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4 **Anaerobic digestibility of up-concentrated organic matter obtained from direct**  
5 **membrane filtration of municipal wastewater**

6

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13

14 **Abstract:** Direct membrane filtration (DMF) process is a promising technology for up-  
15 concentration and recovery of organic matter (OM) from municipal wastewater. This  
16 study investigates anaerobic digestibility of up-concentrated OM obtained from the  
17 DMF process. Effluent from a primary sedimentation basin was used as feed water.  
18 Effect of polyaluminum chloride (PACl) addition to the DMF process on the anaerobic

19 digestibility of obtained up-concentrated OM was also investigated because PACl is  
20 sometimes used as a coagulant in the DMF process for improving effluent quality and  
21 OM recovery, and for mitigating membrane fouling. Batch anaerobic digestion  
22 experiments were conducted to achieve these objectives. Fitting the obtained data to the  
23 reaction curve model revealed that maximum biogas production based on feed volatile  
24 solids ( $VS_{fed}$ ) from up-concentrated OM containing PACl reached 0.56 L-Biogas/g-  
25  $VS_{fed}$ , which was higher than from up-concentrated OM without PACl (0.47 L-  
26 Biogas/g- $VS_{fed}$ ), waste activated sludge (0.35 L-Biogas/g- $VS_{fed}$ ), and mixed sludge  
27 (0.48 L-Biogas/g- $VS_{fed}$ ). Methane yield based on degraded VS ( $VS_{deg}$ ) of up-  
28 concentrated OM containing PACl was 0.56 L- $CH_4$ /g- $VS_{deg}$ , which was also highest.  
29 These results indicate the up-concentrated OM obtained from the DMF process has high  
30 potential for biogas production, and the addition of PACl in the DMF process (~4.3 mg-  
31 Al/L) could enhance the biogas production.

32

33 **Keywords:** Biogas, Methane, Polyaluminum chloride, Batch experiment, Waste  
34 activated sludge

35

36 **1. Introduction**

37 In municipal wastewater treatment, organic matter (OM) is a main substance to be  
38 removed to maintain sanitary conditions and to protect aquatic environments. On the  
39 other hand, OM in municipal wastewater can be regarded as a resource for energy  
40 production, because biogas can be generated from up-concentrated OM via anaerobic  
41 digestion [1]. The activated sludge process is the most widespread wastewater treatment  
42 process and has been used for more than a century [2]. Although the activated sludge  
43 process provides adequate effluent quality, a large amount of energy is consumed by  
44 aeration. Mixed sludge, the mixture of primary sludge and waste activated sludge  
45 (WAS), is generated in the activated sludge process and is often treated by anaerobic  
46 digestion in large-scale wastewater treatment plants [3]. However, the methane yield of  
47 WAS is low because of its low biodegradability [4]. The high rate activated sludge  
48 (HRAS) process and the chemically enhanced primary treatment (CEPT) process have  
49 been proposed for up-concentration and recovery of OM to achieve energy neutrality in  
50 municipal wastewater treatment [1]. Although the HRAS and CEPT processes have  
51 been tested in full-scale applications, aeration is still needed.

52

53 Direct membrane filtration (DMF) of municipal wastewater using microfiltration or

54 ultrafiltration membranes is another option to up-concentrate the OM [5]. In the DMF  
55 process, membranes provide solid-liquid separation based on their pore sizes. Therefore,  
56 OM with a size larger than that of membrane pore size cannot pass through the  
57 membrane and then it is concentrated. As DMF is a membrane-based process, it does  
58 not require intensive aeration. In addition, a high recovery efficiency of OM (70-90%)  
59 based on chemical oxygen demand (COD) was reported for the DMF process [6-9], as  
60 membranes can provide high solid-liquid separation efficiency. The reported COD  
61 concentrations of up-concentrated OM are in the range of 8,000 to 23,000 mg/L [6-9],  
62 which can be supplied for anaerobic digestion process. The main concern of DMF  
63 process is membrane fouling, which decreases filtration flux and increases energy  
64 consumption. Therefore, some studies focusing on control and mitigation of membrane  
65 fouling in the DMF process have been reported [6-9]. Polyaluminum chloride (PACl) is  
66 sometimes used as a coagulant in the DMF process to increase OM recovery and to  
67 mitigate membrane fouling [7-9]. It has been reported that use of PACl also improved  
68 effluent quality by removing OM and nutrient (i.e., phosphorus) [7-9]. On the other  
69 hand, inhibitory effects of PACl on anaerobic digestion of WAS has been reported [10].  
70 So far, anaerobic digestibility of up-concentrated OM obtained from the DMF process  
71 has not been evaluated in detail. Investigation of the anaerobic digestibility of up-

