Szent István University

Intensifying the membrane filtration processes in food industry and environment protection using static mixers

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PhD School

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1. INTRODUCTION AND OBJECTIVES

Oil-in-water emulsions are used in large amounts in food- and mechanical industry, and as an industrial waste water, they have to be treated. Environmental regulations limit the concentration of the oil in these contaminating fluids. In case of oily wastewaters in Hungary, the concentration cannot be bigger than 50 mg/L. These microemulsion oils often cannot be adequately separated by settling, special treatment methods are needed. Evaporation is a very energy consuming technique, this separation can be done more economically with membrane filtration as pre-process of evaporation. With ultrafiltration, content of the oil in permeate can be kept below limit, and fluid can be discharged directly into the sewers. Using this method, water content of the retentate can be significantly reduced, and final purity can be obtained by evaporation, reducing the overall energy cost.

In this membrane filtration research, I also used sweet whey as a by-product of cheese production. Nanofiltration can be used for the concentration of proteins and lactose in whey. In industrial practice, spiral wound membrane module is commonly used due to its high filtration surface. To avoid fast fouling of the spacer, suspended solids and lipids has to be removed. This can be done by microfiltration, a subject of my future research.

The primary aim of my work was to install a static mixer inside tubular ceramic membrane and to analyze its effect on permeate flux, specific energy consumption and solute retention.

My further goals were to model the performance of the membrane filtration and to determine the optimal parameters for filtration of the oily emulsions and whey.

As part of my research on oil-in-water emulsions, I have compared ceramic tube membranes equipped with installed static mixer with ceramic capillary membranes and with polymeric flat-sheet membranes, analyzing their influence on filtration performance.

I investigated the applicability of a commercially available Kenics static mixer. It seems that this turbulence promoter has a positive effect on the permeate flux and the retention of the membrane, but it's main disadvantage is the high pressure drop along the membrane.

On the basis of these results I have studied new geometric configuration of turbulence promoter, which does not generate significant pressure drop along membrane, but it keeps two main advantages of the filtration with Kenics static mixer. Since, I could not find any static mixers with these specifications on the market, I started with analyzing new forms with computational fluid dynamics (CFD) simulation. Final step was to produce novel static mixers and to test them in real conditions.

2. MATERIALS AND METHODS

2.1.Experimental apparatus

I carried out my experiments with a lab-scale conventional membrane filtration apparatus (crow-flow), designed and assembled on the Food Engineering Department, Szent István University, (Fig. 1).

