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Novel Yeast & Oil Drop Microfiltration Equipment

Michael T. Stillwell, Wiwit Sumritwatchasai, Richard G. Holdich, Serguei R. Kosvintsev

Introduction
The conventional microfiltration of yeast and oil is problematic due to irreversible fouling on the membrane surface and within the internal pore structure. These problems result in the need for high shear at the membrane surface, which entails:

- Higher operating costs
- Periodic replacement of deteriorated filters
- Cell or drop damage, affecting product quality and filtration performance

A new type of filter is commercially available, which is a true surface filter with no internal structure, where each pore forms a direct channel of uniform size from one side of the membrane to the other. These membranes can be used in an oscillating filtration system, which provides a high peak shear directly at the membrane surface. The benefits of this system are:

- High permeate fluxes
- Low operating pressures
- A gentle process not involving excessive shear forces
- Greater membrane longevity
- Lower operating costs

The oscillating microfiltration system has recently been tested in a consortium project involving three large oil companies. The aim was to filter Produced Water from oil rigs and reduce the oil concentration in the water from over 1000 ppm to 30 ppm in order to comply with the new environmental pollution limits. The system was capable of meeting the 30 ppm legal requirement and provided all of the above advantages including being a compact lightweight unit that operated at low noise levels.

In this work, filtration tests have been carried out to validate the system at different shear rates by varying the frequency and amplitude of oscillation for calcium carbonate, yeast and oil challenge suspensions. The membrane rejection was determined at different shear rates for each challenge suspension. It was found that high shear rates provide higher critical fluxes because the membrane surface is kept cleaner. However, the rejection decreases at higher shear rates because more of the smaller particles can pass through into the permeate. Experimental data relating the permeate pressure to the filtration time were compared to a predictive pore blocking model for constant rate filtration. The model was found to fit the experimental data well at higher shear rates but was poor at the low shear rates. It is suspected that cake filtration occurs at the lower shear rates, as there is not enough shear stress to keep the membrane surface clean. Simultaneous development work has been performed to develop a composite membrane
coating, which is designed to improve the membrane rejection for the smaller particle sizes, whilst retaining the advantages of the system stated previously.

**The Equipment**

The oscillating system was provided by Micropore Technologies Ltd., Leicestershire, UK. A tubular metal membrane with active area length 65 mm and diameter 14 mm was used with a slotted pore geometry of 400 microns length and 6 microns width (Figure 1).

![Microscope image of the tubular membrane used in the oscillating filtration system with pore dimensions of 400 microns length and 6 microns width.](image)

The membrane had a durable low surface free energy coating in order to minimise fouling during filtration. The membrane was attached to an electrically driven oscillating unit, which could be controlled for frequency and amplitude (Figure 2).

The membrane was inserted into a beaker containing the challenge suspension, which was agitated using a magnetic stirrer. The permeate was drawn through the membrane pores using a peristaltic pump. Flux measurements were made using a measuring cylinder and stopwatch, and pressure measurements were taken from the gauge located upstream of the pump. For each challenge suspension, the shear rate was increased from 249 s\(^{-1}\) to 7761 s\(^{-1}\) by altering the frequency and amplitude.
The feed and permeate size distributions were measured using a Coulter Multisizer. This device allows the mass of particles in each size grade to be calculated and thus the rejection can be calculated according to the following equation:

\[
\text{rejection} = \left(1 - \frac{\text{mass of particles in permeate grade}}{\text{mass of particles in feed grade}}\right) \times 100
\]

**Grade Efficiency Results**

Calcium carbonate suspensions, yeast suspensions and dispersions of crude oil in water were prepared using reverse osmosis water, the details of which are contained in Table 1.

<table>
<thead>
<tr>
<th>Suspension</th>
<th>Mean Size, microns</th>
<th>Concentration, g/litre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium Carbonate</td>
<td>2.1</td>
<td>1.0</td>
</tr>
<tr>
<td>Crude Oil</td>
<td>3.7</td>
<td>1.0</td>
</tr>
<tr>
<td>Yeast</td>
<td>3.9</td>
<td>0.1</td>
</tr>
</tbody>
</table>

The feed and permeate from each experiment were analysed using a Coulter Multisizer to determine the rejection efficiency as a function of particle size.

