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Essential role of trace elements in continuous anaerobic digestion of food waste

Lei Zhang[a], Wenxiang Ouyang[a], Aimin Li[a,*]

[a] *Key Laboratory of Industrial Ecology and Environmental Engineering, School of Environmental Science and Technology, Dalian University of Technology, Linggong Road 2, Dalian 116024, PR China*

Abstract

The purpose of this study is to explore the possibility of long-term sustainable anaerobic digestion of food waste in semi-continuous single-stage reactors by supplementing trace elements. Compared with the failure of anaerobic digestion of food waste after prolonged operation, a clearly enhancement of process performance was observed by supplementing a model trace element solution. Although the sustainable continuous anaerobic digestion of food waste could not be achieved by supplementing a model trace element solution, the correlation of process performance and trace element profile during continuous operating period revealed that the declining performance was highly likely due to the decreasing trace element concentrations, especially Co, Mo, Ni and Fe. This finding was expected to provide a promising strategy for sustainable anaerobic digestion of food waste.

Keywords: Anaerobic digestion; Food waste; Trace element; Volativle fatty acid

1. Introduction

Food waste is defined as the materials resulting from the processing, storage, preparation, cooking, handling, or food residual [1]. The typical food waste contained 69-93% of moisture, volatile solids to total solids ratio (VS/TS) of 85-96%, and carbon to nitrogen ratio (C/N) of 14.6-18.3 [1-3]. In China, a huge amount of food waste was generated, the untreated food waste may cause many environmental problems, such as contaminations of soil, water, and air during collection, transportation and storage due to its easy decomposition [2]. So, the treatment and disposal of food waste is challenge task.

* Corresponding author. Tel.: +86-0411-84707448; fax: +86-0411-84706679.
E-mail address: Leeam@dlut.edu.cn.

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Currently, there are diverse food waste reutilization and disposal choices available, such as landfill, incineration, animal feed, aerobic composting and anaerobic digestion. With the new issued environmental regulation, some disposal methods are more prohibited and becoming less desirable. For instance, Korean environmental law prohibited the sanitary landfill of food waste due to the large amount of leachate release which contaminates the ground and surface water body [4]. The high moisture content in the Korean food waste prevents effective incineration of wastes [2]. Although animal feeding is the most popular method for reutilization of the food waste, the concerns that some food wastes may contain biological and chemical contaminants may limit their reutilization in animal feeding [5]. Aerobic composting was another dominant method for treating food waste. The Korean food waste contained high level sodium [4, 6]. Since the salt content in compost is regulated in Korea, desalting through washing is necessary before compositing fertilizer production [6].

Food waste is a highly desirable substrate for anaerobic digestion with regards to its high biodegradability and methane yield. Heo et al. evaluated the biodegradability of a traditional Korean food consisted of boiled rice (10-15%), vegetables (65-70%), and meat and eggs (15-20%) and reported that after 40 days a methane yield of 489 mL/g VS could be obtained at 35 °C [7]. Zhang et al. also reported that the methane yield was determined to be 435 mL/g VS, respectively, after 28 days of digestion [1]. In addition, the nutrient content analysis showed that the food waste contained well balanced nutrients for anaerobic microorganisms [1].

Nowadays, anaerobic digestion of food waste is an intensive researched field, and many novel anaerobic configurations were developed and applied to treat the food waste, such as two stages, HASL (hybrid anaerobic solid-liquid) system [8-11]. Due to the technical complexity and high operating cost, so far, most of these processes are still under bench- or pilot- scale. In the practical application, single-phase treatment is, generally, the more predominant AD treatment applied in full scale [12]. However, there are rare reports about successfully operating single-stage anaerobic digestion of food waste alone at higher organic loading rates. El-Mashad et al. found the digester treating food waste was not stable at the OLR of 4.0 g VS/L·day or at the reduced OLR of 2.0 g VS/L·day, as indicated by high volatile fatty acid concentrations and low pH in the digester and low biogas production rate [13]. Climenhaga and Banks reported that at organic loading rate (OLR) of 1.45 g VS/L·day, an extended HRT of 180 days was needed to achieve a steady state with ammonia nitrogen exceeding 5.7 g/L and volatile fatty acid levels exceeding 15 g/L [14]. These low organic loading rates (OLR) render anaerobic digestion of food waste alone economically infeasible. Lee et al. and Cho et al. also reached a similar conclusion that single-stage anaerobic digestion is impossible with this easily degradable Korean food wastes [8, 15].

