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Fouling of Inorganic Membrane and Flux Enhancement in Membrane-Coupled Anaerobic Bioreactor

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ABSTRACT

The fouling mechanism of an inorganic membrane was studied during the operation of a membrane-coupled anaerobic bioreactor (MCAB) when alcohol distillery wastewater was used as a digester feed. It was observed that the fouling mechanism of an inorganic membrane was significantly different from that of conventional membrane filtration processes. The main foulant was identified to be an inorganic precipitate, struvite ($MgNH_4PO_4 \cdot 6H_2O$), rather than anaerobic microbial flocs. Struvite appears to be precipitated not only on the membrane surface but also inside the membrane pores. The amount of struvite generated during the bioreaction was estimated to be about 2 g/L alcohol distillery wastewater. The inorganic foulant was not easily removed by general physical cleaning such as depressurization, lumen flushing, and backflushing. Based on these findings, the membrane fouling was alleviated and thus flux was enhanced by adopting a "backfeeding" mode which has dual purpose of feeding and backflushing with particle-free acidic wastewater used as the feed for anaerobic digestion.

Key Words. Membrane-coupled anaerobic bioreactor (MCAB); Inorganic membrane; Struvite; Backflushing; Backfeeding.

INTRODUCTION

Anaerobic digestion is an energy-saving process to produce methane gas using high-strength organic wastewater (1–3). In Asian countries such as

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South Korea, this process has been widely applied to alcohol distillery wastewater treatment. Recently membrane technology has been considered for incorporation in anaerobic digestion in order to improve the efficiency of the anaerobic digester. The so-called membrane-coupled anaerobic bioreactor (MCAB) systems offer advantages such as high efficiency and better quality of effluent due to the complete recycling of microorganisms (4, 5).

However, the major hurdle in the extensive use of MCAB system is the continuous reduction of permeation flux caused by membrane fouling. Membrane fouling in the MCAB system has been attributed to the deposition of colloids such as microorganisms as well as the adsorption of organic species (6). Recently, Choo et al. (7) applied alcohol distillery wastewater to MCAB using an organic ultrafiltration membrane for 200 days. They found an inorganic precipitate, struvite ($MgNH_4PO_4 \cdot 6H_2O$), which formed on the membrane surface, could be a major foulant in MCAB for alcohol distillery wastewater in long-term operation. Several remedial actions have been tried to prevent the deposition of a cake layer as well as for the removal of a cake layer. Periodic backflushing (8), multiphasic flowing (4), and lumen flushing (7) have been traditionally used. Moreover, special kind of modules have been developed such as intermittent suction using external pressure-type membranes (5, 9) and rotating disk membranes (10).

Since most full-scale MCAB plants operate with organic membranes (11, 12), the fouling phenomena of inorganic membranes in the MCAB system have not been well studied. Some results obtained with inorganic membranes in MCABs have been limited to short-term operations (4, 5, 7, 13, 14). An effective method for the suppression of struvite formation in MCAB for alcohol distillery wastewater has not been known.

In this study the long-term fouling mechanisms of an inorganic membrane were studied in the MCAB system for alcohol distillery wastewater, and a new operation mode, so-called "backfeeding," was developed as an effective removal method of the inorganic precipitate struvite which strongly attached to the membrane surface and pores.

EXPERIMENTAL

Membranes

The membrane used was a tubular ceramic membrane (Carbosep-60 kit, Tech-Sep, France) with a pore size of 0.45 μm. The skin and support layer were made of zirconium oxide and carbon, respectively. The inner and outer diameters and length of a membrane were 6 mm, 10 mm, and 600 mm, respectively, with an effective membrane area of 113 cm^2. The used membrane was cleaned with sodium hypochlorite solution and then rinsed with ultrapure water. About 90% of the initial flux was recovered by such a washing procedure.

Pretreatment of the Feed

Raw wastewater was delivered from an alcohol fermentation plant in South Korea and was pretreated to remove suspended solids (SS) because the particles in raw wastewaters might decrease the biochemical reaction rates and cause a failure of digestion. It was pretreated using a 0.08-μm zirconium oxide inorganic membrane. The almost particle-free permeate was used as the digester feed. The major chemical parameters of the feed are listed in Table 1. The organic strength was more than 40,000 mg/L as COD_{Cr}. The wastewater also contains inorganic ions such as Mg^{2+}, NH_4^+, and PO_4^{3-}. Distillery wastewater is usually acidic because it contains various volatile fatty acids (VFA) generated by the fermentation reactions (14).

