



Piezoceramic membrane with built-in ultrasonic defouling

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ABSTRACT

Membranes utilized for water and wastewater treatment are susceptible to fouling. Fouling leads to gradual flux decline, possible loss of selectivity and increased operational costs. Conventional de-fouling strategies such as backwashing and chemical cleaning can be effective, but have inherent disadvantages. Recently, application of ultrasound was shown to be an effective cleaning method but, thus far, has been applied only with the ultrasonic source in close proximity to the membrane. In this work we demonstrate for the first time, a new membrane design in which ultrasound for de-fouling is generated from within the membrane structure. To test the feasibility of this idea, we fabricated porous, poled and unpoled piezoelectric ceramic microfiltration membranes and performed filtration tests with a fouling dispersion containing 10 mg/L of 500 nm latex particles. To generate ultrasound from within the membrane, an alternating voltage was applied across the membrane with the feed side electrode located 1 mm from the membrane surface. In the absence of voltage, the flux decreased by > 20% within 3 h of filtration. Conversely, when an alternating voltage was applied in intermittent pulses, no water flux decrease was observed over 3 h of filtration, demonstrating the feasibility of membranes with built-in de-fouling functionality.

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1. Introduction

Membranes with pore size classifications from reverse osmosis to microfiltration are widely-implemented and time-tested for achieving stringent water treatment requirements [1]. Despite advances in membrane technology and operation, fouling continues to persist as the foremost limitation of membrane filtration [2–4]. Flux reduction resulting from fouling ultimately increases operational costs and can lead to premature failure of membranes. Development of effective and non-destructive fouling mitigation strategies is therefore critical to ensure prolonged and efficient operation of membranes.

Conventional approaches to de-fouling membranes include backwashing and/or chemical cleaning [1,5]. Although backwashing is generally effective, it requires short down-times leading to decreased productivity. Furthermore, irreversible foulants may remain on the membrane after backwashing. Such foulants eventually need to be removed with more aggressive, off-line chemical cleaning procedures which require longer periods of down-time. The use of aggressive reagents may also result in

handling and disposal issues. More recently physical membrane vibration [6] and ultrasound-induced cavitation [7] have been shown to be excellent alternatives to chemical cleaning for de-fouling.

When ultrasound is generated in a liquid, gas pockets form once the negative wave pressure of the sound exceed the liquid's tensile strength. These bubbles rapidly grow and collapse, which results in cavitation and very high localized energy release (temperatures up to 5000 K and pressures up to 100 MPa) [8,9]. If the cavitation is directed to take place near the membrane surface, the released energy has been shown to be highly effective at mitigating or reversing fouling [7,10–17]. These previous studies were performed at the lab-scale with ultrasound generated by a transducer in close proximity to the membrane surface. However, large-scale implementation of a system utilizing separate ultrasonic equipment is believed to be cost-prohibitive. To sidestep the requirement of such equipment, a possible solution is a piezoelectric membrane structure capable of generating ultrasound during filtration.

The concept of piezoelectric membranes has been previously introduced using polyvinylidene fluoride (PVDF), a polymer material commonly used in micro- and ultrafiltration membranes, that is piezoelectric [18]. It was found that application of 0.5–1 kHz alternating voltage to poled PVDF membranes during filtration resulted in an increase of the flux and, simultaneously, a decrease

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of the fouling rate [18–20]. In the present study, we are focusing on a different class of membrane materials, namely piezoelectric ceramics. Ceramic membranes have a controlled pore size distribution, as well as high thermal, mechanical and chemical stability. Ceramic piezoelectrics exhibit stronger piezoelectricity than polymers such as PVDF. We present disk-shaped, strongly piezoelectric ceramic membranes, which respond to frequencies in the ultrasonic spectrum (> 20 kHz). Using ultrasonic frequencies may be advantageous over lower frequencies, as localized cavitation may occur [8], resulting in more rigorous de-fouling. For the sake of demonstration, lead zirconate titanate (PZT) was chosen as the piezoelectric membrane material and ultrasound was generated by application of an external 70–80 kHz alternating voltage.

2. Materials and methods

2.1. Membrane fabrication and poling

Porous PZT ceramic disks were synthesized using a commercially-available powder, APC840 (APC International, Ltd). The supplied powder consisted of 50–150 μm spray-dried granules containing 1% binder with a 0.5 μm particle size. Dry pressing and sintering, a standard method of advanced ceramic processing [21], was selected, and the conditions were optimized for our samples as follows. Circular membrane disks with 20 mm diameter and 1.3 mm thickness were synthesized by dry pressing the APC840 powder at 100 MPa, followed by sintering at 950 $^{\circ}\text{C}$ for 10 h. Lead evaporation from the sample was suppressed by embedding it in loose-packed APC840 powder. The furnace heating and cooling rate was 2 $^{\circ}\text{C}/\text{min}$ and 3 $^{\circ}\text{C}/\text{min}$, respectively.