Trace elements were necessary nutrients for cell growth in microbiology. In the practical application, however, the issue of lack of trace element was ignored due to the diverse sources of trace element in the feedstock. The methanogens exhibited special requirement for some trace elements. In this study, we investigated the effect of trace element on anaerobic digestion of food waste by supplementing a trace element solution according to the literature report Gonzalez-Gil et al. [16].

2. Materials and methods

2.1. Food waste and inoculum

The food waste used in this study was collected from a Korean restaurant. The obtained food waste was crushed using an electrical kitchen blender (HMF-347, Hanil, Korea) and the resulting slurry food waste was sieved (No. 10) to remove coarse particles larger than 2 mm and kept at -18 ºC until used. The seed sludge used in this study was taken from a 20-L bench scale anaerobic reactor treating the piggery wastewater for more than 2 years. The operating conditions were hydraulic retention time (HRT) of 20-30 days and OLR of 1.0-2.3 g VS/L day. The volatile suspended solids (VSS) concentration of the seed culture was approximately 15 g/L.

2.2. Semi-continuous anaerobic digestion

Semi-continuous anaerobic digestion was carried out in a 500-mL Schott Duran bottle with a 150-mL working volume. Initially, the bottle was filled with different amount of seed sludge and substrate according to HRT. After sparged with nitrogen gas, the digester was capped with a rubber septum, and then inversely incubated in a shaking incubator at 37 ºC and 140 rpm, which provided the constant agitation and temperature. Incubation was proceeded in a semi-continuous mode with daily withdrawing and feeding of the same of amount, and the HRT was kept at 20 or 30 days. All the operations were made under nitrogen atmosphere to avoid contacting oxygen.

In this set of experiments, four reactors with a 200-mL working volume were run in parallel. Two were run as controls, which was fed with food waste only through experimental period. In another two trials, 0.05 mL model trace element solution was added in pulse model every five days. In addition to the model trace element solution, 4.0 g/L CaO was added into one of latter two, because CaO addition showed some positive effects in our batch anaerobic digestion of food. The model trace element solution was prepare according to the recipe reported by Gonzalez-Gil et al. [16]. Table 1 shows the composition and content of the trace element solution used in this study. The final desired concentrations of each trace element were also listed in Table 1. Besides the biogas sample and aqueous sample was taken for biogas composition and pH and volatile fatty acids (VFA), the seed sludge at beginning of experiments and the digestate on day 38 was collected and submitted to trace element analysis.

Table 1. Composition of trace element solution used in this study from Gonzalez-Gil et al. [16].

Component	Compound concentration in stock solution (mg/L)	Element concentration in stock solution (mg/L)	Designated element concentration in reactor (μg/L)
$FeCl_2 \cdot 4H_2O$	2000	563 as Fe	563 as Fe
H_3BO_3	50	8.9 as B	8.9 as B
$ZnCl_2$	50	23.9 as Zn	23.9 as Zn
$CuCl_2 \cdot 2H_2O$	38	14.2 as Cu	14.2 as Cu
$MnCl_2 \cdot 4H_2O$	500	138.9 as Mn	138.9 as Mn
$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$	50	27.2 as Mo	27.2 as Mo
$AlCl_3 \cdot 6H_2O$	90	10.1 as Al	10.1 as Al
$CoCl_2 \cdot 6H_2O$	2000	495.8 as Co	495.8 as Co
$NiCl_2 \cdot 6H_2O$	142	35.2 as Ni	35.2 as Ni
$Na_2SeO \cdot 5H_2O$	164	56.1 as Se	56.1 as Se
EDTA	1000		
Resazurine	200		
36% HCl	1 mL/L		

2.3. Analytical methods

Total solids (TS), volatile solids (VS), ammonia-nitrogen and total Kjeldahl nitrogen (TKN) were analyzed according to the Standard Methods. The pH values of the samples were determined using a pH meter (Orion, Model 370). Chemical oxygen demand (COD), total nitrogen (TN) and total phosphorus

(TP) were measured using Standard Methods (HACH digestion system and colorimetric spectrometer, HACH Company).

