Chapter 23
Ultrasonic Membrane Processing

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1 Introduction

A membrane is a semipermeable material that permits the passage of some molecules while retaining others. The membrane can discriminate between species based on their size, concentration, or electrical charge.

Membranes are widely used throughout the food and bioprocessing industries for both separation and concentration purposes. Specific applications include

- microfiltration of milk to concentrate caseins (Le Berre and Daufin, 1994, 1996, 1998),
- microfiltration of fermentation broths and milk products to retain bacteria and spores (Krstic et al. 2001),
- ultrafiltration of dairy whey and skim milk to concentrate solids prior to spray drying (De Boer and Hiddink, 1980; De Boer et al., 1977; Renner, 1984),
- membrane bioreactors for cell culture (Drioli and De Bartolo, 2006) and enzymatic fermentation (Prazeres and Cabral, 1994; Rios et al., 2004),
- concentration of flavors and colors (Babu et al., 2006; Rodrigues et al., 2004),
- membrane affinity methods for protein separation (Charcosset, 1998; Klein, 2000; Zou et al., 2001),
- reverse osmosis to produce purified water for specialized bioprocesses and for steam generation (Noble and Stern, 1995),
- emulsification (Joscelyne and Tragardh, 2000), and
- clarification of beers and fruit juices (Cassano et al., 2003; Gan et al., 1999; Vaillant et al., 2005).

Ceramic membranes are generally provided as tubes or flat discs. Conversely, polymeric membranes may be supplied in a spiral wound format or as hollow

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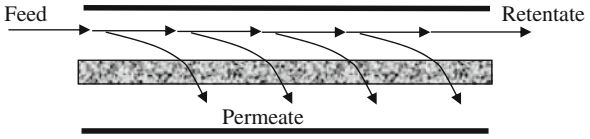

Fig. 23.1 Basic overview of membrane operation

fibers. In all cases, the processing fluid flows tangentially to the membrane surface (Fig. 23.1).

1.1 Concentration Polarization and Membrane Fouling

It is a natural outcome of membrane operation that retained solutes will tend to accumulate near the surface of the membrane, while permeating species are depleted. This is known as *concentration polarization* (Fig. 23.2). The reduced concentration of the permeating species at the membrane surface leads to a reduction in flow through the membrane and thus ultimately restricts membrane performance.

In some cases, the concentration of retained solutes increases to a point where precipitation occurs. Alternatively, the feed solution may contain suspended solids that precipitate continuously. These solids accumulate at the membrane surface. Initially, they will act to partially cover the membrane pores and may also enter the pore structure. This is known as *pore blockage*. Further precipitation leads to *cake formation.* This is a buildup of a layer of precipitated or suspended solids at the membrane surface, which further restricts flow through the membrane and thus further reduces the ability of the membrane to operate.

If the driving force for flow through a membrane is a pressure difference (ΔP), then this flow is described by the following expression:

$$J = \frac{\Delta P - \Delta \pi}{\mu R} \tag{23.1}$$

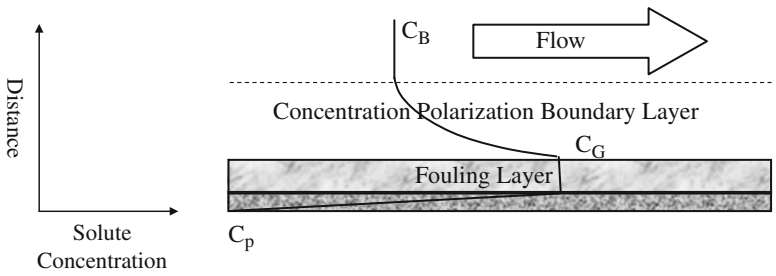

Fig. 23.2 Schematic of membrane fouling. The concentration of the retained species varies from a bulk value in the feed (C_B) to a permeate concentration (C_P). The concentration at the surface of the fouling layer is known as the gel concentration (C_G)

where J is the flow of permeate per unit area, usually referred to as the membrane flux and with units of l/m^2/h; μ is the viscosity of the fluid; and R is the resistance to flow. $\Delta\pi$ represents the difference in osmotic pressure between the two sides of the membrane. Comparable expression can be developed for membrane operations where concentration or electrical potential drives the separation (Noble and Stern, 1995).

