Effect of pH level and acid type on total ammoniacal nitrogen (TAN) retention and fouling of reverse osmosis membranes processing swine wastewater

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ABSTRACT

Wastewaters fed to reverse osmosis (RO) membranes sometimes need to be acidified to prevent inorganic fouling and increase total ammoniacal nitrogen (TAN) retention. In this project, the effect of pH level (6.5–7.1) and acid type (HCl vs. H2SO4) on membrane fouling and cleaning as well as permeate flux and quality during the processing of swine wastewater with a spiral-wound RO membrane was investigated. The use of H2SO4 to lower pH produced slightly higher permeate fluxes than HCl and there was no indication of sulfate precipitates on the membrane. Membrane fouling intensity and flux recovery upon cleaning were not affected by pH level or acid type. Lowering swine wastewater pH from 7.1 to 6.5 with HCl reduced TAN concentration in permeate from 142 to 59 mg/L. Using H2SO4 to lower pH to 6.5 further decreased TAN concentration to 39 mg/L. At pH 6.5 with both acid types, the concentration of unionized NH3 in the permeate was below the Canadian guideline of 0.019 mg/L for the release of wastewater to an aquatic environment. The use of H2SO4 would be recommended with swine wastewater, because of lower cost and volumetric input required to lower pH, as well as higher permeate quality and flux.

Key words | acid type, ammoniacal nitrogen retention, pH level, reverse osmosis, swine wastewater

INTRODUCTION

Acidification of water and wastewater fed to reverse osmosis (RO) membranes is often required to increase the solubility of alkaline scale and prevent inorganic fouling (Singh & Song 2008; Antony et al. 2011). Precipitation of inorganic materials on the membrane can drastically reduce flux and salt crystals can rupture the RO membrane operated under high pressures (Darton et al. 2004). Wastewaters rich in total ammoniacal nitrogen (TAN), such as swine wastewater, also require acidification in order to maximize TAN retention and reduce ammonia volatilization from the concentrated feed. In solution, TAN is partitioned between free ammonia (NH3), a small, unionized and highly volatile molecule that readily diffuses through RO membranes, and ammonium (NH4+), an ionized molecule that is more easily retained by membranes because it forms loose complexes with anions such as HCO3− or PO43− (Masse et al. 2008). Retention of complexed ammonium by RO membranes ranged from 80 to 98%, while the retention of free ammonia varied between 10 and 40% (Awadalla et al. 1994; Koyuncu et al. 2001).

Reducing feed pH and temperature increases TAN retention by decreasing the fraction present as NH3. Reducing the pH of swine wastewater from 8.2 to 6.2 increased TAN retention from 82 to 92% (Pieters et al. 1999). The retention of TAN by RO membranes fed anaerobically digested manure ranged from 75 to 96% at pH 8.0, and was nearly 100% at pH 4.0 (Bilstad et al. 1992). However, swine wastewater has a high buffering capacity and acid requirements to lower pH may become excessive. Masse et al. (2008) concentrated various swine wastewaters with flat RO membrane sheets installed on a laboratory-scale unit and concluded that reducing wastewater pH to 6.5 would maximize TAN retention while minimizing...
acid requirements. At pH 7.0, the recovery rate would have to be reduced to maintain TAN below 100 mg/L in the permeate.

Sulfuric acid is often used to acidify wastewater, because it is economical and its production is more environmentally friendly than that of HCl (Bonné et al. 2000; Dai & Blanes-Vidal 2013). However, the precipitation of sulfate compounds, such as BaSO₄ or CaSO₄ (gypsum), presents a major challenge because it cannot be prevented by lowering feed pH and these precipitates are difficult to clean from membrane surface (Lee et al. 1999; Le Gouellec & Elimelech 2002; Duranceau et al. 2003; Antony et al. 2011). Barium sulfate precipitated on the surface of RO membranes filtering a hard water acidified with H₂SO₄, while no precipitates were observed with HCl (Bonné et al. 2000). Using H₂SO₄ in combination with an antiscalant to control precipitation was still recommended, because operating costs remained lower when HCl was used to acidify the wastewater. Singh & Song (2008) studied the impact of acidifying a silica colloid solution to pH 3 with weak and strong acids on the fouling potential of ultrafiltration (UF) membranes. All strong acids, namely HCl, H₂SO₄, and nitric acid, produced a similar fouling potential, which was higher than that of the weaker acids. Koyuncu (2003) reported a 12% decrease in the flux of a nanofiltration (NF) membrane when using H₂SO₄ instead of HCl to acidify a dye bath wastewater, mainly due to an increase in conductivity with the former acid. However, NaCl retention was lower when the wastewater was acidified with HCl (15%) than with H₂SO₄ (25%).

