Original Article

Effects of Microwave Pretreatment of Dewatered Sludge from an Oxidation-Ditch Process on the Biogas Yield in Mesophilic Anaerobic Digestion

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ABSTRACT

Microwave pretreatment was applied to highly concentrated dewatered sludge produced from an oxidation ditch process (OD sludge) to improve its degradation and biogas production in mesophilic anaerobic digestion. In a batch experiment using methane fermentation, the biogas production potential of OD sludge was increased after microwave irradiation. The increase in the biogas potential depended on the irradiation period and the temperature. Continuous digestion of the pretreated OD sludge was carried out in mesophilic conditions using a laboratory-scale digester at 25 days of hydraulic retention time (HRT). Although the biogas yield in the control digester was 0.12 NL/g-total solids (TS), that fed with OD sludge pretreated with max 100 W of irradiation at 80°C for 60 min was 0.15 NL/g-TS. Subsequently, dewatered OD sludge was pretreated by a novel continuous microwave irradiation device at 400 W for 20 min and fed into a pilot-scale co-digester with six kinds of waste biomass. The biogas yield of the reactor was increased from 0.25 NL/g-TS to 0.28 NL/g-TS. Assuming that the biogas yield of each co-digesting biomass was constant, the biogas yield of the OD sludge was estimated to increase by 42%.

Keywords: anaerobic digestion, pretreatment, microwave, mesophilic, sludge

INTRODUCTION

Anaerobic digestion has been introduced to many large-scale sewage treatment plants to improve the sludge characteristics and to recover energy from the sewage sludge. However, most of the small-scale sewage treatment plants that use the oxidation ditch (OD) process do not use a sludge digestion process, since the amount of excess sludge that is produced by the OD process is too small to use effectively. In addition, the biodegradability of the excess sludge produced by the OD process is low, because the OD process does not have a primary sedimentation basin and is operating at long hydraulic retention time (HRT). For the introduction of anaerobic digestion to small-scale OD plants, high-con-

centration digestion might be useful, using dewatered sludge gathered from one or more treatment plants. We conducted a laboratory-scale high-concentration digestion experiment using OD sludge, and we report that the high-concentration mesophilic digestion was successful when the input total solids (TS) was 10% and the sludge retention time (SRT) was 25 days. However, the gas yield of the OD sludge was only 0.11 NL/g-TS.

The pretreatment of sewage sludge helps increase the gas production of the sludge. For sludge pretreatment, several sludge dissolution methods have been reported including a thermal treatment [1], a hydrothermal treatment [2], an ozone treatment [3], and an ultrasonic treatment [4]. However, most of these studies were conducted using low-concentration

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Table 1 Characteristics of the sewage sludge.

	TS [mg/L]	Hydrogen content	Carbon content	Nitrogen content	Others	C/N
Dewatered OD sludge ^a	152,000	6.4%	44.3%	8.0%	41.3%	5.6
Raw sludge ^b	19,000	6.8%	45.1%	5.4%	42.7%	8.4
Excess sludge ^b	7,000	5.9%	37.2%	8.0%	48.9%	4.7

a ; used in this experiment

b ; collected from Johoku sewage treatment plant

sludge (below 6%). In addition, the application of ozone and ultrasonic treatments may be difficult for dewatered sludge, because the viscosity of dewatered sludge is high. With the thermal treatment and hydrothermal treatment, continuous treatment may be difficult. Recently, several research groups have reported microwave treatments for sewage sludge. For example, Appels *et al.* [5] observed a 50% increase in biogas production following the microwave irradiation of sewage sludge. Eskicioglu *et al.* [6] reported that microwave pre-treated sludge samples consistently produced higher biogas than conventional heating samples at the same temperatures. Tang *et al.* [7] studied the anaerobic digestion using the excess sludge (water content 96.60% – 99.59%) and reported that water content in the sludge was an important factor for microwave irradiation. However, most of their experiments were carried out using low-concentration sludge (<approx. 6% of TS), and they evaluated the pretreatment effect in batch experiments. In addition, the microwave irradiation was conducted using an oven. To apply the microwave pretreatment in the OD plant, continuous irradiation device was necessary.

In the present study, microwave pretreatment was applied to dewatered sludge (approx. 15% of the TS concentration). First, the effects of this pretreatment were examined using a microwave oven in both a batch digestion experiment and a continuous experiment. Next, a novel continuous microwave irradiation device was used, and the effects of microwave pretreatment were also investigated in a pilot-scale continuous digestion experiment.

