane fouling is a major challenge limiting the use of membrane applications. In this study high induced shear rates were utilized at the membrane surface in order to reduce the organic and inorganic scaling by using the torsional vibration of flat sheet membranes. The performances of a vibratory shear-enhanced processing (VSEP) system for the ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) membrane filtration of industrial dairy wastewater were investigated. The vibration and non-vibration methods were compared with the same membrane and operational parameters during the purification of real dairy industrial process wastewater. In the initial experiments, short-term tests were carried out in which the effects of vibration amplitude, recirculation flow rate and transmembrane pressure were measured and compared. The permeate flux, turbidity, conductivity and chemical oxygen demand (COD) reduction of dairy wastewater were investigated by using UF, NF and RO membranes with vibration and non-vibration methods. In the subsequent experiments, concentration tests were also carried out. Finally, scanning electron microscopy (SEM) revealed that the vibration method gave a better performance, which can be attributed to the higher membrane shear rate, which reduces the concentration of solids at the membrane, and the transmission.

Keywords: shear-enhanced membrane filtration; VSEP; dairy wastewater; membrane fouling; SEM pictures

1. Introduction

Increasing industrialization and rapid urbanization have enhanced the rate of water pollution considerably. The European and Hungarian regulations have become more stringent, with the aim of protecting the environment (Sarkar *et al*. 2006). Among the food industries the dairy industry requires and eventually discharges huge volume of water, with wide fluctuations in the quality of effluent (Farizoglu and Uzuner 2011). Since water is used in the steps of dairy technology, including cleaning, sanitization, heating, cooling and floor washing, it generates large volumes of effluents, mainly white waters (Luo *et al*. 2011). Dairy wastewater is distinguished by its high biochemical oxygen demand (BOD) and chemical oxygen demand contents, and its high levels of dissolved or suspended solids, including fats, oils and grease, nutrients such as ammonia or minerals and phosphates, milk components (lactose and proteins) and cleaning chemicals and detergents, and therefore requires appropriate treatment before disposal (Frappart *et al*. 2008).

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If discarded without treatment, this effluent results in water eutrophication, due to the presence of nitrogen and phosphorus (Luo and Ding 2011). Thus, the treatment of dairy effluent is of crucial importance not only for the environment, but also for there cycling of the water. A number of technologies have been applied for the treatment of dairy wastewater, such as coagulation (Sengil and Ozacar 2006), an ecological treatment system (Lansing and Martin 2006), and anaerobic and aerobic reactors (Beszédes *et al*. 2011, Demirel *et al*. 2005). However, all of the biological treatment systems (including aerobic and anaerobic processes) that have been used have their own disadvantages, caused by the considerable operational difficulties (Kushwaha *et al*. 2010). Several works have focused on the treatment of dairy effluents have demonstrated that membrane operation has often been considered to be a promising method: ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) (Vourch *et al*. 2008). A few publications have indicated that NF and RO are convenient operations for the treatment of effluents at source and achievement of the set targets (Balannec *et al*. 2002, Luo *et al*. 2011). The concentrated retentate of dairy industrial wastewater can be precipitated by coagulation to obtain a feeding supplement for animals (Dyrset *et al*. 1998), or can be treated by anaerobic digestion to collect renewable energy sources (Mohan *et al*. 2008), this being regarded as an economical and environment-friendly process for the treatment of dairy wastewater. The significant improvements in the reliability and cost effectiveness of membrane technology have increased the probability of reuse and the extent of recycling. In recent times, the development of membranes with high flux (J) and rejection characteristics has enhanced the possibilities of water reuse and recycling. Unfortunately, membrane fouling and the resulting decline in J, still remains a major bottleneck preventing wide spread application. In order to solve the problem, many researchers have investigated the potential applicability of rotating and vibration modules in wastewater treatment (Akoum *et al*. 2004, Shi and Benjamin 2011). In order to control the decline in J during the concentration of dairy effluents, a vibration method could be use. Vibratory shear-enhanced processing (VSEP) module can be used to prevent deposition with increasing the shear rate close to the membrane surface by vibrating the membrane module. Notable feature of this technique is that the shear applied is independent of the cross-flow velocity (Brans *et al*. 2004). Accordingly, a low cross-flow velocity can be applied, avoiding a decreasing trans-membrane pressure (TMP) along the membrane. Other means of increasing the shear close to the membrane surface are spacers, turbulence promoters, and inserts that create flow instabilities, such as Dean vortices or micro-turbulences (Winzeler and Belfort 1993). The very few reports relating to the treatment of dairy wastewater by VSEP have shown that NF or RO is adequate for the concentration of milk components (Akoum *et al*. 2005, Luo *et al*. 2011). Akoum *et al*. (2005) investigated the treatment of dairy process waters by using VSEP with NF and RO membranes and found that VSEP outperforms conventional cross-flow filtration in NF in terms of J and permeate COD reduction. For UF protein adsorption caused a decline in J, mitigated by the presence of casein micelles and whey proteins.

