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Evaluation of Heavy Metals Influence on Biogas Production

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Heavy metals play a very significant role in the performance and stability of biogas digesters, which are operated with organic fraction of municipal solid wastes or any other type of organic waste. For this reason this paper tries to evaluate the impact of heavy metals on biogas yield and quality. Anaerobic digestion of sewage sludge and rapeseed cake mixture has been carried out for 14 days. The obtained data show that the greatest negative impact on biogas production was made by zinc additive. Meanwhile, methane content in biogas varied from 64.5 to 70%.

Keywords: *biogas, heavy metal, methane, digestion, organic matter*.

1. Introduction

An increased rate of sewage sludge production in developing countries like Lithuania is always a great matter of concern whenever the aspect of final disposal is taken into account. At the moment, most of the municipalities in Lithuania are trying to adopt landfilling and biocomposting as main alternatives for the final disposal of sewage sludge. These methods should be viewed with reserve, considering that sewage sludge potentially carries chemical pollutants, such as heavy metals and persistent organic pollutants.

Anaerobic digestion is the most widely used treatment process for stabilization of resultant sewage sludge from a municipal wastewater treatment plant (Shehu et al. 2012). Anaerobic digestion has been recognized as an efficient technology for treatment of sewage sludge, offering many environmental and economic benefits, such as sludge stabilization, sludge volume reduction, nutrient recycling, and energy production (Bougrier et al. 2005; Luostarinen et al. 2009). Recently, many researchers have investigated various techniques of sludge digestion. Among those reported are chemical methods which use alkaline and acid applications (Li et al. 2012; Chi et al. 2011), mechanical methods which use ultrasound, high pressure homogenizer (Zhang et al. 2012; Jiang et al. 2010; Chang et al. 2011), thermal methods at low and high temperatures (Wang et al. 2009; Carrère et al. 2010). Whereas mechanical and chemical pre-treatment methods are well studied and commonly used in practice to increase methane yields from recalcitrant biomass, little is known about the influence of heavy metals on biogas yield and its composition.

Availability of heavy metals plays a very significant role in the performance and stability of biogas digesters, which are operated with energy crops, organic fraction of municipal solid wastes or any other type of organic waste (Demirel and Scherer 2011). Heavy metals are present in significant concentrations in some industrial wastewaters and municipal sludge, and are often the leading cause for the upset of the wastewater treatment process (Peng et al. 2011; Fang and Chan 1997). Heavy metals can be stimulatory, inhibitory, or even toxic in biochemical reactions depending on their concentrations. A trace level of many metals is required for activation or functioning of many enzymes and co-enzymes (Demirel and Scherer 2011). Excessive amounts, however, can lead to inhibition or toxicity. This is mostly due to the chemical binding of heavy metals to the enzymes, resulting in disruption of enzyme structure and activities (Vallee and Ulmer 1972). Nickel and cobalt are essential co-factors of enzymes involved in the anaerobic digestion of biomass (Goodwin et al. 1990; Zandvoort et al. 2006).

Inhibition of heavy metals commonly found in the electroplating effluent inhibits bioactivity of H_2-producing sludge in the following order: Cu (most toxic) >>Ni~ Zn > Cr > Cd > Pb (least toxic).

Co, Fe, Mo, Ni and Se are important to the stability of anaerobic digestion as micronutrients are essential for the growth and metabolism of anaerobes (Ilangovan and Noyola 1993; Kayhanian and Rich 1995). Critical nickel and cobalt concentrations of 0.6 mg/kg and 0.02 mg/kg FM, respectively, have been reported (Pobeheim et al. 2010; Jarvis et al. 1997). In addition, bioavailability of these obviously essential trace elements is significantly lower than their total content in the fermentation medium (Oleszkiewicz and Sharma 1990). The presence of heavy metals may result in the inhibition and failure of sewage digestion, for example. Several studies have demonstrated the toxic effects of heavy metals on anaerobic digestion (Lin 1993; Yenigun et al. 1996).

This study has been conducted for the purpose of investigating the effect of heavy metals on biogas production using anaerobic sewage sludge microflora in the presence of Ni, Cu, and Zn ions. For this reason, efficiency and toxicity density of biogas production have been analyzed.