Operation of the MCAB System

Figure 1 shows a schematic diagram of the normal operation mode of an MCAB system. The anaerobic bioreactor employed continuous mechanical stirring white it was maintained at a thermophilic temperature of 55°C. The seed sludge was obtained from an anaerobic digestion plant where it had been acclimated to the alcohol distillery wastewater for over 2 years. Excess permeate over the feed volume was discharged by a dual head microperistaltic pump. Consequently, the working volume of the bioreactor was constantly maintained at 5 L. The digester broth in the digester was pumped into the membrane module by a positive displacement pump.

The fluid velocity through the membrane channel was controlled at 3 m/s by regulation of the inverter connected to the main pump (vane-type), while the transmembrane pressure was regulated using a backpressure valve. At this hydrodynamic condition the Reynolds number was calculated to be 34,600, indicating a turbulant state. An operating pressure of 0.6 bar was selected because the limiting flux was reached at this transmembrane pressure under the operating conditions listed above.

TABLE 1
Composition of a Pretreated Alcohol-Distillery Wastewater

Chemical parameters	Value
pH	4.0
VFA	3,230 mg/L
COD_{Cr}	42,600 mg/L
Mg^{2+}	14.9 mmol/L
NH_4^+	5.37 mmol/L
PO_4^{3-}	7.55 mmol/L

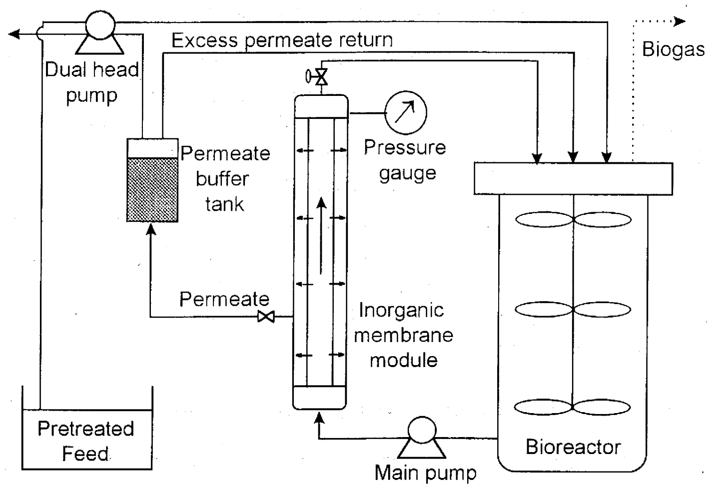

FIG. 1 Schematic of the normal operation mode of the membrane-coupled anaerobic bioreactor (MCAB).

Backflushing was performed periodically using the permeate stored in the permeate buffer tank as shown in Fig. 2(a). In this mode, 30 minutes of normal operation and 30 seconds of backflushing were repeated automatically under a pressure of 1 bar.

The "backfeeding mode," in which acidic digester feed was introduced countercurrently to the normal mode, was performed every 2 hours of normal operation as shown in Fig. 2(b). The backfeeding time increased with operation time because the membrane pore became more and more plugged. The backfeeding time was increased from 1 to 2 minutes with operation time because the backfeeding time was adjusted to supply a fixed amount of feed (70 mL/cycle) to the MCAB system.

Analytical Methods

The analytical methods from *Standard Methods* (16) were adopted for the measurement of chemical oxygen demand (COD_{Cr}), digester broth suspended solids (MLSS), pH of digester feed, and digester broth. Total volatile fatty acids (VFA) were determined by using a spectrophotometric method with the Hach DR-2000 instrument and reagent kits. The sample of digestion broth was pretreated by centrifuging the broth for 15 minutes at 3000 rpm (1200*g*).

Analyses of Mg^{2+}, NH_4^+, and PO_4^{3-} ions in the digester feed, digester broth in the bioreactor, and the precipitated white crystals were performed by the direct injection of aqueous samples to a Dionex 4500 I ion chromatograph (IC) equipped with a sensitive conductivity detector.