72 concentrated OM is important to estimate the energy efficiency of the whole DMF  
73 process. The aim of this study is to first investigate the anaerobic digestibility of up-  
74 concentrated OM and compare it with that of mixed sludge and WAS. The second aim is  
75 to investigate the effect of PACl on the digestibility of up-concentrated OM. Batch  
76 anaerobic digestion experiments were used to achieve these objectives. The maximum  
77 biogas production and maximum biogas production rate were estimated by fitting the  
78 experimental data to the reaction curve (RC) model.

79

## 80 **2. Material and Methods**

### 81 *2.1. Up-concentration of OM in DMF system*

82 Up-concentrated OM was obtained from two laboratory-scale DMF reactors which were  
83 installed at an existing full-scale municipal wastewater treatment plant (Soseigawa  
84 wastewater treatment plant, Sapporo, Japan). Conventional activated sludge process is  
85 used in the plant and the treatment capacity is 144,000 m<sup>3</sup>/d. Effluent (i.e., not primary  
86 sludge but wastewater) from the primary sedimentation basin in the plant was used as  
87 the feed water. The average concentration of COD in the feed water was  $194 \pm 5$  mg/L.  
88 The DMF reactors were set up according to a previously reported configuration with  
89 some modifications [6]. Each DMF reactors was composed of two filtration tanks

90 sequentially connected (working volume: 4.6 L each). Flat-sheet ceramic membranes  
91 (total effective area: 0.147 m<sup>2</sup>) with a nominal pore size of 0.1 μm were immersed in  
92 each tank. The feed water was filtered through the membranes with dead-end filtration  
93 using suction pumps. Up-concentrated OM was continuously obtained from the second  
94 tank. The operational condition of each DMF reactors is shown in Table S1. Intermittent  
95 filtration and intermittent aeration were conducted throughout the operation. Mechanical  
96 scoring with granules and backwashing with tap water and/or sodium hypochlorite  
97 solution (500 mg/L) were conducted to avoid membrane fouling. Granules (BCN,  
98 Nisshinbo Chemical Inc., Tokyo, Japan) were added in each tank and were fluidized  
99 mechanically with stirrers for physical membrane cleaning. The granules were made  
100 from polyethylene glycol and they were cylindrical (diameter: 4 mm; height: 4 mm).  
101 One DMF reactor was operated with PACl addition and the other one was operated  
102 without PACl. The PACl addition was set at 4.3 mg-Al/L, which was the same addition  
103 in a previous study [9]. PACl stock solution (7 mL) was directly added to the first tank  
104 of the DMF reactor every 15 minutes. The hydraulic retention time of the first tank was  
105 1.1 h, which ensured sufficient contact time.

106

107 2.2. *Batch anaerobic digestion experiments*

108 The mixed sludge and WAS were also obtained from the Soseigawa wastewater  
109 treatment plant. The mixed sludge was thickened by centrifuge to reduce the sludge  
110 volume. The digested sludge was obtained from a mesophilic sludge digester of another  
111 full-scale municipal wastewater treatment plant (Ebetsu wastewater treatment plant,  
112 Ebetsu, Japan) and was used as seed sludge. All sludge samples were screened through  
113 a 1 mm mesh before use. The batch anaerobic digestion experiments were conducted in  
114 200 mL glass vials at 35 °C according to previously reported methods [11]. This kind of  
115 small-scale batch experiment is useful to evaluate biogas production potential of  
116 substrates [11-13]. Each vial was filled with 0.66 g-VS of seed sludge (80 mL) and 0.22  
117 g-VS of substrate sludge (60-120 mL). The produced biogas volume was normalized to  
118 standard condition of 101.3 kPa and 0 °C. All samples were prepared in duplicate, and  
119 control samples filled with only seed sludge were also prepared. The maximum biogas  
120 production and maximum biogas production rate based on the feed volatile solids  
121 ( $VS_{fed}$ ) were estimated by fitting the experimental data to the reaction curve (RC) model  
122 which is described in Eq. (1) [14]:

123 
$$B = P \left( 1 - \exp \left( - \frac{R_m(t - \lambda)}{P} \right) \right), (1)$$

124 where  $B$  is the cumulative biogas production (L/g- $VS_{fed}$ ),  $P$  is the maximum biogas

125 production ( $L/g-VS_{fed}$ ),  $R_m$  is maximum biogas production rate ( $L/(g-VS_{fed}\cdot d)$ ),  $t$  is  
126 digestion time (d), and  $\lambda$  is lag time (d). We obtained these values by using Origin Pro  
127 9.65 software. The RC model was reported as a simplified model for anaerobic  
128 digestibility test and the equation has been widely applied for various substrates  
129 including sewage sludge [15].