2. Materials and methods

Heavy metal analysis

Total amount of heavy metals in sewage sludge was determined using concentrated nitric acid. 0.2000 g of air-dry sewage sludge was accurately added into a 50 ml polytetrafluorethene crucible and digested with 5 ml conc. nitric acid in a closed PTFE vessel on a heat plate (block) at the temperature of 170 ^{o}C. Heating stopped after 6 h, and the crucible was kept overnight, allowing a digest to cool slowly till the room temperature. After that, the residue was centrifuged and diluted to 50 ml. The solution was then analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES) with the spectrometer Perkin Elmer “Optima 7000”. The obtained data were calculated in $mg \cdot kg^{-1}$ of dry matter.

Inoculum and defined model substrate

Freshly digested sewage sludge (10 L) used in this investigation was collected at Kaunas Wastewater Treatment Plant (WTP), which processes the majority of domestic and industrial wastewater of Kaunas city, Lithuania. The samples were transferred to the laboratory in a clean 15 L plastic barrel within 4 h. Sewage sludge was used for filling up the biogas production reactor. Rapeseed cake was used as feed material for biogas production. Rape seed cake has been found to be one of the most suitable substrates that enhance gas production (Ramachandran et al. 2007). Moisture of the samples was determined by oven-drying at 105 ^{o}C for 24 h.

Trace elements

Individual aqueous solutions of Ni, Zn and Cu were prepared from their sulfate salts and a required amount of solution was taken for setting a desirable concentration of metal in each reactor. The amount of heavy metals in nine medium samples was adjusted respectively to 1200, 2400 and 4800 mg of nickel, 4000, 6000 and 8000 mg of zinc and 2000, 3500 and 5000 mg of copper per kilogram of sewage sludge and rapeseed cake mixtures (dry matter). All chemicals used in these experiments were analytical reagent grade and purchased from Roth and Merck chemicals, Germany.

Reactor’s set up and operation

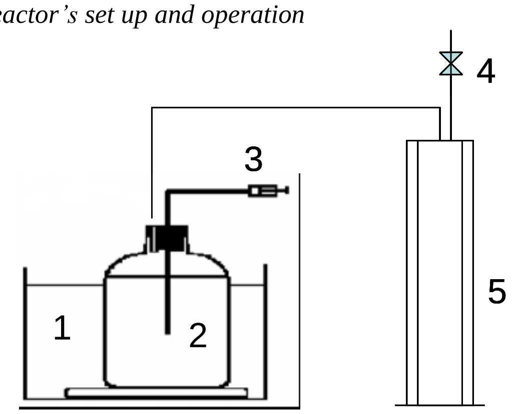

Fig. 1. Experimental device
Here: 1. Bath thermostat; 2. Reactor; 3. Syringe for flushing with nitrogen gas; 4. Syringe for sampling; 5. Biogas holder.

Anaerobic digestion was carried out in a 1000 ml capacity dark glass bioreactor, hermetically closed, Figure 1. Twelve such reactors were operated at 36 ^{o}C (±1 ^{o}C) in a bath thermostat. The reactors were initially filled with 400 g of the inoculum sludge with a concentration of 5% DS and 5 grams of rapeseed cake. Then, the required amount of described heavy metal solutions was specifically added. Thereafter, the fermenters were flushed with nitrogen gas to obtain anaerobic conditions. The fermentation process was analyzed for 14 days. The reactor’s content was mixed manually by shaking fermenters twice a day. Reactors were equipped with a distributor cap to remove generated biogas from fermenters. Fermenters were linked to gas holders for continuous measurement of produced biogas. Furthermore, a 1 L gas-sampling bag was connected to gas holders to collect biogas for determining gas quality.

The methane content in biogas was detected with the gas analyzer GA 2000 PLUS (Geotech, England). Experiments with nickel, zinc, and copper limitation were carried out in triplicate and in analysis data presented in this paper were obtained from the average of triplicate experiments. Samples were collected at 1–3-day intervals.