In the present study, the performances of a VSEP system for the UF, NF and RO of industrial dairy wastewater were investigated, and the vibration and non-vibration modes were compared.

2. Materials and methods

2.1 Feed dairy wastewater

The dairy industry wastewater was provided by Sole-Mizo Ltd. (Szeged, Hungary); its main characteristics are given in Table 1.

Table 1 Wastewater parameters

Conductivity [mS cm^{-1}]	Turbidity [NTU]	COD [mg L^{-1}]	pH
1.3	1150	6175	6.62

2.2 Analytical methods

The COD was determined in test tubes with an ET 108 digester and a PC CheckIt photometer (Lovibond, Germany). The conductivity and pH were measured with a multi-parameter analyser (Consort C535, Belgium). The turbidity of the samples was determined with a HACH2100N turbidimeter (Hach, Germany).

2.3 VSEP experimental set-up and membranes

The filtration module was a VSEP Series L (New Logic Research Inc., Emeryville, CA, USA). In Fig. 1 the VSEP flowing process could be seen. It was equipped with a single circular membrane with a surface area of 503 cm^2 (outer radius 13.5 cm outer radius, inner radius 4.7 cm). The vertical shaft supporting the membrane housing acts as a torsion spring which transmits the oscillations of a lower plate in the base, which is vibrated by an eccentric drive motor. As a result, the housing containing the membrane oscillates azimuthally with displacement amplitude *d* which we adjusted to be 2.54 cm (1 inch) on the outer rim at the resonant frequency of 55 Hz. All experiments with non-vibration method were carried out with the same VSEP L device, membranes and operational parameters. Table 2 shows the characteristics of the examined membranes.

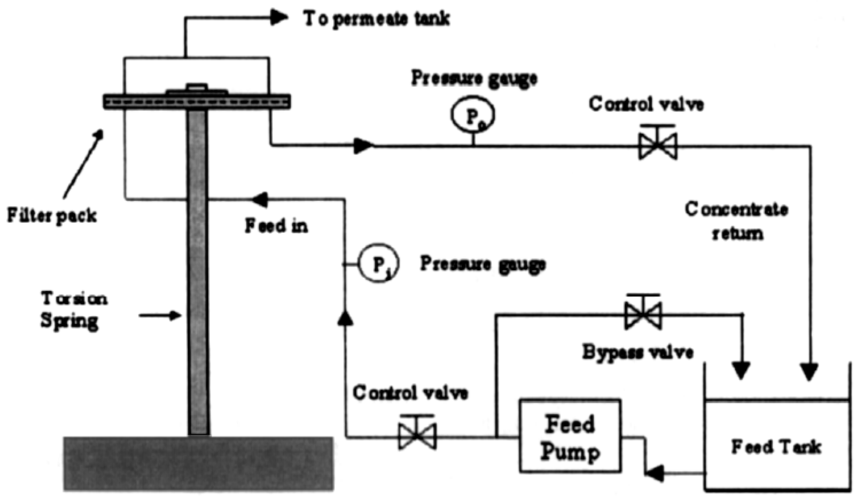


Fig. 1 Flow process diagram of VSEP

Table 2 Membrane characteristics

Membrane surface area	503 cm^2		
Filtration type	UF	NF	RO
Membrane brand name	PES-10 SYN	NF	LFC
Pore size, MW cut-off [Da]	10 000	240	30
Membrane material	polyethersulfone	thin film composite	thin film composite
Vendor	Synder	Filmtec	Hydranautics
NaCl rejection [%]	–	61.7	98.0
$MgSO_4$ rejection [%]	–	93.4	–
pH tolerance	2-12	2-11	2-12
Temp. tolerance [°C]	90	60	60

2.4 Scanning electron microscopy (SEM) measurements

SEM was performed with a Hitachi S-4700 field emission scanning electron microscope operated at an acceleration voltage of 10 kV in ultrahigh resolution mode. To analyse the gel layer after UF and NF concentration tests with the vibration and non-vibration methods, 250 and 10 000 - fold top magnification pictures were recorded and compared.

