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Research Article

Methane yield of paper industry waste in the presence of two compounds from alcohol and aldehyde groups during thermophilic anaerobic digestion

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ABSTRACT

In this study, effect of two chemical compounds (i.e., 1-octanol and hexanal) respectively from the alcohol and aldehyde groups on thermophilic (55±2 °C) anaerobic process digesting the waste produced at a paper industry was investigated. In this scope, possible inhibition was monitored by the cumulative methane (CH_4) yields in the batch reactors digesting the paper waste as the feedstock at concentrations of 0.005%, 0.05%, and 0.5% for each compound. Comparing the effects of the two different groups with the control reactor having only the paper waste as the substrate, the results revealed that adding 1-octanol and hexanal up to 0.05% concentrations had some synergistic effect on biogas yield (i.e., from 3% to 12% enhancement). Accordingly, the highest methane yields were 550 and 567 mL/g-VS_{fed}, respectively on average in the presence of 1-octanol and hexanal at a concentration of 0.05% while the cumulative methane yield was observed as 490 mL/g-VS_{fed} for the control reactor. With the exception of 1-octanol at 0.5%, adding both compounds at each investigated concentration was beneficial for the digestion in the batch process. Therefore, the selected alcohol and aldehyde sources did not cause the expected detrimental effect on the methanogens even at the maximum amounts added in this study. Nevertheless, since the effect of the chemical compounds on methane generation has been generally concentration-dependent, the toxic effects of 1-octanol and hexanal would be better observed at higher concentrations (>0.5%), especially when their threshold levels are exceeded in anaerobic reactors digesting paper wastes.

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INTRODUCTION

Depending on the total solids (TS) content of the substrate, anaerobic digestion has been applied as wet (i.e., TS from 0.5% to 15%) or dry (i.e., TS more than 20%) processes for many decades. However, dry anaerobic digestion is particularly preferred for treating the industrial wastes in order to meet the energy demand of the facilities. In this context, since paper wastes have high carbon content and TS concentrations; these difficult degradable

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feedstocks have been stabilized efficiently by anaerobic reactors also enabling biogas generation as one of the renewable energy sources [1–3]. However, paper industry wastes contain mostly hemicellulose and cellulose and degradation of these materials by the microorganisms are not easy. On the other hand, inhibitory substances found in paper wastes might cause process failure in bioreactors with reduced biogas yields. These inhibitory compounds are sulphuric (e.g., unionized H_2S, SO_3^{2-}, SO_4^{2-}) and chlorinated (e.g., Adsorbable Organic Halides and pentachlorophenol) compounds, wood extractives (e.g., resin acids, fatty acids, volatile terpenes, sterols, juvabiones, and tannins), and some other compounds such as H_2O_2 [4, 5]. Since paper and packaging materials are lignocellulose-based natural polymers; they release acetic acid, aldehydes, alcohol, and ester-based volatile organic compounds (VOCs) due to ageing [6]. Hence, necessary precautions against these toxic substances should be taken in order to keep the digestion process at steady-state condition for achieving an enhanced methane production. For example, some chemical compounds such as aldehydes (e.g., hexanal) and alcohols (e.g., 1-octanol) might be toxic especially to the methanogenic cultures, which adversely affect biogas yields of anaerobic processes with long lag phase periods at their increasing concentrations [3, 7]. In this context, acclimation periods generally allow the microbial cultures to adapt unfavorable conditions and inoculum adaptation plays a key role during anaerobic digestion of the inhibitory substances in bioreactors [8]. Besides available microorganisms, the impact of the chemical compounds on the stabilization process change depending on the operation parameters (e.g., pH, temperature, hydraulic retention time etc.). Moreover, it was reported that the effect of the compounds from alcohol and aldehyde groups on methane generation has been also concentration-dependent [9]. According to Jansson et al. [3], the chemical compounds that could affect biogas production in traditional wet anaerobic digestion processes were hexanal and 1-octanol which have been detected in the wastes/wastewaters of various industries such as food and paper sectors. Accordingly, the main source of odour in many paper with high wood extractives is the auto- and photo-oxidation of linoleic acid, oleic acid, linolenic acid. On the other hand, hexanal is a well-known aliphatic aldehyde and it is a predominant reaction product of linoleic acid (i.e., a fatty acid component in wood extractives) oxidation and accounts for odour nuisance in pulp and paper industry. It was also reported that hexanal was the dominating volatile aldehyde originating from pulp and paper produced for food packaging purposes. On the other hand, a large variety of other aldehydes, ketones and alcohols are also formed [10, 11]. Among them, 1-octanol, known as a fatty alcohol, is described as typical volatile oxidation compound of oleic acid which also contributes to off-flavor development and released to the environment as a metabolite of many plants [10, 12, 13]. Octanol is also found in paper industry wastewaters, due to its usage as an antifoaming agent during paper making [14]. Hence, investigation of the impact of hexanal and 1-octanol on anaerobic digestion is important owing to the fact that anaerobic treatment is mostly used to stabilize the wastes from paper industry [3, 15]. Nevertheless, these two compounds might be beneficial or detrimental to the anaerobic digestion [9]. Although hexanal and 1-octanol could be degraded by some microorganisms which are involved in anaerobic digestion process, high concentrations of these chemicals could reduce the biogas production [3]. Hence, the aim of this work was to investigate the impacts of 1-octanol and hexanal on cumulative methane yield during thermophilic (55±2 °C) anaerobic digestion of the waste from a paper industry.