For calcium carbonate and yeast particles larger than 6 microns, 100% rejection was obtained for all shear rates, which is expected for a 6 micron pore width. However, the affects of oil droplet deformation at the membrane surface were observed as the rejection was only 97% for droplets 6 microns in diameter at the maximum and minimum shear rates. For an additional ten fold increase in the permeate pump setting, the rejection of oil droplets at 6 microns decreased to 90% at the maximum and minimum shear rates, which provides further evidence of droplet deformation. A comparison of other
rejection values for each suspension at a particle size of 2.5 microns is shown in Table 2 for the maximum and minimum shear rates.

Table 2  Summary of the rejection data for each suspension at a particle size of 2.5 microns for the maximum and minimum shear rates.

<table>
<thead>
<tr>
<th>Suspension</th>
<th>Rejection, %</th>
<th>Shear rate, s⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium Carbonate</td>
<td>45</td>
<td>7761</td>
</tr>
<tr>
<td></td>
<td>85</td>
<td>249</td>
</tr>
<tr>
<td>Crude Oil</td>
<td>77</td>
<td>7761</td>
</tr>
<tr>
<td></td>
<td>88</td>
<td>249</td>
</tr>
<tr>
<td>Yeast</td>
<td>0</td>
<td>7761</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>249</td>
</tr>
</tbody>
</table>

Table 2 shows that membrane rejection for the smaller particles is dependent upon the shear rate. For calcium carbonate particles sized 2.5 microns, only a 45% rejection was obtained at the highest shear rate of 7761 s⁻¹, whereas a rejection of 85% was obtained at the lowest shear rate of 249 s⁻¹. This trend occurs also for the crude oil and yeast suspensions. This demonstrates that the membrane surface is kept relatively cleaner at the highest shear rate, which prevents a cake layer from forming and allows more of the smaller material into the permeate. This is demonstrated quite clearly by the 0% rejection of yeast particles at the highest shear rate of 7761 s⁻¹, which had a low feed concentration of 0.1 g/litre and contained only 18% of the total particles in the calcium carbonate suspension.

**Pore Blocking Model**

A pore blocking model has been proposed for constant pressure filtration (Filippov et al. 1994), which has been modified to represent a constant rate process. The model is based on a sieve mechanism that takes into account the membrane pore size and particle size probability distribution functions. By assuming that the permeability at time infinity, \( k_\infty \), is 0, the model can be written as:

\[
J(t) = \frac{k_o}{\frac{1}{\Delta P} + \frac{k_o \cdot y \cdot c \cdot t}{t}}
\]

Where \( k_o \) is the initial permeability (m³/s.kPa), “y” is the blocking area (empirical constant), \( c \) is the concentration of the suspension (kg/m³) and “t” is the time (s).

The model predictions were compared to experimental permeate pressure measurements for a challenge suspension containing 0.2 g/litre calcium carbonate and 0.457 g/litre yeast. The model predictions were obtained by adjusting the empirical value “y” for each shear rate until the best fit was obtained. An example at the highest shear rate of 7761 s⁻¹ is shown in Figure 3.
Figure 3 shows that there is a good agreement between the model and experimental data, which is also obtained for the next two highest shear rates of 6468 s\(^{-1}\) and 5174 s\(^{-1}\). At a shear rate of 4225 s\(^{-1}\), the fit begins to deteriorate and the agreement becomes poor. This indicates that pore blocking occurred at the highest shear rate, but at low shear rates the fouling mechanism probably occurred via a cake filtration mechanism.