2.5 Test protocol

The measurements of VSEP were carried out at 50 ± 1°C with the UF, NF and RO membranes. The short-term tests were performed with different cross-flow velocities ranging from 7.57 to 15.14 L min^{-1}.

2.5.1 Short-term tests

For rapid evaluation of the membrane performance in dairy effluent treatment, short-term tests with full permeate and retentate recycling were performed. Before measurement of the membrane hydraulic permeability, prefiltration was carried out with deionized water for 60 min at the tested parameters (temperature T, recirculation flow rate q_V, transmembrane pressure TMP) to ensure membrane stabilization. The feed tank was then filled with wastewater, the feed pump was started and the vibration amplitude (A) was adjusted by increasing the frequency if the vibration method was used. When the desired parameters were reached and had stabilized, the wastewater J was measured and samples were collected from the permeate 10 min after each parameter increment.

2.5.2 Concentration tests

Before the concentration measurements, the membranes were prepressured with deionized water for 60 min in order to remove the excess of preservation chemicals attached to the new membranes. After stabilization, the pure water flux of the membranes was measured. The thermostatic controlled feed tank was then filled with 10 L wastewater, the feed pump was started and the vibration frequency was adjusted. When the desired parameters (A, T, q_V and TMP) were reached and had stabilized (under a pressure of 0.8 MPa for UF 2 MPa for NF), the concentration test was started with collection of the permeate separately in order to increase the volume reduction ratio (VRR). The tests were stopped when the permeate volume reached VRR = 5. q_V was constant at 15.14 L min^{-1} during the concentration tests.

3. Results and discussion

3.1 Effects of operational parameters, short-term tests

3.1.1 Variation of J with q_V

In order to evaluate the effect of q_V on the membrane separation process, short-term tests were performed with different cross-flow velocities ranging from 7.57 to 15.14 L min^{-1}. Since the applied TMPs were different, 0.8, 2 and 3 MPa for UF, NF and RO, respectively, effective specific permeate fluxes ($J_{sp,\,eff}$) were calculated by using the following equation

$$J_{sp,\,eff} = \frac{\text{Average Flux}}{\text{Effective TMP}} = \frac{J}{\text{TMP} - \Delta\pi} \quad [\text{L m}^{-2}\ \text{h}^{-1}\ kPa^{-1}] \tag{1}$$

where J = average permeate flux [Lm^{-2}h^{-1}]; TMP = transmembrane pressure [MPa] and $\Delta\pi$ = osmotic pressure difference across the membrane [MPa].

The high shear rate on the surface of the membrane is caused by both q_V and membrane vibration. Fig. 2 shows the values of $J_{sp,\,eff}$ as a function of q_V with non-vibration method (A=0). Increasing q_V increased the $J_{sp,\,eff}$ in all cases. Higher q_V causes higher shear-induced back-diffusion and can decrease the concentration of solutes and the precipitation of particles on the membrane, thereby reducing the extent of membrane fouling, with a resulting higher $J_{sp,\,eff}$. Although the shear rate can be increased by elevating q_V, this induces large pressure drops in the modules.

VRR during the concentration tests of the wastewater treatment was determined as the ratio of the feed volume at the beginning of operation (V_{feed}) to the retentate volume after a certain time (V_{conc}) (Krstic *et al*. 2007)

$$VRR = \frac{V_{feed}}{V_{conc}} \tag{2}$$

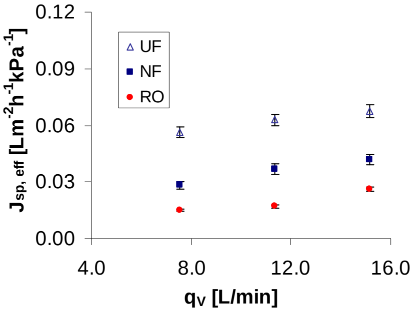


Fig. 2 $J_{sp,\,eff}$ change during increasing q_V at VRR = 1 with vibration amplitude A = 0