The resistance to flow (R) is composed of several parts, i.e., the resistance provided by the membrane itself (R_m), the resistance provided by the cake layer (R_p), and the resistance provided by concentration polarization (R_c) so that

$$R = R_m + R_\mathrm{p} + R_c \tag{23.2}$$

The resistance provided by concentration polarization is often expressed in terms of a mass transfer coefficient, k. In this case, a more appropriate expression for the flux is

$$J = k \ln \frac{C_\mathrm{G}}{C_\mathrm{B}} \tag{23.3}$$

where C_G is the concentration at the surface of the cake layer and C_B is the concentration in the bulk feed solution.

2 Ultrasonic Flux Enhancement

The principle application of ultrasound to membrane processing is to enhance the membrane flux (J) by reducing the impact of the various resistance terms (R) as described above. Flux enhancements between 40 and 800% have been reported (Ahner et al., 1993; Narayan et al., 2002; Teng et al., 2006a, b; Zhu and Liu, 2000).

There are several specific effects of ultrasonic irradiation that can be implicated in this flux enhancement:

(a) The asymmetric collapse of cavitating bubbles can scour the surfaces and this leads to removal or control of the fouling cake layer (Chen et al., 2006a, b, c; Duriyabunleng et al., 2001; Kobayashi and Hosaka, 2003; Latt et al., 2004; Matsumoto et al., 1996). This surface action can also dislodge particles attached to the surface and break down large aggregates into smaller particles (Hagenson and Doraiswamy, 1997; Kost and Langer, 1988; Zhu and Liu, 2000).

(b) Acoustic streaming and/or cavitation causes turbulence, which results in bulk water movement near the membrane surface. This reduces the effects of concentration polarization and thus increases the mass transfer coefficient at the membrane surface (Ahner et al., 1993; Chai et al., 1998; Kobayashi et al., 1999; Kokugan et al., 1995; Muthukumaran et al., 2005a; Simon et al., 2000). Muthukumaran et al. (2005a) found that the mass transfer coefficient (k) increased from 9 to 16 $\times$ 10^{-7} m/s when ultrasound was applied. Similarly,

Simon et al. (2000) found that ultrasound was effective in disrupting the concentration polarized layer.

(c) The added turbulence can lead to a looser, more porous fouling cake layer (Kokugan et al., 1995; Muthukumaran et al., 2005a). The use of ultrasound acts to lower the compressibility of both the initial fouling deposit and the growing cake. In a similar manner, the turbulence associated with ultrasound separates physical aggregates by disrupting the intermolecular forces (Kost and Langer, 1988).

(d) There is evidence that sonication can cause agglomeration of fine particles and thus could potentially reduce pore blockage and cake compaction (Matsumoto et al., 1996). However, both Kokugan et al. (1995) and Muthukumaran et al. (2005a) show that ultrasound is not effective in reducing pore blockage by such mechanisms.

Kobayashi et al. (1999) found that the highest permeate flux in an ultrafiltration application was obtained when the sonication was directed to the retentate side of the membrane. Conversely, Kylloenen et al. (2006) obtained a higher flux when irradiating from the permeate side in a microfiltration experiment. These authors (Kylloenen et al., 2005) argue that when pore sizes are relatively small, sonication is most effective on the retentate side as cake formation is reduced, whereas for larger pore sizes, permeate side irradiation acts as a backflushing mechanism, reversing flow through the pores.

2.1 The Effects of Ultrasonic Intensity

Ultrasonic intensity can be increased by either increasing the applied power or reducing the frequency. Power densities between 0.05 (Muthukumaran et al., 2007) and 83 W/cm^2 have been reported (Li et al., 2002), with frequencies between 1 MHz (Lamminen et al., 2004) and 20 kHz (Chen et al., 2006a). Most workers note that the flux enhancement improves as ultrasonic intensity increases (Kylloenen et al., 2006; Muthukumaran et al., 2007).

