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Trace metal requirements for anaerobic co-digestion of sewage sludge from rural areas and food waste in Japan

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Abstract

For small-scale wastewater treatment plants in rural areas (WWTPRs) of Japan, reducing sludge disposal costs is an important issue. Co-digestion of sludge from WWTPRs and food waste is a promising method of reducing maintenance costs and producing energy resources such as methane. In this study, co-digestion experiments using sludge and kitchen garbage were conducted to investigate the stability of the co-digestion from the perspective of trace metal requirements. The gas production rate and pH were stable in the digester with HRTs of 140–70 days. With a HRT of 30 days, the pH gradually dropped from 7.0 to 6.5, and VFA accumulated up to a concentration of 2,200 mg/L, indicating process failure. Cobalt (Co) concentration was approximately one-tenth of the required amount reported by a previous study. When Co was added to the digester, the pH and gas production rate became stable even with a higher VS loading rate. Therefore, the unstable situation was attributed to a lack of Co in the sludge. The Co concentration of sludge from WWTPRs in Japan is generally low, similar to the sludge used in this study. The addition of Co is required for stable co-digestion of sludge from WWTPRs and food waste.

Key words: cobalt, kitchen garbage, nickel, rural areas, sludge, stability of digestion

INTRODUCTION

Anaerobic digestion (AD) is a technology for decomposing organic matter through biological activity in an oxygen-free environment with concomitant biogas production containing approximately 60% methane (CH_4) and 40% carbon dioxide (CO_2) (Golueke 1977; Ward *et al.* 2008). The methane from AD is a renewable energy source and greenhouse gas emissions can be reduced by using CH_4 as fuel instead of fossil fuel. Sewage sludge discharged from wastewater treatment plants (WWTPs) is often used as a feedstock for AD because a tremendous amount of sludge is generated in WWTPs (Hidaka *et al.* 2015). Seventy-eight million tons of sludge is generated in Japan per year (Ministry of Agriculture Forestry & Fisheries 2016), and it is used as AD feedstock at large-scale plants.

In rural areas of Japan, there are approximately 5,000 small-scale wastewater treatment plants (WWTPRs) supported by the Ministry of Agriculture, Forestry and Fisheries of Japan (Ministry of Agriculture Forestry & Fishery 2017). WWTPRs generally treat the wastewater of 200–1,000 people using an extended aeration process, and the amount of generated sludge is 140–700 tons per plant per year on a thickened sludge basis (water content of approximately 98.5%) (Yuyama *et al.* 1993; Ministry of Agriculture Forestry & Fishery 2017). For WWTPRs, sludge disposal cost reduction is an important issue because it accounts for most of the maintenance cost (63%). Although AD of sludge from WWTPRs is a candidate for reducing maintenance costs, the amount of sludge generated in each plant is small, and it is difficult to build an energy-efficient AD system. Therefore, additional measures are needed to introduce AD into WWTPR systems.

Co-digestion of sludge from WWTPRs and food waste is a promising method for increasing biogas production and improving energy efficiency. Moreover, it can reduce the amount of food waste for disposal and contribute to improving the recycling rate of food waste, which is very low (24%) in Japan (Ministry of Agriculture Forestry & Fisheries 2016). The Ministry of Agriculture, Forestry and Fisheries of Japan started a demonstration project to study the feasibility of this system (Nakamura 2017). The stability of the co-digestion of sludge from WWTPRs and food waste was examined in the project.