Biogas composition was analyzed using a gas chromatography (Hewlett-Packard 6890, Palo Alto, CA, USA) equipped with a thermal conductivity detector (TCD) and an HP-Plot Q column (30 m × 0.32 mm × 20 μm). The operational temperatures of injector, detector and column were kept at 60 °C, 250 °C and 35 °C, respectively. Helium was used as a carrier gas at a flow rate of 0.5 mL/min. Fifty microlitter of biogas sample was withdrawn for GC analysis.

The concentrations of individual volatile fatty acids were analyzed using the same GC equipped with a flame ionization detector (FID) and an HP-INNOWax column (30 m × 0.25 mm × 0.25 μm). The operational temperatures of injector, detector and column were kept at 220 °C, 250 °C and increased from 120 to 210 °C at a rate of 15 °C/min, respectively. Hydrogen and air were used as a flame gas at flow rates of 35 and 300 mL/min, respectively. Helium was used as a carrier gas at a flow rate of 24 mL/min. Ten microlitter of supernatant after centrifugation at 1750 g for 10 min was injected for GC analysis.

An element assay was performed using an Element Analysis Instrument (Flash EA1112, Thermo Electron SPA). Nitrogen, carbon, hydrogen, sulphur and oxygen was the target elements. The metal analysis was performed using an ion coupled plasma atomic emission spectrometer (ICP-AES) (Optima 4300DV, PerkinElmer, Boston, MA).

3. Results and discussion

Fig. 1 presents the performance profile of semi-continuous anaerobic digestion of food waste supplied with a model trace element solution as compared with two controls (anaerobic digestion of food waste only without trace element addition). In all cases, a stimulatory period (day 0-20) was observed, while the methane productivity was increasing. However, after the stimulatory period, quite different performances were observed. For two controls, the methane productivity continuously decreased from highest of 2.0 L/L·day to zero after around 15 days of operation. Concomitantly, a large amount of VFA was accumulated (around 15000 mg/L) on day 35, which resulted in the pH drop to lower than 6.0. The methane content also decreased from 55% to zero. These results indicated that, under these conditions: OLR of 3.6 g VS/L·day, HRT of 20 days and mesophilic temperature (37 °C), anaerobic digestion of food waste was not feasible. We had also attempted the pH control by adding lime, NaOH or $NaHCO_3$ for anaerobic digestion of the food waste alone. But the steady state was not be achieved, as indicated by the methane production rate progressively decreased after about two HRTs, even when pH values were remained in the optimum range (pH 6.8-7.5) (data not shown). In addition, the unstable process performances were also observed in anaerobic digestion of the food waste alone even at a reduced OLR (4.3 g COD/L·day) and a longer HRT (30 days) (data not shown). In many applications, the additives addition could effectively reduce inhibitory effects, thereby enhance the performance of anaerobic digestion[17]. The effects of $CaCl_2$ and bentonite addition on anaerobic digestion of food waste were examined in semi-continuous experiments. The additive addition did not significantly enhance anaerobic digestion of food waste in semi-continuous operation, and the steady states could not be achieved after prolong operating period, and significant VFA accumulations were observed (data not shown). El-Mashad and Zhang reported that, in CSTR system, a steady state of anaerobic digestion of food waste at OLR of 4.0 g VS/L·day could not be reached [18]. Climenhaga and Banks also reported that anaerobic digestion of food waste was successful only at long retention time (HRT 100 days) and lower organic loading rate (1.0 g VS/L·day) with high level VFA concentration [14]. These results further confirmed that anaerobic digestion of food waste was not successful in single-stage reactors.

