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Improving methane production during the codigestion of waste-activated sludge and fatty wastewater: Impact of thermo-alkaline pretreatment on batch and semi-continuous processes

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HIGHLIGHTS

- Pretreatments were applied to a mixture of sludge and fatty wastewater before codigestion.
- Recalcitrant compounds were formed during 170 °C pretreatment of fatty wastewaters.
- Thermo-alkaline pretreatments increased the rate during batch anaerobic digestion.
- Semi-continuous methane production was 58% higher after pretreatment at 80 °C, pH = 8.
- Semi-continuous digestion of a high lipid content mixed waste (73% COD) was stable.

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ABSTRACT

With the objective of maximising methane production from waste-activated sludge (WAS), codigestion with fatty wastewater (FW) collected from restaurants was studied and the application of both a 170 °C thermal pretreatment and thermo-alkaline/saponification to mixed WAS/FW waste was evaluated. Batch anaerobic digestion tests showed that recalcitrant compounds were formed during the 170 °C pretreatment of fatty wastewater and thermo-alkaline pretreatments (80 °C or 120 °C, pH 8, 9 or 10) led to an increased initial methane production rate and to a very slight impact on the methane potential of the mixed waste (+4–7%). Semi-continuous anaerobic digesters were fed over 4 months with WAS, two mixed substrates WAS/FW 90/10 and WAS/FW 60/40, and with WAS/FW 90/10 pretreated at softer conditions (80 °C and pH = 8, 0.14 $g_{KOH}\ g_{VS}^{-1}$). This pretreatment led to a significant increase in the methane production of semi-continuous reactors (+58%). Finally, this study showed the feasibility of the codigestion of waste activated sludge with highly concentrated fatty wastewater (40% in volume, 49% in VS and 73% in COD, equivalent to 7 g L^{-1} of lipids). It resulted in specific methane production of 362 mL $CH_4\ g^{-1}$ VS whereas WAS alone produced only 116 mL $CH_4\ g^{-1}$ VS.

1. Introduction

In France, the transport sector is the main origin of greenhouse gases, responsible for about 27% of total emissions of greenhouse gases and 34% of CO_2 emissions in 2004 [1]. A key short-term measure for reducing greenhouse gases emissions consists in increasing the use of alternative fuels such as biomethane which displays very favourable environmental indicators [2]. In the context of sustainable development, the example of Lille, a city in the northern France, offers a relevant example of the biomethane used by a municipality. The Urban Community of Lille (UCL) is seeking to profitably use the sewage sludge from its Wattrelos wastewater treatment plant as biofuel for city buses. The average energy requirement is 100 Nm^3 CH_4 per bus per day which replaces 110 L diesel/day or 28,500 L diesel/year. Indeed, as early as 1994, UCL implemented the first European pilot plant for biomethane fuel production at the Marquette wastewater treatment plant and four buses run on biomethane [3].

Pursuing their commitment to sustainable development, a further objective of UCL is to increase biogas production and make profitable use of the sewage sludge from the Wattrelos wastewater treatment plant. However, the activated sludge treatment process is currently operated under extended aeration conditions, resulting in waste-activated sludge (WAS) with a low methane potential. An option for increasing biogas output from a WAS digester is

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codigestion with other residues. Fatty wastewater is particularly interesting because it presents high methane potential, is liquid and can be easily mixed with sludge. However fatty wastewaters has disadvantages: lipids may be poorly accessible to microorganisms and long-chain fatty acids have inhibiting properties.

In earlier research, Kabouris et al. [4] studied the codigestion of primary sludge and waste-activated sludge with polymer-dewatered FOG (fat, oil and grease) from a wastewater treatment plant; Fountoulakis et al. [5] proposed the codigestion of sewage sludge with glycerol; Davidsson et al. [6] and Luostuarien et al. [7] investigated the codigestion of sewage sludge with grease trap sludge. In fact, semi-continuous codigestion was shown to be feasible with an addition of 10–30% of grease-trap sludge [6] or up to 46% [7] of organic feed, but too excessive lipid content (55% and 71% of VS) in the feed mixture resulted in incomplete degradation and the acidification of the reactor with a subsequent decrease in the biogas production rate [7]. This was explained by the partial inhibition of the methanogenesis step by long-chain fatty acids [7] and may also have been caused by overloading.

Additionally, the anaerobic digestion of fatty wastes may be enhanced by different preteatment processes such as enzymatic hydrolyse (mainly lipase [8]), acid treatment (addition of HCl for pH = 2–2.5 [9]) or saponification. During saponification, glycerides are first hydrolysed into glycerol and fatty acids then neutralised to form soaps with higher solubility and surface-active properties. The conversion of insoluble lipids into soluble soaps should improve the contact between the substrate and microorganisms [10]. Saponification has been carried out in different experimental conditions: at 80 °C with KOH in excess [11], at 80 °C with KOH at pH = 9 [12] or at 60–120 °C and 150 °C with NaOH 0.3 wt.% [13]. On the other hand, alkali treatment at room temperature (NaOH, 50–400 meq L^{-1}) did not lead to a significant solubilisation of slaughterhouse waste containing approximately 3 g L^{-1} of pork fat particles [14]. Finally, different types of thermal pretreatment have been applied for sanitising slaughterhouse waste and by-products from the meat-processing industry at, respectively, 145 °C, 5 bar for 25 min [15] or 70 °C for 60 min [9]. In both cases, subsequent methane output fell due to the formation of recalcitrant compounds.

