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Structural Analysis and Dewatering Characteristics of Waste Sludge from WWTP MBR

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Abstract: A pilot-scale UF membrane bioreactor (MBR) of 1 m^3/day capacity was set up in an industrial wastewater treatment plant to evaluate its performance. This study mainly focused on testing the dewaterability and structural analysis of MBR sludge. MBR had 14% reduction of excess sludge production in relative to the conventional activated sludge process (CAS sludge). For dewatering, MBR sludge had comparable dewaterability with the CAS sludge but required nearly 20% less flocculant to reach the highest filterability χ and lowest specific filtration resistance (SRF). This could reduce the cost for running the dewatering facilities and final disposal. Meanwhile the chemical and morphological analyses on MBR sludge exhibited lower EPS (exocellular polymeric substances) content, slightly smaller flocs and more compact morphology. Additionally, to estimate the appropriate polyelectrolyte dose prior to dewatering, we measured the hysteresis loop area of the sludge rheogram (shear stress vs. shear rate) using a co-axial cylinder viscometer. For both sludges, the area dramatically increased at some critical flocculant dosage and then plateaued off. The critical dosage, though not optimal, still led to an acceptable dewatering performance for the sludge.

Keywords: Membrane bioreactor, sludge dewaterability, floc structure, rheogram, microtome slicing, fractal dimension

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INTRODUCTION

The membrane bioreactor (MBR), combined with conventional activated sludge process and membrane filtration, has been proposed as a new wastewater treatment unit. The membrane is submerged in the aeration tank and retains sludge biomass in the reactor. Solid-liquid separation is effectively achieved without the secondary settler. After filtration by microfiltration or ultrafiltration membrane, satisfactory effluent quality can be obtained, including the removal of SS, COD, and pathogens (2). Sludge reduction is the other advantage of MBR. Long sludge age (mean cell retention time) in the bioreactor enhances endogenous respiration and is favorable to the growth of high trophic level organisms (protozoa and metazoan). Both factors will result in the reduction of excess sludge (3).

On the other hand, when comparing the sludge dewaterability wasted from conventional activated sludge process (denoted as "CAS sludge") and MBR system ("MBR sludge") tested in parallel, diverged results were reported in literature. Many studies showed that the dewaterability of the MBR sludge became worse than the CAS sludge mainly because the long sludge retention time in MBR led to high EPS in the bioreactor and increased the filtrate viscosity accordingly (4, 5). Crossflow to mitigate membrane fouling and the enhanced aeration also caused strong turbulence and disrupted the sludge flocs. The fraction of small flocs and the amount of dispersed microorganisms increased in supernatant accordingly and deteriorated the dewaterability of raw sludge (6–9). Some reports, however, observed different dewatering results. Murakami et al. observed no significant difference of the dewaterability between the MBR sludge and the CAS sludge at the CST test (10). Bouhabila et al. noticed that increasing the sludge age in MBR would improve the wasted sludge dewaterability accordingly (11). Holbrook et al. found that the MBR sludge would have better dewaterability and less conditioner requirement than those of CAS sludge if both were digested (either aerobically or anaerobically) followed by conditioning (either $FeCl_3$ or polymer) (9). Merlo et al. reported that lower CST values were measured for the MBR sludge than the CAS sludge. The authors stated that the dewaterability improvement was possibly because of the lower EPS content in MBR sludge, though higher soluble microbial products and colloidal material were measured in their test (12).

Summarizing the preceding studies, several factors may influence the MBR sludge dewaterability, such as the source of wastewater (synthetic, municipal, or industrial), organic loading, sludge age, and aeration intensity. These factors influence the amount of EPS, soluble microbial product (SMP) and sludge floc size, and lead to the deterioration of MBR sludge dewaterability if smaller floc size and higher EPS/SMP was found. On the other hand, the detailed structure in sludge floc of MBR sludge may also be changed and different from the CAS sludge, and may also be one factor to control the MBR sludge dewaterability (13). In this study, we sampled the

MBR sludge and the CAS sludge for conditioning and dewatering tests. The morphological properties, such as floc size, EPS, rheological behavior, and floc structure were measured. These measurements would help reveal the structural difference of the two sludges, and provide more advanced information for evaluating the dewatering performance of sludge.