There are very few publications on the effect of acid type on membrane fouling and permeate quality. The objective of this research project was to investigate the effect of acidification level and acid type on membrane fouling and cleaning as well as permeate flux and quality during the processing of swine wastewater with a spiral-wound RO membrane installed on a semi-commercial scale unit. Based on previous work by Masse et al. (2008), a narrow range of pH values, from 6.5 to 7.1, was tested, in order to determine the pH at which a commercial system would have to be operated in order to optimize TAN retention while minimizing acid addition. Two types of acid were compared, namely HCl and H₂SO₄.

### MATERIALS AND METHODS

#### Swine wastewater

About 4 m³ of swine wastewater was collected from a mechanical, in-barn, solid–liquid separator installed on a commercial farrow-to-finish swine farm in St-Isidore, Quebec, as described in Masse et al. (2012). Prior to each filtration run, 500 L of swine wastewater was acidified from pH 7.8 to the required pH level (6.5, 6.8, or 7.1) with HCl. At pH 6.5, acidification was also conducted with H₂SO₄. Both acids were used at 10 N. The characteristics of the acidified swine wastewater fed to the membrane are presented in Table 1.

#### Membrane and RO unit

The SW30 membrane (DOW FILMTEC™) and the semi-commercial scale RO unit used for the project were described in Masse et al. (2013). The spiral-wound SW30 element had an active surface area of 7.4 m² and a nominal NaCl retention of 99.4%. It was used for 12 24-h filtration cycles at 55 bar with highly charged swine wastewater prior to this project (Masse et al. 2013). Previous usage ensured that the membrane was fully compacted and irreversible fouling of vulnerable sites had occurred prior to this project.

The RO unit was equipped with a 10-μm cartridge pre-filter to remove large particles that could damage the membrane, a piston pump to build pressure, and a patented turbo pump (Dominion & Grimm, Montréal, QC) designed to increase turbulence in the spiral-wound element. The system was operated at a crossflow velocity of 12.7 cm/s (1.24 m³/hr). Permeate flux was continuously measured with an impeller flow meter (FPR301, Omega Engineering Inc.). Feed temperature was maintained at 20.5 ± 0.5 °C by keeping the swine wastewater in a refrigerated bulk tank.

#### Experimental run

Before each cycle, water flux through the membrane was measured at 34.5 bar using tap water at an average conductivity of 500 μS/cm and a temperature of 20.3 °C. Ionic
retention efficiency of the membrane was also determined by measuring the conductivity of the feed, concentrate, and permeate, as explained in Masse et al. (2015).