At low ultrasonic intensities, that is, at either low power densities or high frequencies, the flux enhancement principally arises through acoustic streaming effects. Thus, Muthukumaran et al. (2005a), working at about 0.05 W/cm^2 and 50 kHz, found that the main mechanisms involved in flux enhancement were due to acoustic streaming. However, as the acoustic intensity increases, either through a lowering of ultrasonic frequency or through an increase in power, bubbles begin to form through the process known as acoustic cavitation. Thus, Lamminen et al. (2004) found that at 0.2–0.5 W/cm^2 and 205–620 kHz, particles were loosened from the membrane surface by cavitational mechanisms (microstreaming and microjets), although acoustic streaming assisted in the movement of particles away from the surface (see Fig. 23.3). Latt et al. (2004), working at 28 kHz and comparable power

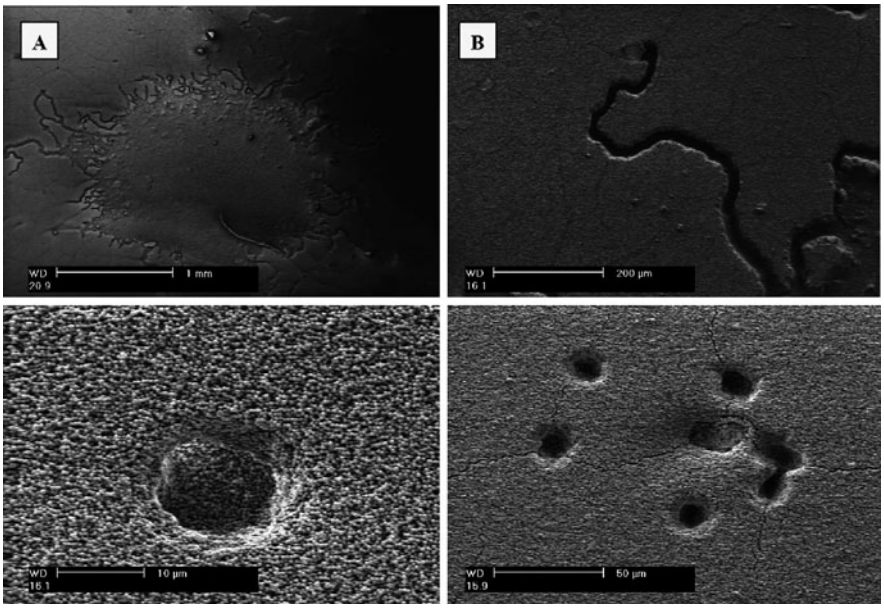

Fig. 23.3 Scanning electron microscope (SEM) images of circular patches of cake layer removal and channel-like formations along the edges of the circular patches attributed to microstreaming/microstreamers: (**a**) 620 kHz for 5 s, 0.21 W/cm^2; (**b**) 620 kHz for 5 s, 0.12 W/cm^2, SEM images showing evidence of microjet impacts on the surface of the cake layer; (**c**) 1062 kHz for 5 s, 0.21 W/cm^2; (**d**) 620 kHz for 5 s, 0.12 W/cm^2 (reproduced from Lamminen et al., 2004)

densities, concluded that violent collapse of cavity bubbles was the source of enhanced permeability.

In general, increases in acoustic intensity result in an increase in the number of bubbles formed (Sivakumar and Pandit, 2001) and increase the size of the cavitation zone (Suslick, 1988). However, cavitation efficiency increases with increasing power only up to a critical power level. Further increase beyond this critical power level can result in a decrease in cavitational activity (Thompson and Doraiswamy, 1999). A possible explanation for the observed decrease at high power is the formation of a cluster of bubbles due to Bjerknes forces (Leighton, 1994). This clustering shields the inner bubbles from the transmission of acoustic energy.

The optimization of ultrasonic intensity is important for a number of other reasons. The use of high ultrasonic intensities has the potential to lead to changes in the feed solution itself. Chai et al. (1998) reported that there was a slight change in the gel permeation chromatograms of dextran molecular size before and after sonication, when the solution was directly exposed to ultrasonic irradiation of 248 W for 30 min. However, they found no changes in the dextran molecular size when the dextran solution was exposed through a crossflow ultrafiltration cell and suggested that this may be due to the shielding of ultrasonic power by the ultrafiltration module. Residence times would also be much shorter in a crossflow unit. Villamiel and

De Jong (2000) found some evidence for the denaturation of whey proteins when sonication was performed at temperatures in excess of 60°C.