EXPERIMENTAL

MBR Pilot Plant

The MBR pilot plant with a capacity of 1 m^3/day was installed in the unified wastewater treatment plant (WWTP) of an industrial park in northern Taiwan. As depicted in Fig. 1, the pilot plant included an influent tank (250 L), an ultrafiltration membrane tank (250 L, PES hollow fiber membrane with pore size 0.036 μm and surface area 0.93 m^2), a backwash tank (30 L), and a chemical tank (15 L, filled with sodium hypochlorite solution). The influent of MBR was taken from the buffer tank after the primary treatment of WWTP using a submersible pump (Pump A in Fig. 1) and then stored in the influent tank. A circulating pump (Pump B) drew the wastewater from the influent tank to the membrane tank (Figure 1). The CAS sludge was used as the seeds for the MBR. Two aeration diffusers were set in the membrane tank: one providing sufficient dissolved oxygen for bioreaction

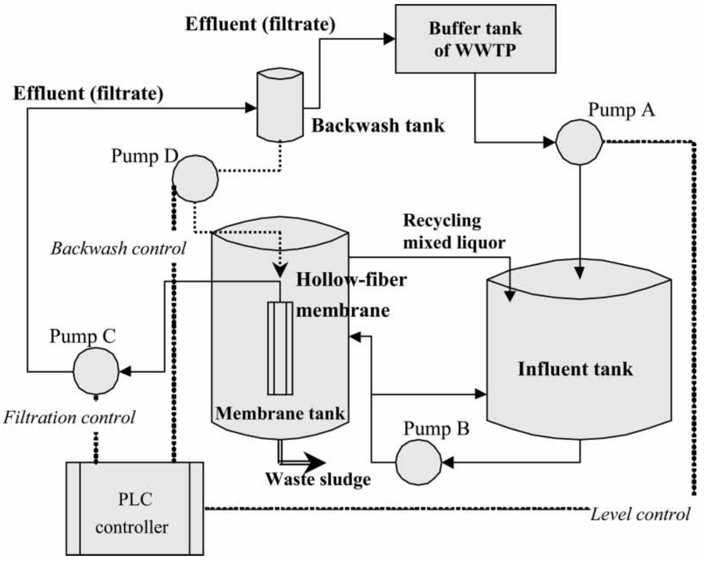

Figure 1. MBR pilot plant, (a) the flow chart (Pump A: influent; Pump B: feeding to the membrane tank and circulating; Pump C: filtrate suction; Pump D: backwash).

Figure 2. Photos of the MBR pilot plant (from left to right: PLC controller, membrane tank and influent tank).

with tiny bubbles, and the other providing large bubbles for scrubbing and shaking the membrane to mitigate fouling. An effluent pump (Pump C) sucked the filtrate from the membrane, and some filtrate was then stored in the backwash tank for membrane cleaning. The backwash was programmed by the PLC controller and was set to apply for every 15 minutes with a dose of 10 ppm NaClO (Pump D). Pressure sensors and flow meters were also installed to monitor the membrane performance. Figure 2 shows the appearance of the MBR pilot plant.

Sludge Characteristics

MBR sludge was sampled from the sludge wasting pipes in the bottom of the membrane tank (MBR sludge). The suspended solid (SS) was measured to be 6,570 mg/L. We took the microphotographs (Microscope CX41, Olympus) of the sludge flocs in the initial stage and the mature stage after acclimation (demonstrated in Fig. 3), respectively. In the initial stage, the sludge flocs were of $100 \sim 200\ \mu m$ in size with open and loose structures (Fig. 3a), similar to the seeding biomass of the CAS sludge. In the mature stage, the sludge flocs became smaller (less than $100\ \mu m$) and more compact (Fig. 3b). A quantitative structural analysis is given later.