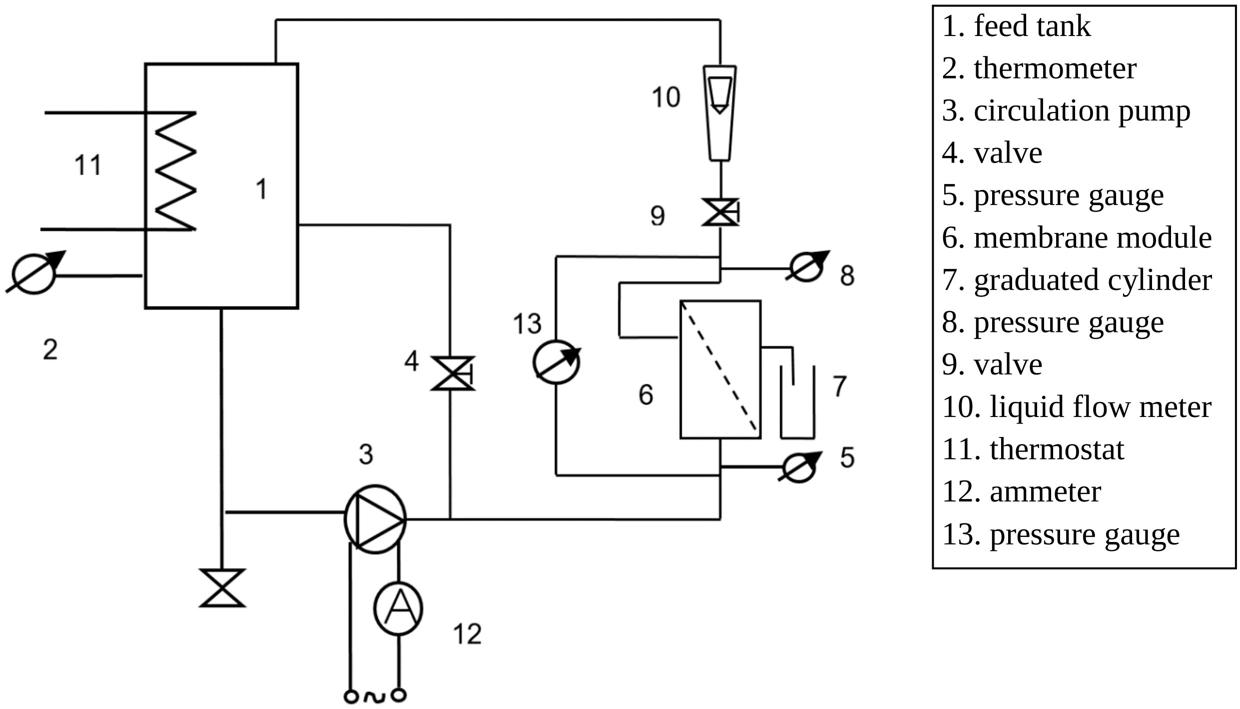

Fig.1 *Schematic diagram of the experimental set-up*

2.2.Materials

Concentration of the stable oil-in-water emulsion used as a model solution, was 5 m/m % of oil, and it was prepared by MOL Unisol oil (MOL Plc., Hungary) and distilled water.

In my experiments with whey, I used sweet whey, a by-product of cheese production. Composition of raw whey is shown in Table 1.

Table 1 *Composition of raw whey [m/m %]*

Solid content	Fat	Protein	Lactose	Other
7.55%	0.18%	0.67%	5.45%	1.25%

2.3. Used membranes

For ultrafiltration of oil-in-water emulsion I used polymeric flat-sheet-, ceramic tube- and ceramic capillary membranes. The main characteristics of membranes can be seen in Table 2.

Table 2 *Membranes used for filtration of oil-in-water emulsions*

Manufacturer	**Notation**	**Type**	**Material**	**Pore size**	**Area**
BFM Germany	BFM 70100-P	Flat-sheet	PAEK*	100 kDa	470 cm^2
Pall Exekia	TI-70-20-Z	Tube	Zirconia	20 nm	50 cm^2
Hyflux	M20-011-0.04	Capillary	Alumina	20 nm	400 cm^2

*PAEK – Polyaryletherketone

During the microfiltration of whey, 50, 200 and 1400 nm pore size ceramic tube Membralox (Pall, USA) membranes with a filtration area of 50 cm^2 and of 6.8 mm inner diameter were used.

2.4. Static mixers

A commercially available Kenics static mixer was used in experiments carried out with ceramic tubular membranes. Main characteristics: diameter ¼” (6.35 mm), length: 250 mm. Further 5 static mixers were designed, produced and tested.

2.5. Experimental design

For analyzing the effect of the examined parameters (recirculation flow rate (RFR), transmembrane pressure (TMP), static mixer effect (SM)) on permeate flux (J) and retention (R), I used 2^P and 3^P type full factorial designs. For data processing I used Statistica 6.0 software.

2.6. Analytical methods

Concentration of the oil in retentate and permeate were determined by a spectrophotometric assay, measuring the absorbance at a wavelength of 600 nm.

Concentration of whey components were also measured with a spectrophotometric method.

2.7. Computational Fluid Dynamics (CFD)

For computational fluid dynamics I used an open source lattice Boltzmann algorithm (http://www.openlb.org – retrieved 11th June, 2012.). The input for the algorithm was a 3D matrix carrying information of the rectangular space surrounding the membrane with a resolution of 0.1 mm. Other input parameters for simulation included the recirculation flow rate (RFR=50 L/h) and the transmembrane pressure (TMP=200 kPa). Output of the algorithm was also a 3D matrix, where each element contains the velocity and normal vorticity vectors for the specific particle. To analyze and to visually represent this huge amount of data, I used ParaView 3.6.1, an open-source, multi-platform data analysis and visualization application (http://www.paraview.org/ – retrieved 11th June, 2012.).

3. RESULTS

3.1. Intensification of ultrafiltration of oil-in-water emulsion using static mixers

In research with oil-in-water emulsions, I proved that inserting a Kenics static mixer inside ceramic tube membrane has a positive effect on permeate flux and retention of the oil. My experiments confirmed that it is important to choose the recirculation flow rate properly, because using a Kenics static mixer only on lower flow rates has smaller specific energy consumption needed for the filtration. Increasing the recirculation flow rate exponentially increases the frictional pressure-drop along the membrane.