3.1.2 Variation of $J_{sp, eff}$ with membrane vibration *A*

The creation of a more effective high shear rate on the membrane without a pressure drop would be possible with a moving part such as the high tangential vibration of the membrane module with increasing *A*. Fig. 3 represents the values of $J_{sp, eff}$ versus *A*. It can be seen that increasing *A* increased $J_{sp, eff}$. This was more pronounced in the case of UF. Higher *A*, resulting in a higher shear rate on the surface of the membrane, can reduce the accumulation of solutes on the surface, leading to a higher $J_{sp, eff}$ at constant *TMP*. Bian *et al*. (2000) have also found that the concentration polarization of humic substances in membranes can be reduced by increasing the shear rate, due to the increase in mass transfer coefficient and the decrease in the concentration on the membrane surface with increasing shear rate (Bian *et al*. 2000). In UF, however, the higher shear rate changed the transfer of solutes through the membrane pores, because UF involves a much higher pore size than for NF or RO. However, a higher shear rate will waste much energy and increase the abrasion of the equipment.

3.1.3 Variation of J with TMP

Variations in *J* a function of *TMP* are displayed in Fig. 4 with non-vibration method (*A* = 0). For the UF membrane, *J* increased distinctly with increasing *TMP*, from 0.2 to 1.2 MPa, whereas for NF and RO *J* increased on a much smaller scale. Furthermore, the *J* values of NF and RO membranes become pressure-independent (mass transfer-limited regime) at a critical *TMP* of about 2.5 MPa for NF and at 3 MPa for RO.

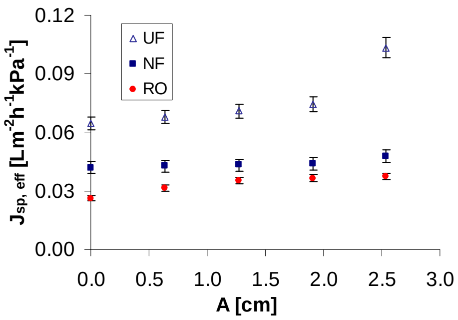


Fig. 3 $J_{sp, eff}$ change during increasing vibration amplitude *A* at *VRR* = 1 with q_V of 15.14 L min^{-1}

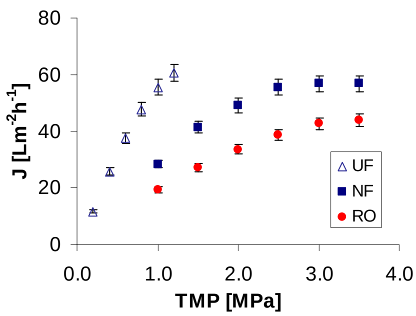


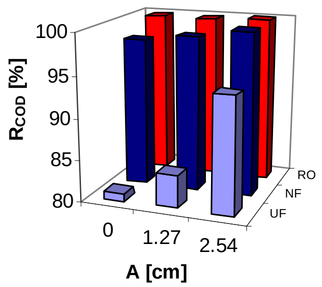
Fig. 4 Variation of *J* with *TMP* at *VRR* = 1 with vibration amplitude *A* = 0

3.1.4 Variation of retention with membrane vibration A

After measurement of the COD, turbidity and conductivity of the permeate, the membrane retention was calculated by means of Eq. 3 and shown as in Figs. 5 and 6. The selectivity of a membrane for a given solute was expressed by the average retention (R) (Akoum *et al*. 2002)

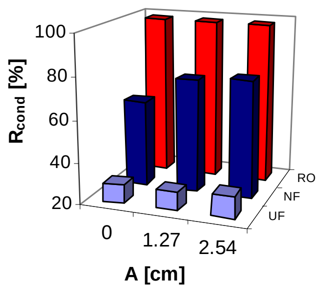
$$R = \left(1 - \frac{c}{c_0}\right)100 \qquad [\%] \tag{3}$$

where c is the average concentration of the solute in the permeate phase, and c_0 is the concentration of the solute in the bulk solution. Turbidity analysis demonstrated that the membrane retention was always higher than 99.33% (with a permeate value of 7.7 NTU); it was lowest for UF, resulting in transparent, visually clear permeates. The R_{COD} values obtained with the three different membrane systems (Fig. 5) showed that the organic content was almost



	0	1.27	2.54
UF	80.86	84.00	93.85
NF	97.82	98.59	99.64
RO	99.74	99.74	99.95

Fig. 5 Removal efficiency of COD with *A* at *VRR* = 1



	0	1.27	2.54
UF	29.01	29.11	30.71
NF	61.90	75.01	76.62
RO	97.80	98.09	97.99

Fig. 6 Removal efficiency of conductivity as a function of *A* at *VRR* = 1

completely retained by the NF (> 97.82%) and RO membranes (> 99.40%) and largely retained by the UF membrane (> 80.86%), resulting in levels of 135, 37 and 1182 mg O_2 L^{-1} permeate COD, respectively.

