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Comprehensive monitoring of a biogas process during pulse loads with ammonia

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ABSTRACT

A thermophilic pilot scale anaerobic digester treating chicken litter was subjected to pulse loads of ammonia of increasing concentration. A micro gas chromatograph (μ-GC) measured CO_2, CH_4, N_2, H_2 and H_2S in the biogas. In the liquid, NH_4-N was measured manually, volatile fatty acids (VFA) were measured manually and with Near Infrared Spectroscopy (NIRS). Within the first 7–24 h after the pulse, the concentrations of iso-butyric and iso-valeric acid increased rapidly and were the best indicators of process stress due to ammonia pulses, confirmed by multivariate analysis. NIRS was not capable of accurate prediction of VFA iso-forms most likely due to low concentrations. Propionic acid was persistent. The ammonia pulses caused an overall decrease of biogas production. The biogas composition was not a good indicator of imbalances; little correlation with VFA measurements was observed.

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1. Introduction

Commercial-scale biogas plants digesting organic waste combine waste treatment with energy recovery (production of biogas). Often, anaerobic digestion processes do not achieve optimal performance because of toxic or inhibiting compounds introduced by mistake in the digester. This results in decreased methane productivity and in some cases it can cause process failure. Parameters to be used as indicators of the biogas process have to be identified to develop reliable monitoring and control of a biogas reactor.

The complexity of the biogas process makes it difficult to find simple and reliable control parameters. In anaerobic processes, the mineralization of organic material to methane and carbon dioxide takes places in four main steps and imbalances in the process can be detected when concentrations of some of the intermediates formed during the four main steps are seen to change. These changes depend on the type of imbalance occurring (e.g. overload, toxins or inhibitors) and on the parameters contributing to the selection of the microbial population in the digester, such as temperature and substrate [1,2]. To identify parameters suitable for monitoring and to study their response, an imbalance can be induced in the reactor. Individual volatile fatty acids (VFA) are often considered to be good indicators of process imbalances. Although literature shows that VFA concentration seems to be among the best indicators to detect imbalances in biogas processes, it also shows that there seem to be differences

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about which of the VFAs are the most suitable depending on the substrate. Iso-butyric, butyric and iso-valeric acids were found to be good indicators of ammonia inhibition in thermophilic digesters fed with cattle and pig manure [1,3]. However, it has been argued that the effectiveness of VFA isoforms in process monitoring is limited due to the complicated interactions between the VFA species [4]. The main source of VFA isoforms is from the breakdown of proteins, with iso-butyric and iso-valeric acids derived from valine and isoleucine, respectively [2]. Yet a later study has shown that butyrate and iso-butyrate can also form through reversible isomerisation and that butyrate is an intermediate in the degradation pathway of iso-butyrate [5]. It was found [4] that pulses of butyrate, iso-butyrate and iso-valerate produced only acetate in a digester fed with cattle manure, whereas valerate produced acetate and propionate and that all these processes showed some product inhibition. The reactions between butyrate and iso-butyrate, iso-valerate and acetate, valerate and acetate and valerate and propionate are reversible, when propionate, butyrate or hydrogen concentrations are high, but there is no evidence that valerate or iso-valerate can form from each other. In some more recent publications, propionate was found to best indicate imbalance when lipids or meat and bone meal were added to laboratory scale reactors treating mixed cattle and pig manure [6] and propionate was suggested [7] to indicate process stress due to gradual organic overload in a continuous reactor digesting manure. Process indicators for digesters fed with 100% chicken litter may not be those of processes treating substrates such as manures, brewery waste [8] or mixtures of chicken litter and other substrates [9].

Gas phase measurements, including both flow rate and composition, have also shown success as the basis for monitoring systems [10]. However, hydrogen concentration cannot be considered as a universal indicator for early warning against imbalances [11] and it was found [3] that methane concentration did not decrease following inhibition due to successive pulse additions of ammonia.

pH is an important factor in biological systems and very simple to measure, but it has been suggested that pH alone is not sufficiently sensitive to detect sudden changes in the process state [12] due to the high buffering capacity of solid digestion systems.