130

### 131 *2.3. Analytical methods*

132 Selected physical and chemical properties of sludge samples were analyzed before and  
133 after the batch digestion experiments. The pH of the sludge samples was measured  
134 using a pH meter (D-73, Horiba, Ltd., Kyoto, Japan). Concentrations of total solids (TS)  
135 and VS were determined according to standard methods [16]. Concentrations of total  
136 nitrogen (T-N), ammonium ( $NH_4^+-N$ ), total phosphorus (T-P), and orthophosphate  
137 ( $PO_3^{4-}-P$ ) were determined according to previously reported protocols using Hach  
138 reagent kits [17]. Following appropriate sample dilution, T-N and T-P concentrations  
139 were measured according to a Hach methods (Methods 10127 and 10072, respectively)  
140 with a spectrophotometer (DR 3900, Hach Co., Loveland, USA). Concentrations of  
141  $NH_4^+-N$  and  $PO_3^{4-}-P$  concentrations in the samples were also determined using Hach  
142 methods (Method 10031 and 8114, respectively) after centrifugation and filtration

143 through 0.45  $\mu\text{m}$ -pore size membranes (25HP045AN; Toyo Roshi Kaisya, Ltd., Tokyo,  
144 Japan). Microwave digestion was performed before determination of total aluminum (T-  
145 Al) concentrations. Each sludge sample (1 mL), concentrated  $\text{HNO}_3$  (2 mL), and  
146 concentrated HCl (6 mL) were added into digestion vials and samples were then  
147 digested on a microwave digestion system (ETHOS TOUCH CONTROL; Milestone  
148 General K.K., Kawasaki, Japan). Concentrations of T-Al were determined by an  
149 inductively coupled plasma atomic emission spectrometer (ICPE-9000; Shimadzu  
150 Corporation, Kyoto, Japan). The methane ( $\text{CH}_4$ ) and carbon dioxide ( $\text{CO}_2$ ) content of  
151 the biogas were determined using a gas chromatograph (GC-14B; Shimadzu  
152 Corporation, Kyoto, Japan) equipped with a thermal conductivity detector and a 6.0 m  $\times$   
153 3.0 mm stainless-steel-packed column (Shincarbon St, Shinwa Chemical Industries,  
154 Ltd., Kyoto, Japan). The capillary suction time (CST) of the digested sludge samples  
155 was measured using a CST apparatus (Type 304B CST; Triton Electronics Ltd., Essex,  
156 England).

157

### 158 **3. Results and Discussion**

159 Physical and chemical properties of sludge were shown in Table 1. The TS  
160 concentrations of the sludge varied from 2.3 to 4.9 mg/L because of differences between

161 the operational conditions of the wastewater treatment plant and laboratory-scale DMF  
162 reactors. The VS/TS ratio of the up-concentrated OM containing PACl was 73%, which  
163 was low compared with another substrate sludge due to PACl addition. Compared with  
164 other substrate sludge, the T-P and T-Al concentrations of the up-concentrated OM  
165 containing PACl were high because of the added PACl in the DMF process. In the DMF  
166 reactor without PACl addition, the trans-membrane pressure (TMP) which reflects  
167 membrane fouling reached 20 kPa after 100 hours of operation. On the other hand, TMP  
168 was below 10 kPa in the DMF reactor with PACl addition (data not shown). This result  
169 indicates the PACl addition was effective for mitigating membrane fouling.