MATERIALS AND METHODS

Batch experiment

In the batch experiment, we used a microwave oven equipped with power generators with 5 – 1,000 W output power and 2.45 GHz frequency (Shikoku keisoku, Kagawa, Japan, μ Reactor Ex). Dewatered OD sludge was collected from Kashima Chubu Sewage Treatment Plant in Nakanotocho, Ishikawa Prefecture, Japan, in which wastewater was

treated by an OD process and the excess sludge produced thereby was dewatered by a screw press. **Table 1** shows the characteristics of the dewatered OD sludge compared to raw sludge (a mixture of primary sludge and excess sludge) and compared to the excess sludge in a conventional process collected from Johoku Sewage Treatment Plant in Kanazawa City, Ishikawa Prefecture, Japan. There was no major difference in the carbon content between the OD sludge and the raw sludge, but the nitrogen content in the OD sludge was higher than that in the raw sludge. Togari *et al.* [8] reported that the biogas production potential of OD sludge was almost the same as that of excess sludge produced in the conventional activated sludge process.

In the present study, 50 g of dewatered OD sludge was spread in a plastic dish and placed in the center of the microwave oven. The sludge sample was 0.5 – 1 cm thick. The microwave oven was operated in various conditions. After the measurement of the total weight of the microwave-treated sludge in the dish, 12 – 16 g of the treated sludge was mixed with 50 mL of water and centrifuged. The supernatant was filtered through a 0.2- μ m membrane filter, and the concentrations of dissolved organic carbon (DOC), dissolved total nitrogen (DTN), and organic acids were analyzed. Using the microwave-treated sludge, the biogas production potential by conducting a batch experiment in a plastic syringe were measured. Mesophilic digested sludge was collected from Johoku Sewage Treatment Plant, at which sewage is treated in a conventional activated sludge process. A 0.5 – 0.7-g (dry weight) sample of microwave-pretreated sludge was mixed with 30 mL of digested sludge and cultivated for 30 days at 35°C, with shaking at 150 rpm. The sample's biogas production was recorded daily according to the syringe calibration. After the 30-day cultivation was completed, the gas composition was analyzed by a gas chromatograph.

Laboratory-scale continuous experiment

An 82 – 95-g sample of dewatered sludge spread in a beaker was pretreated in the microwave oven for 60 min below 100 W. During the irradiation, the temperature at the center

Fig. 1 Laboratory-scale continuous digester.

part of the sludge was controlled at 80°C. The biogas production potential of the pretreated sludge was then evaluated in a batch experiment.

The two continuous digesters shown in **Fig. 1** were run in parallel for 126 days in mesophilic conditions (35°C). One digester was fed untreated dewatered OD sludge as a blank (Run 1), and the other digester was fed microwave-pretreated dewatered OD sludge (Run 2). The digesters were installed in a room maintained at a constant temperature of 35°C, and the sludge was stirred at 150 rpm. First, 3 L of digested sludge (15 g-TS/L) collected from Johoku plant was put into each reactor as inoculums. Once a day, 120 mL of digested sludge in the digester was pulled out, and an equal amount of feed sludge was added into the digester to adjust the sludge retention time (SRT), for 25 days. The concentration of feed sludge was adjusted to 7% by dilution with an inorganic substrate (NaHCO_3 : 71 mg/L, KCl : 74 mg/L, CaCl_2 : 51 mg/L, KH_2PO_4 : 91 mg/L, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$: 130 mg/L, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$: 0.25 mg/L). The TS and volatile solids (VS) concentrations of the sludge were measured once a week. The pH and the concentrations of DOC, DTN, organic acids and ions in the supernatant were analyzed.

Pilot-scale continuous experiment

Figure 2 shows our novel continuous microwave irradiation device and the pilot-scale digester used in this experiment. The microwave irradiation device was equipped with 5 – 1500 W power generators and 2.45 GHz frequency. Dewatered OD sludge was introduced to the irradiation column continuously by a pump at 42.5 g/min. The irradiation conditions were set as 400 W for 20 min. The working volume of the pilot-scale digester was 1 m³. Before the experiment, the digester had been operated for 294 days using a mixture of the dewatered OD sludge and six types of waste biomass. The mixing ratios of each biomass were as follows: OD sludge, 69%; two types of wasted fried tofu, 18%; kitchen waste, 3%; night soil, 3%; Jokaso sludge, 4%, and waste sludge from a contact aeration plant, 3%. The input biomass mixture concentration was 9%. The SRT was set at 25 days, and the temperature was maintained at 37°C. After the 294th day, the pretreated dewatered OD sludge (42.5 g/min, 400 W, 20 min) was added to the digester instead of OD sludge, the changes in the gas generation, sludge concentration and supernatant composition were evaluated.