The acidified swine wastewater (500 L) was recirculated without pressure through the RO unit for 5 min before an initial feed sample was collected. Pressure was then gradually increased from 20 to 55 bar over 50 min. The swine wastewater was first concentrated to 45% of the initial volume (concentration factor of 2.2) by removing the permeate in a separate vessel. The concentration period, which lasted about 2.2 h, would be representative of the initial period of a bleed and feed system, when feed is progressively concentrated to a set value, before steady-state operation. During the concentration period, feed, concentrate, and permeate were sampled after the removal of 25, 150, and 275 L. The permeate collected during the entire concentration period was also mixed and sampled. At the end of the concentration period, the concentrate and permeate lines were returned to the refrigerated bulk tank, and the system was operated in full-recycling mode for the rest of the 24-h cycle. This period would be representative of steady-state operation of the unit. The pH of the swine wastewater was regularly measured and adjusted.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>HCl pH 7.1</th>
<th>HCl pH 6.8</th>
<th>HCl pH 6.5</th>
<th>H₂SO₄ pH 6.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Eₜ (mS/cm)</td>
<td>27.4 ± 0.1b</td>
<td>27.9 ± 0.2b</td>
<td>28.9 ± 0.3c</td>
<td>26.5 ± 0.2a</td>
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<tr>
<td>pH</td>
<td>7.13 ± 0.06b</td>
<td>6.82 ± 0.04b</td>
<td>6.54 ± 0.00a</td>
<td>6.53 ± 0.00a</td>
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<tr>
<td>DM (g/L)</td>
<td>11,616 ± 142a</td>
<td>11,797 ± 7a</td>
<td>12,417 ± 182a</td>
<td>11,256 ± 143a</td>
</tr>
<tr>
<td>VDM (% DM)</td>
<td>36.1 ± 0.8a</td>
<td>35.6 ± 1.0a</td>
<td>39.2 ± 6.5a</td>
<td>35.4 ± 0.8a</td>
</tr>
<tr>
<td>SS (mg/L)</td>
<td>1,513 ± 74a</td>
<td>1,561 ± 72a</td>
<td>1,469 ± 33a</td>
<td>1,594 ± 11a</td>
</tr>
<tr>
<td>VSS (% SS)</td>
<td>82.4 ± 1.7a</td>
<td>81.4 ± 0.1a</td>
<td>79.1 ± 1.9a</td>
<td>77.7 ± 6.3a</td>
</tr>
<tr>
<td>TAN (mg/L)</td>
<td>2,873 ± 25a</td>
<td>2,839 ± 129a</td>
<td>2,886 ± 53a</td>
<td>2,757 ± 99a</td>
</tr>
<tr>
<td>P (mg/L)</td>
<td>44 ± 6.8a</td>
<td>39 ± 5.9a</td>
<td>40 ± 6.1a</td>
<td>33 ± 5.1a</td>
</tr>
<tr>
<td>K (mg/L)</td>
<td>2,522 ± 192a</td>
<td>2,355 ± 177a</td>
<td>2,188 ± 170a</td>
<td>2,318 ± 177a</td>
</tr>
<tr>
<td>Ca (mg/L)</td>
<td>96 ± 3.8a</td>
<td>93 ± 3.7a</td>
<td>89 ± 3.5a</td>
<td>99 ± 3.9a</td>
</tr>
<tr>
<td>Cl (mg/L)</td>
<td>2,343 ± 35b</td>
<td>3,108 ± 128c</td>
<td>4,304 ± 11d</td>
<td>1,358 ± 20a</td>
</tr>
<tr>
<td>S (mg/L)</td>
<td>234 ± 77a</td>
<td>228 ± 61a</td>
<td>198 ± 44a</td>
<td>1,526 ± 17b</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parameters</th>
<th>HCl pH 7.1</th>
<th>HCl pH 6.8</th>
<th>HCl pH 6.5</th>
<th>H₂SO₄ pH 6.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Eₜ (mS/cm)</td>
<td>54.7 ± 1.0ab</td>
<td>57.5 ± 0.5b</td>
<td>61.0 ± 0.8c</td>
<td>52.4 ± 0.6a</td>
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<tr>
<td>pH</td>
<td>7.21 ± 0.01c</td>
<td>6.89 ± 0.00b</td>
<td>6.56 ± 0.01a</td>
<td>6.62 ± 0.05a</td>
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<tr>
<td>DM (g/L)</td>
<td>30,954 ± 3,705a</td>
<td>31,791 ± 948a</td>
<td>33,549 ± 2,402a</td>
<td>33,164 ± 1,013a</td>
</tr>
<tr>
<td>VDM (% DM)</td>
<td>48.8 ± 7.1a</td>
<td>52.7 ± 0.7a</td>
<td>56.1 ± 3.7a</td>
<td>47.5 ± 2.2a</td>
</tr>
<tr>
<td>SS (mg/L)</td>
<td>6,281 ± 78a</td>
<td>6,226 ± 108a</td>
<td>6,237 ± 47a</td>
<td>6,414 ± 148a</td>
</tr>
<tr>
<td>VSS (% SS)</td>
<td>77.2 ± 0.7a</td>
<td>79.7 ± 0.6a</td>
<td>83.1 ± 4.5a</td>
<td>83.0 ± 0.1a</td>
</tr>
<tr>
<td>TAN (mg/L)</td>
<td>6,281 ± 78a</td>
<td>6,226 ± 108a</td>
<td>6,237 ± 47a</td>
<td>6,414 ± 148a</td>
</tr>
<tr>
<td>P (mg/L)</td>
<td>93 ± 33a</td>
<td>74 ± 27a</td>
<td>55 ± 20a</td>
<td>75 ± 2.5a</td>
</tr>
<tr>
<td>K (mg/L)</td>
<td>5,542 ± 135a</td>
<td>5,448 ± 133a</td>
<td>5,354 ± 131a</td>
<td>5,385 ± 277a</td>
</tr>
<tr>
<td>Ca (mg/L)</td>
<td>193 ± 36a</td>
<td>171 ± 32a</td>
<td>148 ± 28a</td>
<td>193 ± 11a</td>
</tr>
<tr>
<td>Cl (mg/L)</td>
<td>6,145 ± 158b</td>
<td>9,706 ± 178c</td>
<td>13,359 ± 226d</td>
<td>3,131 ± 46a</td>
</tr>
<tr>
<td>S (mg/L)</td>
<td>429 ± 71a</td>
<td>417 ± 62a</td>
<td>392 ± 53a</td>
<td>5,000 ± 56b</td>
</tr>
</tbody>
</table>