Some researchers have also found that if membranes are used at higher power levels, membrane damage may occur (Juang and Lin, 2004). In the study of Wang et al. (2005) polymeric membranes were immersed in an ultrasonic bath at 40 kHz and 1–4 W/cm^2. They reported the formation of large cracks resulting from the interconnection of neighboring large pores. Similarly, Masselin et al. (2001) indicated that polyethersulfone membranes could be damaged after as few as 5-min exposure at 47 kHz. The ultrasonic power intensity in this work is not reported. While working at 3.8 W/cm^2 and 20 kHz Chen et al. (2006a) observed pitting and cracking of ceramic membrane material if the acoustic irradiation was within the cavitation region. Other researchers have found no evidence of membrane damage, but those researchers typically work at lower ultrasonic intensity levels (Chai et al., 1998; Lamminen et al., 2004; Muthukumaran et al., 2004).

Membrane damage will lead to a reduction in membrane life and this is likely to have a significant impact on the economic viability of the process. Similarly, the capital costs of transducers and the operating costs arising from operating at higher power levels can make such an operation economically unfeasible. A careful balance is required between the positive effects of ultrasound on system performance and the negative effects of capital and electricity expenses.

Many researchers have considered the use of intermittent or pulsed ultrasound to mitigate the potentially high energy consumption (Chen et al., 2006a; Matsumoto et al., 1996; Muthukumaran et al., 2007; Simon et al., 2000; Yuk and Youm, 2003). Yuk and Youm (2003) observed that intermittent ultrasound was very effective in maintaining the high flux and high permeability of BSA during crossflow ultrafiltration. Similarly, Matsumoto et al. (1996) found that alternate operation of an ultrasonic generator and the feed pump was effective in removing BSA fouling layers from a membrane during crossflow microfiltration. However, Chen et al. (2006a) argued that the relative permeate flux improvement decreased with long pulse intervals of ultrasound and only approached that of continuous sonication when the pulse interval was short (1.0 s on/0.1 s off). This is consistent with our own results (Muthukumaran et al., 2007) and those of Simon et al. (2000), which show very little flux enhancement when the pulse interval was of the order of minutes.

It is common in the food industry to operate a sequence of membrane modules in series, with the concentration of solids increasing across the system. In this instance, it may also be optimal to provide ultrasonic enhancement only to the final module, where fouling is the greatest. Use in this mode could significantly enhance productivity for minimum energy expense.

2.2 Operating Pressure Effects

In the absence of ultrasound, membrane permeate flux usually increases as the feed side pressure increases and then plateaus, as the effects of a greater driving force

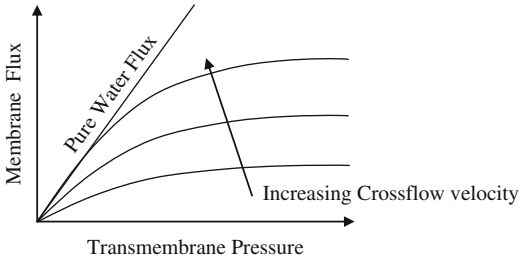

Fig. 23.4 A schematic of how membrane flux changes in the absence of ultrasound, as both transmembrane pressure and crossflow velocity are increased

are balanced by compaction of the fouling cake (see Fig. 23.4). Conversely, increasing the fluid pressure under ultrasound will increase the cavitation threshold and thus fewer bubbles will form (Henglein, 1987; Leighton, 1994). While this may reduce the number of cavitational collapse events, the pressure in the cavitating bubble will also be greater at the moment of collapse, and this can lead to more rapid and violent collapse events (Lorimer and Mason, 1987). These effects can also lead to unevenness of the ultrasonic field (Kylloenen et al., 2005). Conversely, a reduced number of bubbles can lead to greater penetration of the sound field, and this may increase the extent of cavitation at the membrane surface itself (Chen et al., 2006a).

There is thus likely to be an optimum operating pressure at which the membrane performance is maximized under ultrasound. Both Duriyabunleng et al. (2001) and Matsumoto et al. (1996) report such an optimum transmembrane pressure difference (Fig. 23.5). Chen et al. (2006a) found that at higher operating pressure, the cavitational intensity at the membrane surface increased, but that the ultrasonic enhancement declined. They argued that in this case, the fouling cake compaction caused by the higher transmembrane pressure was more significant than the turbulence due to ultrasound. Other researchers note only that increasing operating