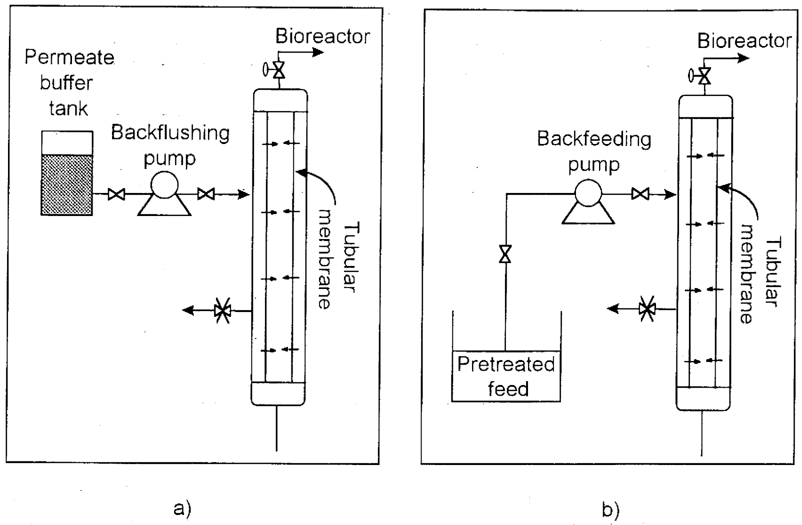

FIG. 2 Schematic diagrams of backflushing (a) and backfeeding (b) modes.

ICP analysis of the precipitate sample for Mg and P was also made on a Shimadzu ICPQ-1000 spectrometer. The sample was prepared by dissolving 5.83 mg of the ignited precipitate in a solution acidified by the addition of 1 mL HNO_3 and 5 mL HCl, and diluting to 50 mL with ultrapure water.

The methane gas content in the biogas produced during the anaerobic decomposition of organic matter was analyzed by a gas chromatograph (HP 5890 Series II model, Hewlett-Packard, USA) equipped with thermal conductivity detector (TCD). The cross sections of the used membrane were examined without further treatment by using a scanning electron microscope (JSM-35 model, Jeol Co., Japan).

RESULTS AND DISCUSSION

Digester Performance

During the long-term operation of the MCAB system, digester performance was tested at two different organic loading rates: 2 and 7 kg COD_{Cr}/m^3/day. The results of the digester performances are shown in Table 2. With the increase of the feeding rate, the pH of the digester broth decreased from 7.7 to 7.3 due to the larger production of volatile fatty acids at the higher organic loading rate. However, COD removal was quite stable but changed from 95 to 90%. The methane contents in the biogas were 58–62%.

TABLE 2
Performance of MCAB System According to the Organic Loading Rate

Parameters	COD loading rates 2 kg COD_{Cr}/m^3/day	7 kg COD_{Cr}/m^3/day
pH	~7.7	~7.3
In digester:		
MLSS (mg/L)	~2,800	~5,000
VFA (mg/L)	~400	~2,000
COD (mg/L)	<2,000	~4,400
COD removal	>95%	~90%
Methane contents (%)	58–62%	

In both cases the total population of anaerobic microbes (MLSS) decreased gradually from the outset but increased again after the acclimation period. Finally, even without any withdrawal of excess cells, it leveled off at 2800 or 5000 mg/L according to COD loading rates. The mechanical shear stress generated by the high-speed vane-type pump was considered to be the cause of the lysis of the microorganisms. The growth and death rates of anaerobic microbes became steady at each organic loading rate.

Membrane Performance

Figure 3 shows the flux decline curves during the operation of the MCAB system. Reduced flux normalized by the initial flux was used to compare the experimental results because the initial fluxes of each operation were slightly different. As shown in Fig. 3, the fluxes declined with time whether periodic backflushing with permeate was carried out or not, except at the initial stage for example, the initial flux of 190 L/m^2/h decreased to 58 L/m^2/h without backflushing after 10 days of operation. Backflushing turned out to be slightly effective only at the initial stage, which led to a further investigation of flux recovery by backflushing as described in the following section.

Effects of Physical Cleaning

Backflushing is known to be one of effective ways for flux recovery in the microfiltration of microorganisms. Some cake layer on the membrane surface or particle in the membrane pore can be removed by reversing the permeate flow. In this experiment, backflushing was performed for 30 seconds every 30 minutes under 1 bar. The flux was recovered immediately, almost to the ini-