Pretreatment has been more widely studied to improve the anaerobic digestion of waste activated sludge and several review papers have been published recently [16–18]. Among the different pretreatment techniques, thermal hydrolysis at 160–180 °C has been shown to increase both the efficiency and the rate of degradation of waste activated sludge [17]. Moreover, thermal treatment induces sludge sanitation and the improvement of sludge dewaterability while energy costs can be covered by the biogas produced [19]. Few papers have reported on the thermal–alkaline pretreatment of sewage sludge but the conditions involved were quite different from those for fatty residue saponification, with either higher temperatures (121 °C [20], 130 °C [21,22] or 175 °C [23]) or a longer reaction time (6 h at 55 °C [24] or 10 h at 90 °C [25]). According to Valo et al. [22], sludge pretreatment at 130 °C and pH = 10 with the addition of potassium hydroxide led to the same increase in methane production as with 170 °C thermal treatment.

To the best of our knowledge, studies are lacking on the pretreatment of mixed waste prior to its codigestion. The aim of this paper was thus to evaluate thermal pretreatment at 170 °C (generally used for waste activated-sludge) and thermo-alkaline/saponification pretreatment (generally used for fatty residues) applied to mixed waste composed of waste activated sludge (WAS) and fatty wastewater (FW) collected from restaurants.

Batch anaerobic digestion tests were first carried out to optimise the pretreatment conditions (temperature and pH) on mixed waste composed for FW/WAS 67/33 vol/vol, and then thermo-alkaline pretreatment performances were evaluated during semi-continuous codigestion. Semi-continuous anaerobic digestion was carried out with pre- and untreated mixed substrates: WAS/FW 90/10 vol/vol. Another semi-continuous digestor was fed with the mixed waste WAS/FW 60/40 to study codigestion with high lipid content and a control reactor was fed with WAS only.

2. Materials and methods

2.1. Materials

Waste activated sludge was collected at the Wattrelos wastewater treatment plant after thickening by flotation. WAS originated from an extended aeration process with a sludge age of 21 days. Fatty wastewater was collected during truck discharging at the wastewater treatment plant. The wastewater was collected from several restaurants and was composed of grease tank washing water. The composition of WAS and of FW is shown in Table 1. WAS and FW were stored at 4 °C for 2–6 months.

The anaerobic inoculum was a mesophilic anaerobic sludge from a sugar factory in Marseille. The sludge contained 26±2 g_{TS} L^{-1} and 18±1 g_{VS} L^{-1}.

2.2. Analysis

The soluble fraction of samples is referred to as the supernatant and the particulate fraction as the pellets obtained by centrifugation (Beckman J2 MC, 42,000 g, 15 min, 5 °C).

Measurements of total solids (TSs) and volatile or organic solids (VSs) were done for the waste samples; total suspended solids (TSSs) and volatile suspended solids (VSSs) were measured as solids after centrifugation, in accordance with Standard Methods [26].

Chemical oxygen demand (COD) was determined using Spectroquant® test kits (Merck, Darmstadt, Germany) and a HACH DR/2000 spectrophotometer (Hach Company, Loveland, CO., USA) at 620 nm.

Lipid concentration was assessed by the amount of matter which was extracted by heptane (HEM, heptane extractable matter). This measurement is usually carried out using hexane but heptane was preferred because of its lower toxicity [27]. A volume *V* of waste sample was first acidified (pH < 2) by sulphuric acid in order to maintain fatty acids in their non-ionic form. A volume *V* of heptane and *V*/2 of methanol were added to the sample in an extraction funnel. Methanol was used to maintain lipoproteins in the aqueous phase. The funnel was set on an oscillating mixer (40 oscillations per minute) for 30 min. The heptane phase was afterwards recovered and put into a flask. Heptane extraction was repeated until it remained colourless, indicating exhaustion of the sample. Heptane was removed by a roto-evaporator (rotavapor R, Büchi) at 80 °C under partial vacuum. Extracted lipids were weighed after drying at 105 °C for 24 h.

Biogas volume was measured by acidified column displacement, the liquid being water at pH = 2 and 10 g L^{-1} of NaCl. The accuracy of this measurement was ±1 mL for batch tests and ±10 mL for semi-continuous reactors.

Table 1
Composition of waste activated sludge and fatty wastewater.

	Waste activated sludge	Fatty wastewater
TS (g L^{-1})	39.65	42.66
VS (g L^{-1})	23.57	32.73
COD (g L^{-1})	37.9	92.3
HEM[a] (g L^{-1})	1.46	21.49

[a] Heptane extractable matter.