MATERIALS AND METHODS

Substrate, Inoculum, and Chemical Compounds

The paper waste (PW) with a total solids (TS) and volatile solids (VS) contents of about 28% and 27% was supplied by a pulp and paper industry located in Varberg, Sweden. The inoculum was obtained from a dry anaerobic digester plant treating municipal solid waste at thermophilic conditions (Borås, Sweden). TS content of the inoculum was 3.6% with a volatile content of about 65% [16].

The chemical compounds examined were from two groups i.e. alcohol (1-octanol) and aldehyde (hexanal); all provided by Sigma-Aldrich (Germany). 1-Octanol ($C_8H_{18}O$), also known as N-octan-1-ol or capryl alcohol, is a clear and colorless liquid which is a fatty alcohol lipid molecule with a molecular weight of 130.23 g/mol [12]. Besides, hexanal ($C_6H_{12}O$) is also a clear and colorless liquid with a molecular weight of 100.16 g/mol [17]. Both chemical solutions were prepared by diluting pure liquid chemical compounds with distilled water in order to achieve concentrations of 0.005%, 0.05% and 0.5% (w/v). Hence, 0.27–27 and 0.3–30 mg COD were added to each bottle by adding hexanal and 1-octanol, respectively.

Batch Reactors and Operating Conditions

The experiments were carried out at thermophilic conditions (55±2 °C) using 118 mL serum glass bottles at which anaerobic digestion took place. The reactors were kept in an incubator and operated as batch systems. In order to measure the methane production of only the inoculum, the blank reactors were set-up with only inoculum (Control_Seed) to be incubated without the addition of substrates and chemical compounds. On the other hand, the control reactors were also set-up only with the inoculum and the used paper waste (Control_PW) with-

Table 1. The toxic concentrations of some alcohols and aldehydes in anaerobic digestion

Compound class	Value	Unit	Reference
4-Methylbenzaldehyde	4.25 (IC_{50})*	mmol/L	Sierra-Alvarez and Lettinga [20]
Benzaldehyde	5.03 (IC_{50})	mmol/L	
Formaldehyde	10	mg/L	Qu and Bhattacharya [21]
2-Phenylethanol	46.53 (IC_{50})	mmol/L	Sierra-Alvarez and Lettinga [20]
Phenylmethanol	31.74 (IC_{50})	mmol/L	
Allyl alcohol	3000	mg/L	Demirer and Speece [22]

*: The inhibitory concentration (IC_{50}) that reduces 50% of methane production.