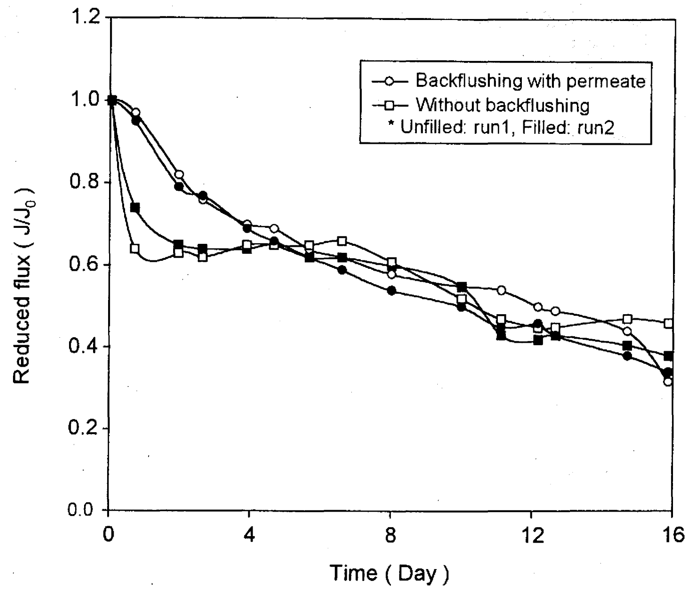

FIG. 3 Flux decline with and without backflushing. The initial flux, J_0, is 190–230 L/m^2/h.

tial value, by backflushing with permeate at the initial step as shown in Fig. 4. However, backflushing effects gradually decreased with time and were significantly decreased after 7 days. Almost no flux recovery was observed after 14 days. Other physical cleaning methods such as lumen flushing, flow stopping, and depressurization showed similar trends.

It is worth noting that only a negligible microbial cake layer was observed on the surface of the membrane after 14 days in spite of a drastic decrease in flux. Figure 5 are scanning electron micrographs (SEM) of the cross section of the fouled inorganic membrane used for 14 days. The zirconium oxide skin layer and the carbon layer are shown in white and black, respectively, in Fig. 5(a). Instead of a microbial cake layer, some irregular and sharp-shaped precipitates were observed in the cross-section micrograph of the membrane surface. This led to a further investigation of unusual membrane fouling in an MCAB system using an inorganic ceramic membrane.

Membrane Fouling by Inorganic Precipitate

During the operation of an MCAB system, white crystals were observed on the inner wall of the transparent permeate tube after 1 day's operation. They

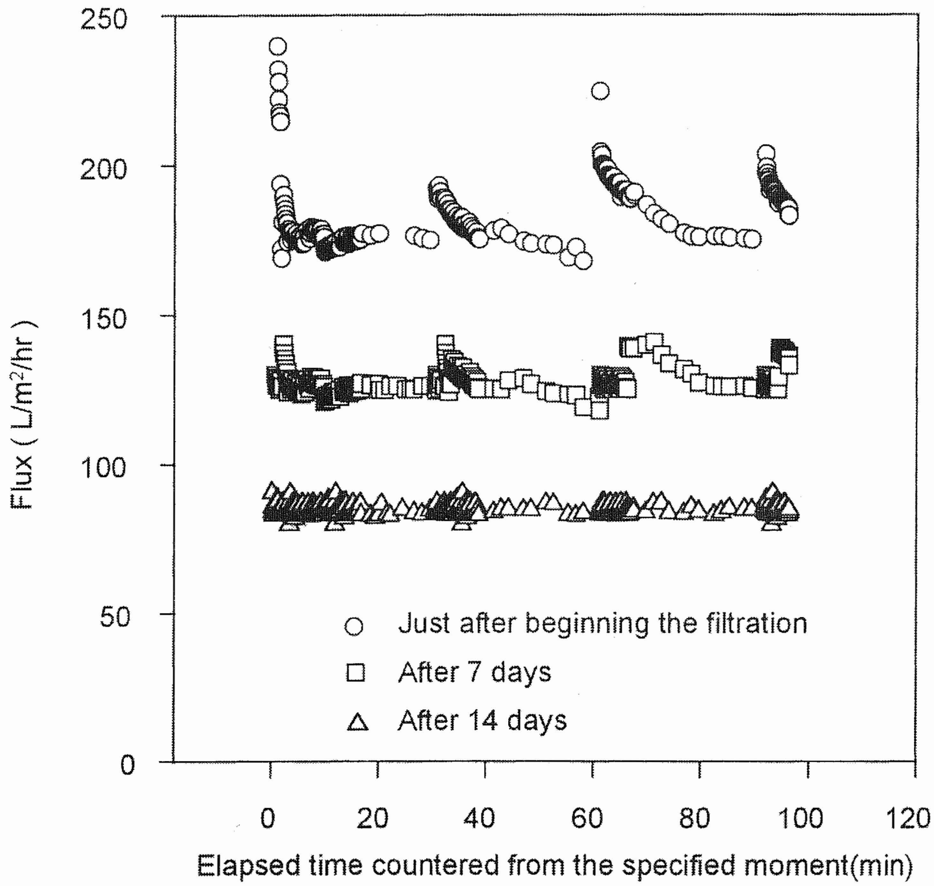

FIG. 4 Backflushing effects with time. The three flux curves correspond to the flux for a short period just after the indicated operation time.

became larger and numerous over time. When the membrane module was disassembled following long-term operation, a large quantity of white crystals were also found at the shell side of the inorganic membrane unit.