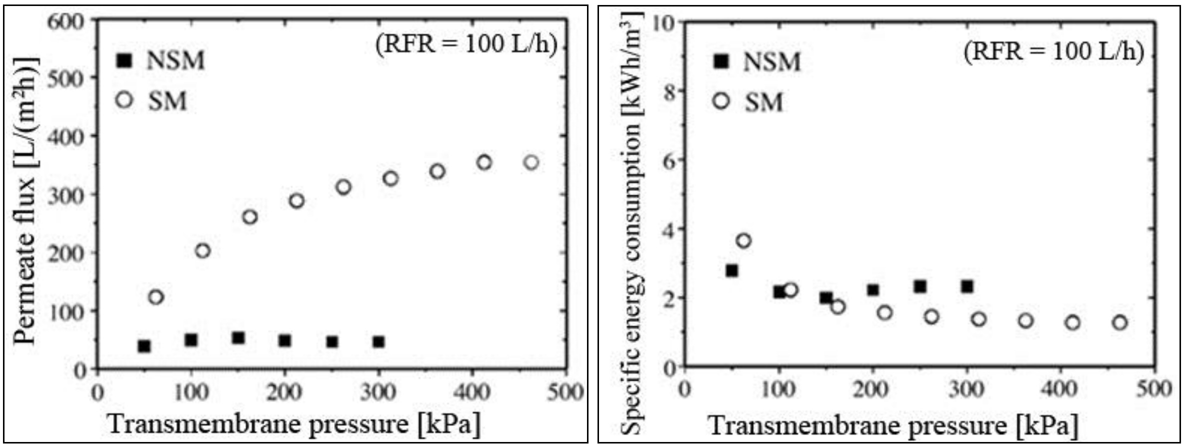

Fig. 2 *Permeate flux (left) and specific energy consumption (right) as function of transmembrane pressure, measured at RFR=100 L/h with- (SM) and without (NSM) static mixer*

Comparative experiments with ceramic tube membranes (with inserted static mixers) and polymeric (PAEK) membranes confirmed that both membrane filtration processes have similar permeate flux (under similar operating parameters). Specific energy consumption was approximately twice due the effect of the static mixer. Much better retention of the oil justifies the additional energy requirement. Overview of the comparison is shown in Table 3.

Table 3 *Overview, comparison of filtration with ceramic membrane equipped with static mixer and polymeric membranes*

	v [m/s]	TMP [kPa]	J [L/(m^{2}h)]	E [kWh/m^3]	Cp [mg/L]	
Ceramic + SM	**1,2**	**150**	**90**	**2,2**	**21**	←stabilized J
	1,8	200	120	3,3	39	
Polymeric	1,2	150	60	1,6	110	
	1,8	**200**	**100**	**1,8**	**151**	←stabilized J

where v: flow velocity, TMP: transmembrane pressure, J: permeate flux, E: specific energy consumption, Cp: concentration of the oil in permeate, “stabilized J”: means those parameters, where raising of the TMP doesn’t have significant effect on permeate flux changing.

My further investigation on oil-in-water emulsion focused on the comparison of ceramic tubular membranes with inserted SM with ceramic capillary membranes. Recirculating flow rate (RFR) ranges were chosen to obtain identical flow velocity range (v) for both filtration processes. The most pronounced difference was observed in permeate fluxes. In Table 4, I show those operating parameters (TMP, RFR), at which the largest permeate flux (J) was reached. Specific energy consumption (E) and retentate of the oil (R) measured under identical circumstances are also included in table.

Table 4 *Highest permeate fluxes and corresponding operating parameters, specific energy consumption and retention of the oil.*

	v [m/s]	RFR [L/h]	TMP [kPa]	**J [L/(m^{2}h)]**	E [J/m^3]	R[%]
Tube m.+SM	1.2	150	400	**300**	$2.6 \cdot 10^6$	99.99
Capillary m.	1.2	1200	400	**45**	$2.3 \cdot 10^6$	98

As justified by the data results shown in Table 4, capillary membrane is not suited as a proper replacement for ceramic tube membranes equipped with static mixers. However due the higher (filtration area / membrane volume) ratio, further research is recommended to investigate the industrial application of capillary membranes, for example by installing static mixers in capillaries of the filter.