Table 2. Average methane yields and standard deviations (mL CH_4/g-VS)

Chemical conc. (%)	Incubation time (d)							
	0	2	9	13	23	29	35	45
Inoculum (Control_Seed)								
0	0±0	13±1.0	38±1.6	89±4.0	102±4.2	107±5.4	119±7.1	128±8.8
Paper waste + Inoculum (Control_PW)								
0	0±0	34±2.5	145±10.8	461±36.3	537±89.8	581±92.3	600±93	618±92.1
Paper waste + Inoculum + 1-octanol								
0.005	0±0	39±5.1	137±1.5	400±7.8	596±13.5	628±8.9	657±12.2	675±9.1
0.05	0±0	35±4.2	133±2.3	395±14	567±36	628±13.8	655±11.8	678±12.5
0.5	0±0	34±1.1	136±8.9	381±106	527±87	563±91	583±91	599±96
Paper waste + Inoculum + Hexanal								
0.005	0±0	37±1.6	143±11	414±51.4	615±32.7	651±45	673±40.4	685±43
0.05	0±0	34±2.0	143±10	439±33.5	607±18	646±25.4	673±19.7	695±17.4
0.5	0±0	30±2.3	130±10.2	408±42.8	540±42.1	578±44.4	607±35.5	638±43.5

out the addition of chemical compounds. Only distilled water was added in the blank and control flasks. All assays were running with the substrate to inoculum ratio of 1:2 (VS basis) in reactors corresponding to 0.5 g-VS for the substrate and 1.0 g-VS for the inoculum ($VS_{substrate}$ to $VS_{inoculum}$). Besides, each serum bottle except the control ones, included 2 mL of the single selected compound. On the other hand, the control reactors were prepared with 2 mL tap water instead of hexanal or 1-octanol solution. Hence, the working volume for each reactor was about 45 mL with the headspace of about 75 mL. After the addition of all necessary components; each serum bottle was sealed with a rubber septum and with an aluminum cap. Then, the headspace of each reactor was flushed with the gas mixture of 80% N_2 and 20% CO_2 for 2 minutes to maintain anaerobic conditions inside the reactors. The reactors were then incubated at 55±2 °C while they were shaken manually once a day during the incubation period of 45 days. All experimental set-up was done in triplicates (i.e., with a total of 24 flasks) and average methane gas values were calculated.

Analytical Procedure

Gas samples were taken three times a week at the first weeks of the study, then were gradually reduced as twice a week and once a week towards the end of the digestion period. The total solids (TS) and the volatile solids (VS) of the substrate and inoculum were determined according to Standard Methods [18]. On the other hand, the biogas samples were taken from each reactor's headspace using a 250-µL pressure-lock gas syringe (VICI, Precision Sampling Inc., Baton Rouge, LA, USA). Gas measurement and analysis were conducted as described in a previous study by Teghammar et al. [19] and each methane (CH_4) by volume was obtained at standard conditions (0 °C and 1 atm). The compositions [methane (CH_4), hydrogen (H_2), carbon dioxide (CO_2)] of the produced biogas were determined using a gas chromatography (Perkin-Elmer Clarus 590, Perkin-Elmer Inc., Waltham, MA, USA) equipped with a packed column (6' x 1.8" OD, 80/100, Mesh, Perkin Elmer Inc., Waltham, MA, USA) and a thermal conductivity detector (Perkin-Elmer Inc., Waltham, MA, USA) at an inject temperature of 150 °C. The carrier gas was nitrogen operated with a flow-rate of 20 mL/min at 60 °C.