The shell side white crystals were scratched off of the used membrane and analyzed by ion chromatography. They were dried at room temperature or ignited at 550°C, dissolved into a slightly acidic solution (HNO_3), and the concentrations of Mg^{2+}, NH_4^+, and PO_4^{3-} were measured, respectively. The results are given in Table 3. It can be seen that the composition closely corresponds to that of struvite. It was further identified as struvite through examinations of the XPS and IR spectra of the white crystal.

We believe that the struvite crystals were formed inside the inorganic membrane pores during filtration of the digester broth, and thus they blocked the pores which played the major role in the flux decline. This is the reason why the flux declined by 60% of the initial flux even though the accumulation of microbial flocs was negligible on the surface of the membrane.

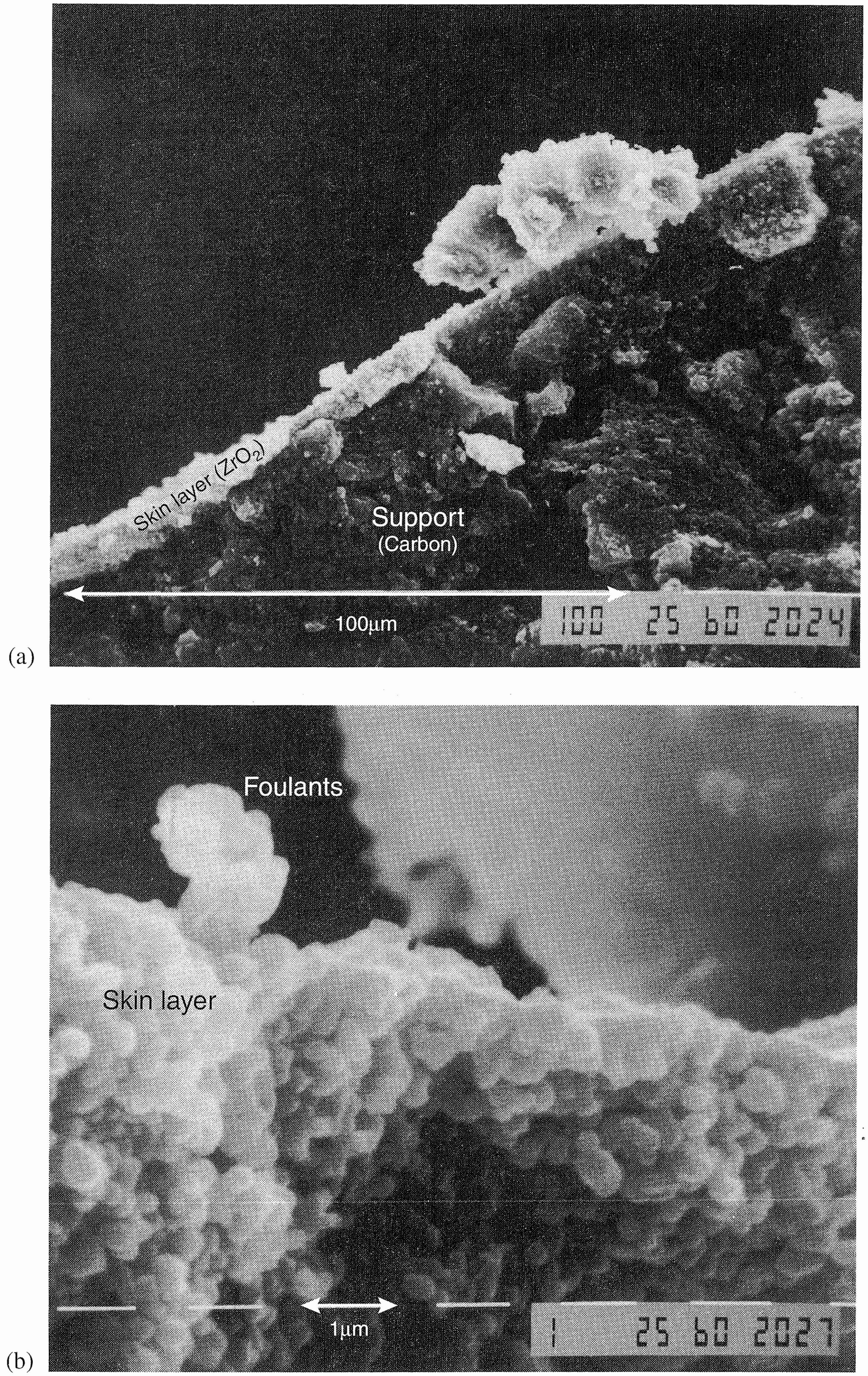

FIG. 5 Scanning electron micrographs of the cross section of the inorganic membrane used for 14 days in the normal mode with backflushing: (a) 2,000×, (b) 10,000×.

TABLE 3
IC and ICP Analyses of the Inorganic Precipitate

	Dried at room temperature			Ignited at 550°C	
Ion	Actual value (mg/L)	Theoretical value[a] (mg/L)	Element	Actual value (%)	Theoretical value[b] (%)
Mg^{2+}	48.1	54.0	Mg	21.9	21.9
NH_4^+	38.3	40.1			
PO_4^{3-}	183.9	210.9	P	27.6	27.8
Fe^{3+}	2.3	—			

[a] Calculated based on the MW of $MgNH_4PO_4 \cdot 6H_2O$ (109 mg crystals dissolved in 200 mL acidic solution).
