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Production and characterization of piezo-electric membranes

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It is shown that it is possible to impart piezo-electric properties to polyvinylidene fluoride (PVDF) membranes. This was achieved by "poling" the membranes in an intense electric field. Out of the plane surface displacements were produced when AC signals were applied to the membrane. Flux and separation performance measurements performed in a cross flow membrane module demonstrated that piezoelectric induced vibrations out of the plane of the membrane (i.e. in the direction of the flux) increased the flux by an order of magnitude and delayed membrane fouling. The relative antifouling effect of piezoelectric induced vibration was enhanced with increasing cross flow velocity.

1. Introduction

Accumulation of unwanted materials onto membrane surfaces and into membrane pores results in a decline in the permeate flux during filtration. Attachment of the accumulated material may make it difficult to remove even with severe chemical treatments. Such fouling of membranes, resulting also in increased power consumption and a reduction in membrane lifespan is recognized as a major operational and economic factor in determining the viability of membrane separation plants [1]. Approaches commonly used to combat fouling include pretreatment of the feed [2], modification of membrane bulk or surface properties [3], modification of the operating parameters and various cleaning procedures [4]. The latter include procedures to destabilize the fluids in contact with the membranes [5], using, for example, magnetic [6], or ultrasonic fields [7] and turbulence promoters [8]. During membrane separation processes, concentration polarization at the membrane surface occurs and this plays a major role not only in modulating the driving forces but also in the development of fouling of the membrane. In turn, the fouling can exacerbate the concentration polarization; the so called cake enhanced polarization [9]. It has been suggested that fluid instability might ameliorate concentration polarization and membrane fouling [5].

Ultrasonic agitation produced by piezoelectric transducers to overcome fouling during filtration has been described [10] but this technique has not been implemented in industrial plants. Here we describe the construction of piezo-electric membranes that can be made to undergo internal vibrational deformations normal to the plane so that itself becomes the source of agitation [11]. For this study PVDF membranes were chosen, because of the potential piezoelectric properties of PVDF. PVDF is used in the fabrication of actuators and sensors [12]. It is also used to manufacture membranes because it lends itself to commonly used methods of membrane manufacture that involve phase-inversion techniques as well as its superior chemical resistance [13].

1.1. Piezo-electric properties of PVDF

PVDF is a semicrystalline polymer that has at least four known crystalline structures (α, β, γ and δ) but the all-trans (β) phase is mainly responsible for its piezoelectric properties [12]. Common melt or solution processing techniques do not yield the β-phase due to thermodynamic limitations [14]. One method of converting the other crystalline structures into the β form is by electrical "poling". This involves the application of an intense electric field whilst the sample is held at an elevated temperature (just below the melting temperature) and then cooled whilst still in the intense electric field. This, in the case of PVDF, converts the other forms into the β form with permanent electric dipoles aligned with the field and imparts piezo-electric properties to the material [15,16].

Poling in an intense electric field for changing the crystalline configuration of PVDF and fabrication of piezoelectric films is a common technique [15–17]. However, to the best of our knowledge this technique has not been applied to PVDF membranes or other porous structures of this polymer such as foams. This may be related to the fact that the presence of pores, voids or defects tends to

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decrease the electric breakdown strength of dielectrics and hence increase the probability of electric breakdown during poling in an intense electric field [17–19]. For poling membranes, therefore, particular care was needed to obtain a satisfactory degree of poling without electrical breakdown.

When a voltage is applied across an electrically poled sample of a piezo-electric material, the material contracts or expands depending on the polarity of the voltage applied. When an AC signal is applied, the material will undergo vibrational contraction and expansion.

2. Materials

2.1. Membranes

The starting materials for the present study were PVDF microfiltration membranes (Pall Fluoro Tran® W supplied by PALL Life Sciences Australia)). The thickness and nominal pore size of the membranes were 123 and 0.22 μm, respectively. These membranes were then modified to impact the desired piezo-electric properties as described below.