3. Results and Discussion

Heavy metal concentration in sewage sludge

Heavy metal concentrations in sludge samples of this study, data from the literature concerned, and comparison to the recommended maximum heavy metal levels for land filling/agricultural use in Lithuania and EU legislation are given in Table 1. In general, all samples of sludge contained relatively higher concentrations of Zn and Cu, but lower concentrations of Cr, Ni, Pb, and Cd. The concentration of Zn and Cd in sludge samples from all listed sources exceeded the permissible limits, restricting their use in agriculture, according to the Lithuanian legislation. High concentrations of Zn can be related to the treatment (coagulation and sterilization) of sludge in the WTP. Sludge samples show an increase in the concentrations of heavy metals in the following order: Cd ~ Ni< Cr < Pb < Cu < Zn.

Table 1. Heavy metal content of sewage sludge of this work compared to that found in different works and to the requirements of Lithuanian and the EU legislations for heavy metals in sludge for agricultural use

Source	Heavy metal concentration, mg/kg					
	Cr	Cd	Ni	Pb	Cu	Zn
Sewage sludge from Kaunas (Lithuania)	196.00	3.63	44.06	56.00	201.73	1503
Sewage sludge from Torun (Poland) (Kosobucki et al. 2008)	17.54	4.26	138.32	31.78	23.40	-
Sewage sludge from Hangzhou (China) (Peng et al. 2011)	-	-	-	35.13	296.4	3756.2
Sewage sludge from Guangzhou (China) (Deng et al. 2009)	99	2	-	751	286	1258
Sewage sludge from Rio de Janeiro (Brazil) (Bridle et al. 1990)	615	44	198	267	1187	1999
LAND 20:2005 (I class sewage sludge)	<140	<1,5	<50	<140	<75	<300
EC Directive 86/278/EEC	–	20–40	300–400	750–1200	1000–1750	2500–4000

According to the Lithuanian legislation, a likely final deposition of such sewage sludge would be landfilling. Therefore, it is useful first of all to gain energy from such sewage sludge by producing biogas. At the same time, the amount of landfilled sewage sludge will be reduced. Biogas is an alternative energy resource which could be obtained from **o**rganic materials, waste, as well as sewage sludge. Therefore, it is an important task to get more information on the influence of heavy metals on biogas quantity and quality.

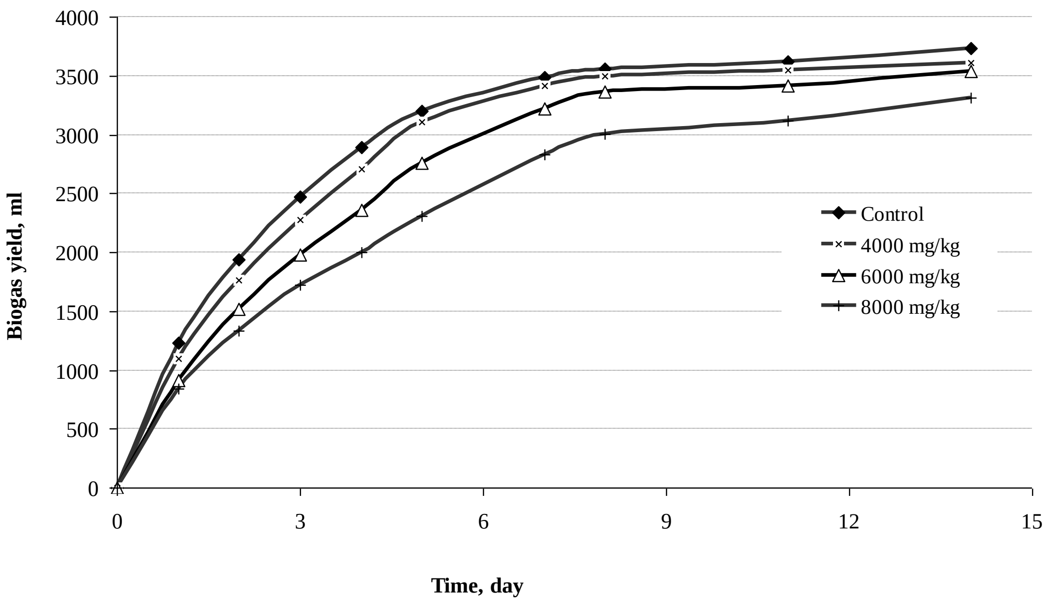

Fig.2. Biogas yield during 14 days of anaerobic digestion at various Zn(II) concentrations

