Waste Management xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Waste Management

journal homepage: www.elsevier.com/locate/wasman

Biochemical methane potential from sewage sludge: Effect of an aerobic pretreatment and fly ash addition as source of trace elements

César Huiliñir [a,*], Paula Pinto-Villegas [a], Alejandra Castillo [a], Silvio Montalvo [a], Lorna Guerrero [b]

[a] Departamento de Ingeniería Química, Universidad de Santiago de Chile, Santiago, Chile
[b] Departamento de Ingeniería Química y Ambiental, Universidad Técnica Federico Santa Marıa, Valparaıso, Chile

ARTICLE INFO

Article history:
Received 12 October 2016
Revised 8 February 2017
Accepted 13 March 2017
Available online xxxx

Keywords:
Aerobic pretreatment
Fly ash
Combined effect
Methane generation
Mixed sludge

ABSTRACT

The effect of aerobic pretreatment and fly ash addition on the production of methane from mixed sludge is studied. Three assays with pretreated and not pretreated mixed sludge in the presence of fly ash (concentrations of 0, 10, 25, 50, 250 and 500 mg/L) were run at mesophilic condition. It was found that the combined use of aerobic pretreatment and fly ash addition increases methane production up to 70% when the fly ash concentrations were lower than 50 mg/L, while concentrations higher than 250 mg/L cause up to 11% decrease of methane production. For the anaerobic treatment of mixed sludge without pretreatment, the fly ash improved methane generation at all the concentrations studied, with a maximum of 56%. The removal of volatile solids does not show an improvement compared to the separate use of an aerobic pre-treatment and fly ash addition. Therefore, the combined use of the aerobic pre-treatment and fly ash addition improves only the production of methane.

1. Introduction

Nowadays the continuous production of large amounts of sewage sludge is becoming a worldwide environmental problem (Zhao et al., 2010). Thus, emphasis in sludge management has shifted from its disposal to the beneficial use of itself and its products.

Among sludge treatment technologies, anaerobic digestion (AD) is a well-established and proven method. It transforms organic matter into biogas (60–70 vol% of methane, CH_4), destroys most of the pathogens present in the sludge, limits possible odor problems associated with residual putrescible matter, and reduces the amount of final sludge solids for disposal (Appels et al., 2008). However, the application of AD to sewage sludge is often limited by very long retention times (20–30 days) and a low overall degradation efficiency of the organic dry solids (30–50%). These limiting factors are generally associated with the hydrolysis stage (Tiehm et al., 2001). In order to improve this step, several sewage sludge pretreatments have been proposed, which include biological (largely thermal phase anaerobic), thermal hydrolysis, mechanical (ultrasound, high pressure, and lysis), chemical with oxidation and alkali treatments (Ennouri et al., 2016; Neumann et al., 2016). Among the biological methods, micro-aerobic pretreatment has been proposed (Girotto et al., 2016; Mata-Alvarez et al., 2000; Montalvo et al., 2016). It was reported that the limited oxygen supply caused an increase of the hydrolysis rates in the case of complex organic matter in batch tests (Johansen and Bakke, 2006), an improvement in digester performance, and a reduction of volatile fatty acid concentration (Diaz et al., 2011). Furthermore, the micro-aerobic step can be directly performed in the same unit prior to anaerobic digestion. This pretreatment has been used successfully to treat sewage sludge at the laboratory scale (Montalvo et al., 2016), increasing methane generation as well as volatile solids degradation.

In addition to pretreatment challenges, existing trace elements are insufficient for anaerobic microorganisms when solid waste, such as organic fractions of municipal solid waste or sewage sludge, is used alone as a feedstock for anaerobic digestion (Moestedt et al., 2016). The literature has emphasized the importance of supplementing trace elements for maximum methanogenic activity (Choong et al., 2016; Romero-Guiza et al., 2016; Thanh et al., 2016). Although the importance of this parameter is clear, its control is limited mainly by the high cost of the trace salts used for this purpose. In this context, the use of a cheaper source of trace elements could help control this parameter without additional expenses (Huilinir et al., 2015). A source of trace metals is fly ash (FA), which has been applied to the anaerobic digestion of municipal solid waste (Lo et al., 2010; Romero-Guiza et al., 2016) and recently to sewage sludge coming from the pulp and paper industry (Huilinir et al., 2015). The inclusion of fly ash in both cases

* Corresponding author.
E-mail address: cesar.huilinir@usach.cl (C. Huiliñir).

http://dx.doi.org/10.1016/j.wasman.2017.03.023
0956-053X/

increases methane production and also reduces the concentration of volatile solids (VS). However, the use of fly ash in the anaerobic digestion of sewage sludge has not been presented yet. Therefore, an analysis of the effect of fly ash on the anaerobic digestion of sewage sludge in the presence of trace elements is necessary.

Even though the use of a pretreatment and the use of fly ash as trace elements source had shown positive effects separately, the use of a combination both in the same substrate has not been studied yet. Only Mansour et al. (2012) compared the effect of an aerobic pretreatment and addition of bottom ashes (as pre-treatment) separately on anaerobic digestion of municipal solid waste, focusing on the kinetic of the process, without using the aerobic pretreatment and the bottom ashes combined. On the other hand, Liu et al. (2015) studying the anaerobic digestion of corn stover indicated that NaOH pretreatment and the addition of trace elements (Fe, Co and Ni) improve the biodegradability of corn stover and enhance biomethane production, and the present study is the only one that has dealt with the combined effect of pretreatment and trace element supplementation. Thus, the aim of the present work was to determine the combined effect of an aerobic pretreatment and the addition of fly ash as trace element source on methane production and VS reduction in a batch system. To the best of our knowledge, the combined effect of an aerobic pretreatment and fly ash as trace element source have not been reported previously. The effect of fly ash as the sole source of trace elements was also determined.