2.2. Polyethylene glycol (PEG)

PEG was chosen as the model foulant for filtration tests because it is a synthetic material and can readily be obtained with reproducible average molecular weight and molecular weight distribution. The low adsorption of PEG on most polymer surfaces [20] was another reason for using this water soluble polymer as a model foulant in the membrane filtration tests. Fig. 1 shows the molecular weight distribution of PEG measured using gel permeation chromatography (GPC). The molecular weight distribution was broad with a polydispersity index (PDI) of 2.86. Molecular weights as great as 1323 KD were detected by GPC and about 40% of PEG molecules had molecular weights greater than the average molecular weight of 112 KD. For dilute solutions of PEG in water the gyration radius (Rg) of the polymer chain has been measured to be $\mathrm{Rg}=0.215\ \mathrm{M}^{0.583}$ [21], $\mathrm{Rg}=0.35\ \mathrm{M}^{0.55}$, or $\mathrm{Rg}=0.458\ \mathrm{M}^{0.55}$ [22]. In addition, the high concentration of PEG used in the present study increased the chance of molecular aggregation [21]. The insert of Fig. 1 shows the diameter of the PEG molecules estimated using these radii of gyration without considering the possibility of molecule aggregation. Molecules as great as 0.21 μm were actually detected in the solution. Such molecules could block the pores of the PVDF microfiltration membranes and form a cake layer on the surface of the membrane.

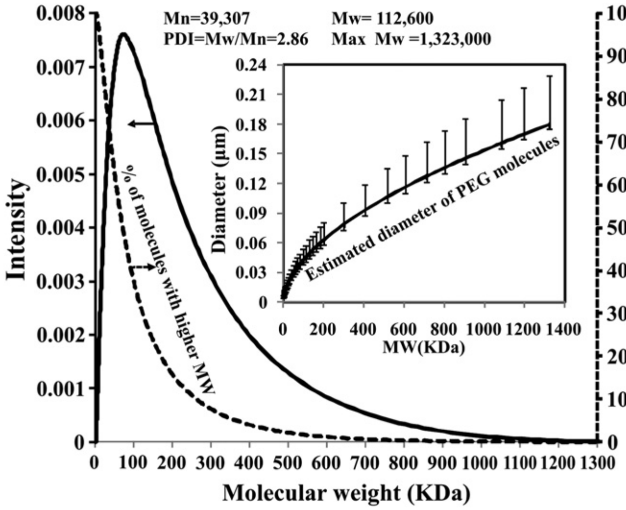

Fig. 1. Molecular weight distribution and diameter of PEG molecules used for the feed solution in the membrane flux experiments.

3. Methods

3.1. Electrical poling

Electrical "poling" was used in the present study to impart piezoelectric properties to PVDF membranes. Poling is achieved by placing the sample material in an intense electric field whilst the sample is held at an elevated temperature (just below the melting temperature). This aligns permanent electric dipoles in the material. The sample is then cooled whilst still in the intense electric field. Poling in the present context was performed using 220 × 220 mm membrane samples sandwiched between two electrodes as illustrated in Fig. 2. The specimen was heated from room temperature to 90 ± 5 °C, whilst the voltage applied between the electrodes was increased gradually from zero to 2000 V, at a rate of ~50 V/min. The applied voltage of 2000 V corresponds to field strength of 16.3×10^{6} V/m within the membrane sample. The sample was then kept at 90 °C, with the voltage of 2000 V applied, for 2 h. It was then cooled to room temperature (20–25 °C), whilst the 2000 V was still applied (that is with the 16.3×10^{6} V/m electric field) still present. To prevent electrical arcing between the electrodes when the high voltage was applied, the top electrode was made smaller (a 200 × 200 mm rectangular) than the bottom electrode (a 250 × 250 mm rectangle). Thus not the entire area of the membrane was poled and only the section of membrane which was exposed to the intense electric field, between the top and bottom electrode was used in the experiments.

3.2. Piezoelectric vibration measurements

To test the piezo-electric properties of the poled membranes, they were sputter coated, on both sides, with gold and mounted in the set-up shown in Fig. 3. In this apparatus the membranes were mounted in a chamber fitted with a transparent end window so that a laser beam could sample the surface of the membrane, even when the chamber was filled with water. AC signals were applied to the membranes by connecting the gold coatings to an AC variable frequency signal generator (Topward TFG-8114). The vibration of the membrane surface when the electric signal was applied, was measured using a laser Doppler movement detector system (Polytec PDV 100 Laser Doppler Vibrometer with VibSoft software). The vibrometer was located at a fixed distance (about 300 mm) from, and perpendicular to, the surface of the membrane. The vibrational response of the membranes was measured for sinusoidal signals of various frequencies, amplitudes and DC offsets applied to the membranes.