Imbalances can be induced in different ways. Among them, are the increase of the loading rate [13] and addition of ammonia [14,15]. Urea was added to digesters treating cattle manure to simulate ammonia shock loads [16] whereas ammonium chloride and pig manure were used in other studies [3]. Ammonia is often the nitrogen source, but this compound can be toxic if present in high concentration [17]. The concentration of ammonia at which inhibition occurs has been reported at a wide range of values [15,18,19]. The factors responsible for the variation in ammonia inhibition thresholds have been reviewed [20] and include pH, temperature, the presence of other ions and acclimation of the organisms.

Poultry litter presents difficulties as a substrate for biogas production because of the risk of ammonia inhibition [21,22]. Poultry litter as monosubstrate for biogas processes is gaining increasing attention and a number of process design solutions have been suggested [23,24]. Although a high number of studies have been done from lab- to commercial-scale, most of them were conducted at mesophilic conditions [25]. Thermophilic processes in continuous stirred tank reactors are effective treatment facilities thanks to their higher productivity compared to mesophilic processes, but are more subject to instability, which has been attributed to reduced microbial diversity at thermophilic conditions compared to mesophilic conditions [26]. Thus, thermophilic biogas processes treating chicken litter as monosubstrate need dedicated studies and comprehensive monitoring to identify the parameters that are most suitable for indicating imbalances at an early stage.

In this work, imbalances were induced with ammonia pulses to increase the ammonia concentration in a biogas digester and to study the effect on the process parameters. NH_4HCO_3 in increasing amounts was added artificially to a thermophilic pilot-scale digester fed with chicken litter as monosubstrate which was monitored over 65 days in both the gas and liquid phases. By use of multivariate data analysis for examination of the large amount of data from digester monitoring, the effect on biogas productivity and composition and on the VFA concentration in the liquid was examined for signs of imbalance.

2. Materials and methods

2.1. Substrate

Chicken litter (total solids TS 62.4 ± 1.8%, volatile solids VS 52.8 ± 1.8%) mixed with water was used as substrate. The diluted substrate contained 6.2 ± 0.2% TS, 5.3 ± 0.2% VS, 2.1 ± 0.2 g dm^{-3} NH_4-N, total Kjeldahl nitrogen (TKN) 3.0 ± 0.2 g dm^{-3}, pH 6.0 ± 0.3. Volatile fatty acids (VFA) were acetic acid 6800 ± 2000 mg dm^{-3}, propionic acid 2800 ± 800 mg dm^{-3}, iso-butyric acid 170 ± 80 mg dm^{-3}, butyric acid 1700 ± 580 mg dm^{-3}, iso-valeric acid 270 ± 170 mg dm^{-3}, valeric acid 450 ± 230 mg dm^{-3}. Averages and standard deviations are calculated from measurements repeated over the 65-day experiment.

2.2. Reactor set-up and operation

The digester was a 300 dm^3 pilot scale semi-continuous (effluent removal and feeding every 12 h) stirred tank reactor with 200 dm^3 working volume, operated at thermophilic conditions (52 °C) as described in Ref. [13], Fig. 1. The organic loading rate (OLR) on VS basis and the hydraulic retention time (HRT) were maintained constant at 3.5 g dm^{-3} d^{-1} and 15 d, respectively. During effluent removal, air was drawn into the digester, diluting the gas in the headspace. The results have been corrected for the air dilution by assuming that CH_4 and CO_2 constitute very close to 100% of the biogas and that the H_2, H_2S and other trace components of the biogas constitute considerably less than 1%. The data presented here is based on the relative concentrations of CH_4 and CO_2, i.e. the two components together have been adjusted to equal 100%. The H_2 and H_2S data were corrected using the ratio between the measured CH_4 and the corrected CH_4.

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