When the UF membrane was used, the organic content (mainly protein, lactose and casein) could pass through the membrane completely due to their much higher molecular weight cut-off (MWCO) (10 000 Da) than the MW of lactose (342.30 Da), but the proteins were almost completely rejected (Luo *et al*. 2011). Furthermore, the average size of casein micelles is about 110 nm, while the average pore size is less than 10 nm, even for the UF membrane, and thus casein micelles cannot pass through these membranes (Akoum *et al*. 2002). This was confirmed by the direct observations, as all the permeate samples were very clear, because of the low turbidity values. The vibration increased the retention in all cases, but this was more apparent with the highest porous UF membrane. In the short-term UF experiments, the highest J was 83 L m^{-2} h^{-1} (at 0.8 MPa, 50°C) and 380 mg O_2 L^{-1} permeate COD (R_{COD} = 93.85). In NF, the highest J was 96 L m^{-2} h^{-1} (at 2 MPa, 50 °C) and 22 mg O_2 L^{-1} permeate COD (R_{COD} = 99.6), and in RO the highest J was 112 L m^{-2} h^{-1} (at 3 MPa and 50°C) with 3 mg O_2 L^{-1} permeate COD (R_{COD} = 99.95). From the permeate electrical conductivity and Eq. (3), the retention of salt was calculated; this is depicted in Fig. 6.

The retention increased, due to the lower salt content on the permeate side when A increased, resulting in a higher shear rate on the membrane surface. The retained salts led to higher osmotic pressure on the retentate side of the NF and RO membranes resulting in higher retentions.

3.2 Concentration tests

In this series, the permeate was not returned to the feed tank, and 10 L of industrial dairy wastewater was concentrated to 2 L with the UF and NF membranes detailed in Table 2. For concentration tests a single membrane treatment was used to concentrate dairy effluents.

To know the flux decline, the relative flux (J_{rel}) was calculated via the following equation

$$J_{rel} = \frac{J}{J_{water}} \tag{4}$$

where J = average permeate flux [L m^{-2} h^{-1}] and J_{water} = water permeate flux [L m^{-2} h^{-1}].

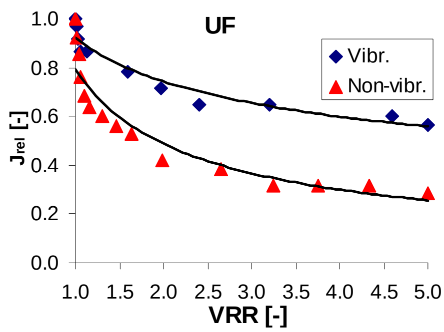


Fig. 7 The effect of vibration on the profiles of the decline in J for UF treatment

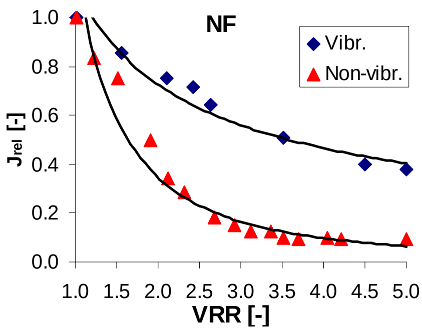


Fig. 8 The effect of vibration on the profiles of the decline in *J* for NF treatment

3.2.1 Variation of J_{rel} with VRR

In the concentration tests (Figs. 7 and 8) the vibration amplitude was set to 24.5 mm in order to ensure the possibly highest shear rates at the membrane surface. Using vibration methods, due to higher shear rates, almost two times larger values of *J* were observed. This trend was independent of membrane type.

In NF, *J* was pressure-limited and its decay was mainly due to the rise in osmotic pressure.

The decay in *J* is shown in Figs. 7 and 8.