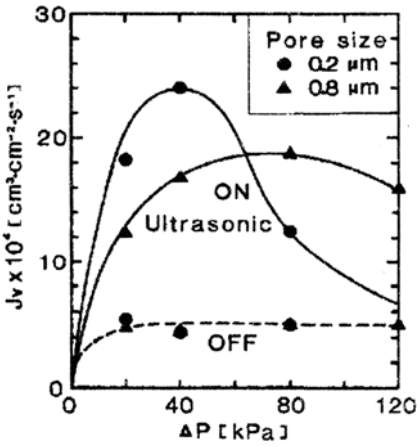

Fig. 23.5 Relationship between steady-state permeate flux (J_v) and transmembrane pressure (ΔP) for microfiltration of a yeast solution of 10 g/l (crossflow velocity 0.46 m/s, nominal ultrasonic power of 240 W) (reproduced from Matsumoto et al., 1996)

pressures leads to improved performance (Kobayashi et al., 1999; Yuk and Youm, 2003). Our own work showed that the effect of pressure was minimal when continuous low-frequency ultrasound was used (50 kHz), but that performance declined at high pressures when intermittent high-frequency ultrasound (1 MHz) was used (Muthukumaran et al., 2007). Indeed, in this work the permeate flux in the presence of ultrasound dropped below that without ultrasound at the highest operating pressure used.

2.3 The Influence of Crossflow Velocity

As shown in Fig. 23.4, in the absence of ultrasound, permeate flux generally increases as the flow velocity across the membrane increases. High fluid velocities reduce the thickness of the fouling cake by increasing the shear forces across the cake surface. Greater turbulence also reduces concentration polarization and thus increases mass transfer in the boundary layer. Some researchers have found that ultrasound has a proportionately greater effect at lower crossflow velocities when these effects are minimized (Duriyabunleng et al., 2001). However, our results (Muthukumaran et al., 2005a) showed that the effects of ultrasound were independent of crossflow velocity. This work also found that ultrasound remained effective even when hydrodynamic turbulence across the membrane was enhanced by the use of spacers. Hacias et al. (1997) suggest that the physical effects of cavitation can be more pronounced in fluid flow conditions. A moving fluid can assist the free flow of particles dislodged from the membrane, away from the membrane area.

High crossflow velocities will also assist in the movement of cavitation bubbles. In all cases where polymeric membrane damage due to sonication has been observed, there has been no crossflow across the membrane. In a stagnant environment it is possible for a cavitation bubble to become trapped at a point on the membrane surface and physically erode the surface by repeated oscillations at this point. Such a failure mechanism fits with the discussion of membrane damage reported in these papers.

2.4 Solids Concentration

A high concentration of particulate solids in the solution will attenuate the sound waves through acoustic impedance (Duriyabunleng et al., 2001; Wakeman and Tarleton, 1991). The degree of attenuation varies with the type of solids and experimental conditions. Furthermore, increasing feed concentrations can lead to higher viscosities and this is well known to dampen the effects of cavitation. Several studies report the effects of feed concentration (Duriyabunleng et al., 2001; Muthukumaran et al., 2005a; Wakeman and Tarleton, 1991; Yuk and Youm, 2003), but results of those studies conflict, with some research showing greater flux enhancement at high feed concentrations (Yuk and Youm, 2003), others showing no effects

(Muthukumaran et al., 2005a), and still others showing a loss of performance at high feed concentrations (Tarleton and Wakeman, 1992; Wakeman and Tarleton, 1991).

3 Membrane Cleaning

In all membrane operations, the processing operation must be halted regularly and the membranes cleaned for sanitation purposes and to remove the fouling cake. A typical cleaning cycle might include rinsing with an alkaline surfactant solution to remove protein or other organic deposits and/or an acid solution to remove inorganic contaminants, as well as a series of water flushes. Chlorine-derived cleaning agents may also be used to sanitize the membrane, and enzyme cleaning agents for protein removal are also gaining in popularity.

There has been considerable work on the use of ultrasound to supplement or replace some of these cleaning cycles (Chai et al., 1999; Feng et al., 2006; Kahler, 2004; Kawai et al., 2006; Kobayashi et al., 2003; Lamminen et al., 2004, 2006a, b; Muthukumaran et al., 2004, 2005b). There is particular interest in using this approach for membrane bioreactors, as ultrasonic cleaning can be completed in situ (Hoehn, 1998; Huang et al., 2005).