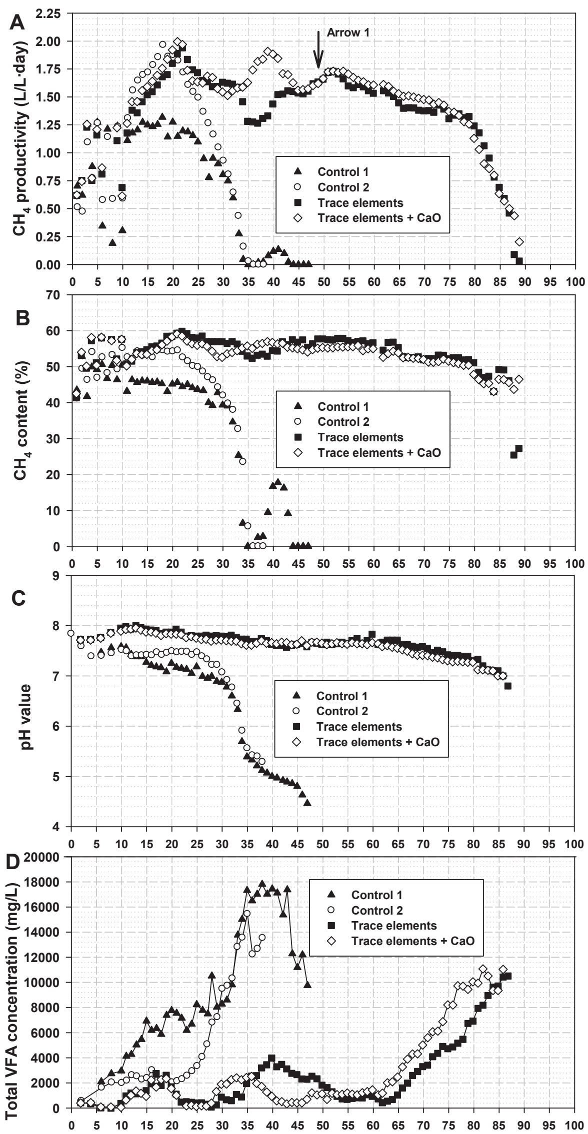

Fig. 1. Performance profile of single-stage anaerobic digestion of food waste in semi-continuous anaerobic digestion of food waste in the presence or absence of a model trace element solution. Arrow 1 (↓) indicates the last time point for supplementing trace element solution in the case of "Trace elements + CaO".

Interestingly, the two cases fed with a model trace element solution showed superior performance in terms of higher and stable methane productivity, high methane content, constant pH value, and relative lower VFA level (Fig. 1). In the initial period, both trace element supplemented digesters showed stable conditions. During the period of day 50-60, it seems that temporal pseudo-steady states were achieved in both cases. These results clearly revealed the important role of trace elements in enhancement of anaerobic digestion of food waste.

Unfortunately, with the prolonged operation, noticeable decreases of methane productivity and methane content were observed after day 60, even though pH was still in the optimal range. Concomitantly, the total VFA concentrations rose from 1500 mg/L and then to 10000 mg/L (see Fig. 1C). All the volatile fatty acids (C2-C5) were showed in Fig. 2, which was expected to provide more deep insights on anaerobic metabolism. Surprisingly, acetate concentration did not significantly increase as compared with propionate and iso-butyrate and iso-valerate were accumulated in large amount. The accumulation of propionate and iso-form fatty acids indicated the upset of anaerobic process [19]. These results also suggested that the acetogenesis might be limited step other than aceticlastic methanogenesis during the biomethanization of food waste.