At the end of the concentration tests, J_{rel} had decreased to 0.57 and 0.29 for UF, and to 0.38 and 0.09 for NF with the vibration and non-vibration methods, respectively, as VRR increased from 1 to 5. With increasing VRR, the permeate COD soared to 512 mg O_2 L^{-1} and 90 mg O_2 L^{-1} for UF and NF, respectively.

3.2.2 Variation of conductivity with VRR

The permeate and concentrate conductivities changing were shown during UF (Fig. 9) and NF (Fig. 10) concentration tests as a function of VRR. Good overall salt retention, as measured by the difference in conductivity between the permeate and the retentate, was achieved in NF concentration tests (Fig. 10). It can be seen that the conductivity slowly increased continuously and only a small difference was observed between the values of the vibration and non-vibration methods. During the NF test, the permeate conductivities with the non-vibration method were higher than those with the vibration method, due to the lower retention rates. Since the vibration changed the structure of the gel layer on the surface of the membrane, the ion transmission through the pores also changed (see Fig. 11).

3.3 Membrane fouling examination by SEM

SEM images of the scale that formed on the membrane surface during the concentration tests of dairy wastewater are shown in Fig. 11 at 250 - fold magnification. With the non-vibrating method, the membrane was almost uniformly covered with scale (Fig. 11 NV_UF; NV_NF), but with the vibration method the morphology of the scale layer changed (Fig. 11 V_UF; V_NF). The scale in UF appeared more compacted structure relative to NF in the cases of without vibration. However in the cases of vibration the NF gel layer structure seen in Fig. 11 is seem to be more compact, which could be explained by the higher salt rejection results. To examine the differences in the NF

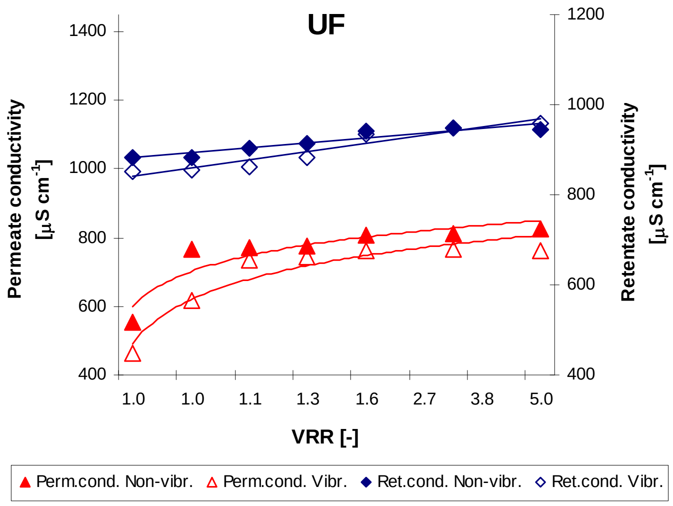


Fig. 9 Retentate and permeate conductivity profiles for UF treatment

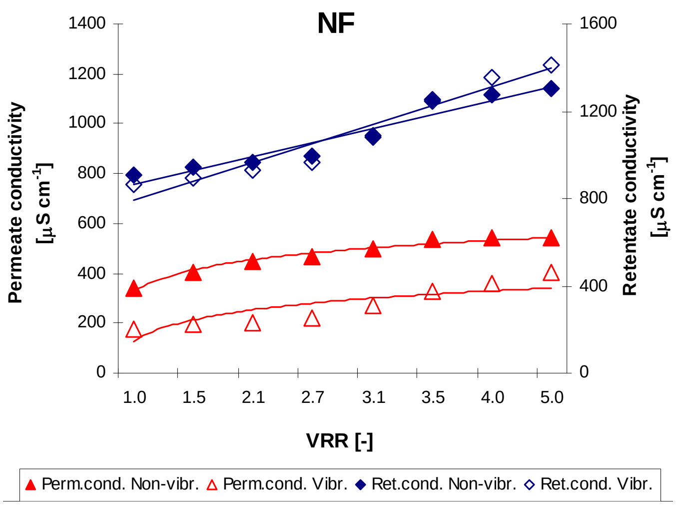


Fig. 10 Retentate and permeate conductivity profiles for NF treatment

gel layer structure with the vibration and non-vibration methods, SEM pictures at higher magnification (10 000 – fold) were also taken (Fig. 12). The scale on the membrane after NF treatment of the dairy wastewater with the vibration (V_NF_2) and the non-vibration (NV_NF_2) methods was examined. The entire membrane surface was covered by a scale layer that had approximately uniform morphology for both the vibration and non-vibration methods, but the structures and shapes of the scale differed: the scale in the non-vibration method formed a more aggregated and continuous, overcrowded layer, whereas the scale in the vibration method comprised a lower number of smaller and mainly roundish particles in only one layer. This shows that the vibration changed the structure and average particle diameter of the gel layer.