2. Materials and methods

2.1. Experimental setup

A 1-L glass reactor operating with an effective volume of 900 mL was used for the aerobic pretreatment. In each pretreatment air was injected continuously into the bioreactors by mini-compressors (0–10 l per minute, lpm) and distributed by small diffusers. The air flow rate (AFR) was calibrated before the pretreatment assays using the volume variation method. The conditions used for pretreatment were 0.35 vvm (air volume per liquid volume per minute) for 48 h at 35 °C, according to Montalvo et al. (2016). After aerobic pretreatment, pretreated sludge was fed to several batch anaerobic digesters of 280 mL of total volume.

For the anaerobic process, every experimental run considered the installation of twelve 280-mL anaerobic mini-digesters with effective operating volumes of 250 mL each. Eight of them were meant for measuring parameters in the liquid phase (chemical oxygen demand (COD) and volatile solids, and four were used to measure methane by liquid displacement according to Huilinir et al. (2015). The mini-digesters operated in discontinuous mode for 43 days, at which time the system reached a steady state. The digesters were stirred manually once or twice per day before reading the volume displaced by methane.

The volume of the digesters was completed with distilled water, they were stoppered with rubber stoppers and sealed with white silicone to ensure anaerobiosis, and they were covered with aluminum foil to prevent the growth of photosynthetic organisms. All the runs were carried out in duplicate.

2.2. Inocula, substrate and experimental design

The inoculum was obtained from an anaerobic reactor of the La Farfana Water Treatment Plant of Aguas Andinas in Santiago, Chile. The inoculum had 0.59 ± 0.01 g VS/g of total solids (TS), 2.2 ± 0.42 g of soluble COD/L, and a pH of 7.23 ± 0.12. The substrate was mixed sludge (a mixture of primary (60%) and secondary (40%) sludge) also from the La Farfana Water Treatment Plant. The substrate presented a ratio of 0.76 ± 0.01 g VS/g TS, 5.87 ± 0.87 g/L of soluble COD, and a pH of 5.69 ± 0.32. The main trace elements found in the mixed sludge were: Cd (1.2 mg/kg), Zn (3.19 mg/kg), Fe (1.80 mg/kg), Al (1.58 mg/kg), Zn (3.19 mg/kg), Cr (1.80 mg/kg), Cu (2.06 mg/kg), Ni (2.24 mg/kg), Mn (2.51 mg/kg), Ba (2.09 mg/kg), Co (0.8 mg/kg), B (0.74 mg/kg), Pb (2.09 mg/kg) and Se (2.77 mg/kg).

The sewage sludge was refrigerated at 4 °C for no more than 3 days until its use.

The fly ash was obtained from a thermoelectric power plant, with a particle diameter between 0.12 and 0.2 mm. The ash was taken from electrostatic precipitators used to collect particulate matter generated by the combustion of bituminous coal, placed before the gaseous effluents leave the plant. The main trace elements found in them were Fe (4.24 g/kg), Al (1.37 g/kg), Zn (5.8 mg/kg), Cr (3.93 mg/kg), Cu (2.32 mg/kg), Ni (3.85 mg/kg), Mn (37.7 mg/kg), V (26.4 mg/kg), Ba (64 mg/kg), Co (0.9 mg/kg), and B (25.4 mg/kg). Since the fly ash used in all the experiments came from the same sample obtained *in situ* from the thermoelectric power plant, the mass of trace elements used in all the experiments was proportional to the mass reported here.

In each anaerobic treatment, the substrate/inoculum ratio (F/I) was 1.5 g VS substrate/g VS inoculum according to Tomei et al. (2008). For each assay two weekly measurements of soluble and total COD and suspended VS were made in duplicate. The temperature in the container was kept at 35 °C using three automatically controlled aquarium heaters. Three assays were made in order to see the effect of fly ash and aerobic pretreatment, both separately and combined, as shown in Table 1. The first and second assays were ran on pretreated sludge (P) and different fly ash (FA) concentrations. The third assay was only meant to see the effect of fly ash on the anaerobic digestion.

2.3. Chemical analyses

The following parameters were determined: soluble COD and VS. COD was measured by a colorimetric method according to APHA (2012) and volatile solids (VS) were also measured according to APHA (2012).

Methane production was measured by volumetric displacement, connecting inverted Falcon tubes containing 3% w/w NaOH solution to remove CO_2 and H_2S as main impurities from the biogas, displacing only the methane volume. The resulting biogas travels through the flexible tubing to the Falcon tube, where it comes in contact with the NaOH solution, forming Na_2CO_3 and Na_2S and bubbling only methane. The methane collected in the

Table 1
Experimental conditions used in the anaerobic digestion assays.

Assay 1	Assay 2	Assay 3
Not pretreated, No FA[*] (NP-NFA1)	Not pretreated, No FA (NP-NFA2)	Not pretreated, No FA (NP-NFA3)
Pretreated, No FA (P-NFA1)	Pretreated, No FA (P-NFA2)	Pretreated, No FA (P-NFA3)
–	Pretreated, 10 mg/L FA (P-10)	Not pretreated, 10 mg/L FA (NP-10)
–	Pretreated, 25 mg/L FA (P-25)	Not pretreated, 25 mg/L FA (NP-25)
–	Pretreated, 50 mg FA/L (P-50)	Not pretreated, 50 mg/L FA (NP-50)
Pretreated, 250 mg/L FA (P-250)	–	Not pretreated, 250 mg/L FA (NP-250)
Pretreated, 500 mg/L FA (P-250)	–	Not pretreated, 500 mg/L FA (NP-500)

[*] FA: Fly Ash.