3.3. Membrane filtration performance measurements

To measure the flux performance of the piezo-electric membranes, the poled membranes were mounted in a cross flow membrane module. The cross-flow chamber was fitted with two porous steel

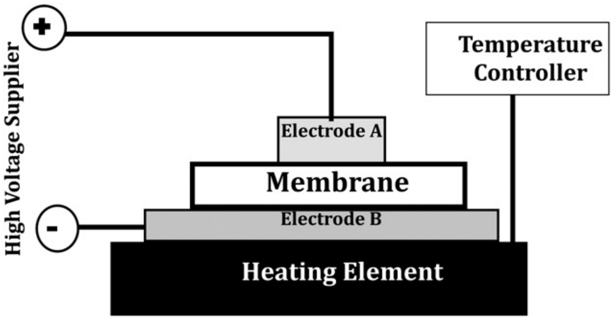

Fig. 2. The electrical poling set-up: The membrane was sandwiched between two electrodes connected to a high voltage DC power supply, whilst it was heated using a temperature controlled hot plate.

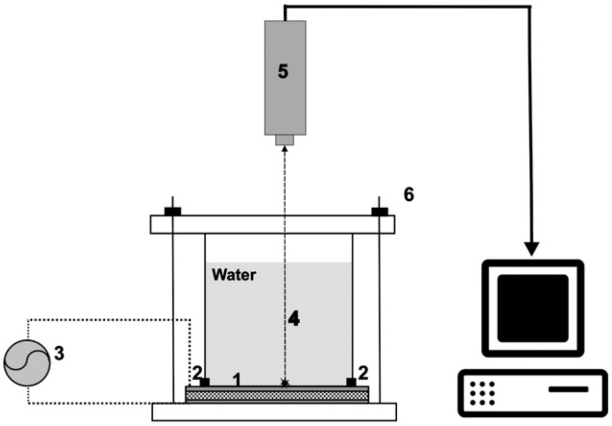

Fig. 3. The setup to measure the vibration of a poled membrane. The membrane (1) was sealed in the chamber using the O-rings (2). The AC signal (3) was applied to the membrane via the gold sputter coat on each side of the membrane. The laser beam (4) reflected from the membrane surface was used to measure the membrane vibration using a Laser Doppler Vibrometer (5).

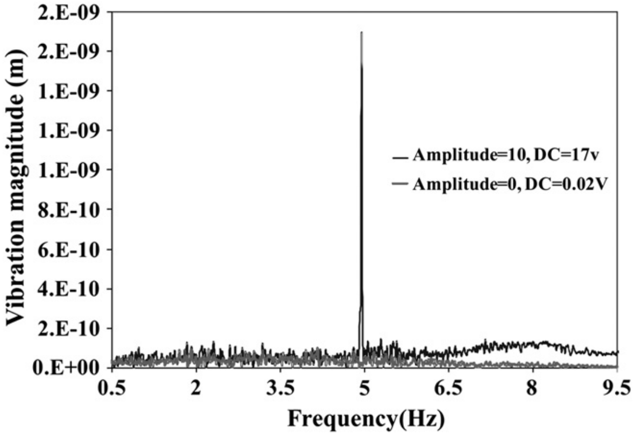

Fig. 5. An example of the vibration amplitude measured in air by a Laser Doppler Vibrometer at 5 Hz when a signal at 5 Hz was applied to the poled (piezoelectric) membrane (full line). For comparison, the response for an un-poled membrane is shown in gray.

electrodes that were used to apply the AC signal to the membrane. The bottom electrode also acted as the membrane support spacer as can be seen in Fig. 4. In these experiments the AC signal was applied to the two steel fabric electrodes and the membrane was not coated with a gold film. The effective area of the membrane was 36.08×10^{-4} m^2.