Analytical methods

The pH of the digested sludge was measured by a pH meter (HM-21P, DKK-TOA, Tokyo, Japan). The DOC and DTN concentrations were measured by a TOC/TN analyzer (TOC-V_{CPN}, Shimadzu, Kyoto, Japan). The concentrations of organic acids were analyzed by an ion chromatography system (HPLC Organic Acid Analysis System, Shimadzu). The ammonium concentration was analyzed by ion chromatography (HIC-SP, Shimadzu). The biogas volume was measured by a wet gas meter (W-NK, Shinagawa, Tokyo, Japan), and the gas composition was analyzed by gas chromatography (GA-8A, Shimadzu) in the laboratory-scale experiment.

Fig. 2 Microwave irradiation device and pilot-scale digester.

Fig. 3 Typical profile of temperature and power input during microwave irradiation.

Fig. 4 Effects of irradiation time on DOC and biogas production potential.

Fig. 5 Effects of control temperature on DOC and biogas production potential.

RESULTS AND DISCUSSION

Batch experiment

Figure 3 shows a typical profile of the temperature and power output during the irradiation period when 50 g of dewatered sludge was irradiated for 40 min at 80°C (Experiment 2-1). The temperature of the sludge immediately increased to the target temperature, and it was maintained by the low power output. The total output energy was calculated by the integration of output power.

Figure 4 shows the effects of irradiation time on the DOC concentration and the biogas production potential after irradiation, when the temperature was controlled at 80°C. When the irradiation time increased, the DOC concentration increased remarkably. On the other hand, although the biogas production increased until 60 min, further extension of the irradiation time was not needed. Figure 5 shows the effects

of the control temperature on the eluted DOC and the biogas production potential at 60 min of irradiation time. When the temperature rose, both the biogas production and the DOC increased. However, there was no clear correlation between input energy and the increase in biogas production potential as shown in Figure 6. The effects might depend on the absorbed energy. Solyom *et al.* [9] measured the input energy and absorbed energy in microwave irradiation and reported that these values were not in accord and that the efficiency was changed by the irradiation conditions. Figure 7 shows the relationship between DOC and the increase in biogas production potential. The biogas production tended to increase by the elution of DOC, suggesting that biodegradable organic substances were eluted by microwave irradiation and that eluted organic substances converted to biogas.

The results of the present batch experiment are summarized in Table 2. The biogas production of dewatered sludge was increased by 7 – 60% through the microwave irradiation.

Fig. 6 Effects of input energy on biogas production potential.

Fig. 7 Relationship between DOC and biogas production potential.

Table 2 Summary of the batch irradiation experimental results.

Series	No.	Maxpower [W]	Time [min]	Temperature [°C]	TS [%]	Gas production [NmL/g-TS]	DOC [mg/L]	Input energy [kJ/g-TS]	Note
1	1-1	300	10	80	13.11	164.5	331.2	5.9	
	1-2					135.7	115.1	6.4	
	1-3		60			165.9	548.4	18.4	
	1-4	173.8				888.0	16.2	covered with cling film	
	1-5	100	10			163.7	385.0	5.7	
	1-6		60			161.5	634.4	26.1	
	1-7	untreat	127.4			20.9	0.0		
2	2-1	300	40	80	12.59	169.7	935.8	10.3	covered with cling film
	2-2		60			175.1	1,020.0	12.4	covered with cling film
	2-3		120			180.5	1,310.0	23.4	covered with cling film
	2-4		60	60		147.5	227.6	10.4	covered with cling film
	2-5		60	95		214.8	1,400.2	20.8	covered with cling film
	2-6	untreat	134.0	53.1		0.0			

The maximum biogas production was 215 NmL in No. 2–5. It was reported that the biogas production rose by 41 – 52% in a microwave irradiation experiment using waste sludge (TS was approx. 5%) [10]. In No. 1–4, when the dish was covered with cling film, the biogas production amount increased by 4.8%, implying that the heat dissipation by evaporation was suppressed, and the amount of absorbed energy increased. The biogas production amount decreased by 17.5%, when the same amount of sludge was spread onto two dishes at half thickness. These results suggest that the effects of microwave irradiation depend on the absorbed energy.

Laboratory-scale continuous experiment

Figure 8 shows the cumulative biogas production, $\text{NH}_4\text{-N}$ concentration and VS concentration in the continuous experiment. Stable biogas generation was observed in both runs. The gas production of Run 2, in which pretreated sludge was used as a substrate, was greater than that of Run 1 (Control), meaning that the biogas production was increased by microwave treatment. The $\text{NH}_4\text{-N}$ concentration was increased in the digester by the sludge decomposition in both runs, but the biogas production was not inhibited at this concentration. The VS concentration was decreased in both runs by the sludge decomposition. The increase in ammonium and

Fig. 8 Results of the laboratory-scale continuous digestion experiment. Run 1: untreated sludge, Run 2: pretreated sludge.

the decrease in VS in Run 2 were higher than those in Run 1, indicating that the sludge decomposition was accelerated by the microwave pretreatment.