Ec, electrical conductivity; DM, dry matter; VDM, volatile (organic) dry matter; SS, suspended solids; VSS, volatile (organic) suspended solids; TAN, total ammoniacal nitrogen.

In each line, values with the same letters are not different at p < 0.05.
to the target value by adding HCl or H₂SO₄ using a peristaltic pump.

At the end of the 24-h cycle, feed was again sampled and the RO unit was rinsed by passing tap water through the system at the same crossflow velocity (12.7 cm/s) as that used during swine wastewater filtration. The unit was then cleaned successively with permeate and a solution of ethylenediaminetetraacetic acid (2 mM EDTA), sodium tripolyphosphate (2.7 mM STPP), and NaOH to adjust solution pH to 10.9. Each cleaning operation lasted for 1 h, as cleaning solution temperature increased from 20 to 40°C, and was conducted at a high crossflow velocity of 31.8 cm/s. The unit was then filled with deionized water and left to soak for 3 days before a new cycle was initiated. After each cleaning operation, tap water flux was measured as described above. Each pH/acid combination was tested in duplicate cycles, randomly distributed over eight cycles.

### Samples and analyses

Initial feed and concentrate samples collected at the end of the concentration process and at the end of the filtration cycles were analyzed for conductivity, pH, dry matter (DM), volatile dry matter, suspended solids (SS), volatile suspended solids, TAN, phosphorus, potassium, calcium, chloride, sulfdide, magnesium, and alkalinity using standard methods as described in Masse et al. (2010). Total permeate was analyzed for conductivity, pH, and TAN. The fraction of unionized ammonia (NH₃) in permeate was calculated using the equilibrium equations presented in Masse et al. (2008). Permeate, feed, and concentrate samples collected after the removal of 25, 150, and 275 L during the concentration process were analyzed for TAN concentration and were used to determine TAN retention by the membrane.

Flux recovery was determined by comparing tap water fluxes measured after swine wastewater filtration, cleaning with permeate or the EDTA-STPP solution, and soaking to the tap water flux measured at the beginning of each cycle. Tukey’s multiple comparison test in SPSS® (version 21) was used to test the significance \( p \leq 0.05 \) of pH level and acid type on feed, permeate, and concentrate characteristics as well as flux recovery.

### RESULTS AND DISCUSSION

#### Effluent characteristics and acid requirements

Effluent characteristics are presented in Table 1. The four effluents fed to the membrane had similar characteristics except for conductivity and pH, as well as chloride and sulfide contents. Chloride (Cl⁻) concentration in the feed increased from 1,358 mg Cl⁻/L in raw wastewater to 4,304 mg Cl⁻/L in effluents acidified to pH 6.5 with HCl. Lowering wastewater pH to 7.1, 6.8, and 6.5 required the addition of 27 mmoles, 50 mmoles, and 84 mmoles of HCl/L of wastewater, respectively. Three times more acid was thus necessary to lower wastewater pH from 7.1 to 6.5. This pH range corresponds to a region of high bicarbonate buffer capacity in the manure system (Georgacakis et al. 1982), and typically consumes high volumes of acid.