Problems such as low CH_4 yield and instability are often encountered during AD. Inhibitory substances that cause instability include ammonia, sulfide, light metal ions, heavy metals, and organics (Chen *et al.* 2008; Hagos *et al.* 2017). Many researchers have indicated that the availability of trace metals is a significant factor affecting the performance and stability of biogas digesters (Speece 1988; Takashima *et al.* 2011; Qiang *et al.* 2012). Requirements for iron (Fe), nickel (Ni), cobalt (Co), molybdenum (Mo), selenium (Se) and tungsten (W) of various methanogens have been reported (Demirel & Scherer 2011). Among these, Fe, Ni and Co are the most important trace elements in an anaerobic digestion system (Choong *et al.* 2016). Mono-digestion of food waste often leads to digester instability and even failure at higher organic loading rates, and one of the causes was shown to be a limitation in trace metals (Xu *et al.* 2018). Although it is necessary to add trace metals for stable digestion of food waste, it is well known that co-digestion with sewage sludge, which is originally rich in trace elements, is an effective measure (Okuno *et al.* 2001; Choong *et al.* 2016; Xu *et al.* 2018).

However, co-digestion of sludge from WWTPRs and food waste still faces issues for stable implementation. WWTPRs are generally in rural areas and treat only domestic wastewater and no industrial discharges. Moreover, many WWTPRs are not equipped with dehydrators for sludge. Therefore, total solids (TS) and trace metal concentrations of the sludge may be low and insufficient for stable co-digestion with food waste. Detailed data concerning the concentrations of some trace metals in sludge from WWTPRs are not available, and no data are available concerning Co, an important trace metal for AD, because previous reports have focused on safety for utilization of sludge as a fertilizer.

The aim of this study was to investigate the stability of the co-digestion of sludge from WWTPRs and food waste (kitchen garbage) from the perspective of trace metal requirements.

MATERIALS AND METHODS

Characteristics of sewage sludge and kitchen garbage

The feedstocks were sewage sludge and simulated kitchen garbage. Sewage sludge was obtained from WWTPR A in Ibaraki Prefecture, Japan. The composition of the simulated kitchen garbage followed a recipe reported by Li *et al.* (2003) (Table 1), which was designed based on the results of a garbage composition survey in Japan. The kitchen garbage was chopped into 2-cm cubes and shredded with a high-speed shredder. The TS content was adjusted to 12% by adding tap water. Substrate characteristics are summarized in Table 2.

Continuous fermentation experiment

Two continuous anaerobic digestion experiments were conducted.

Table 1 | Composition of simulated kitchen garbage (%)

Apple	10
Grapefruit peel	5
Orange peel	5
Banana peel	10
Cabbage	12
Potato	12
Carrot	12
Ground meat	5
Fish with bone	5
Egg	4
Cooked rice	10
Bread	5
Japanese noodle	2.5
Chinese noodle	2.5

Table 2 | Compositions of feedstocks

	Sewage sludge	Kitchen garbage
TS (mg/L)	16,200	118,000
VS (mg/L)	13,100	113,000
COD_{Cr} (mg/L)	17,400	123,000
T-N (mg/L)	1,180	3,130
S (mg/L)	300	200
Fe (mg/L)	139	4.14
Co (mg/L)	0.02	<0.01
Ni (mg/L)	0.21	0.02

AD experiment 1

To investigate the stability of co-digestion of sludge from WWTPRs and kitchen garbage, Experiment 1 was conducted using a continuous stirred tank reactor (CSTR) with a working volume of 10 L, as shown in Figure 1. The reactor was operated under mesophilic (37 °C) conditions and agitated at

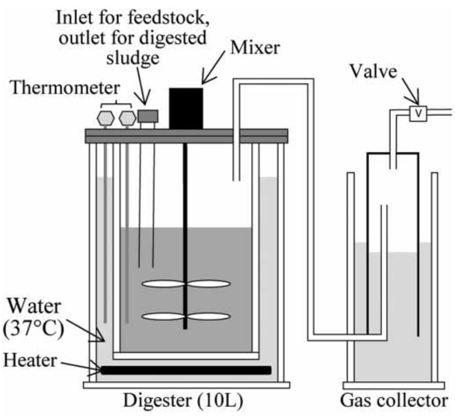

Figure 1 | Schematic diagram of the CSTR (Experiment 1).