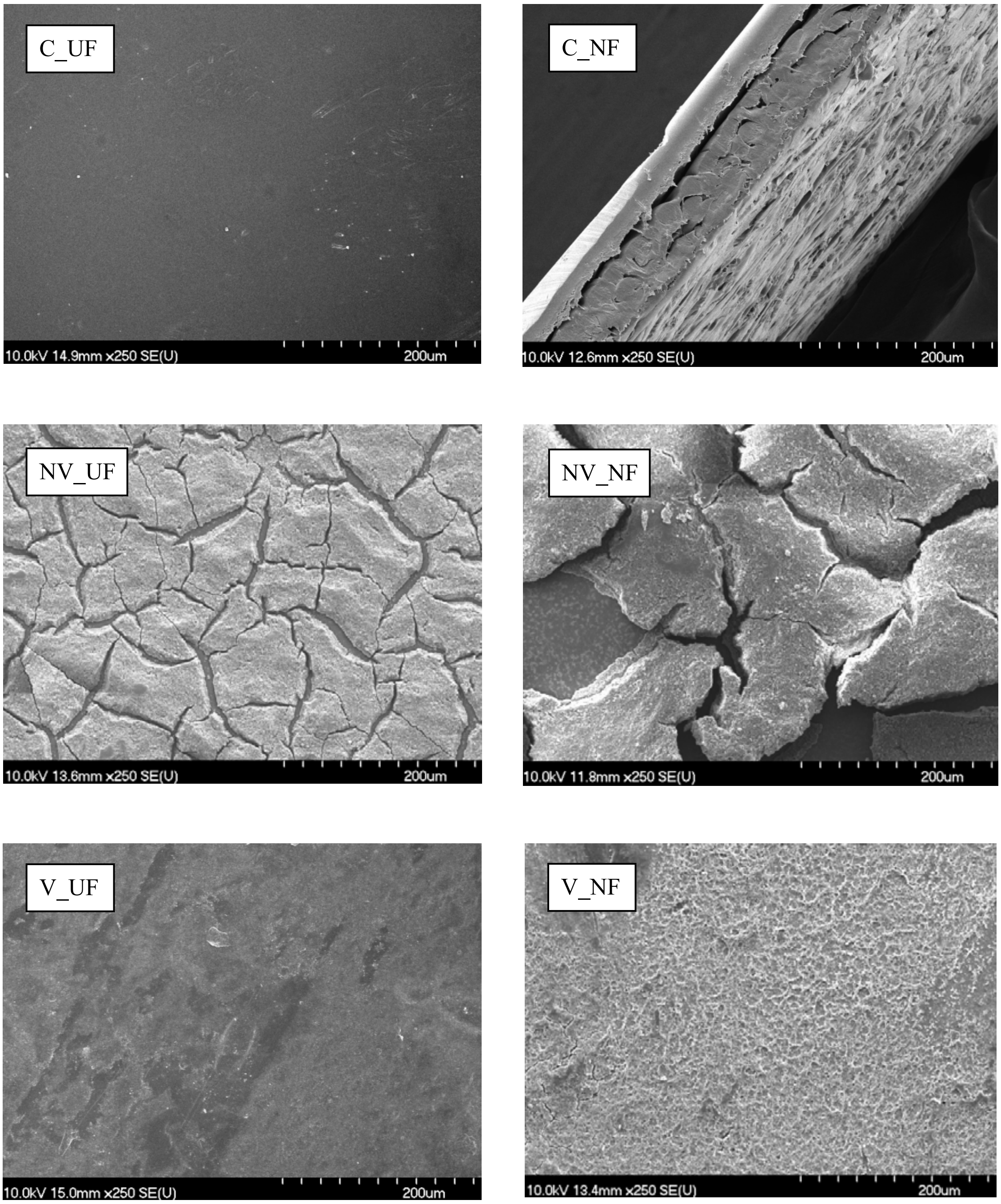


Fig. 11 SEM images of clean UF (C_UF) and NF membrane (C_NF), UF and NF membrane without vibration (NV_UF; NV_NF) and UF and NF membranes fouled at a vibration amplitude of 2.54 cm (V_UF; V_NF) and after the long-term testing of dairy wastewater treatment. All specimens were taken from a location 10 cm from the center of the membrane (250-fold top magnification, except C_NF, cross-section)