170

171 Fig. 1 shows the temporal change in the cumulative biogas production from each type of  
172 sludge. Each data point represents the average value of two replicates. The RC model  
173 was fitted to the experimental data and the solid lines in Fig. 1 were obtained. The RC  
174 model was well fitted to the experimental data and the obtained parameters are  
175 summarized in Table 2. A long lag time was not observed because of the good  
176 acclimatization of the seed sludge to the substrate sludge. The biogas production from  
177 up-concentrated OM containing PACl was the highest among the four types of sludge,  
178 and the maximum biogas production reached 0.56 L/g-VS<sub>fed</sub>. The biogas production

179 from up-concentrated OM was comparable to that from mixed sludge. The maximum  
180 biogas production from WAS was the lowest among all sludge types because of the  
181 sufficient stabilization with long solid retention time in the plant. The maximum biogas  
182 production rates from up-concentrated OM, up-concentrated OM containing PACl, and  
183 mixed sludge were comparable, between 0.076 and 0.079 L/(g-VS<sub>fed</sub>·d). These results  
184 indicate that the up-concentrated OM containing PACl has the highest potential for  
185 biogas production. In this study, the mixed sludge contained primary sludge, but the OM  
186 obtained from the DMF process did not contain the primary sludge. Therefore, further  
187 increase in biogas production could be expected by anaerobic digestion of primary  
188 sludge and OM obtained in primary sedimentation basin-DMF process.

189

190 Although Chen *et al.* [10] reported that PACl has inhibitory effects on the anaerobic  
191 digestion of WAS, no inhibitory effects were observed in this study because of the low  
192 PACl dosage (4.3 mg-Al/L) in the DMF reactor. In previous studies about DMF, the  
193 range of PACl dosage was between 4 to 15 mg-Al/L [7-9]. In fact, the T-Al  
194 concentration (190 mg/L) of up-concentrated OM containing PACl was almost equal to  
195 that of the seed sludge (120 mg/L; Table 1). On the contrary, biogas production of the  
196 up-concentrated OM was enhanced by PACl addition in the DMF process. Gong *et al.*

197 [7] also reported the bio-methane potential of OM obtained from membrane-based pre-  
198 concentration (i.e., DMF) and reviewed the bio-methane potential of WAS from other  
199 studies. However, the anaerobic digestibility of sludge is largely affected by the  
200 characteristics of raw wastewater, and there are significant differences in the anaerobic  
201 digestibility of sludge among treatment plants. In our study, we successfully conducted  
202 a direct comparison of the biogas production from up-concentrated OM, mixed sludge,  
203 and WAS obtained from the same full-scale municipal wastewater treatment plant.

204

205 There was no significant difference in the methane content in the biogas produced from  
206 each sludge (69–73%; Table 3). The VS degradation rate of mixed sludge was the  
207 highest (80%) because it contained primary sludge. The VS degradation ratios of up-  
208 concentrated OM with and without PACl were 72% and 62%, respectively. The methane  
209 yields of up-concentrated OM were higher than those of mixed sludge and WAS, which  
210 means that up-concentrated OM obtained from DMF of municipal wastewater has high  
211 potential for methane production. The difference in the methane yields was probably  
212 come from the difference of constituents (i.e., carbohydrates, protein, and lipid) in the  
213 substrates [18]. Further research is needed to confirm this hypothesis. Considering the  
214 maximum biogas production and the methane content, the maximum methane

215 production of up-concentrated OM containing PACl was 407 mL-CH<sub>4</sub>/g-VS<sub>fed</sub>, which is  
216 slightly larger than previously reported value (367 mL-CH<sub>4</sub>/g-VS<sub>fed</sub>) [7]. Finally, we  
217 investigated the dewaterability of digested sludge (i.e., degree of ease in dewatering of  
218 sludge) by measuring the CST/TS value which is one of the index parameters of  
219 dewaterability of sludge [19]. Improved dewaterability of sludge is one of the  
220 advantages, as actual digested sludge is usually dewatered after anaerobic digestion.  
221 Although we expected better dewaterability of up-concentrated OM containing PACl  
222 because it contained a coagulant, there is no significant decrease in the CST/TS value.  
223 This result also suggested that the PACl content was low and the dewaterability was not  
224 improved. In a future study, we will scale up a reactor and conduct continuous operation  
225 of the reactor treating OM obtained from the DMF process. Investigation of energy  
226 efficiency and economic feasibility in an OM recovery process containing the DMF is  
227 also needed considering costs for membrane filtration and PACl use.

228

#### 229 **4. Conclusions**

230 This study investigated the anaerobic digestibility of up-concentrated OM obtained  
231 from DMF of municipal wastewater. We compared the biogas production from up-  
232 concentrated OM, mixed sludge, and WAS obtained from the same source of municipal

233 wastewater. The maximum biogas production and the methane yield of up-concentrated  
234 OM containing PACl were higher than those of up-concentrated OM without PACl,  
235 mixed sludge, and WAS. The presence of PACl in the up-concentrated OM did not show  
236 an inhibitory effect on the anaerobic digestibility, but instead enhanced the biogas  
237 production from up-concentrated OM. These results indicated that the up-concentrated  
238 OM obtained from DMF of municipal wastewater has high potential for biogas  
239 production. The obtained values in the batch anaerobic digestion experiments will be  
240 useful for estimation of energy efficiency and economic feasibility of the DMF process.