100 rpm using a motor mixer. Temperature inside the reactor was maintained by an on/off control outside the reactor to operate the heater in the water bath. Gas production was measured using the water replacement method. To prevent dissolution of CO_2, 2% vol H_2SO_4 was used instead of water (Cha *et al.* 1992).

Anaerobic sludge taken from a mesophilic anaerobic digester treating sewage sludge in Sendai City, Japan, was used as the seed sludge. The digested sludge was withdrawn, and substrates were fed every weekday (five times a week) using the inlet illustrated in Figure 1. Feeding was conducted quickly to prevent O_2 from entering the reactor. The reactor hydraulic retention time (HRT) was 140 days at the beginning of the experiment and gradually shortened to 30 days. Substrates were prepared every 20 days and stored in a refrigerator at 4 °C until use. Only sludge was used as the substrate until Day 48, and a mixture of sludge and kitchen garbage in a weight ratio of 1:1 was used after Day 49. The detailed operation conditions are shown in Table 3. Gas production and COD removal rates, pH, alkalinity and ammonia, COD_{Cr}, VFA, Fe, Co, and Ni concentrations were monitored during the experimental period.

Table 3 | Operating conditions of co-digestion (Experiment 1)

Period (Day)	0–48	49–76	77–101
HRT(d)	140	70	30
VS loading rate (g/L/d)	Sludge 0.00 Garbage 0.76	Sludge 0.09 Garbage 0.76	Sludge 0.22 Garbage 1.79

AD experiment 2

Experiment 2 was conducted to investigate the effects of the addition of Co or Ni to stable digestion. Four 0.3-L glass triangular flasks with a working volume of 0.2 L and rubber stoppers were used as the anaerobic digestors. The gas outlet port was connected to an aluminum-coated gas bag (AAK1; GL Science, Tokyo, Japan). The volume of biogas produced was measured using a glass syringe. The reactors were placed in an incubator to maintain mesophilic (37 °C) conditions. They were agitated at 100 rpm using a multi-purpose shaker.

The seed sludge used in Experiment 2 was the digested sludge taken from the CSTR on Day 104 during Experiment 1. The seed sludge was stored in an incubator for 7 days at 37 °C before use in Experiment 2. The substrate was the mixture of sludge and kitchen garbage in a weight ratio of 1:1. Four experimental conditions with or without $CoCl_2$ or $NiCl_2$ additions were designed as shown in Table 4. The amounts of added Co or Ni were determined based on Qiang *et al.* (2012). The detailed operating conditions are shown in Table 5.

Substrates were fed and digested sludge was withdrawn every weekday (five times a week). When feeding substrates, the reactor was opened, and digested sludge was withdrawn from the reactor. Then, the reactor was replenished with the same amount of fresh substrate. Feeding was conducted quickly to prevent O_2 from entering the reactor. The reactors were flushed with N_2 gas for 0.5 min

Table 4 | Experimental conditions (Experiment 2)

Experimental plot	Amount of added Co (mg Co/COD of kitchen garbage)	Amount of added Ni (mg Ni/COD of kitchen garbage)
Co+Ni+	6.00	5.70
Co+Ni−	6.00	Not added
Co−Ni+	Not added	5.70
Co−Ni−(Control)	Not added	Not added

Table 5 | Operating conditions during co-digestion (Experiment 2)

Period (d)	0–7	8–42	43–56	57–70
HRT(d)	60	30	20	15
VS loading rate (g/L/d)	Sludge 0.11 Garbage 0.89	Sludge 0.22 Garbage 1.79	Sludge 0.33 Garbage 2.69	Sludge 0.44 Garbage 3.58

to maintain anaerobic conditions in the reactor after each feeding. Feeding was stopped for reactors when pH reached 5. Gas production and pH were monitored during the experimental period. Alkalinity and ammonia, COD_{Cr}, Fe, Co and Ni concentrations were measured after the experiment.