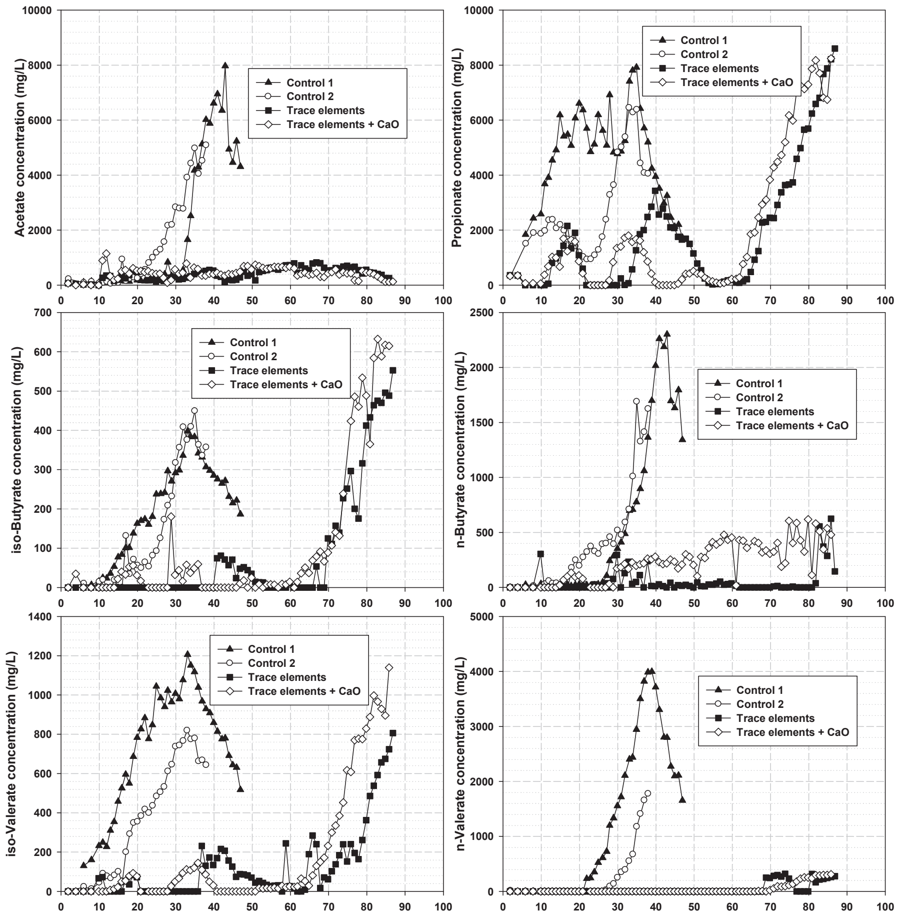

Fig. 2. Individual VFA profile in the presence or absence of a model trace element solution.

In order to elucidate the different performances in the presence and absence of a model trace element, the selected trace elements prolife was predicated and depicted in Fig. 3 based on the analytical data in the seeding sludge and calculation. The trace element contents in the cases of Control 2 and trace element and trace element + CaO on day 30 were quantified for confirmation of predicted the values. The selected trace elements included Co, Ni, Mo and Fe, which might be insufficient for stable and efficient anaerobic digestion of the food waste alone due to the relative lower concentration as compared with seeding sludge. It was also considered that the important roles of these trace elements (Co, Ni, Mo, Fe) for activating and maintaining enzyme activities of anaerobic microorganisms [4, 20-23].

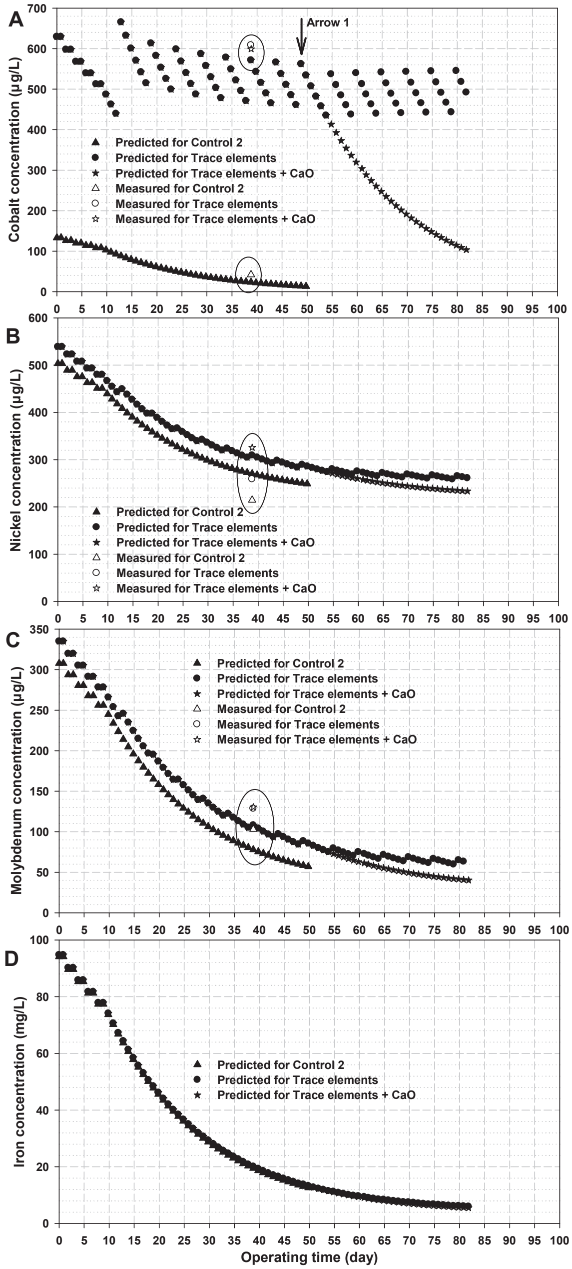

Fig. 3. Trace element profile of predicated and measured values in semi-continuous anaerobic digestion of food waste in the presence or absence of a model trace element solution. Arrow 1 (↓) indicates the last time point for supplementing trace element solution in the case of "Trace elements + CaO".