At pH 6.5, concentrate chloride concentration reached 13,339 mg Cl⁻/L at the end of the concentration period (Table 1) and 15,099 mg Cl⁻/L at the end of the 24-h cycle. The high chloride content was due to volumetric concentration of the wastewater as well as additional acid input during the 24-h filtration cycle to maintain pH at the target value. At all three pH levels, an additional 80 mmoles HCl/L of wastewater had to be added to the feed because swine wastewater pH tended to increase during the concentration and filtration cycles, probably due to CO₂ volatilization. Sørensen & Eriksen (2009) also reported increases in the pH of stored acidified manure, especially when the manure was aerated. Frequent recirculation of the feed through the system, as was the case during the experimental cycles, increased aeration of the wastewater as well as CO₂ volatilization. In a commercial unit with a larger filtering surface area, the wastewater would not recirculate as often through the system, which would reduce acid requirements during processing of the wastewater.

Lowering swine wastewater pH to 6.5 required 47 mmoles H₂SO₄/L of wastewater, with an additional 37 mmoles/L during the 24-h filtration cycle. The volume of acid was thus reduced by about half compared to HCl, as expected since H₂SO₄ has twice the acid equivalency of HCl. Sulfide (S⁻²) concentration was increased from 220 mg S⁻²/L in raw wastewater to 1,526 mg S⁻²/L in...
acidified feed (Table 1). Sulfide concentration reached 5,000 mg S\(^{-2}\)/L in the concentrate at the end of the concentration period (Table 1), and 5,773 mg S\(^{-2}\)/L at the end of the 24-h cycle. This value is slightly above the concentration (5,000 mg/L) at which Lee et al. (1999) observed a drastic decline in the flux of NF membranes, due to CaSO\(_4\) precipitation on the membrane surface.

Hydrochloric acid addition increased conductivity of the feed from 25.5 mS/cm in the raw swine wastewater to 27.4 mS/cm, 27.9 mS/cm, and 28.9 mS/cm at pH 7.1, 6.8, and 6.5, respectively (Table 1). With H\(_2\)SO\(_4\), conductivity was only increased to 26.5 mS/cm, because of lower requirements to reach pH 6.5 (164 mmoles and 84 mmoles of HCl and H\(_2\)SO\(_4\), respectively, per L of wastewater) as well as the lower conductivity of H\(_2\)SO\(_4\) compared to that of HCl.

**Permeate flux**

Figure 1 presents transmembrane pressure, permeate flux, and feed conductivity during the 24-h filtration cycle. Periods I and II correspond to the 2.2-h filtration cycle. During Period I (the initial 50 min), pressure was gradually increased from 20 to 55 bar as swine wastewater was slowly concentrated from 26–29 to 32–35 mS/cm. During that period, the flux steadily increased because the transmembrane pressure was increased at a faster rate than osmotic pressure. In Period II, the feed was further concentrated at a constant pressure of 55 bar. Flux decreased proportionally to the increase in conductivity, mainly due to increases in osmotic pressure and concentration polarization. During Period III, the system was...
operated in full-recycling mode and at constant pressure. Flux further decreased by 15.7% ± 1.4% for all effluents acidified with HCl and 12.6% ± 0.1% for feeds acidified with H2SO4. Part of the flux reduction was attributable to an increase of about 8 mS/cm in the conductivity of all effluents acidified with HCl, because of acid addition during Period III. With H2SO4, however, conductivity remained nearly constant throughout the period.

The swine wastewater acidified with H2SO4 maintained a slightly higher flux that the three other effluents (Figure 1(b)), mainly due to the lower conductivity of this effluent throughout the filtration cycle (Figure 1(c)). The swine wastewater acidified with H2SO4 exhibited no drastic decreases in flux, which would indicate that sulfate-containing compounds had precipitated on the membrane surface. Le Gouellec & Elimelech (2002) reported that the presence of bicarbonates, magnesium ions, and humic acid in solution retarded the onset of CaSO4 precipitation by tying up calcium ions and thus reducing the amount available for gypsum formation. The swine wastewater had low magnesium concentrations (28.7 ± 2.9 mg/L and 51.4 ± 14.2 mg/L in feed and concentrate, respectively), but high concentrations of bicarbonate, with an alkalinity (before acidification) of 11,306 ± 33 mg CaCO3/L, and high levels of organic (volatile) matter, representing, on average, 80% of the SS and 44% of DM in the feed and concentrate (Table 1). The presence of these charged species may have prevented excessive sulfate-containing precipitates on the membrane.