For the sake of comparison, the excess waste activated sludge was also sampled from the wastewater treatment plant (CAS sludge). The suspended solid was 3,580 mg/L. After sampling, both sludges were stored at 4°C and gravitationally thickened to increase the SS value to 10,000 mg/L for conducting subsequent tests. The volume average floc size $d_f[4,3]$ was

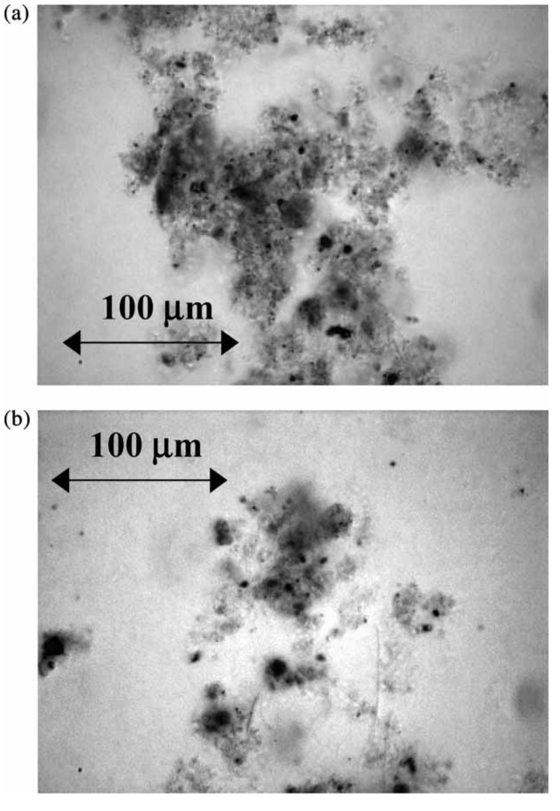

Figure 3. The microphotographs of sludge flocs, (a) initial stage; (b) mature stage.

measured using light scattering techniques (LS230, Coulter). The measurement of EPS in sludge flocs followed the mild heat extraction method suggested by Foster et al. (14). Capillary suction time (CST) and specific resistance of filtration (SRF) were measured to evaluate the sludge dewaterability. Experimental details can be found in the studies of Vesilind (15) and Christensen et al. (16). The CST is converted to filterability χ according to the method of Vesilind by considering the effects of filtrate viscosity and suspended solids content (15). Cationic polyelectrolyte flocculant T3052 (cationic polyacrylamide, with $MW = 10^7$ and charge density = 2.27 meq/g) was used for the sludge conditioning.

Rheological Tests

The rheogram of the sludge was reported as a possible index for evaluating the sludge dewatering efficiency because it can evaluate the response of sludge subjected to physical shearing (1). In this study, we selected a programmable rotational rheometer (Brookfield model RV-III+, USA). Model RV can measure a higher torque, and also can record the shear stress variation more accurately when measuring the rheogram of sludge with large flocs. A spindle of 25.2 mm diameter and 90.9 mm length is used to construct the rheogram curves (shear stress $\tau_{r\theta}$ vs. shear rate $\dot{\gamma}$). The inside diameter of the Rheometer cell (fluid container) was 27.6 mm. The rotational speed increased linearly from 0 to 120 s^{-1} in 6 minutes (step 1), and then decreased linearly from 120 s^{-1} down to 0 in 6 minutes (step 2). The rheogram produced by steps 1 and 2 forms a hysteresis loop due to the thixotropic behavior of the sludge. A typical plot is demonstrated in Fig. 4. The area of the loop is calculated and denoted as A ($\mathrm{nt/m^{-2} \cdot s^{-1}}$).