[b] Calculated based on the MW of $Mg_2P_2O_7$ (5.83 mg ignited crystals dissolved in 50 mL acidic solution).

Quantitative Calculation of Precipitated Amount

Although the formation of struvite is well known in other biological waste treatments (17, 18), quantitative analysis has rarely been attempted in MCAB systems because it involves complex biological reactions. In order to estimate the amount of precipitated struvite and to elucidate struvite formation for alcohol distillery wastewater, a pH–solubility diagram for struvite was constructed as shown in Fig. 6 (19, 20).

Several elemental reactions of struvite formation can be written as follows. The constant for each reaction at 55°C is also given.

$$MgNH_4PO_4 = Mg^{2+} + NH_4^+ + PO_4^{3-}; \qquad \log K_{sp} = -11.7 \quad (1a)$$

$$NH_4^+ = NH_3\,(aq) + H^+; \qquad \log K_a = -8.4 \quad (1b)$$

$$H_3PO_4 = H_2PO_4^- + H^+; \qquad \log K_{a,1} = -2.23 \quad (1c)$$

$$H_2PO_4^- = HPO_4^{2-} + H^+; \qquad \log K_{a,2} = -7.13 \quad (1d)$$

$$HPO_4^{2-} = PO_4^{3-} + H^+; \qquad \log K_{a,3} = -12.06 \quad (1e)$$

$$Mg^{2+} + H_2O = Mg(OH)^+; \qquad \log K_{d,1} = -10.4 \quad (1f)$$

The ionization constant, α, for each ion is defined as the ratio of the free ion

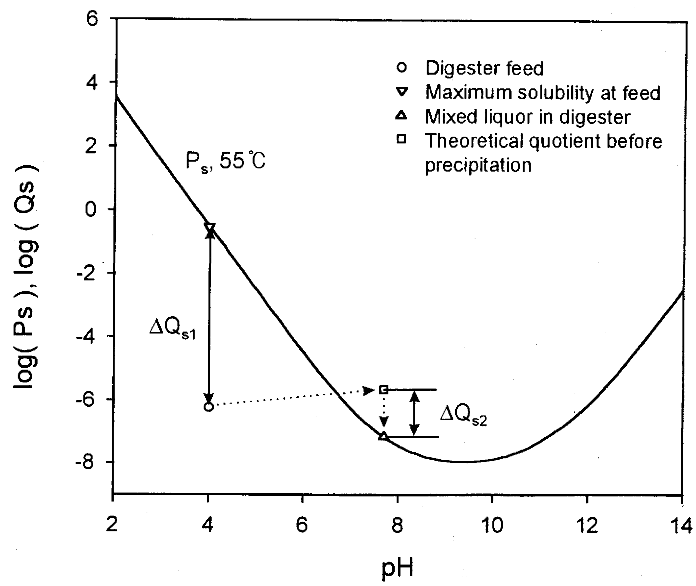

FIG. 6 Conditional solubility product (P_s) and reaction quotient (Q_s) for struvite as a function of pH. ΔQ_{s1}: additional solubility in the digester feed, ΔQ_{s2}: struvite formation potential in the digester as a result of increased ion concentrations and decreased conditional solubility product.