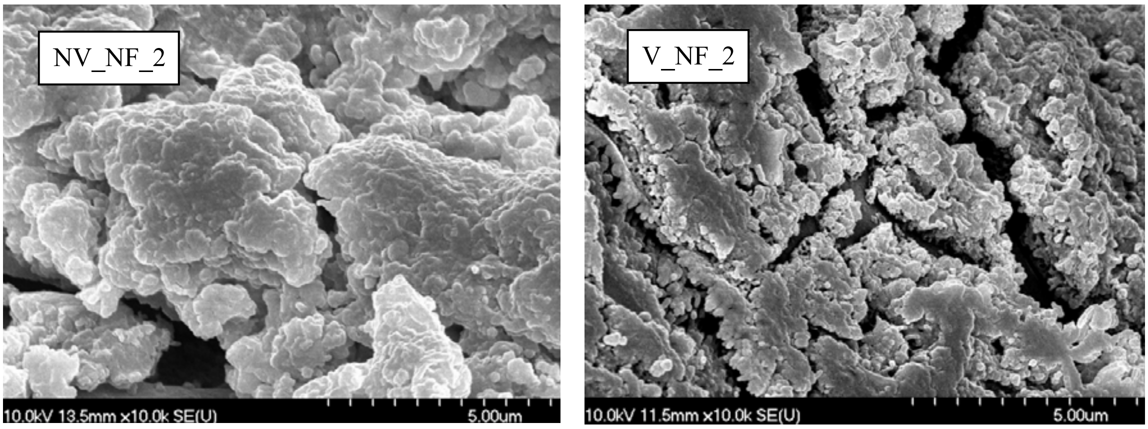


Fig. 12 SEM images of NF membrane fouled without vibration (NV_NF_2) and at a vibration amplitude of 2.54 cm (V_NF_2) after long-term testing of dairy wastewater treatment. All specimens were taken from a location 10 cm from the centr of the membrane (10 000-fold magnification top)

Table 3 COD values of single membrane filtration

COD [mg O_2 L^{-1}]	UF	NF	RO
non-vibration	1181.9	134.6	16.1
vibration method	380.0	22.0	3.0

4. Conclusions

In this work the performance of a VSEP system for the UF, NF and RO membrane filtration of industrial dairy wastewater was investigated. Vibration of UF, NF and RO membranes in an L-mode VSEP system reduced the level of fouling in the treatment of dairy industrial wastewater. In short-term tests vibration reduced the rates at which *J* declined, and increased the practical recovery. Treatment with vibration led to the retention of most ions: > 30.7% for UF, > 76.6% for NF and > 98% for RO. It may be concluded from these studies that each individual UF, NF and RO treatment could improve the treatability of dairy industrial wastewater, but NF and RO could generate treated effluents that meet the strict requirement of general European COD threshold limit, i.e., below 150 mg O_2 L^{-1}.

In concentration tests of UF and NF the vibration method greatly reduced the membrane fouling, mainly with gel layer reduction. SEM images indicated that the membrane surface was almost uniformly covered with a scale-forming gel layer with the non-vibrating method in both the UF and the NF systems, but in the methods with vibration, the morphology of the scale layers differed. The scale in UF appeared mostly continuous as, compared with that in NF, and it became more scattered with more open spaces between individual clumps. A higher magnification of the SEM pictures showed that the scale in the NF non-vibration method formed a more aggregated and continuous, overcrowded layer, whereas the scale in the vibration method comprised a lower number of smaller and mainly roundish particles in only one layer. The results showed that NF and

RO single membrane operations allowed the purified water to be released into the environment, but the UF permeate water did not reach the requirements of the COD threshold limit.

Membrane technology is quickly expanding to all industries, including the producers of some very challenging wastewaters, which should be the major target of VSEP applications. Our future aim is to investigate the possible hybrid membrane processes, coupling of VSEP with various pre-treatment and post-treatment processes, to further explore the benefit of module vibration.

Acknowledgments

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