Floc Structure Analysis

To further reveal the sludge floc structure, a drop of sludge sample was embedded in the paraffin media and then sliced to thin layers for microscopic observation. The experimental details of the microtome slicing are given in Chu and Lee (17). The slicing microphotograph was obtained using a microscope (Microscope CX41, Olympus) mounted with a digital camera for further image analysis, following the algorithms suggested by Chu and Lee (18). The porosity ε (using iterative method), volume average pore size $d_p[4,3]$ (using maximum convex-perimeter method, MCPM) and 2-D box-counting fractal dimension $D_{P,2}$ (also using MCPM) are then obtained. In other words, a two-dimensional surface with smooth perimeters has a $D_{P,2}$ of 2. The more

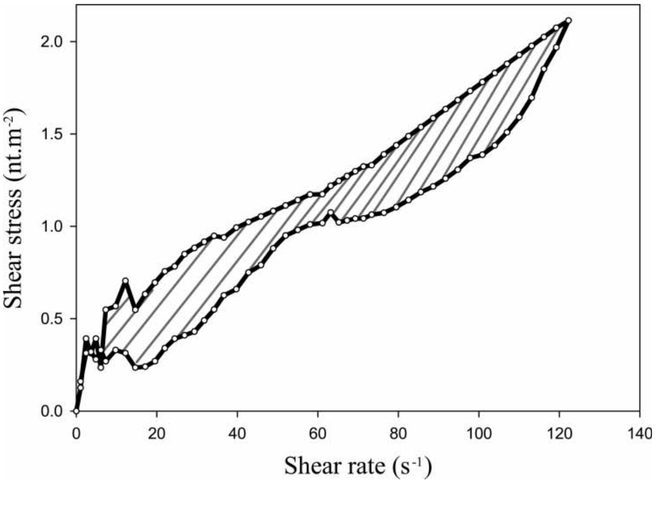

Figure 4. An example of hysteresis loop of sludge rheogram curves. The area in the loop is denoted as A.

rugged the perimeters are, the lower the $D_{P,2}$ will be (generally between 1 and 2), which indicated more significant fractal behavior.

RESULTS AND DISCUSSION

Wastewater Treatment Efficiency

An MBR pilot test was conducted during March 2005 to January 2006. After the initial phase acclimation (the first two months), the MLSS (mixed liquor suspended solids) in the reactor was stably kept to 5,000~6,000 mg/L. This value is higher than the MLSS of the conventional activated sludge process (2,000~2,500 mg/L) but is lower than the generally suggested MLSS of MBR (8,000~15,000 mg/L). Due to the low MLSS, it still requires a secondary settler after the MBR to increase the solids content of sludge for subsequent dewatering. One possible reason is that the low biodegradability of the organics in industrial wastewater might be unfavorable to the growth of microorganisms. The other reason is that strong aeration caused foaming in the reactor, which resulted in an unexpected overflow of sludge biomass. Though performed at a relatively low MLSS, the system still maintained reasonable treating efficiency. The COD of the influent fluctuated largely from 62 mg/L to 505 mg/L (average 157 mg/L). The effluent COD, however, was stable and kept to 30 ~ 40 mg/L. Since the pore size of UF is 0.036 μm, all suspended solids in the filtrate can be removed theoretically. The SS of the effluent ranges from ND (non-detected) to 5 mg/L. The tiny amount of suspended solids in the filtrate may come from the atmosphere or the impurity in NaClO solutions for membrane backwash. In the mature stage, the membrane flux is $0.5\ m^3 \cdot m^{-2} \cdot d^{-1}$, hydraulic retention time is 8.1 hours, and F/M ratio is $0.35\ g\ COD \cdot (g^{-1}\ VSS) \cdot d^{-1}$.