The feed solution was pressurized to 175kPa. The permeate was collected in a reservoir placed on an electronic balance (Shimadzu Corp.), that recorded the weight of the permeate. The cross flow was adjusted using a positive displacement pump. Because PVDF is a hydrophobic material the membranes were pre-wetted for 1 h in ethanol [23], followed by thorough washing with water to remove ethanol. The removal is particularly important as polyethylene glycol (PEG) was used a model foulant in the feed and PEG is insoluble in ethanol [24] and any residue ethanol could result in precipitation of PEG in membrane and premature blocking. The PEG used in the experiments had a nominal (average) molecular weight of 100 KD (product number 181986, Sigma Aldrich, Australia). A solution of 1 wt.% PEG in distilled water was used in the filtration tests.

4. Results and discussion

4.1. Piezoelectric vibration studies

The experiments with membranes with gold sputtered surfaces were aimed at measuring the piezo-electric response of the membranes when AC signals were applied to the membranes. Fig. 5 gives an example of the vibration measured with the membrane in air using the laser vibrometer. This figure shows the vibration responses in the absence (gray line) and presence (full line) of a 5 Hz sinusoidal stimulus of 10 volt amplitude. The manifestation of a sharp peak of 1.7 nm amplitude in the vibration response at 5 Hz arising from the application of the 5 Hz stimulus indicates that the poling was effective and had yielded a piezoelectrically active PVDF membrane.

The dependency of the vibration amplitude on the amplitude of the stimulus, for various frequencies and DC offset of the stimulus are shown in Fig. 6. The results shown indicate that the vibration did not increase linearly with the stimulus, contrary to trends reported in some of the literature [25–27], but in agreement with the results of Zhang [28]. Such a non-linear response has also been reported for lead zirconate/titanate ceramic composite structures [29,30] and for piezoelectric polymer foams [31]. The vibration-stimulus characteristic did not depend substantially on the frequency of the stimulus; an observation consistent with that reported for other piezoelectric materials [32–34]. The amplitude of vibration of the piezo-electric membranes increased substantially with the application of a DC offset in addition to the AC signal, as is evident from the results shown in Fig. 6.

The effect of submerging the membrane in a fluid, such as water, on the piezo-electric vibration was also investigated. Fig. 7 shows the vibrational responses when the membrane was submerged in water. These results showed that the water in the membrane pores and on either side of the membrane did not substantially damp the displacements of the surface of the membrane when the electrical signals were applied. Like the results obtained in air, the amplitude of vibration increased with increasing amplitude of the AC stimulus

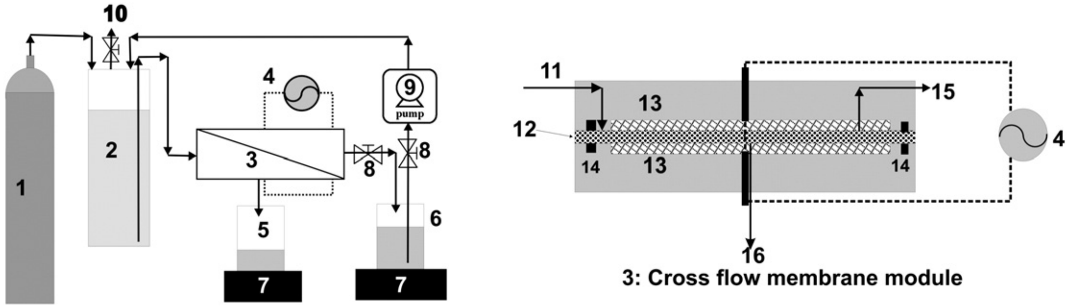

Fig. 4. Filtration test set-up and cross flow membrane module used for piezoelectric membrane filtration tests. Compressed air (1) was used to pressurize the feed solution reservoir (2), fed into the cross flow module (3). The AC signal generator (4) was used to apply signals to the porous steel fabric electrodes (13). The permeate reservoir (5), and the cross-flow collected in the reservoir (6), were monitored using balances (7). The cross flow and permeate flows could be adjusted using the valves (8), pump (9) and pressure relief valve (10). The inlet for the feed solution is shown as (11), the membrane (12), sealing O-rings (14), the cross flow outlet (15) and permeate outlet is (16).