**Membrane Coating Development**

The minimum slotted pore width is currently 4-5 microns due to manufacturing limitations, which means that it is not possible to achieve a 100% particle rejection below 5 microns. The data shown previously also suggests that as the shear rate at the membrane surface increases, the rejection decreases because a cleaner membrane surface allows more of the smaller particles through into the permeate. In order to address these problems, a composite membrane has been developed which involves attaching a thin layer of particles onto the surface of the filter to create a permanent secondary membrane. Filtration is therefore performed entirely by the secondary membrane and the metal filter acts purely as a support to the coating. To demonstrate this concept, a metal filter with relatively large slot dimensions of 35 microns width and 800 microns length was used in filtration studies.

Commercially available glass micro-spheres shown in Figure 4a were deposited onto 42 mm diameter flat disc membranes and sintered under pressure to create a thin and robust porous coating (Figure 4b).
Figure 4 (a) the commercially available glass microspheres used to form the top coating and (b) an example of a sintered coating ready for filtration tests.

The filtration performance of the sintered discs were tested using a dead-end filtration device known as a stirred cell. Two types of calcium carbonate challenge suspension and a yeast challenge suspension were prepared using reverse osmosis water, the details of which are contained in Table 3.

Table 3 Details of the challenge suspensions used with the composite membranes.

<table>
<thead>
<tr>
<th>Suspension</th>
<th>Mean Size, microns</th>
<th>Concentration, g/litre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium Carbonate (Large)</td>
<td>7.5</td>
<td>0.5</td>
</tr>
<tr>
<td>Calcium Carbonate (small)</td>
<td>3.0</td>
<td>0.5</td>
</tr>
<tr>
<td>Yeast</td>
<td>4.3</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Low concentrations were used to minimise fouling in order to determine the approximate pore size of the membrane sample. A high shear rate of 5500 s\(^{-1}\) was used in each experiment in order to minimise fouling. The permeate pressure and flux trends are shown in Figures 6a and 6b.
Figures 6a and 6b show that the large calcium carbonate suspension had a constant permeate pressure of less than 0.07 bar and a constant permeate flux of 6000 litres/m²/hr. This indicates that a cake layer was not formed on the membrane surface during the filtration. The permeate was clear and a microscope analysis could not detect any particles indicating a 100% rejection of suspended material.

Under the same conditions of pump speed setting and shear rate, the permeate pressure for the small calcium carbonate suspension gradually increased to 0.3 bar. The permeate flux was lower than for the large calcium carbonate and showed a slight decline over time with a value of approximately 4500 litres/m²/hr. This indicates that particles in the feed became trapped inside the internal pore structure of the sintered membrane coating during the filtration. The measured rejection was approximately 90% for particle sizes down to 2.7 microns, but the feed particles deposited inside the sintered pore...
structure would have contributed to this rejection value. This result still provides useful information about the internal pore size of the sintered coating prepared under the particular set of bake conditions including temperature, time and pressure. Composite coatings that were not sintered to the same extent as the one reported here could easily pass all of the small calcium carbonate particles in the feed and provided no resistance to their flow.

The permeate pressure and flux trends for yeast are similar to those of the small calcium carbonate, but they also indicate that less internal blocking of the sintered coating occurred compared to the small calcium carbonate. This could be attributed to the deformable nature of the yeast under the high flux conditions, which pulled more yeast particles into the permeate. The measured rejection was approximately 85% for particle sizes down to 2.7 microns.

These results suggest that the concept behind the composite coating is working, because the internal pore structure of this particular membrane sample could trap particles down to 2.7 microns using a 35 micron slotted pore substrate as a support. Work is currently underway to reduce the sintered pore size further in order to carry out more extensive tests on yeast filtration, which is known to be a problematic challenge suspension even for certain types of surface filter (Chandler, Zydney 2006).

Conclusions
This investigation has shown that high shear rates are successful at minimising surface fouling, but this also leads to a decrease in the membrane rejection. It is also possible for deformable material larger than the slotted pore width to enter the permeate. At high shear rates, a pore blocking model for constant rate filtration can accurately represent the membrane fouling. At low shear rates, membrane fouling is attributed to a cake filtration mechanism. Initial development work to overcome the problems of membrane rejection has focused on the membrane coating. A composite coating that involves forming a permanent secondary membrane over the top of the metal surface filter shows good signs for future development work.

References