As result of my further research, a model equation was obtained, and by knowing the transmembrane pressure (TMP) and Reynolds (Re) number, the polarization layer resistance and membrane resistance ratio (R_P/R_M) can be estimated (under the circumstances). The equation describing the correlation reads as

$$\left(\frac{R_P}{R_M}\right) = f\left(\mathrm{Re}, \frac{TMP}{p_0}\right) \quad => \quad \left(\frac{R_P}{R_M}\right) = a \cdot (\mathrm{Re})^b \cdot \left(\frac{TMP}{p_0}\right)^c$$

where ***a***, ***b*** and ***c*** are constant values listed in Table 5:

Table 5 Constants of the equation in filtration with static mixer (SM) and without it (NSM)

const.	**With static mixer (SM)**	**Without static mixer (NSM)**
a	13196	146726
b	-1,212	-1,425
c	1,274	1,126

Emulsion concentration experiments were performed under optimized conditions. As a result, I proved that in case of 5m/m% starting oil concentration and filtering with 20nm pore size membranes, the 50 mg/L oil concentration in permeate can be kept until volumetric concentration factor (VCF) is below 4. At this VCF value, the permeate flux will be half of the starting flux.

Using experimental data, I created a linear model equation for the prediction of initial flux of permeate as function of transmembrane pressure (TMP) and flow velocity (v). Between two observed parameters, the effect of the flow velocity (v) was approximately twice greater than the effect of the TMP.

3.2. Analyzing the effect of static mixer to microfiltration of whey

At the beginning of research related to microfiltration of whey (as pre-process of nanofiltration), I tested effect of membrane pore size (50-, 200- and 1400 nm) to permeate flux and retention of lactose and protein. According to these results, from tested membranes, the 200 nm pore size membrane was the most appropriate.

In my next experiment, I tested the effect of a Kenics static mixer on permeate flux and specific energy consumption. The influence of turbulence promoter on permeate flux was concrete. Inside investigated range, in every measured point the improvement was 60-80%.

In term of specific energy consumption, similar results were obtained for whey microfiltration as previously for oily wastewaters. The improvement achieved by the employment of static mixer is positive only at lower recirculation flow rate, as shown in Table 6.

Table 6 *Effect of Kenics static mixer on permeate flux and energy saving using 200 nm ceramic tube membrane (positive values predict improvement effect of SM)*

RFR [L/h]	**TMP [kPa]**	**J [%]**	**E [%]**
50	200	64	+10
100	200	70	-428
150	200	73	-982

Influence of static mixer inside tube ceramic membrane on the retention of whey components (protein, lactose, fat) was very variable. For better understanding of the process, the experimental plan was carried out according to 2^P factorial design. Data processing was realized using Statistica 6.0 software. This method gives exact description about the examined effects or their cross-reactions. I proved that from examined effect (TMP, RFR, SM) to retention of whey components, the effect of static mixer was the biggest. Among the analyzed components, the effect of SM was approximately 2-3 times greater than the effect of RFR and TMP. Using the set-up of the models, initial retention of whey components can be estimated.

3.3. Optimizing static mixer geometry using computational fluid dynamics

My experiments confirmed a positive effect of Kenics static mixer on permeate flux and membrane retention. Unfortunately, the significant pressure drop along the membrane has direct effect to raising of specific energy consumption. For a better understand of the underlying phenomenon inside the tubular membrane, I performed computational fluid dynamic (CFD) simulations. The geometrical properties of the Kenics static mixer served as input for the three-dimensional matrix, were the input for an open source lattice Boltzmann algorithm. Further 5 static mixers were designed and tested with CFD. The simulations were carried out at a recirculation flow rate of RFR=50 L/h and transmembrane pressure of TMP=200 kPa. The results can be represented visually too, but more precision can be reached with numeric processing. The comparison of the simulated vorticity normal values as shown in Fig 3.