**Flux recovery and membrane salt retention capacity**

Flux recoveries after various cleaning operations are presented in Figure 2. After the 24-h filtration cycle, the membrane was rinsed by passing tap water through the system at the same crossflow velocity as that used during swine wastewater filtration. The objective was to remove the wastewater from the membrane casing without excessive disturbance to the fouling layer on the membrane surface. Rinsing would nevertheless remove part of the gel layer, which has been found to be a main contributor to fouling during swine wastewater processing with RO as well as UF membranes (Zhang et al. 2007; Masse et al. 2012). Flux recovery of the rinsed membrane ranged from 71.4 to 77.5% without significant differences (p < 0.05) between the four effluents (Figure 2), suggesting that pH level and acid type had no effect on membrane fouling.

Successively cleaning with permeate, cleaning with the EDTA-STPP solution, and soaking the membrane in permeate water for 3 days increased tap water flux to 90.2% ± 2.11%, 97.5% ± 1.7%, and 99.0% ± 0.7% of initial flux, respectively. Again, there was no significant effect of pH level or acid type on flux recovery. Finally, the salt retention capacity of the membrane remained at 99.5 ± 0.1% throughout the experimental period, indicating no structural damage to the membrane during the experiment.

**TAN in permeate and retention by the RO membrane**

TAN concentration in permeate and retention by the SW30 membrane are presented in Figure 3(a) and 3(b), respectively. Reducing pH from 7.1 to 6.5 with HCl significantly decreased TAN concentration in permeate from 142 to 59 mg/L (Figure 3(a)). Acidifying the wastewater to pH 6.5 with H2SO4 further decreased TAN concentration to 39 mg/L. The retention of TAN by the RO membrane ranged from 96.5% when the effluents were acidified to 7.1 with HCl to 99.0% when the effluents were acidified to 6.5 with H2SO4 (Figure 3(b)). At pH 6.5, retention was significantly higher when the wastewater was acidified with H2SO4 than HCl, which may be explained by the lower retention by RO membranes of monovalent ions such as Cl− than divalent ions such as SO42− (Richards et al. 2011). The passage of Cl− anions through the membrane would...
increase the passage of NH₄⁺ to equilibrate charges in the permeate when feed was acidified with HCl.

Permeate pH ranged from 6.2 when the effluents were acidified to 7.1 with HCl to 5.3 when the effluents were acidified to 6.5 with H₂SO₄ (Figure 3(c)). At these pH levels, the concentration of unionized ammonia (NH₃) would be 0.082 mg/L, 0.017 mg/L, and 0.006 mg/L in the permeate of effluents acidiﬁed with HCl to pH 7.1, 6.8, and 6.5, respectively, and 0.003 mg/L for the effluents acidiﬁed to pH 6.5 with H₂SO₄. Unionized ammonia concentration in permeates from swine wastewater acidiﬁed to 6.5 would thus be well below Environment Canada’s guideline for the release of wastewater to the environment. The use of H₂SO₄ would be recommended with swine wastewater because of lower cost, lower volumetric input required to lower pH, and higher permeate quality.

CONCLUSION

Swine wastewater was acidiﬁed from pH 7.8 to 7.1, 6.8, and 6.5 with HCl and to pH 6.5 with H₂SO₄. The effect of pH level and acid type on membrane ﬂux, membrane fouling, and permeate quality was investigated. Acidiﬁying the effluents with H₂SO₄ produced slightly higher ﬂux, because of lower increase in feed conductivity than when using HCl to decrease pH. However, there was no acid type or pH level effect on membrane fouling or ﬂux recovery after cleaning the membrane. Lowering pH from 7.1 to 6.5 with HCl reduced TAN concentration in permeate from 142 to 59 mg/L. Lowering swine wastewater pH to 6.5 with H₂SO₄ further decreased TAN concentration to 39 mg/L. At a pH of 6.5, the concentration of unionized NH₃ in the permeate was well below Environment Canada’s guideline.

REFERENCES


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