241

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246

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318

319 **Table and figure captions**

320 **Table 1.** Characteristics of sludge used for the batch anaerobic digestion experiments.

321 **Table 2.** Parameters obtained with the RC model.

322 **Table 3.** Methane content, VS degradation ratio, methane yield, and CST/TS value after

323 30 days digestion in the batch experiments.

324 **Fig. 1.** Cumulative biogas yield from each type of sludge in the batch experiments. The

325 solid lines are predictions calculated using the RC model.

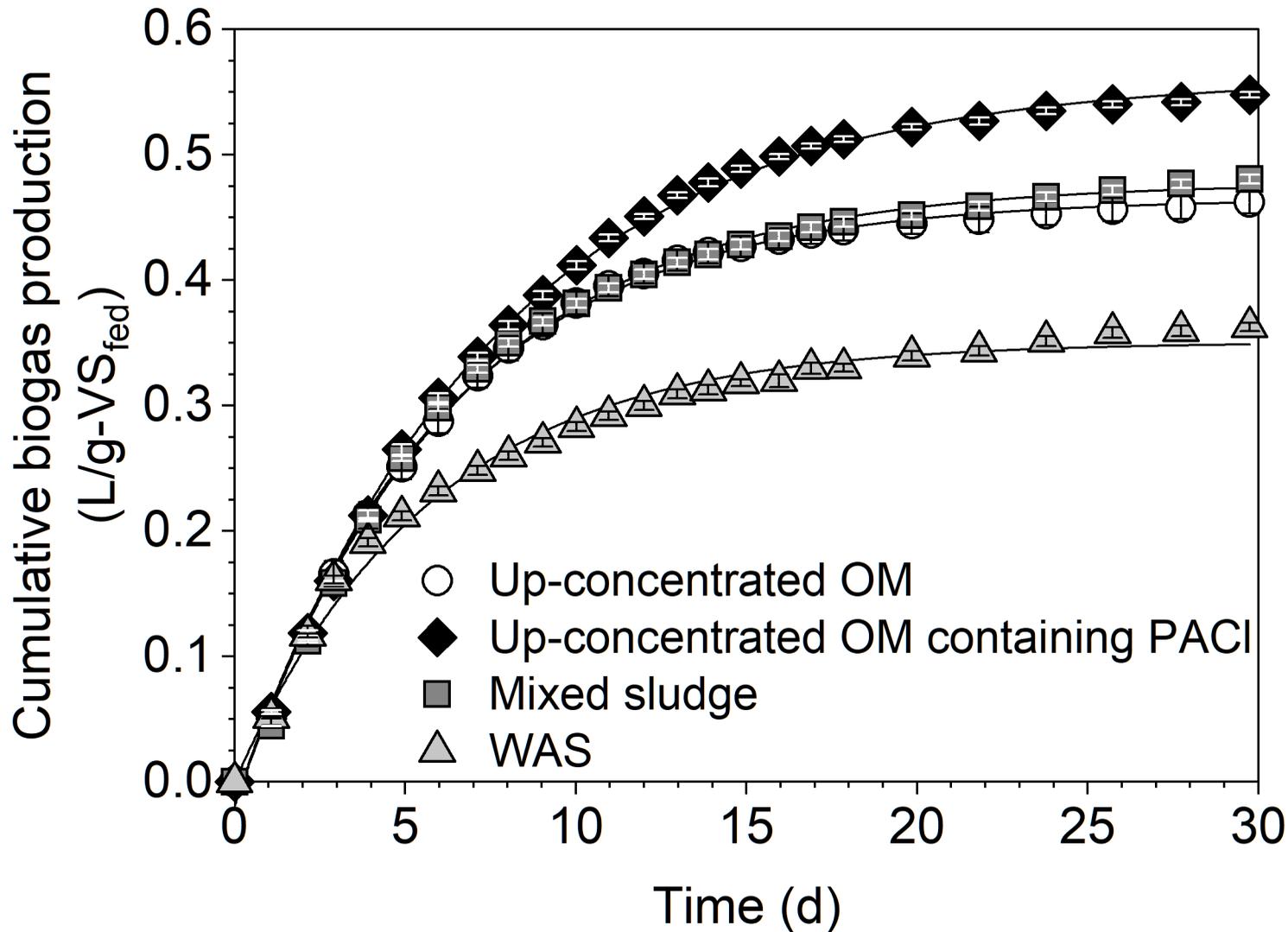
Type of sludge	pH	TS	VS	VS/TS	T-N	NH <sub>4</sub> <sup>+</sup> -N	T-P	PO <sub>4</sub> <sup>3-</sup> -P	T-Al
	(-)	(g/L)	(g/L)	(%)	(mg-N/L)	(mg-N/L)	(mg-P/L)	(mg-P/L)	(mg/L)
Seed sludge	7.4	11.4 ± 0.1	8.3 ± 0.0	73 ± 0	1700 ± 0.0	1030.0 ± 5.0	410 ± 13	290 ± 15	120 ± 0.65
Up-concentrated OM	7.0	3.4 ± 0.1	2.8 ± 0.1	84 ± 0	240 ± 5.0	23.5 ± 0.0	58 ± 4.0	5.1 ± 1.9	7.3 ± 0.06
Up-concentrated OM containing PACl	6.9	3.7 ± 0.2	2.7 ± 0.1	73 ± 0	210 ± 13	18.5 ± 0.5	96 ± 7.5	3.3 ± 1.7	190 ± 0.91
Mixed sludge	6.9	2.3 ± 0.0	1.9 ± 0.0	84 ± 0	150 ± 2.5	23.0 ± 0.0	34 ± 0.8	7.4 ± 1.6	8.8 ± 0.36
WAS	6.7	4.9 ± 0.0	3.9 ± 0.0	80 ± 0	330 ± 30	5.0 ± 3.0	88 ± 4.0	16.3 ± 7.7	22 ± 0.67