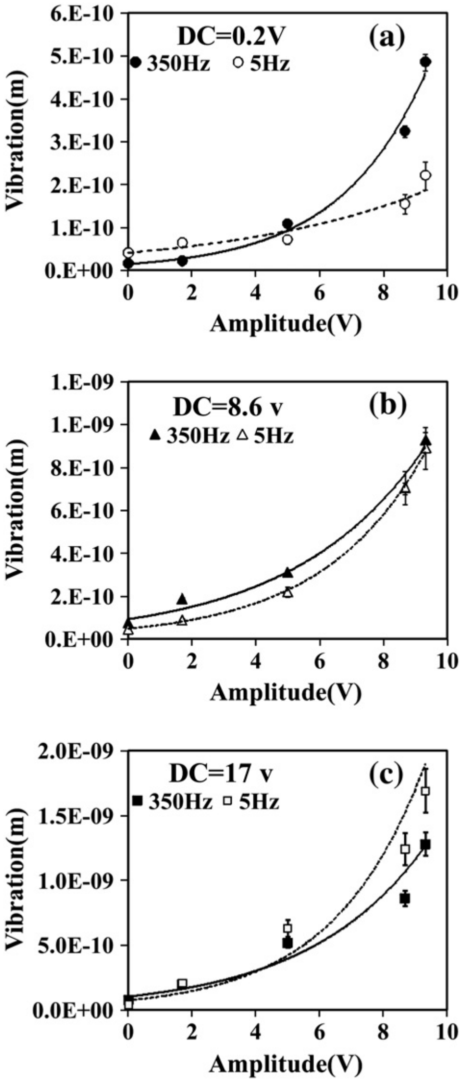

Fig. 6. The vibration amplitude measured by the Laser Vibrometer at 350 Hz and 5 Hz as a function of the AC signal amplitude, in the presence of DC offsets of 0.2 V (a), 8.6 V (b) and 17 V (c).

signal as well as the DC offset. It should be pointed out here that the application of large electrical signals to the electrodes, particularly at very low frequencies (< a few hundred Hz) could result in electrolysis of water and this might present problems in some applications. However, by applying smaller stimuli at high frequencies, electrolysis may be avoided.

4.2. Piezoelectric membrane filtration performance

4.2.1. Filtration properties of non-piezoelectric membranes

As a control experiment, the fouling behaviour of un-poled PVDF membranes was studied, both with and without an applied electrical signal. The un-poled membrane was not expected to undergo piezoelectric vibration, even when a signal was applied, but the possibility that the applied signal itself might affect the flux performance needed to be investigated.

The permeate flux of the un-poled membranes with an applied electrical signal consisting of a 9 V AC sinusoidal waveform at 5 Hz plus a 17 V DC offset were compared with those of the un-poled membranes without such an applied electrical signal. The cross flow in both experiments was maintained at 7 ml/min. The results for these experiments are shown in Fig. 8. It is immediately clear that for the un-poled membrane the application of the electrical stimulus did not noticeably change the permeate flux and the fouling behavior of the membranes.

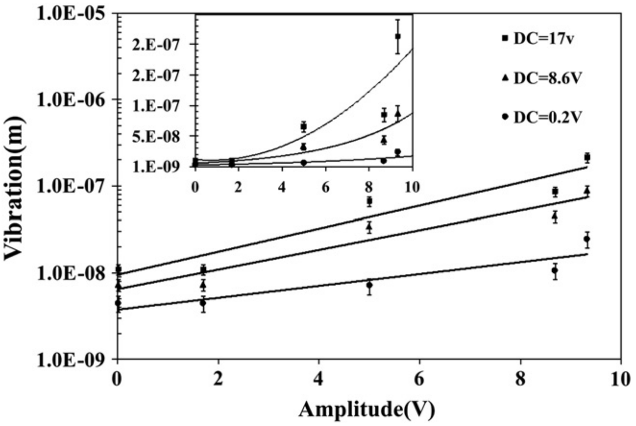

Fig. 7. Peak vibration amplitude of the piezo-electric membrane submerged in distilled water (detected by the Laser Vibrometer) as a function of the amplitude of a 5 Hz signal applied to the piezoelectric membrane.