PZT grains contain ferroelectric domains having a random orientation in compacted and sintered disks made from powder form. Selected sintered disks were poled by being subjected to a strong direct electric field, so that the domain orientations became aligned, with a net polarization vertical to the disk bases. To achieve this, the disks were mounted between two flat aluminum electrodes (face to face) and placed inside a heating mantle. The electrodes were connected to a high voltage power supply (Gamma series ES) as shown in Fig. 1. Poling conditions which yielded optimum results were found to be an electric field of 2.5×10^6 V/m at a temperature of 140 $^{\circ}\text{C}$ for 1 h. This temperature is high enough to increase the mobility of the domains, but much lower than the Curie temperature of 320 $^{\circ}\text{C}$, where the piezoelectric effect is no longer present.

When higher fields were applied at conditions near the dielectric breakdown of air ($\sim 3 \times 10^6$ V/m [22,23]), discharge through and around the sample occurred. These discharges were detected by visible electrical arcs, audible noise, and in some cases led to degradation of the micro-structure including visible holes. Filtration experiments with membranes that experienced discharge revealed substantial break-through of latex particles as evidenced by high flux and visibly cloudy permeate (see Section 2.3).

Piezoelectric ceramics are often poled while immersed in oil; this allows the application of higher voltages (e.g. $4\text{--}6 \times 10^6$ V/m [24] or $10\text{--}12 \times 10^6$ V/m [25]) and results in a more uniform temperature throughout the sample. However, in the present work, samples that were poled in oil exhibited substantial hydrophobicity due to oil penetrating the pore surfaces. Oil immersion during poling led to membrane wetting problems and the inability to produce permeate, even when feed pressure was increased to over 200 kPa. Consequently, the method of poling in air was preferred, although it imposed a limitation on the maximum applicable electric field.

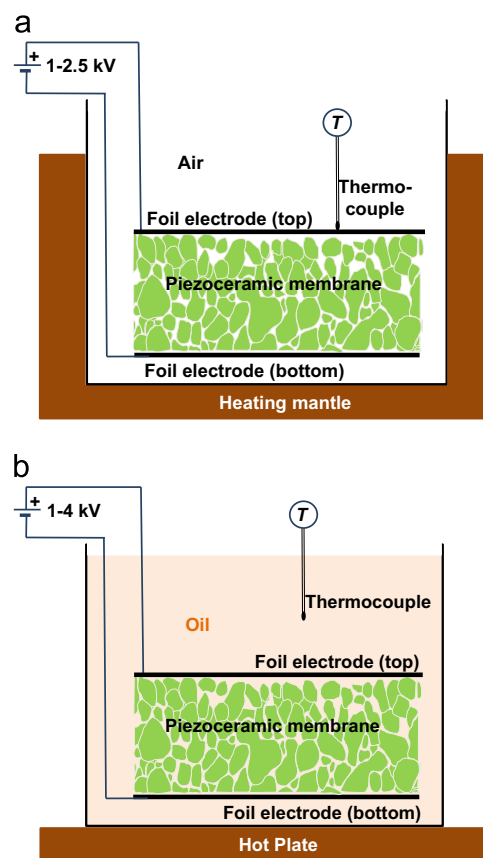


Fig. 1. Schematic of the poling setup (a) in air, (b) in oil.

2.2. Ultrasound generation and detection-resonant frequency measurement

Prior to filtration experiments, the resonant mechanical frequencies and ultrasonic emission of the PZT membranes, submerged in water, were determined in a 61 cm \times 61 cm plexiglass tank filled with MilliQ water ($\rho^0 = 1.82 \times 10^5 \Omega\text{m}$) to a height of 9 cm (total volume of 33.5 L). To obtain an ionic strength of 1 mol/m³ and circumneutral pH, potassium chloride and potassium bicarbonate were added to the water at concentrations of 0.95 and 0.05 mol/m³, respectively. During testing, the membrane was placed on a stainless steel mesh electrode and submerged into the solution. A 6 mm diameter stainless steel bolt was installed through a polyvinyl chloride (PVC) support to a height of 1 mm above the center of the membrane surface to act as a remote electrode. Both electrodes were wired to a 10 V waveform generator (Hewlett Packard) capable of providing alternating voltages at frequencies between 1.0 kHz and 15 MHz. A sinusoidal voltage of 10 V amplitude was applied across the membrane, while sweeping the frequency over the 1–200 kHz range. The mechanical pressure waves (sonic or ultrasonic) generated by the membrane and propagating in water were detected by a hydrophone (Teledyne Reson) submerged and affixed approximately 3 cm from the membrane surface. The hydrophone converted the detected waves to electrical signal (voltage), which was monitored by a digital oscilloscope (Tektronix) (Fig. 2a and b).

Fig. 2c shows the equivalent electrical circuit between the electrodes. The equivalent circuit consists of a space charge capacitance, two resistances and another space capacitance. In this application the Faradaic capacity can be ignored. The non-linear space charge capacitance is caused by alternating ionic accumulation near the electrode surfaces. The resistances are associated with the alternating ionic transport that occurs between the space

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