Note: TS = total solids; VS = volatile solids; T-N = total nitrogen; NH<sub>4</sub><sup>+</sup>-N = ammonium; T-P = total phosphorus; PO<sub>4</sub><sup>3-</sup>-P = orthophosphate; T-Al = total aluminum.

Type of sludge	$P$ (L-Biogas/g-VS <sub>fed</sub> )	$R_m$ (L-Biogas/(g-VS <sub>fed</sub> ·d))	$\lambda$ (d)	$r^2$ (-)	RSS (-)
Up-concentrated OM	0.47	0.079	0.2	0.998	$8.62 \times 10^{-4}$
Up-concentrated OM containing PACl	0.56	0.076	0.2	0.999	$8.73 \times 10^{-4}$
Mixed sludge	0.48	0.077	0.2	0.997	$1.41 \times 10^{-3}$
WAS	0.35	0.062	0	0.991	$1.97 \times 10^{-3}$

Note:  $P$  is the maximum biogas production.  $R_m$  is maximum biogas production rate.  $\lambda$  is lag time. RSS is residual sum of squares.

Type of sludge	Methane content (%)	VS degradation ratio (%)	Methane yield (L-CH <sub>4</sub> /g-VS <sub>deg</sub> )	CST/TS ((s·L)/g)
Up-concentrated OM	71.2 ± 0.1	62 ± 5	0.54 ± 0.05	17 ± 1
Up-concentrated OM containing PACl	72.7 ± 0.2	72 ± 7	0.56 ± 0.06	18 ± 0
Mixed sludge	71.9 ± 0.4	80 ± 3	0.43 ± 0.01	13 ± 0
WAS	69.0 ± 0.0	53 ± 12	0.50 ± 0.13	21 ± 3



**Table S1.** Operational condition of each DMF reactor.

	DMF reactor without PACl addition		DMF reactor with PACl addition	
	First tank	Second tank	First tank	Second tank
Working volume (L)	4.6	4.6	4.6	4.6
HRT (h)	1.5	4.9	1.1	4.2
PACl addition (mg-Al/L)	-	-	4.3	-
Filtration flux (LMH)	15.6	8.8	24.1	9.7
Intermittent filtration (filtration (min)/pause (min))	4/1	4/1	12/3	12/3
Intermittent aeration (aeration (min)/pause (min))	1/4	1/4	1/14	1/14
Granule volume (vol%)	30	30	30	30
Frequency of backwashing with tap water (backwashing (s)/operating time (h))	60/1.0	50/0.5	50/0.5	50/0.5
Frequency of backwashing with sodium hypochlorite (backwashing (s)/operating time (d))	120/2	120/2	-	-