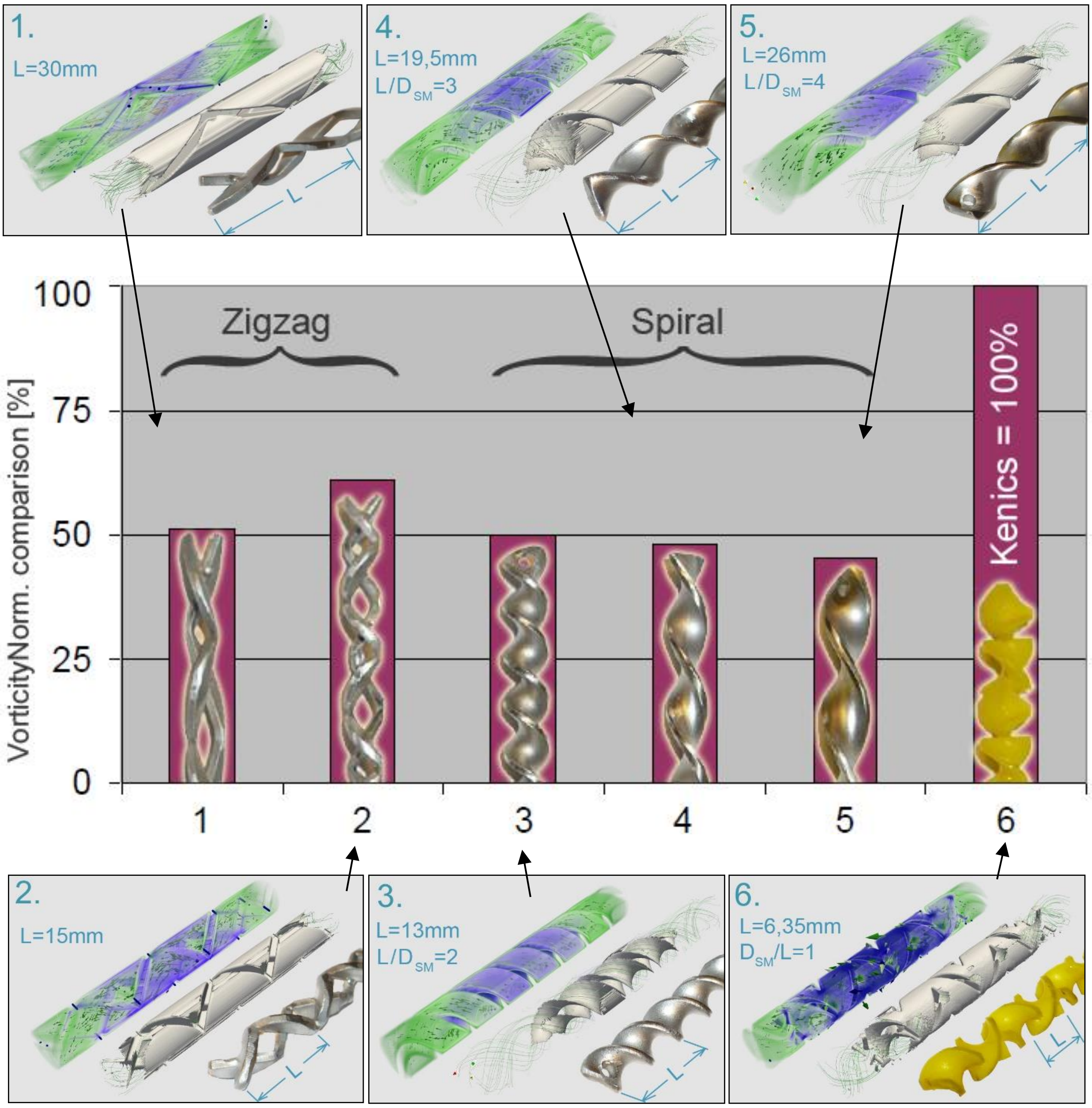

***Fig 3** Comparison of simulated vorticity normal values resulted by static mixers*

Vorticity normal of Kenics static mixer was the biggest, this was the base for my next comparison (100%). It can be seen, that even at lower recirculating flow rate (50 L/h), the vorticity of new static mixers is approximately half size. My assumption is that this difference will be even greater at higher flow rate. In order to prove this hypothesis, I had to produce these novel turbulence promoters and test them under real conditions. The current version of CFD simulation cannot be used for prediction of retention, only the frictional pressure-drop along the membrane (and calculated specific energy consumption) can be estimated. Fig 4 illustrates the effect of recirculation flow rate (RFR) on initial permeate flux, on pressure drop and on retention of oil.