Table 3. Effect of the additions of chemical compounds on biogas production at different concentrations

Reactor content	Chemical compound	Chemical concentration (%)	Net methane yield (mL CH_4/g-VS)	Variation[a,b] (%)
Inoculum (Control_Seed)	–	0	128	–
Paper waste + Inoculum (Control_PW)	–	0	490	–
Paper waste + Inoculum	1-octanol	0.005	547	(+)9
		0.05	550	(+)10
		0.5	471	(-)3
Paper waste + Inoculum	Hexanal	0.005	557	(+)11
		0.05	567	(+)12
		0.5	510	(+)3

a: [(CH_4 produced by feedstock with chemical compound-CH_4 produced by control)/(CH_4 produced by control)x100]; b: Enhancement is indicated by (+) whereas retardation is indicated by (-) in net methane yield.

RESULTS AND DISCUSSION

Since some alcohols and aldehydes found in paper wastes might cause process failure in bioreactors; necessary precautions against these toxic substances should be taken in order to keep the digestion process at steady-state condition for achieving an enhanced methane production [7]. The toxic concentrations of some alcohols and aldehydes in anaerobic digestion are presented in Table 1.

Since Jansson et al. [3] performed a similar study with hexanal and 1-octanol at mesophilic (37±1 °C) condition; this particular study was performed in order to investigate the effects of these selected compounds on biogas production at the same concentrations at thermophilic (55±2 °C) condition. Hence, 1-octanol and hexanal were added into the reactors to achieve concentrations of 0.005, 0.05, and 0.5% (w/v). The results showed that addition of 1-octanol and hexanal at concentrations of 0.05% resulted in increased biogas production (Table 2) after 45-days incubation period. The highest net methane yields were 550 and 567 mL/g-VS_{fed}, respectively in the presence of 1-octanol and hexanal at a concentration of 0.05%. These results corresponded to about 10% and 12% enhancement in cumulative methane production, respectively compared to the control reactor (490 mL CH_4/g-VS_{fed}) digesting only the paper waste. Hence, adding both compounds to 0.05% was beneficial for the digestion process. Even at 0.5%, hexanal was somewhat beneficial; however, 1-octanol at this concentration showed a slight effect with a retardation in biogas generation of about 3% decrease. Accordingly, cumulative methane yield for 1-octanol was 471 mL CH_4/g-VS_{fed} whereas it was 510 mL CH_4/g-VS_{fed} for hexanal compared to the control reactor (490 mL CH_4/g-VS_{fed}). Hence, it could be concluded that increasing concentrations of both chemicals from 0.05% to 0.5% indicated not significantly different methane yields than that obtained from only paper waste in the control reactor. Effect of the additions of chemical compounds on net methane yields are presented in Table 3. In this context, continued