Investigation of sludge from WWTPRs

To evaluate the repeatability of the results of the AD experiments, sludge was collected from four WWTPRs other than WWTPR A, where the sludge used in Experiments 1 and 2 was collected. The treatment process at these WWTPRs is the extended aeration process, which is the same as that of WWTPR A. The TS, VS, COD_{Cr} and Fe, Co and Ni concentrations of the collected sludge were analyzed.

Analytical methods

Routine laboratory analyses were carried out. Most of the analyses were performed using standard methods (APHA 2005). The VFA content was determined by high performance liquid chromatography (LC-20AD; Shimpack-SCR-102H or LC-10A Shimpack-SCR-101). Total acid concentrations were calculated by summing the individual VFAs. Fe, Co and Ni concentrations were determined with ICP-AES (Agilent Technologies 5,110 ICP-OES) after pretreatment (incineration at 600 °C and heating with nitric acid and hydrochloric acid).

The COD removal rate was calculated by Equation (1):

$$\text{COD removal rate (\%)} = (COD_{in} - COD_{out})/COD_{in} \times 100 \tag{1}$$

where COD_{in} (mg/L) is the COD concentration in the substrate, and COD_{out} (mg/L) is the COD concentration in the digested sludge.

RESULTS AND DISCUSSION

Experiment 1

Figure 2 shows the gas production and COD removal rates, pH, alkalinity and ammonia, COD_{Cr}, VFA, Fe, Co and Ni concentrations during the experimental period. The typical operating parameters, gas production rate, pH and VFAs were stable in the digester at HRTs of 70–140 days. This indicated that the experiment started successfully.

From Day 79 (HRT 30 days), the pH gradually dropped from 7.0 to 6.5, and VFAs accumulated to a concentration of 2,200 mg/L, indicating process failure. Okuno *et al.* (2001) conducted a co-digestion experiment using night soil sludge and the same simulated kitchen garbage as this study. In their study, the mixing ratio of sludge and kitchen garbage was 1:9 by TS base and TS, VS and COD_{cr} of the mixed substrate were 97,600, 87,700, and 141,600 mg/L. They did not report digestion instability despite a much higher VS loading rate (5.8 g/L/d) than the one in this study. This showed that the VS loading rate in this study was not excessively high. Ammonia did not exceed 1,700–1,800 mg/L, which is the concentration at which anaerobic digestion is inhibited (Chen *et al.* 2008; Zhang *et al.* 2014).