The experimental results are summarized in **Table 3**. The gas generation of Run 1 (Control) was 0.12 NL/g-TS, which is almost the same value as that of the average gas potential of the OD sludge that was used. In contrast, the biogas production of Run 2 was 0.15 NL/g-TS, suggesting that the biogas production was increased by 27% through microwave irradiation. In the batch experiment, the biogas production potential of the irradiated sludge in this condition was 0.14 NL/g-TS. Biogas production might be increased by the continuous cultivation. The effect of the microwave

Table 3 Summary of the laboratory-scale continuous experimental results.

	Run1 untreated	Run2 pre-treated
Total biogas yield [NL]	92.5	118.2
Biogas yield [NL/g-TS added]	0.12	0.15
CH_4 [%]	65	65
VS removal ratio [%]	26	34

irradiation was thus confirmed by the laboratory-scale continuous experiment. However, the microwave effect in this irradiation condition was relatively low, because the irradiation temperature was 80°C. Further experiments under several conditions are necessary to find a more effective irradiation condition.

Pilot-scale continuous experiment

Although pretreated sludge instead of un-treated sludge was added in the pilot-scale continuous experiment, the biogas production was stable and no accumulation of organic acids or ammonium was recognized. The experimental results are summarized in **Table 4**. The biogas yield of the biomass mixture changed from 0.25 NL/g-TS to 0.28 NL/g-TS. The biogas production was increased by 12% through the pretreatment of OD sludge. Assuming that the biogas yield of each waste biomass (except the OD sludge) is the same as that of the biogas production potential reported by Togari *et al.* [11], we estimated that the biogas yield of the OD sludge was increased by 42% through the microwave irradiation shown in **Table 5**.

CONCLUSIONS

The effects of the microwave pretreatment of dewatered OD sludge on high- concentration mesophilic anaerobic digestion was examined. The results can be summarized as follows.

- 1) The results of our batch experiment confirmed that the increase in the biogas production of dewatered OD sludge was due to the microwave irradiation. Although the increase in the biogas production depended on the irradiation time and the control temperature, a correlation with the input energy was not confirmed. A strong correlation between the eluted DOC and the increase in biogas production by microwave irradiation was recognized.
- 2) In the laboratory-scale continuous experiment, the

Table 4 Results of the pilot-scale continuous experiment.

	Period 1 (untreated)	Period 2 (MW pre-treated)
Days	222	52
Mixed sludge average TS [%]	8.8	8.3
Mixed sludge average VS [%]	7.3	7.1
Average organic loading rate [kg/m ³ day]	2.5	2.4
Biogas yield [NL/g-TS added]	0.25	0.28
CH ₄ yield [NL/g-VS added]	0.19	0.21
Average VS removal ratio [%]	37	40
Average CH ₄ [%]	65	64

Table 5 Estimation of biogas yield of OD sludge based on the biogas production potential of each waste biomass.

	Period 1 (untreated) [NL/g-TS]	Period 2 (MW pre-treated) [NL/g-TS]
Biomass mixture	0.25	0.28
Kitchen waste	0.21	0.21
Night soil	0.22	0.22
Jokaso sludge	0.28	0.28
Waste sludge from a contact aeration plant	0.07	0.07
Wasted fried tofu (raw)	0.97	0.97
Wasted fried tofu (dry)	0.74	0.74
OD sludge	0.12	increased by 42% → 0.17(calculated)

biogas gas production, ammonium production and VS removal ratio were increased by 27%, 28% and 33% respectively, through microwave irradiation for 60 min at 80°C.

- 3) Using a novel continuous microwave irradiation device and a pilot-scale high-concentration mesophilic co-digester, the effects of the pretreatment of OD sludge was examined. The biogas production of the biomass mixture in which OD sludge was comprised 69%, was increased by 12%.

Our findings demonstrate the usefulness of microwave irradiation for the acceleration of the anaerobic digestion of dewatered sludge. Since the continuous microwave irradiation device can treat the sludge continuously, its introduction to digestion plants could be easy. Further experiments are needed to identify the suitable irradiation conditions. In this

study, a clear relationship between the eluted DOC and the biogas production was demonstrated, but the mechanism underlying the irradiation effects on the elution of DOC is not yet clear.

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