Our work shows that the use of ultrasound can increase the effectiveness of the alkaline cleaning step for removing protein deposits (Muthukumaran et al., 2005b). However, Fig. 23.6 shows that this step should still be conducted at a comparable pH, as a sharp optimum in cleaning effectiveness is apparent at this hydroxide concentration.

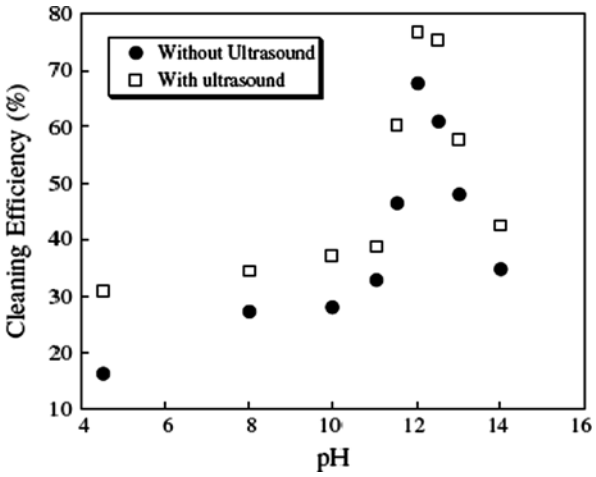

Fig. 23.6 Effect of solution pH on cleaning efficiency at 25°C of an ultrafiltration membrane fouled with a dairy whey solution in the presence and in the absence of ultrasound (50 kHz, nominal power 300 W) (reproduced from Muthukumaran et al., 2005b)

Alkaline cleaning is most effective at elevated temperatures where protein solubility is increased. However, high temperatures lead to high vapor pressures inside cavitation bubbles, which results in a dampening in collapse events. As a result, ultrasound has only a marginal impact when used to supplement a hot alkaline cleaning cycle for protein removal (Muthukumaran et al., 2005b).

Membrane cleaning is ideally conducted at low transmembrane pressures and high crossflow velocities so as to loosen the fouling cake. Several researchers (Li et al., 2002; Muthukumaran et al., 2005b) have found that the cleaning effect of ultrasound is not decreased by a higher crossflow velocity. As low pressures are generally associated with maximum cavitational activity, these conditions are also conducive to ultrasonic cleaning. Indeed, Bayevsky (2004) suggests that a vacuum pressure should be applied during cleaning to further reduce the cavitation threshold. This allows cavitational activity at lower acoustic pressures, which reduces the energy demand and the potential damage to sensitive filter membranes.

Many authors also indicate that lower frequencies (between 20 and 50 kHz) produce better membrane cleaning effectiveness than higher frequencies (between 100 and 200 kHz) (Kobayashi et al., 2003; Lamminen et al., 2004; Wakeman and Tarleton, 1991).

It is sometimes argued that it is environmentally attractive to use ultrasound to reduce the high chemical and water usage of classical membrane cleaning. However, a true environmental analysis must also include the greenhouse gas release associated with the power requirements of ultrasound. Caution must also be applied to ensure that the use of ultrasound for cleaning is economically viable. Our current calculations suggest that membrane cleaning applications may not provide sufficient return on investment unless deployed in conjunction with flux enhancement, as discussed in Section 2.

4 Industrial Scale Module Design

While most laboratory studies of membrane processing utilize baths or horns to supply ultrasonic power, such devices are not practically scalable to industrial operation. The only documented study of the large-scale use of ultrasonics in the food industry is a pilot scale study where a 94 × 10 cm spiral wound plastic microfiltration membrane was installed in a working wine plant (Bjorno and Bjorno, 2001). In this case, the membrane module was assembled with 24 standard transducers placed evenly around the unit (four rows of six transducers each). The 25–27 kHz transducers drew a total power of 3.6 kW. These authors observed no damage to the filter structure as determined by optical microscopy. The flux rate was observed to increase by between 3.5 and 10%, and the time between filter cleans was increased by 100%. The mining industry also uses ceramic microfilters at a commercial scale which are cleaned by ultrasound in an off-line process (Heikkinen et al., 2000).

Similarly, pilot scale studies have been completed on a range of pulp and paper mill waste streams, where ceramic microfiltration membranes were used with in situ ultrasonic cleaning. In this case, the tubular membranes were placed within a large feed vessel and ultrasound was provided by a bank of transducers along one wall of the vessel. The tubular membranes could be rotated within the acoustic field to both increase the crossflow velocity and provide an even exposure to the ultrasonic field (Kylloenen et al., 2005).