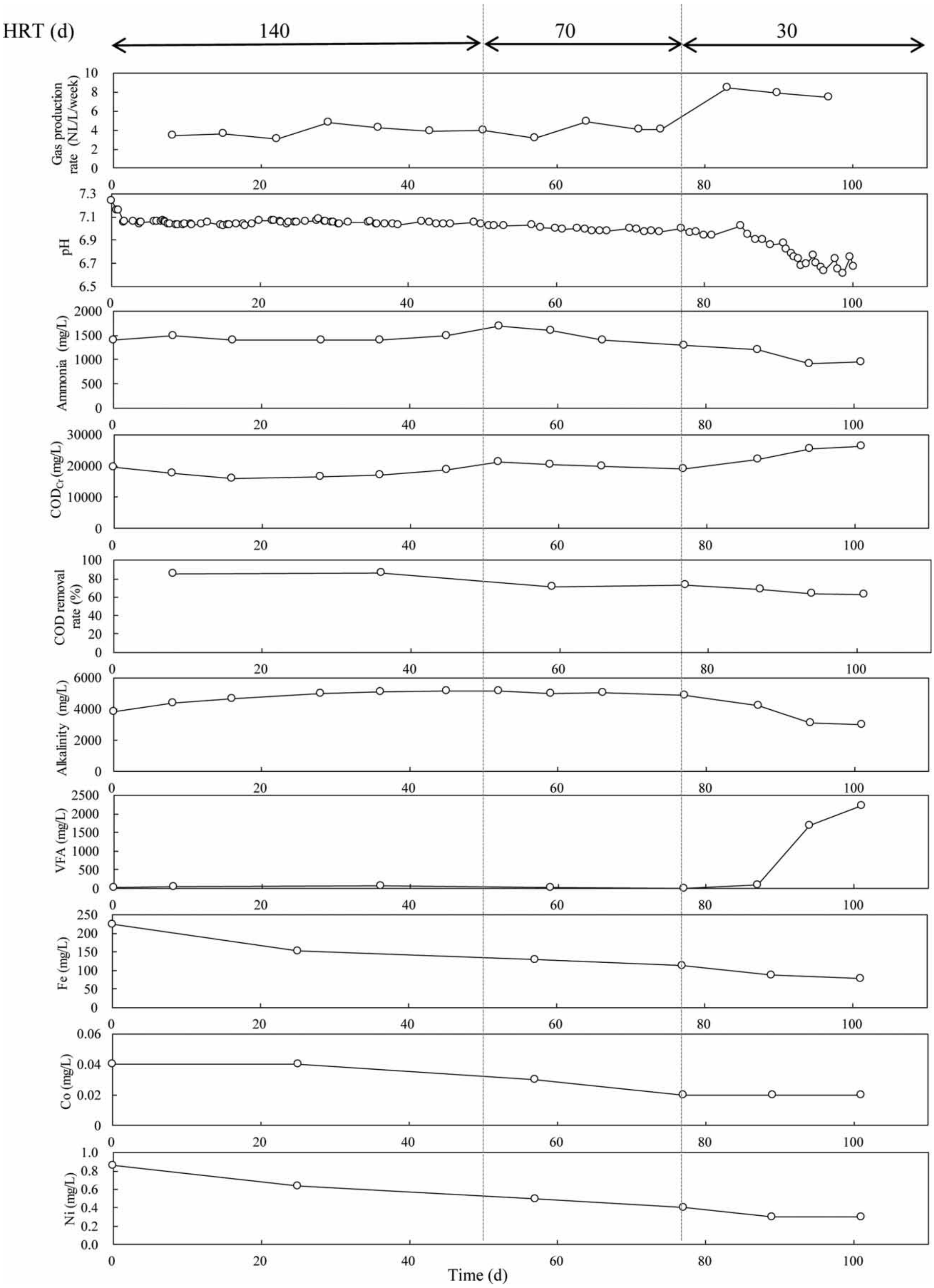

Figure 2 | Gas production rate, pH, ammonia and COD_{Cr} concentrations, COD removal rate, alkalinity and VFA, Fe, Co and Ni concentrations during the experimental period (Experiment 1).

Free ammonia concentration, which is a main cause of inhibition (Chen *et al.* 2008), was very low considering the pH (6.7–7.0) and temperature (37 °C). Therefore, this unstable situation was also unlikely to be attributed to inhibition by high ammonia.

Another possibility was trace metal limitation. Fe, Ni and Co are the most important trace elements in an AD system (Choong *et al.* 2016). Qiang *et al.* (2013) reported that reactor efficiency deteriorated due to limited Fe, Co and Ni during AD of food waste. In this study, the concentrations of Fe, Co and Ni decreased to 77.5, 0.020 and 0.30 mg/L, respectively, during the experimental period, even though their concentrations were relatively high at the start of the experiment due to Fe, Co and Ni derived from the seed sludge. The Co concentration in this study was almost the same as that reported in Qiang *et al.* (2013).

Fe, Ni and Co concentrations per COD removed on Day 101 in this study are shown in Table 6. Takashima *et al.* (2011) and Qiang *et al.* (2012) reported the requirements for Fe, Co and Ni in a mesophilic system. Fe concentration in this study far exceeded the value reported by Takashima *et al.* (2011) and Qiang *et al.* (2012). This indicated that sufficient Fe was supplied from the sludge, even though the Fe concentration in kitchen garbage was low. However, the Ni concentration was similar to the required amount reported, and the Co concentration was much lower and only approximately one-tenth of the required amount. Therefore, the unstable situation in this study seemed to be attributed to a lack of Co supplied from the kitchen garbage or the sludge. However, the possibility of a lack of Ni could not be denied because of the relatively wide range of values for trace metal requirements reported in previous studies, and the requirements were highly dependent on HRT (Climenhaga & Banks 2008).