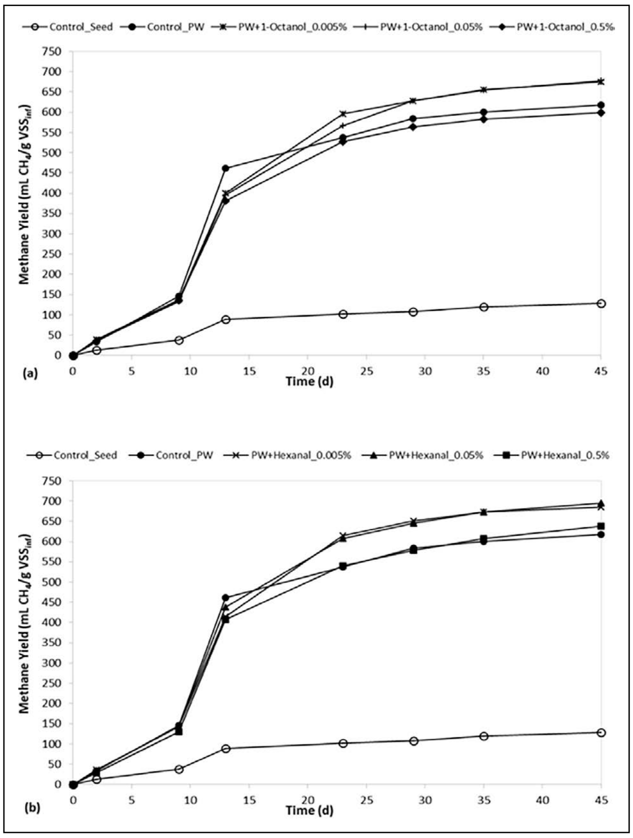

Figure 1. Biogas generation during thermophilic anaerobic digestion of paper waste with **(a)** 1-octanol and **(b)** hexanal at different concentrations in comparison to control reactors (without chemical compounds) .

biogas productions revealed the probable consumption of both chemicals up to a certain concentration by the anaerobic microorganisms available in thermophilic batch reactors. The effect of the selected compounds from alcohol and aldehyde groups on the initial digestion rate is presented in Figure 1a for 1-octanol and in Figure 1b for hexanal.

Although adding 1-octanol and hexanal did not indicate any apparent inhibition on anaerobic digestion performance of the paper waste up to 0.5% at thermophilic (55±2 °C) condition, Jansson et al. [3] who performed a similar study with the same hexanal and 1-octanol concentrations reported contrary findings at mesophilic (37±1 °C) condition. Accordingly, they reported substantial inhibition and lower methane yields at all concentrations irrespective of the chemical compound used. On the other hand, long lag phase was not observed in this particular study unlike Jansson et al. [3]. Accordingly, they previously reported more than 20 days for hexanal and 80 days for 1-octanol during mesophilic digestion of paper waste in similar batch assays at S/I ratio (VS basis) of 1.0 with a TS of 16%. Therefore, since the addition of the same alcohol and aldehyde sources up to a concentration of 0.05% improved the performance of the anaerobic process with an increase in methane yield in this particular study, it was probably due to the difference in initial VS content fed [23] into the batch reactors as well as due to higher (i.e., thermophilic) operating temperature. Meegoda et al. [24] also reported that reaction rates are increased at higher temperatures which result in a possibility of higher loading rates and improved biogas yields.

In conclusion, contrary to expectation, the selected chemical compounds did not indicate notable toxic effect on methane production in the batch assays even the concentration increased to 0.5% for 1-octanol and hexanal. However, preliminary results revealed that the toxic effects of both selected compounds would be observed at different operating conditions [3]. Moreover, higher concentrations of the chemical compounds from alcohol and aldehyde groups would exert inhibitory effect especially when their threshold levels are exceeded in anaerobic reactors digesting the wastes from industries such as food and paper facilities.

CONCLUSIONS

According to the results, cumulative methane yields measured in the batch assays showed that the degradation process was not slowed down and the externally added compounds from the alcohol (1-octanol) and aldehyde (hexanal) groups up to a concentration of 0.05% made it possible for the methanogens to produce a higher amount of ultimate biogas. Hence, addition of the selected compounds was beneficial for the digestion and improved the performance of the anaerobic process with an enhancement in methane yield of 10% for 1-octanol and 12% for hexanal. Accordingly, compared to the control reactor which only included the paper waste; net methane yield for 1-octanol was 550 mL CH_4/g-VS_{fed} whereas it was 567 mL CH_4/g-VS_{fed} for hexanal at 0.05% concentration of each compound. When it comes to the effects of the selected chemical compounds at 0.5% concentration of each, no remarkable toxic effect on methane production was observed even the concentration increased up to 0.5% for both compounds. Accordingly, methane yields indicated a slight increase for hexanal whereas a slight decrease for 1-octanol even at 0.5% concentration. Contrary to expectations, the reason why methane yields did not decrease even with increasing concentrations of both chemical compounds, was probably due to the fact that threshold levels were still not exceeded in the batch assays. In conclusion, continued biogas productions also revealed the probable consumption of both chemicals up to a certain concentration by the anaerobic microorganisms available in the reactors digesting paper waste at thermophilic temperature.

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DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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