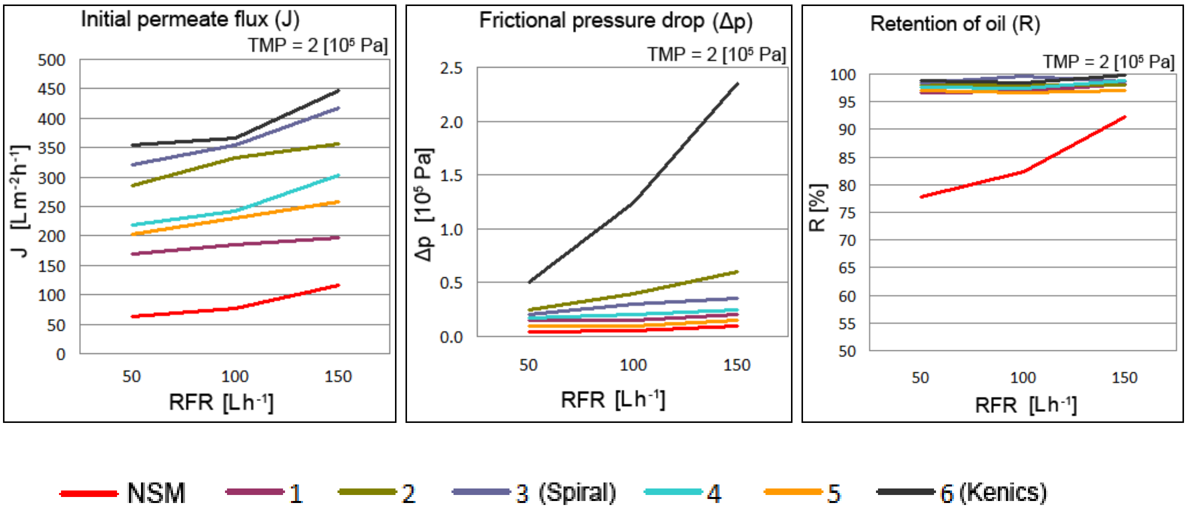

***Fig 4** Initial permeate flux, frictional pressure-drop and retention of the oil values in function of recirculation flow rate.*

My laboratory experiments also proved, that the spiral turbulence promoter (marked with number 3) kept the positive properties of Kenics static mixer, but it can be used at higher flow rates, because the frictional pressure drop in this case is sufficiently low. Main features of Spiral static mixer (No.3) are: thread pitch and diameter ratio: 2; twisted sheet thickness: 1 mm; pitch angle: 32 °, material stainless steel.

3.4.NEW SCIENTIFIC RESULTS

1.

I have proved, that installing Kenics FMX static mixer inside the ceramic tube membrane (Pall, Schumasiv) will result a specific energy saving and greater permeate flux of the cross-flow ultrafiltration, compared to filtration without static mixer.

Laboratory experiments were carried out with stable oil-in-water emulsion (MOL, Unisol) with 5 m/m% of oil. Operating temperature was 50°C, Reynolds number is below 4000±10%.

According to results of experiment, at transmembrane pressure range of TMP = 150-300 kPa and recirculation flow rate of RFR = 100 L/h, the specific energy saving was 10-42% and permeate flux increased by 3.3 – 6 times.

2.

My experiments confirmed, that ceramic tube membranes (Pall, Schumasiv) equipped with Kenics FMX static mixer can produce similar permeate flux as polymeric (PAEK) flat-sheet membranes, while oil concentration in permeate will be lower and there is no significant difference in specific energy consumption. These results also confirm that ceramic tube membranes (with longer lifetime, better pH- and temperature resistance) are proper replacement for polymeric membranes.

The permeate flux produced by ceramic tube membrane equipped with Kenics FMX static mixer is equal to the flux generated by polymeric PAEK membranes, however the oil concentration in permeate filtrated with ceramic membrane was an order of magnitude lower compared to the concentration of permeate filtrated by polymeric membrane. Ceramic tube membranes in combination with static mixer can produce permeate, where oil concentration complies with environmental regulations (oil concentration in the permeate is below 50 mg/L).

For both membranes (at compared parameters) the specific energy consumption was 2±10% kWh/m^3. In case of ceramic tube membrane with equipped static mixer, the transmembrane pressure was TMP = 150 kPa, recirculation flow velocity v=1.2 m/s, permeate flux J = 90 L/(m^2h) and concentration of the oil in permeate C_P=21 mg/L. Same parameters in case of polymeric membranes were: TMP = 200 kPa, v=1.8 m/s, J=100 L/(m^2h) and C_P=151 mg/L.

3.

Results of comparison experiments of ceramic tube membranes equipped with static mixer and ceramic capillary membranes (Hyflux) confirmed that in investigated range (TMP = 200-400 kPa and v=0.4 – 1.2 m/s), the permeate flux generated by Kenics FMX static mixer and ceramic tube membrane is an order of magnitude higher.

In experiments with stable 5 m/m% oil-in-water emulsion the maximum permeate flux measured with the tube membrane combined with Kenics FMX static mixer was 300 L/(m^2h) (at TMP = 400 kPa and RFR = 150 L/h), meanwhile the flux generated by capillary membrane was 45 L/(m^2h) (at TMP = 400 kPa and RFR = 1200 L/h). According to these data, ceramic capillary membrane is not proper replacement for compared ceramic tube membrane equipped with static mixer.