4.2.2. Filtration properties of piezoelectric (poled) membranes

Fig. 9 shows the normalized flux for poled membranes at a cross flow of 6–7 g/min, in the presence and absence of a sinusoidal signal of 9.5 V amplitude at frequencies of 5, 322 and 3000 Hz and a DC offset of 17 V. The normalized fluxes of the membranes subjected to an electrical signal were clearly greater than that of the membranes which were not subjected to electrical signals. This suggested that the vibration generated by these membranes delayed fouling. These results also appear to indicate that over the frequency range of the AC signals used in these experiments (5–3000 Hz), the anti-fouling action was not dependent significantly on the frequency of the AC signals.

The amplitude of vibration of the surface of the membrane with the signals applied in these experiments was in the range of 10–100 nm as determined from the laser Vibrometer measurements. Whilst this might at first appear as a very modest agitation of the surface, it should be pointed out that it is comparable to the dimensions of the initial diffusion polarization layer where fouling initiates. We suggest that the piezoelectric vibration of the membrane introduces fluidic instabilities that allow the cross flow to more effectively sweep away the particles and delay formation of cake

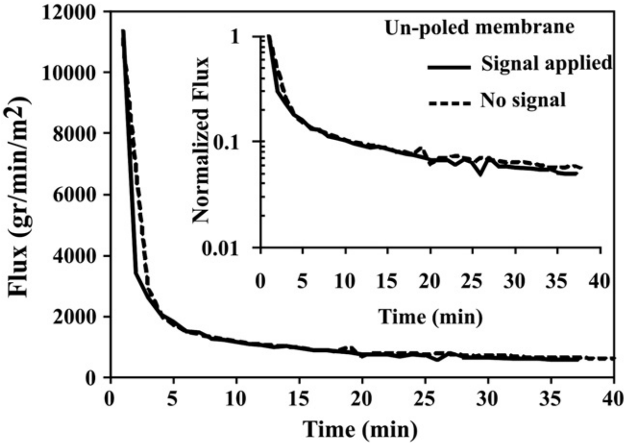

Fig. 8. Effect of electrical signal on flux and normalized flux of a non-piezoelectric (un-poled) membrane during filtration of 1% PEG at 175 kPa pressure and 7grams/min cross flow. For these un-poled membranes the application of the electrical signals did not appear to affect the flux performance.

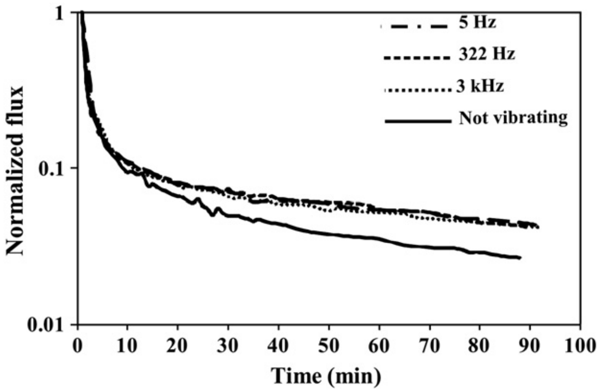

Fig. 9. Effect of AC electrical signals of 9 V amplitude on the normalized flux through piezoelectric membranes during filtration of 1% PEG at 175 kPa pressure and 6–7 ml/min cross flow. The full line is the control when no signal was applied and the membrane was not vibrating.

Table 1
Filtration test results using piezoelectric membranes obtained during filtration at two different cross flow rates.

	Initial Flux(F0) (gr/min/m^2)	Final flux(F) (gr/min/m^2)	F/F0 (%)	Average Flux (gr/min/m^2)	t(10%)* (min)
Cross flow = 30 ± 5 ml/min					
Not vibrating	12,472	119	0.96	563	1.5
Vibrating , subjected to sinusoidal signal of 9.5 V AC amplitude + 17 V DC offset					
Frequency = 5 Hz	11,862	543	4.6	1596	14.3
Frequency = 322 Hz	11,641	421	3.6	1174	10
Frequency = 3 kHz	8869	486	5.5	1217	23.5
Average change due to vibration	− 1681	+ 364	+ 3.6	+ 766	+ 14.4
	− 13.5%	+ 306%	+ 377%	+ 136%	–
Cross flow = 6–7 ml/min					
not vibrating	15,327	406	2.7	1366	8.5
Vibrating , subjected to sinusoidal signal of 9.5 V AC amplitude + 17 V DC offset					
Frequency = 5 Hz	12,639	549	4.3	1327	12.5
Frequency = 322 Hz	13997	581	4.1	1547	12
Frequency = 3 kHz	13,525	562	4.1	1362	11.5
Average change due	− 1940	157.3	1.6	45.5	+ 3.5
to vibration	− 12.7%	+ 39%	+ 59%	+ 3.3%	–