Noticeably, due to the low MLSS in the bioreactor, the excess sludge was not wasted periodically. To maintain the bioactivity, the oxygen uptake rate (OUR) of sludge biomass was measured using the respiratory meter. If the OUR was low ($0.05\ mg \cdot L^{-1} \cdot min^{-1}$, say), 10% of mixed liquor (25 liters) would be discharged manually to remove the aged sludge with low bioactivity. According to our evaluation, the wasted excess sludge produced from the MBR system (0.064 kg SS/m^3 wastewater) is less than that of the conventional activated sludge system (0.055 kg SS/m^3 wastewater, sludge age 6 days), where a 14% sludge reduction can be achieved.

Sludge Dewatering and Morphological Parameters

The results of the sludge dewaterability are demonstrated in Fig. 5. The sludge after polymer conditioning exhibited improved dewaterability (Figs 5a and 5b). For both sludges, an optimal dewaterability (highest χ and lowest SRF) could be found at some critical dose, though the two

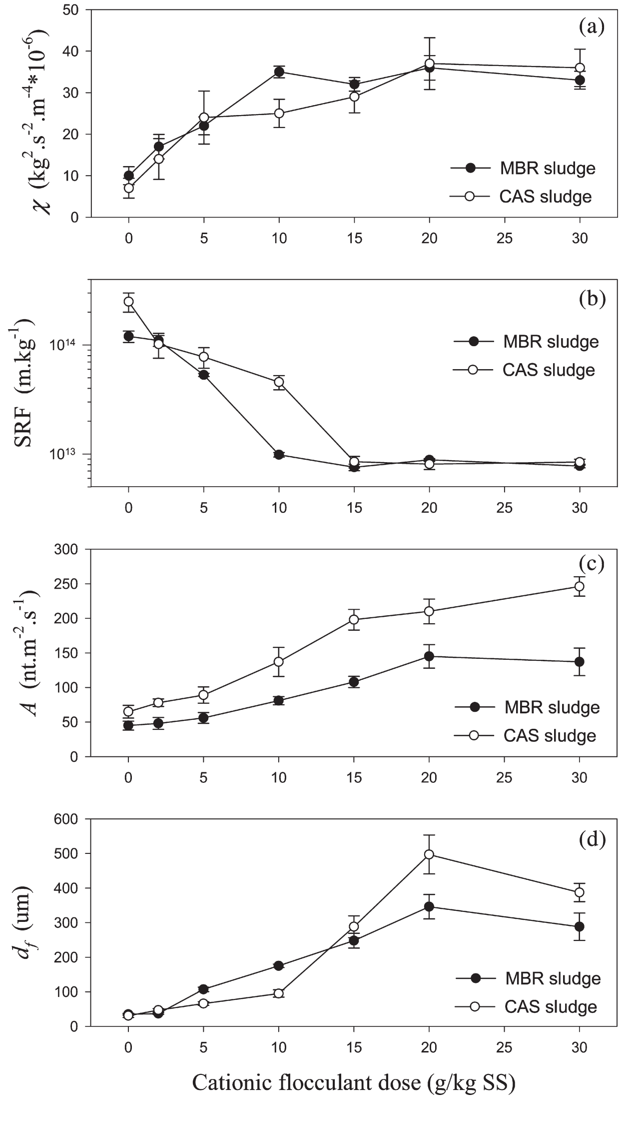

Figure 5. The sludge characteristics of sludge after polymer conditioning, (a) filterability; (b) SRF; (c) floc size; (d) the area of the hysteresis loop of sludge rheogram.

corresponding doses are not identical due to their distinct dewatering mechanism. Though charge neutralization is commonly proposed as an important factor for flocculant conditioning, the zeta potential remained negative for all of the conditioned sludge in this case and showed no correlation with the dewaterability (data not shown here). Comparing the two sludges, the MBR sludge has a comparable dewatering performance with