However, compared to tube membranes, capillary membranes have greater filtration surface per unit volume and further research is recommended, for example with installing turbulence promoters into capillaries of the membrane.

4.

I developed a model equation to estimate a polarization layer resistance (R_P) as function of transmembrane pressure (TMP), Reynolds number (Re), Membrane resistance (R_M) and atmospheric pressure (p_0). The equation describing the correlation reads as:

In case with inserted static mixer (SM):

$$R_P = 13196 \cdot \mathrm{Re}^{(-1.212)} \cdot \left(\frac{\mathrm{TMP}}{p_0} \right)^{1.274} \cdot R_M \qquad [1/\mathrm{m}]$$

In case without static mixer (NSM):

$$R_P = 146726 \cdot \mathrm{Re}^{(-1.425)} \cdot \left(\frac{TMP}{p_0}\right)^{1.126} \cdot R_M \qquad [1/m]$$

where R_P: polarization layer resistance [1/m], Re - Reynolds number (-), TMP – transmembrane pressure [kPa], R_M – membrane resistance [1/m], p_0 – atmospheric pressure [kPa]. Conditions of validity: TMP: 100-300 kPa, temperature: 50°C, membrane: ceramic tube membrane with 20 nm pore size, Reynolds range: 2408 - 7223 (with static mixer, SM) and 2019-6057 (without static mixer, NSM). Measured with 5m/m% oil-in-water emulsion. Accuracy $\leq$ 15%.

5.

I created a model equation for the prediction of initial permeate flux generated by the ceramic tube membrane equipped with Kenics FMX static mixer.

$$J = (2{,}619+0{,}733\cdot v-0{,}371\cdot TMP+1{,}817\cdot v\cdot TMP)\cdot 10^{-5} \quad [m^3/(m^2s)]$$

where J – permeate flux $[m^3/(m^2s)]$, v – flow velocity (m/s), TMP – transmembrane pressure $[10^5$ Pa]. The range of validity: v = 0.36 – 1.08 (m/s), TMP=1 - 3 $[10^5$ Pa]. The other conditions of validity: 20 nm pore size ceramic tube membrane with inserted Kenics static mixer. Fluid temperature of 50°C. Test material: 5 m/m% oil-in-water emulsion. For the shown model the significance level of 95% was applied.

Based on measured data and on developed model, I determined the optimal operation parameters (primary aspect were proper permeate flux and retention of the oil). I stated that the optimal operating parameters of membranefiltration process with ceramic tube membrane supplied with static mixer, from aspect of retention of the oil, are: TMP=200 kPa and v=0.6 m/s flow velocity (permeate quality comply with environmental regulations). I also determined, that by using the optimal operating parameters, emulsion can be concentrated to VCF = 4 where average concentration of oil in permeate will stay below 50 mg/L.

6.

I proved that the permeate flux of microfiltration of whey can be significantly increased by installing Kenics FMX static mixer into ceramic tube membrane.

I determined that within the investigation range (v = 0.36 – 1.08 m/s, TMP = 100 – 300 kPa), the optimal flow velocity is v = 0.36 m/s and transmembrane pressure is TMP = 300 kPa. Using the optimal operating conditions, the permeate flux improvement is 64-65%. I also concluded that the average flux improvement value, using 200 nm pore size membrane is 30%, compared to the 50 nm pore size membranes.

7.

I created a model equation to estimate the initial retention of whey components (protein, lactose and fat) as function of operational parameters (transmembrane pressure (TMP), recirculation flow rate (RFR) and effect of inserting Kenics FMX static mixer (SM) or (NSM)) using the 200 nm pore size ceramic tube membrane.

Equation of the estimated initial retention of protein:

$$R_{prot} = 29.33 - 0.95 \cdot \left(\frac{TMP - 200}{100} \right) - 1.77 \cdot \left(\frac{RFR - 100}{50} \right) - 6.79 \cdot M \, [\%]$$

where, R_{prot} – retention of protein [%], TMP – transmembrane pressure [kPa], RFR – recirculation flow rate [L/h], M – static mixer included, SM (M=1) no static mixer NSM (M=-1).

Equation of the estimated initial retention of lactose:

$$R_{lact} = 19.5 - 1.61 \cdot \left(\frac{TMP - 200}{100} \right) - 0.32 \cdot \left(\frac{RFR - 100}{50} \right) - 6.4 \cdot M \, [\%]$$

where, R_{lact} – retention of lactose [%], TMP – transmembrane pressure [kPa], RFR – recirculation flow rate [L/h], M – static mixer included, SM (M=1) no static mixer NSM (M=-1).