Table 6 | Fe, Ni and Co concentrations in the digested sludge on Day 101 (Experiment 1)

	This study	**Requirement reported**	
		Takashima *et al.* (2011)	**Qiang *et al.* (2012)**
Fe (mg/L)	77.5	–	–
Co (mg/L)	0.02	–	–
Ni (mg/L)	0.30	–	–
Fe/COD removed (mg/kg)	1,650	200	200
Co/COD removed (mg/kg)	0.43	6.3	6.00
Ni/COD removed (mg/kg)	6.4	1.7	5.70

Experiment 2

Figure 3 shows the gas production rate and pH during the experimental period. Table 7 shows the COD removal rate, alkalinity and ammonia, COD_{Cr}, Fe, Co and Ni concentrations in the seed sludge and digested sludge after the experiment. In the Co − Ni− condition (Control), the pH gradually dropped when the HRT was 30 days, similar to the results in Experiment 1, and reached 5 on Day 40. In addition, the COD removal rate decreased and reached 40% on day 40. This suggested that process failure occurred, similar to Experiment 1.

However, in the Co + Ni+ and Co + Ni− conditions with Co additions, the pH and gas production rates were stable during the whole experimental period, including the period with a high VS loading rate. The COD removal rate was high (~70%) under both conditions. This indicated that the addition of Co contributed to the stability of the digester. Digestion under the Co + Ni− condition was stable even though the Ni concentration in the digested sludge after the experiment was much lower than that in the Co + Ni+ condition. This suggested that the Ni requirements were satisfied in Experiment 1.

Under the Co − Ni+ condition, the gas production rate and pH were more stable than under the Co − Ni− condition, but the pH gradually dropped when the HRT was 15 days and reached 5 on Day 63. This phenomenon indicated the possibility of another methanogenesis pathway that required

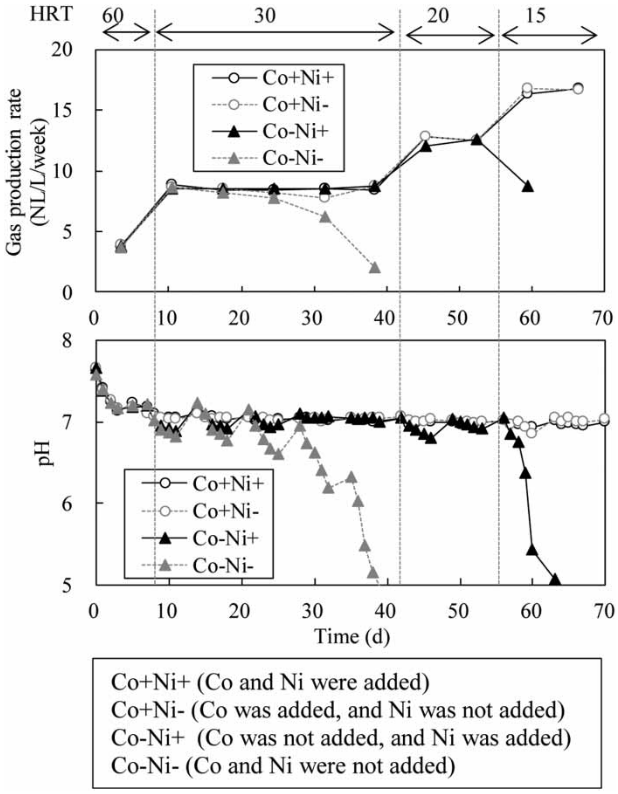

Figure 3 | Gas production rate and pH during the experimental period (Experiment 2).