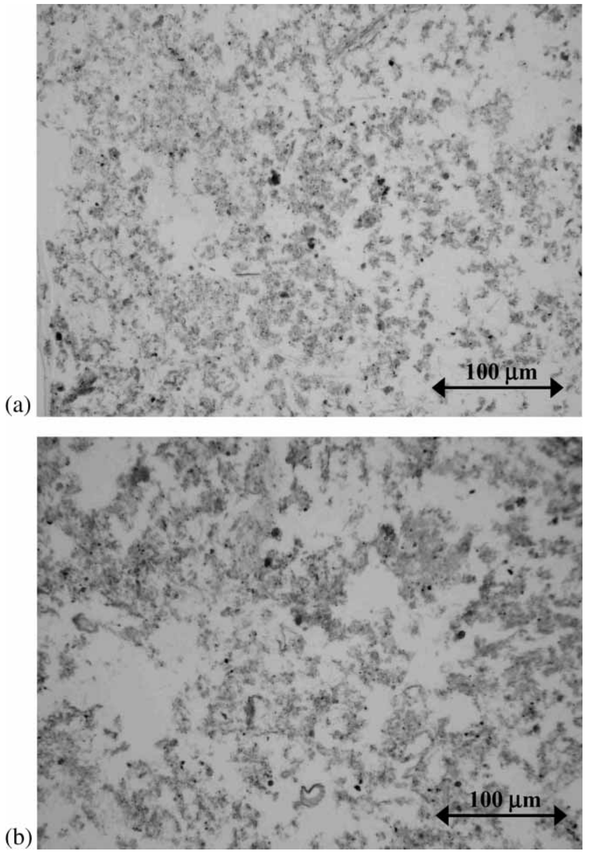

Figure 6. The paraffin microtome slicing photographs of the sludge, (a) MBR sludge; (b) CAS sludge.

the CAS sludge. However, for reaching the highest χ or the lowest SRF value, the MBR sludge requires cationic flocculants at dose 10∼15 g/kg SS, where CAS sludge requires 15∼20 g/kg SS. We may notice that the aforementioned benefits of MBR help reduce the operation/maintenance cost of sludge management because it requires less flocculant (approximately 20% lower), and lower capacity of dewatering facilities since less sludge is produced in the MBR system than the conventional activated sludge process (14% lower).

With regard to the rheological properties of sludge, all sludge has similar patterns on their rheogram curves as demonstrated in Fig. 4 and thus were not shown here for the sake of brevity. For both sludges, the loop area *A* reached a maximum at some critical dose (20 g/kg SS) and then kept a plateau until the highest applied dose (Fig. 5c). When comparing with the optimal dose for sludge dewatering (10∼15 g/kg SS for MBR sludge and 15∼20 g/kg SS for CAS sludge), the dose leading to the maximum A is obviously higher. That is, if the optimal dose for dewatering is estimated using the rheogram method, it may result in overestimation. Though these critical doses did not match well, the results indicated that when A reached the maximum, the sludge had an acceptable dewaterability. This method is still applicable for estimating the sludge dewatering performance, either for the MBR or the CAS sludge. Noticeably, the MBR sludge flocculated at higher dose had a lower A value than the CAS sludge. This is probably due to the smaller floc size of MBR sludge (Fig. 5d). Restated, the filterability, SRF, and the rheological method are all laboratory tests. A more reliable evaluation should be based on the full-scale operation of the dewatering equipment, where the authors are now conducting the next-stage study.

Figure 6 depict the paraffin-embedded microtome slicing photographs. The results of image analysis and the other floc properties are summarized in Table 1. When comparing the floc structure of MBR sludge and CAS sludge, the former has a smaller floc size (45 μm), less EPS content (3.3 mg/g SS), and more compact floc structure, including the lower porosity ε (0.68), the smaller pore size $d_p[4,3]$ (8.74 μm), and the higher $D_{p,2}$ (1.79, indicating a less rugged surface). Unlike most studies on treating municipal wastewater by MBR, the decreased EPS found in this study might be resulted from the low biodegradability of the industrial wastewater. A more solid and sphere-like floc would be favorable for forming a rigid cake structure for efficient dewatering. This correlated well with the better dewatering performance of MBR sludge in Figs. 5a and 5b. In MBR, the longer sludge age (more than 30 days) and extended aeration probably lead to the partial decomposition and reconstruction of the flocs. In the mature stage, the sludge with compact floc structure would be achieved.