concentration and the total ion concentration:

$$\alpha_{Mg} = [Mg^{2+}]/C_{T,Mg} \tag{2a}$$

$$\alpha_{NH_4^+} = [NH_4^+]/C_{T,NH_4^+} \tag{2b}$$

$$\alpha_{PO_4^{3-}} = [PO_4^{3-}]/C_{T,PO_4^{3-}} \tag{2c}$$

The solubility product, K_{sp}, is

$$K_{sp} = (\alpha_{Mg^{2+}} \cdot C_{T,Mg^{2+}})\,(\alpha_{NH_4^+} \cdot C_{NH_4^+})\,(\alpha_{PO_4^{3-}} \cdot C_{T,PO_4^{3-}}) \tag{3}$$

From the above equations, the conditional solubility product, P_s, can be defined by

$$P_s = C_{T,Mg^{2+}} \cdot C_{T,NH_4^+} \cdot C_{T,PO_4^{3-}} = \frac{K_{sp}}{\alpha_{Mg} \cdot \alpha_{NH_4} \cdot \alpha_{PO_4}} \tag{4}$$

Finally, the equation which describes the pH dependence of the conditional solubility product was obtained:

$$\log P_s = -11.7 + \log(1 + 10^{-10.4 + \mathrm{pH}}) + \log(1 + 10^{-8.4 + \mathrm{pH}}) + \log(1 + 10^{12.06 - \mathrm{pH}} + 10^{19.19 - 2\mathrm{pH}} + 10^{21.6 - 3\mathrm{pH}}) \quad (5)$$

The solid line in Fig. 6 shows the conditional solubility product (P_s) of struvite ($MgNH_4PO_4{\cdot}6H_2O$) as a function of pH, which corresponds to its saturation curve. Consequently, the upper part of the curve represents the supersaturation area and the lower part the under saturation area. The reaction quotient (Q_8) is defined by the product of the concentrations of ions making up struvite under certain conditions and is given by

$$Q_s = [Mg^{2+}]\,[NH_4^+]\,[PO_4^{3-}] \quad (6)$$

If the calculated reaction quotient, Q_s, is larger than the conditional solubility product, P_s, then struvite should be precipitated.

Struvite should not precipitate in the feed solution because the reaction quotient in acidic feed solution (pH $\cong$ 4) is much smaller than the conditional solubility product as shown in Fig. 6. In the digester, however, the reaction quotient would increase due to the increase of NH_4^+ and PO_4^{3-} concentrations due to biological degradation of organic compounds contained in the feed. As the degradation of biological organic acids increases the pH in the digester, the conditional solubility product decreases drastically as shown in Fig. 6. As a result of the decrease of the conditional solubility product together with the increase of the reaction quotient, struvite will precipitate by the amount ΔQ_{s2} during anaerobic digestion.

The amount of struvite precipitated per unit volume of feed solution, ΔQ_{s2}, can be calculated by comparing the magnesium ion concentrations in the feed and in the digester because most magnesium ions in the feed exist as free ions.

Table 4 shows the concentrations of each ion in the feed and in the digester broth. As expected, the concentration of soluble magnesium ion decreases with struvite precipitation in the digester broth. On the other hand, the concentrations of soluble ammonium and phosphate increase due to continuous biodegradation despite the precipitation. The molar amount of the struvite precipitated, ΔQ_{s2}, should be equivalent to the precipitated amount of Mg^{2+}. The hypothetical molar concentrations of the ammonium and phosphate ions before precipitation can be deduced by adding the molar concentration of the precipitated magnesium ion to each ion concentration in the digester broth. Based on this procedure, the amount of struvite precipitated was evaluated to be about 2 g/L of digester feed.

TABLE 4
Q_s and P_s for Struvite in the Digester Feed and Digestion Broth

Elements	Digester feed ($\times 10^{-3}$ mol/L)	Digester broth ($\times 10^{-3}$ mol/L)	Before precipitation ($\times 10^{-3}$ mol/L)	Amount of precipitate ($\times 10^{-3}$ mol/L)
$[Mg^{2+}]$	14.9	0.122	14.9	14.9–0.122
$[NH_4^+]$	5.37	65.1	65.1 + (14.9 − 0.122)	14.9–0.122
$[PO_4^{3-}]$	7.55	8.43	8.43 + (14.9 − 0.122)	14.9–0.122
$\log Q_s$	−6.22	−7.17	−4.56	0.0148 mol/L × 135.3
$\log P_s$	1.01 at pH 4.0	−7.15 at pH 7.7	−5.64 at pH 7.7	g/mol = 2 g/L

Flux Improvement through a Backfeeding Mode

Inorganic membrane fouling was mainly caused by pore blocking caused by struvite formation. The blocked pores were not cleared by backflushing. A particle-free, acidic digester feed solution was considered an effective liquid to prevent struvite precipitation or to remove struvite deposited in membrane pores because its acidic character (pH $\cong$ 4) could raise the P_s for the struvite inside the pore by ΔQ_{sl}, as indicated in Fig. 6.