One of the greatest challenges facing this technology is the generation of a uniform acoustic field across the entire membrane surface in a full-scale module. An uneven application of the acoustic field is likely to result in membrane damage and will also cause inefficient energy consumption. Weavers et al. (2006) propose a number of novel configurations that might meet this objective (see Fig. 23.7). In one example, a cylindrical transducer is mounted around the outside of a membrane module, forming a shell that radiates ultrasonic energy inward. In a second example, a ring-shaped transducer is mounted externally or internally around the membrane element in a way that it can be moved longitudinally along the length of

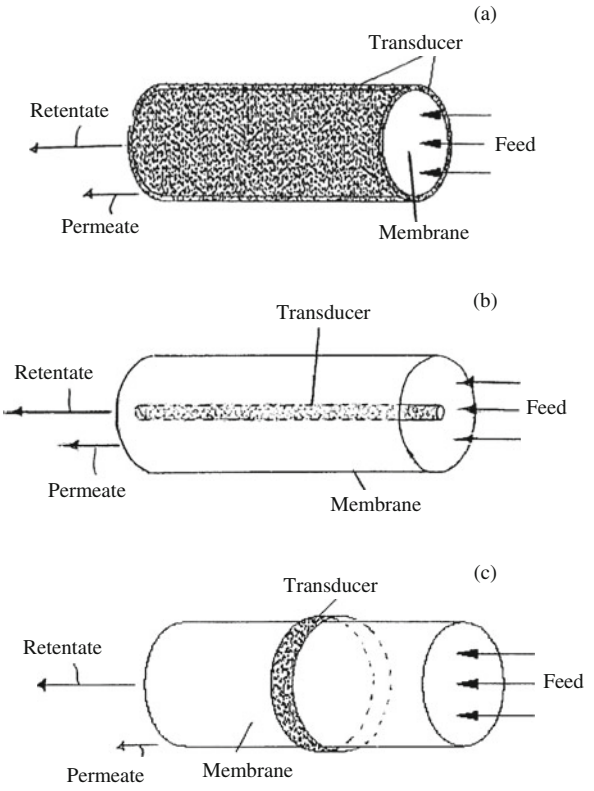

Fig. 23.7 Possible transducer configurations of a spiral wound or hollow fiber membrane module. Adapted from Weavers et al. (2006)

the membrane to provide a sweeping mode of operation. In yet a further example, a smaller diameter cylindrical transducer is mounted at the center of the membrane unit, providing a radial ultrasonic field that extends outward.

5 Related Technologies

There are a number of technologies that have been proposed for membrane flux enhancement that share common features with the use of ultrasonics. Most notable is the use of lower frequency vibration (around 50–60 Hz) in a direction tangent to the face of the membrane (New Logic International, 2001; Choi, 2003; Petala and Zouboulis, 2006). Vibratory shear-enhanced filtration (VSEP) is a commercialized technology that uses a sequence of parallel disc membranes located above a torsion spring that moves the stack back and forth between 1.9 and 3.2 cm (New Logic Research, 2007). Flux enhancements of 300–400% have been observed in these systems relative to a standard spiral wound membrane (Akoum et al., 2003, 2004). The disadvantage of this system is that the use of disc membranes limits the membrane area that can be provided (up to 151 m^2) (Akoum et al., 2005) and leads to a relatively large processing volume per unit of membrane area. The membrane unit must be placed vertically to accommodate the drive arrangement underneath. Rotating disc units produce similar effects (Bouzerar et al., 2003; Frappart et al., 2006; Jaffrin et al., 2004).

Researchers have also considered the injection of gas bubbles concurrently with the feed solution (Cui and Wright, 1994) and pulsatile flow (Finnigan and Howell, 1989). For membranes and/or foulants that are charged, the use of a pulsed electric field can also be used to create similar effects (Bowen and Sabuni, 1992; Tarleton and Wakeman, 1990, 1992; Wakeman and Tarleton, 1991).

6 Conclusions

Ultrasonic technology is now well established at a laboratory scale for membrane flux enhancement. However, the technology uptake to a commercial scale has been slow, and it is this area where future research must be focused. The particular issues that must be addressed in this respect are ensuring that a larger scale process is economical and provides an even ultrasonic intensity across the entire breadth of the membrane unit.

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