Hypothetical Tests

Hypothetical tests were conducted for comparing the preceding parameters of the MBR sludge and the CAS sludge depicted in Fig. 5 and Table 1,

Table 1. Summary of floc properties and structure analysis results

	EPS (mg/g SS)	$d_f[4,3]$ (μm)	ε (−)	$d_p[4,3]$ (μm)	$D_{p,2}$ (−)
Waste sludge of MBR	3.3 ± 1.56	45 ± 6.5	0.679 ± 0.025	8.74 ± 2.15	1.79 ± 0.03
Waste activated sludge of WWTP	18.6 ± 2.80	65 ± 9.2	0.712 ± 0.027	13.1 ± 3.82	1.64 ± 0.01

respectively. The hypotheses, $\vec{X}_M > \vec{X}_C$ (some property on average of MBR sludge is larger than that of CAS sludge) or $\vec{X}_C > \vec{X}_M$ (some property on average of CAS sludge is larger than that of MBR sludge) were tested, which could be accepted at a confidence level of $1 - \alpha$ if

$$H_> = \frac{\bar{X}_M - \bar{X}_C}{t_{1-\alpha} S_p \left(\frac{1}{n_M} + \frac{1}{n_C}\right)^{1/2}} > 1$$

or

$$H_> = \frac{\bar{X}_C - \bar{X}_M}{t_{1-\alpha} S_p (1/n_M + 1/n_C)^{1/2}} > 1$$

respectively (19). $\bar{\mathrm{X}}$, s, and n are the mean value, standard deviation, and data number, respectively, while $t_{1-\alpha}$ were the corresponding value of t-distribution in $1 - \alpha$ with the degree of freedom ($n_M + n_C$-2), and

$$S_p = \left[\frac{(n_M - 1)S_M^2 - (n_C - 1)S_C^2}{n_M + n_C - 2}\right]^{1/2}$$

In this study, every measurement was repeated for three times, and the degree of freedom would be four ($n_M = n_C = 3$).

The hypothetical test in Table 2 reveals that at a confidence of interval of 90% ($\alpha = 0.1$), the filterability of the MBR sludge was not apparently higher than that of CAS sludge since $H_>$ ranged from -0.336 to 3.069 at different flocculant doses. Similarly, at the same confidence of interval, the hypothesis that MBR sludge has lower SRF and A values than those of CAS sludge was not statistically meaningful since $H_>$ was not always larger than 1. This implies that the MBR sludge has comparable dewaterability to the CAS sludge before and after polymer flocculation. The finding in this study supported the results of Murakami et al. (10), and no serious deterioration of MBR sludge dewaterability was observed here.

For other morphological parameters, at a confidence level of 99%, the MBR sludge has lower particle size (Table 2) and less EPS content than those of CAS sludge (Table 3). Also $D_{p,2}$ (2-D box-counting fractal dimension) of the MBR sludge was higher than those of the CAS sludge (Table 3). On the other hand, the hypothesis that the porosity and the pore size of the MBR sludge was lower for the MBR sludge than those of the CAS sludge can be accepted at a confident level of 90% (Table 3), where larger data scattering was found in these morphological measurements using microtome slicing and image analysis.