Equation of the estimated initial retention of fat:

$$R_{fat} = 83.56 - 2.15 \cdot \left(\frac{TMP - 200}{100} \right) - 0.76 \cdot \left(\frac{RFR - 100}{50} \right) - 4.78 \cdot M \, [\%]$$

where, R_{fat} – retention of fat [%], TMP – transmembrane pressure [kPa], RFR – recirculation flow rate [L/h], M – static mixer included, SM (M=1) no static mixer NSM (M=-1).

The range of applicability of equations: TMP = 100 – 300 [kPa], RFR = 50 – 150 [L/h]. Further conditions of validity: Ceramic tube membrane with pore size of 200 nm and inner diameter of 6.8 mm. If M=1 Kenics static mixer has to be installed into membrane (SM). Temperature of the whey: 40°C. In all three formulas, the number subtracted from TMP (200 kPa) is the mean value of measuring range. The number in TMP denominator (100 kPa) is half the value of the measuring range. Similar, the number subtracted from RFR (100 L/h) is the mean value of measurement range, and the number in RFR denominator (50 L/h) is half value of measuring range. 95% significance level was applied.

8.

From the laboratory experiment data and the computational fluid dynamics (CFD) simulation, I concluded that there is a correlation between the vorticity values (generated as output from lattice-Boltzmann algorithm) and the measured frictional pressure drop along the membrane. As shown in laboratory measurement data, frictional pressure drop generated by novel turbulence promoters is half of the size compared to pressure drop generated by Kenics static mixer (at RFR = 50 L/h and TMP = 200 kPa). Similar ratio can be seen in the comparison of vorticity values simulated by CFD.

In the current state of my research, a simulated velocity and vorticity data cannot be used for direct calculation of pressure drop, however the method can be used for comparison of effect on frictional pressure drop, generated by different static mixer forms.

9.

Using a modern computational simulation and laboratory experiments, I designed and tested new static mixer geometries with optimal efficiency on ultrafiltration of oil-in-water emulsion.

Static mixer geometry can be considered as optimal if it generates similar permeate flux and retention but the frictional pressure drop along the membrane is significantly lower (compared to Kenics static mixer).

In case of 250 mm length Kenics mixer, the pressure drop along the membrane was Δp = 230 kPa (at RFR =150 L/h and TMP = 200 kPa), and at same operating conditions, with the new optimal turbulence promoter this value was only Δp = 30 kPa.

Out of 5 new static mixers developed by me, the turbulence promoter with most optimal geometry was: Spiral static mixer with thread pitch and diameter ratio (L/D_{SM}) =2, pitch angle: 32°, the twisted metal strip thickness: 1 mm.

4. CONCLUSIONS AND SUGGESTIONS

During my research with ultrafiltration of oil-in-water emulsion and microfiltration of whey, I proved that integration of a Kenics static mixer into the ceramic tubular membrane have positive effect on permeate flux and specific energy consumption (at lower flow rates). I proved that installing Kenics static mixer in tube membrane will increase the retention of the oil, and in case of 5 m/m% starting oil content in emulsion, the 50 mg/L oil concentration in permeate can be kept until volumetric concentration factor (VCF) is below 4, which meets the requirements of environmental regulations. In case of both test materials, I determined the optimal operating parameters, where combined effect of a ceramic tubular membrane and a Kenics static mixer has best performance and lowest specific energy consumption. With developed model equations, some parameters of the membrane filtration can be estimated without laboratory experiments.

I designed a new turbulence promoter, which kept the positive features of the Kenics static mixer (better retention and permeate flux), however it does not generate high pressure drop along the membrane, so it can be used on the higher recirculation flow rates to delay the fouling of the membrane.

Suggestions:

- Determining the optimal operating parameters of ultrafiltration using new (spiral) static mixer, but with extended measuring range to higher recirculation flow rates.
- Testing the influence of Spiral static mixer on parameters of whey microfiltration.
- Observing the effect of novel turbulence promoter on membrane filtration with other substances
- CFD and laboratory testing of the filtration process using ceramic capillary membranes with inserted turbulence promoters.

5. LIST OF PUBLICATIONS RELATED TO THE DISSERTATION

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Krisztina Albert, András Koris, Igor Gáspár, Gábor Rácz, Gyula Vatai (2014): Production of microemulsion by ceramic tube membrane equipped with static turbulence promoter. Acta Alimentaria Volume 43 Vol.43, p. 1-8, Supplement 2014 (IF 2013: 0,427)

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