In this context, a new operating mode for an MCAB system was anticipated. A "backfeeding" mode was carried out by feeding the acidic digester feed through the shell side of a tubular inorganic membrane, via the lumen, to the digestion reactor. The backfeeding was done intermittently for 1–2 minutes every 2 hours of membrane filtration in order to maintain a constant organic loading rate of 3–3.5 kg COD_{Cr}/m^3/day.

As shown in Fig. 7, the backfeeding mode gave rise to a much higher flux compared with the normal mode, at least for 6 days. After 6 days, however, the flux of the backfeeding mode returned to the same level as that with the normal mode. After 10 days the flux of the backfeeding mode decreased to below the level of the normal mode.

We believe precipitation of struvite inside the pores is suppressed by the backfeeding mode during the initial period of MCAB operation. Struvite precipitation, however, is not completely prevented, so struvite gradually accumulates with time inside the pores would be. As a result, pores decrease in size and the flux decreases because the permeate flow rate is proportional to 4th power of pore size under constant pressure.

Pure acid solutions such as HCl and HNO_3 were also used but their effects were negligible. Because a pure acid solution does not contain any buffer, it is more easily neutralized than a feed solution immediately after mixing with the remaining digester broth in the shell side of membrane. Therefore it quickly lost its ability to dissolve struvite inside the pores.

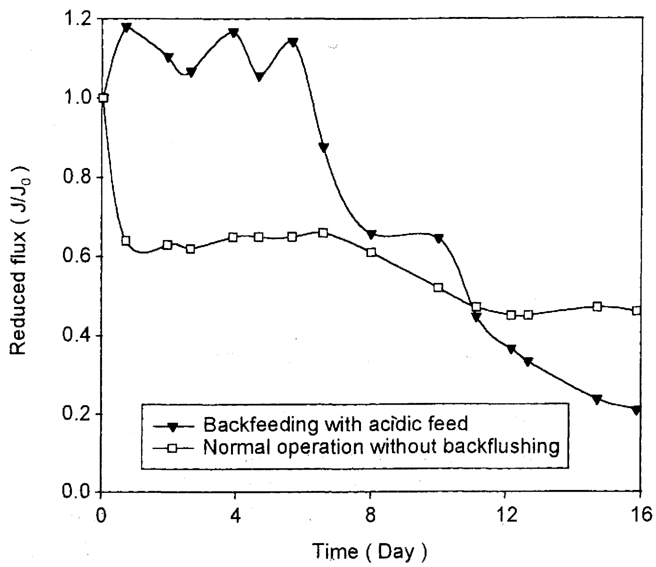

FIG. 7 Flux enhancement by a backfeeding mode with acidic feed wastewater. The backfeeding was performed for 1–2 minutes every 2 hours.

In summary, the backfeeding mode is an effective method to enhance the flux in the MCAB system if it is occasionally accompanied by membrane cleaning with acid and alkaline solutions.

CONCLUSIONS

In this study the fouling mechanism of an inorganic membrane was studied in the membrane-coupled anaerobic bioreactor (MCAB) system for alcohol distillery wastewater. The following conclusions could be drawn.

(1) Almost no microbial cake layer was deposited on the surface of the used inorganic membrane, which was identified by SEM. The white crystals collected from the shell side of the membrane were identified to be struvite ($MgNH_4PO_4 \cdot 6H_2O$). Struvite precipitates formed inside the membrane pores and played the major role in membrane fouling and thus in flux decline.

(2) The amount of struvite formed during anaerobic digestion was evaluated by using a pH–solubility product diagram. About 2 g struvite crystals was calculated to form from 1 L alcohol distillery wastewater.

(3) A "backfeeding" mode with acidic digester feed was devised as a new approach to suppress struvite formation inside membrane pores. It was

possible to maintain the initial flux effectively for at least 6 days, but it then declined rapidly. This suggests that an appropriate combination of backfeeding and cleaning may maintain greater flux.

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