*t10% is the time at which the flux was 10% of the initial flux.

material in the diffusion polarization layer. Another way of perhaps describing that is that the piezoelectric vibration of the surface increases the magnitude of the critical flux [35,36] at which fouling occurs.

To evaluate this hypothesis further, filtration experiments with the piezoelectric (poled) membranes were carried out with different cross flow velocities. Fig. 10 shows the results of such an experiment in which the cross-flow was increased to 30 ml/min from the value of 6–7 ml/min for the results shown in Fig. 9. Note, however, that for this particular membrane sample the flux for the non-vibrating situation (no signal applied) was somewhat lower that that shown in Fig. 10.

The results illustrated in Fig. 10 show that with the larger cross flow a 10-fold increase in the normalized flux was obtained for the vibrating membrane over the non-vibrating membrane.

Details of the results obtained during the filtration experiments at the two different cross flow rates are shown in Table 1. Although the initial flux decline of about 13% was independent of the cross flow rate, the larger cross flow rate substantially reduced the subsequent decline in flux when signals were applied to the piezoelectric membrane; with the *average* flux over the 80 min filtration period being 100% higher than that flux at the lower cross flow rate. For comparison, only 3% improvement in the flux was achieved with a cross flow of 6–7 ml/min. The effect of piezoelectric vibration on the flux at longer times (80 min) was even more dramatic; an 38% increase in this flux was observed when the piezoelectric membrane was excited and the cross flow was ~7 ml/min. Increasing the cross flow to 30 ml/min boosted this enhancement to ~300%. The ratio of the flux at 80 minutes to the initial flux, which indicates a level of fouling, was also significantly affected by the cross flow. This ratio for the piezoelectrically excited membranes at a cross flows of 30 ml/min was almost 400% larger than that for the non-excited membranes under the same conditions. When a 7 ml/min cross flow rate was used, about a 60% improvement in this ratio was found.

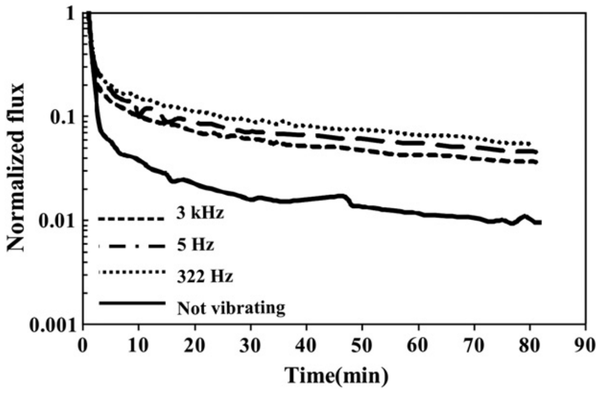

Fig. 10. Effect of AC electrical signals of 9 V amplitude on the normalized flux through piezoelectric membranes during filtration of 1% PEG at 175 kPa pressure with a cross flow 30 ± 5 ml/min. c.f. Fig. 9.

5. Conclusion

Piezoelectric PVDF microfiltration membranes were fabricated by electrically poling the membranes in an intense electrical field. Laser displacement measurements confirmed the vibration of the piezoelectric membranes when AC signals were applied to the membranes both in air and submerged in water. Membrane filtration experiments showed that excitation of the piezoelectric (poled) membranes reduced fouling dramatically. An increase in the cross flow rate further enhanced the relative antifouling effect of piezoelectric membranes. The average and the long time (80 min) fluxes were significantly larger when the piezoelectric membranes were electrically excited.

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