Table 7 | Ammonia and COD_{Cr} concentrations, COD removal rate, alkalinity and Fe, Co and Ni concentrations in the seed sludge and digested sludge after the experiment (Experiment 2)

	Seed sludge	Co+Ni+	Co+Ni−	Co−Ni+	Co−Ni−
Sampling		Day 70	Day 70	Day 63	Day 42
Ammonia (mg/L)	1,100	740	790	660	770
COD_{Cr} (mg/L)	21,200	21,400	23,200	42,400	40,000
COD removal rate (%)	–	70	67	40	43
Alkalinity (mg/L)	5,010	3,510	3,510	1,100	1,100
Fe (mg/L)	66.7	46.2	45.6	46.8	49.1
Co (mg/L)	0.02	0.24	0.26	0.008	0.02
Ni (mg/L)	0.22	0.35	0.09	0.25	0.12

little Co when the Ni concentration was relatively high. This indicates that Ni addition also contributed to the stability of the digester when the amount of Co was insufficient.

Recent research has shown that not only the total concentration but also the bioavailable concentration of trace elements is important to the trace elements requirement (Demirel & Scherer 2011; Choong *et al.* 2016; Takashima 2018). Thus, it was necessary to examine the bioavailability of Co and Ni in the sludge and kitchen garbage to accurately quantify the amounts of Co and Ni added.

Experiments with other sludges

Table 8 shows the compositions of sludge from the WWTPRs. The Fe and Ni concentrations of the sludge used in this study were similar to those in the sludges from WWTPRs B to E and to the average

Table 8 | Compositions of sludges from WWTPRs

	This study	Plant B	Plant C	Plant D	Plant E	Average in Japan
TS (mg/L)	16,200	14,000	19,100	26,700	16,200	18,900
VS (mg/L)	13,100	11,100	15,900	22,100	12,600	14,500
COD_{cr} (mg/L)	17,400	13,600	20,700	30,900	16,400	–
Fe (mg/L)	139	220	80.5	165	359	112
Co (mg/L)	0.02	0.02	0.05	0.06	0.03	–
Ni (mg/L)	0.21	0.31	0.60	0.71	0.24	0.32

for sludge from 66 WWTPRs in Japan (Ministry of Agriculture Forestry & Fisheries 2017). This suggests that the conclusion from this study – that a sufficient amount of Fe and Ni was supplied by the sludge – would be a generally acceptable assumption for WWTPRs in Japan.

However, the Co concentrations of sludges from WWTPRs B to E were low, similar to that from the sludge used in this study. In addition, the Co concentration was approximately one-tenth of the amount required. This showed that a sufficient amount of Co could not be supplied by increasing the sludge ratio in the feedstock, and the addition of Co would be required for stable co-digestion of sludge from WWTPRs and food waste in Japan.

CONCLUSION

This study investigated the stability of the co-digestion of sludge from WWTPRs and food waste (kitchen garbage) from the perspective of trace metal requirements. The following conclusions could be drawn.

Co-digestion of sludge from WWTPRs and kitchen garbage without the addition of trace metals resulted in an unstable performance with an HRT of 30 days and a VS loading rate of approximately 2 g/L/d.

Although sufficient amounts of Fe, Ni and other trace metals for stable digestion were supplied from sludge, the Co in the sludge was only one-tenth of the required amount reported in previous studies. Therefore, the unstable performance in this study was attributed to a lack of Co, and the addition of Co contributed to the stability of the digester.

The Co concentration of sludge from other WWTPRs was also generally low, like the sludge used in this study. The addition of Co is required for stable co-digestion of sludge from WWTPRs and kitchen garbage in Japan.

The co-digestion of sludge and garbage in this study aimed to reduce the operational costs of WWTPRs. From the results of this study, it was clear that Co addition is necessary for stable co-digestion, which may lead to an increase in cost. For practical use of the co-digestion of sludge and kitchen garbage at WWTPRs, it is necessary to minimize the Co addition by examining the required amount of Co in detail and verifying the possibility of supplying Co by co-digestion with various wastes in rural areas to avoid an excessive cost increase.

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