Effect of Zn^{2+} on biogas production

Stimulation or inhibition was quantified by determining the dose of metal ions that caused an increase or decrease in biogas production over a fixed period of 14 days compared to the control. As shown in Figure 2, biogas yield was found to be different among different contents of heavy metals in the substrate. An increased Zn content in the mixtures has a negative impact on biogas production. It is evident that biogas yield was the lowest (3300 ml) when heavy metal concentration increased to 8000 mg/kg. A decrease in biogas yield at a higher range of heavy metal could be attributed to a higher phytotoxity rate of bacteria. Nevertheless, all reactors showed a rapid increase in biogas production from the beginning of the fermentation process. The main amount of biogas was produced during the first 7 days of an experiment (80-90%). Furthermore, after 14 days of anaerobical digestion an approximately 12% lower biogas amount was observed in the reactor containing the highest studied amount of zinc compared to the control reactor without any addition of heavy metal.

Effect of Ni2+ on biogas production

Figure 3 depicts the biogas yield as a function of fermentation duration at the constant temperature of 36 °C and a different amount of nickel. Nickel was added with increasing concentrations up to the maximum of 4800 mg/kg. The results presented in Figure 3 show a trend toward decreasing the biogas yield with an increase in nickel amount rates from 1200 to 4800 mg/kg. It seems that nickel with the amounts found in sewage sludge does not produce a negative effect on the fermentation process.

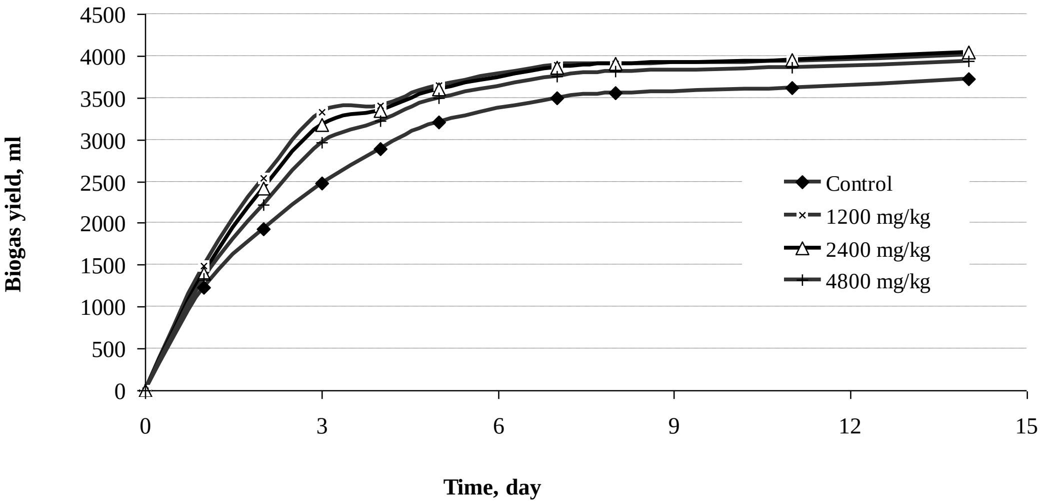

Fig.3. Biogas yield during 14 days of anaerobic digestion at various Ni(II) concentrations

Effect of Cu^{2+} on biogas production

To assess the possibility of using sewage sludge containing a high copper concentration in biogas digesters, the study was carried out by using 2000-5000 mg/kg additive of copper. Surprisingly, during the period of fermentation no significant differences in biogas production and methane yield were detected between the reactors containing various added concentrations of copper and the control (Figure 4). Moreover, it seems that the tested amounts of copper are sufficient for a stable fermentation of the defined model substrate up to a loading rate up to 5000 mg/kg.