Table 2. Hypothetical tests of the results in Figure 5

Dose (g/kg SS)	MBR sludge n_M	MBR sludge $\bar{X}_M$	MBR sludge s_M	CAS sludge n_C	CAS sludge $\bar{X}_C$	CAS sludge s_C	Hypothetical test
Filterability ($kg^2 \cdot s^{-2} \cdot m^{-4} * 10^{-6}$)							$H_{>, 90\%}$ ($\bar{X}_M > \bar{X}_C$)
0	3	10	2.2	3	7	2.4	1.052
5	3	17	2.9	3	14	4.9	0.591
10	3	22	2.2	3	24	6.4	−0.336
15	3	35	1.4	3	25	3.4	3.069
20	3	32	1.6	3	29	3.9	0.809
30	3	36	2.9	3	37	6.2	−0.164
SRF (10^{14} m/kg)							$H_{>, 90\%}$ ($\bar{X}_M > \bar{X}_C$)
0	3	1.209	0.143	3	2.504	0.499	2.83
5	3	1.104	0.127	3	1.020	0.262	−0.31
10	3	0.532	0.019	3	0.778	0.166	1.667
15	3	0.099	0.005	3	0.457	0.068	5.94
20	3	0.076	0.005	3	0.085	0.010	0.911
30	3	0.088	0.003	3	0.081	0.009	−0.901
A ($nt/m^2 \cdot s$)							$H_{>, 90\%}$ ($\bar{X}_M > \bar{X}_C$)
0	3	35	2.9	3	31	4.9	−0.788
5	3	37	2.2	3	47	4.5	2.265
10	3	107	6.5	3	66	5.2	−5.528
15	3	175	4.9	3	95	10.5	−7.795
20	3	248	21	3	288	31.6	1.186
30	3	346	35	3	497	56.1	2.574
d_f (μm)							$H_{>, 90\%}$ ($\bar{X}_M > \bar{X}_C$)
0	3	45	6.5	3	65	9.2	1.111
5	3	48	8.6	3	78	5.4	1.839
10	3	56	7.7	3	89	12	1.467
15	3	81	5.9	3	137	21	1.605
20	3	108	8.2	3	198	15	3.292
30	3	145	17	3	210	18	1.644

CONCLUSIONS

An MBR pilot plant equipped with hollow-fiber UF membrane was used to treat the industrial wastewater discharged from an industrial park in

Table 3. Hypothetical tests of the Results in Table 1

	MBR sludge			CAS sludge			Hypothetical test
	n_M	$\overline{X}_M$	s_M	n_C	$\overline{X}_C$	s_C	
EPS (g/kg SS)	3	3.3	1.56	3	18.3	2.80	$H_{>,99\%} = 2.159$ $(\overline{X}_C > \overline{X}_M)$
Porosity, ε (–)	3	0.679	0.025	3	0.712	0.027	$H_{>,90\%} = 1.026$ $(\overline{X}_C > \overline{X}_M)$
Pore size, $d_p[4,3]$ (μm)	3	8.74	2.15	3	13.1	3.82	$H_{>,90\%} = 1.134$ $(\overline{X}_C > \overline{X}_M)$
2-D box-counting fractal dimension, $D_{p,2}$ (–)	3	1.79	0.03	3	1.64	0.01	$H_{>,99\%} = 1.266$ $(\overline{X}_C > \overline{X}_M)$

Taiwan. When comparing with the behavior of CAS sludge, the MBR sludge exhibited comparable dewaterability but required less flocculant to achieve the highest χ and the lowest SRF. For the floc structure, the MBR sludge has less EPS content, smaller floc size, lower porosity, smaller pore size, and higher fractal dimension $D_{P,2}$ than those of the CAS sludge. The porous floc structure might be partially decomposed and reconstructed to form a relatively compact one in the environment of long sludge retention time and extended aeration of the MBR system. The low EPS in the MBR sludge, however, might be resulted from the low biodegradability of the industrial wastewater and poor microorganism growth in the piloting test. This conversion is favorable to reduce the overall cost of the sludge solid-liquid separation. Further investigation is required to reveal the control strategy of MBR for achieving the compact floc structure.

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