Table 2. Concentrations of trace elements in the digestate on day 38 (ppb = μg/kg in wet basis).

Trace element	Control 2 (Food waste only)	Trace elements	Trace elements + CaO
Cadmium (Cd)	nd [a]	nd	nd
Chromium (Cr)	93.3	91.7	177.0
Cobalt (Co)	41.9	608.3	598.9
Copper (Cu)	7976.2	7218.8	7069.4
Lead (Pb)	233.0	121.9	121.2
Manganese (Mn)	5910.7	5071.2	4886.9
Molybdenum (Mo)	103.0	128.5	130.3
Nickel (Ni)	214.3	258.8	325.8
Silver (Ag)	nd	nd	nd
Tungsten (W)	nd	nd	nd
Zinc (Zn)	84014.2	68636.2	68581.4

[a] lower than detection limit, and the detection limit: Ag, Cd, Co > 30 ppb, W > 300 μg/L
[b] All samples were analyzed three times and averaged.

Comparing the measured values with the predicted ones, it can be seen that changes of the trace element concentration followed the values predicted by the mass balance during experimental period (Fig. 3 and Table 2). During the continuous operation, the original active biomass and trace element was washed out in CSTR. Due to the low trace element content in the feeding food waste, resulting in the level of the trace element in digestate decreased. For example, the Co concentration in two control reactors was decreased from 140 ppb to 30 ppb on day 35, while showing negligible methane productivity. The low Co concentration was reported as the limiting level resulting in process disturbance [23]. By contrast, the two cases with a model trace element addition, due to the relative high Co content in the recipe, the Co concentration was even higher that the value in seeding sludge, which was considered enough. For other trace elements, like Mo, Ni and Fe, due to the relative lower content in the model trace element solution, on day 35, those differences seems not big enough to cause significant different between process performance. Based on these results and discussion, it is highly likely that the difference of Co concentration caused the different process performance after 35 days of operation.

The upset of two model trace element supplemented cases was found, which might be explained by the trace element profile as shown in Fig. 3. Although the Co concentrations were maintained in high level (higher than 130 ppb in seeding sludge), due to the relative lower trace element content in the model trace element solution, the concentrations of other trace elements continuously decreased and reached a relative lower level, like Mo, Ni and Fe. The limitation of trace element other than Co might be responsible for the two cases with the model trace element solution. The different individual VFA accumulating patent, for controls, acetate accumulated together with propionate, and other longer VFAs, for model trace element supplemented cases, the no obvious acetate accumulation other than propionate and other iso-form VFAs. The different patent might be due to Co level in these upset. This speculation was supported by Florencio et al., who stated that the deprivation of Co in methanogenic systems was reported to cause loss of acetate degradation capacity for the medium, resulting in wash-out of the acetotrophic microorganisms [24].

Originally, the model trace element solution was adopted to prepare synthesized wastewater for UASB reactor [16]. Except for Co, most the trace elements in the model solution were greatly lower than that in inoculum. In this type reactor, the trace elements could be adsorbed by the biomass, and accumulated in the system [25]. On the contrary, in the continuous experiment (CSTR), the active biomass was washed out, and the growth of biomass is a necessary step to maintain the robust methanogenic activity. When trace element was the limiting factor, due to many trace elements were included in co-enzymes, for example, Co, Mo, Ni and Fe, the cell synthesis was seriously affected by the deficiency, or cell become more sensitive to inhibitory substances. This could be more important for highly biodegradable food waste in CSTR at the conditions of high OLR and short HRT.

4. Conclusions

In summary, it was concluded that anaerobic digestion of food waste was not sustainable in semi-continuous single-stage reactor. By supplementing a model trace element solution, a clearly enhancement of anaerobic digestion of food waste was observed. Although the sustainable continuous anaerobic digestion of food waste could not be achieved by supplementing a model trace element, the correlation of process performance and trace element profile during continuous operating period revealed that the declining performance was highly likely due to the decreasing trace element concentrations, especially Co, Mo, Ni and Fe.

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