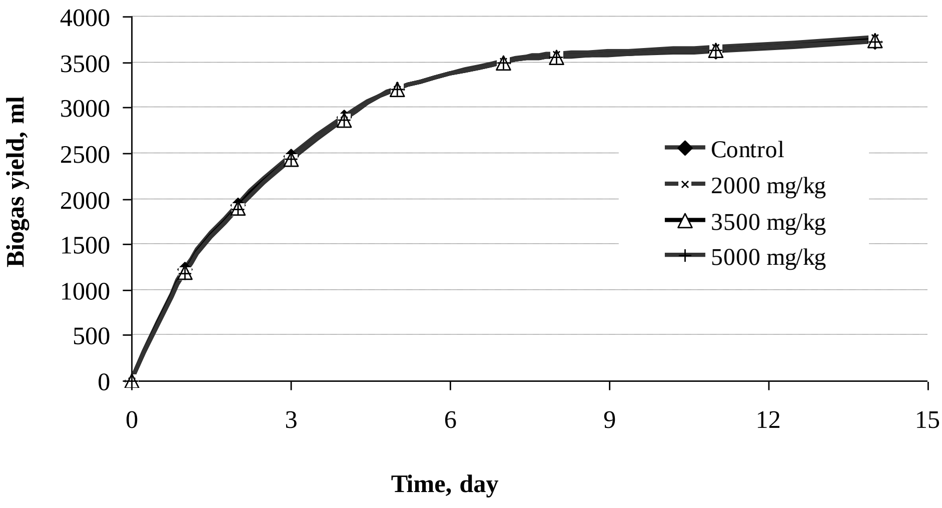

Fig.4. Biogas yield during 14 days of anaerobic digestion at various Cu(II) concentrations

Effect of heavy metal on methane production

Biogas production changes with the change in the waste composition, as it depends on the available organic biodegradable carbon content. Typical composition of biogas is: CH_4 (50-75 %), CO_2 (25-45 %), N_2 (0-10 %), H_2 (1-2 %), H_2S (0-0,5 %), O_2 (0-2 %) (Karellas et al. 2010). The maximum methane content (70%) was recorded in biogas produced after 14 days of incubation of the mixture containing Zn (4000 mg/kg) and Cu (5000 mg/kg) (Table 2). The biggest influence on the methane content was found after addition of nickel. The minimum methane content of biogas was found by using Ni with the concentration of 2400 mg/kg.

Table 2. Qualitative comparison of methane generation by using different amounts of heavy metals after 14 days of incubation at 36 ±1 °C

Zn		Ni		Cu	
Concentration, mg/kg	CH4, %	Concentration, mg/kg	CH4, %	Concentration, mg/kg	CH4, %
Control	68.5	Control	68.5	Control	68.5
4000	70.0	1200	65.0	2000	69.0
6000	69.0	2400	64.5	3500	69.0
8000	69.5	4800	66.0	5000	70.0

No variation of the methane content in biogas was observed with an increase in heavy metal content in the mixtures.

Nevertheless, to evaluate these lab scale experiments, the tests in full-scale biogas plants should be conducted. In addition, investigations should be extended to the other elements which could be limiting in certain substrates the effect on which they may be interdependent

4. Conclusions

In anaerobic semi-continuous fermentations of a defined model substrate for sewage sludge and rapeseed cake, high concentrations of zinc show a negative impact on process stability and biogas production. After 14 days of anaerobic digestion an approximately 12% lower biogas amount was observed in the reactor containing the highest amount of zinc compared to the control reactor containing no addition of heavy metal. Nickel whose amounts could be found in sewage sludge does not produce any negative effect on the fermentation process. Additive of nickel has increased the biogas production. However, copper additive has not enhanced further biogas production. After 14 days of anaerobic digestion of sewage sludge and rapeseed cake mixture the methane content in biogas has varied from 64.5 to 70%. The results obtained need to be confirmed in long term experiments.

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Sunkiųjų metalų įtakos biodujų gamybai įvertinimas

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(gauta 2012 m. liepos mėn.; atiduota spaudai 2012 m. gruodžio mėn.)

Sunkieji metalai turi didelę įtaką biodujų reaktorių, kuriame skaidomos įvairios kilmės organinės medžiagos, organinės atliekos, efektyvumui ir stabilumui. Straipsnyje pateikiami tyrimų rezultatai, kurie rodo, kokią įtaką biodujų išeigai ir kokybei daro sunkieji metalai. Anaerobinis nuotekų dumblo ir rapsų išspaudų mišinio skaidymas vykdytas 14 dienų. Tyrimas parodė, kad didžiausią neigiamą įtaką biodujų susidarymui turėjo cinko priedas. Metano kiekis biodujose svyravo nedaug: 64,5–70 proc.