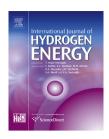


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# Automatic process control for stable bio-hythane production in two-phase thermophilic anaerobic digestion of food waste



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#### ARTICLE INFO

Article history:
Received 17 June 2014
Received in revised form
23 August 2014
Accepted 29 August 2014
Available online 22 September 2014

Keywords:
Bio-hythane
Hydrogen
Process control
Ammonia
Food waste
Anaerobic digestion

#### ABSTRACT

The paper reports the results of a long term (310 days) pilot-scale trial where food waste as sole substrate was treated in a two-phase thermophilic anaerobic digestion process. This was optimized for concurrent hydrogen and methane production. First phase's optimization for hydrogen production was obtained recirculating the effluent coming from the methanogenic phase and without the addition of external chemicals. A drawback of such approach is the recirculation of ammonia into the first phase reactor for hydrogen production with possibility of consequent inhibition.

Therefore this study was focused on the development of a control protocol based on ammonia concentration. The first part of this paper illustrates how the use of a variable recirculation flow makes possible to control the whole process, preventing the ammonia inhibition in the system. In order to lay down the groundwork for an automatic control of the process, in the second part of the study a preliminary statistical study is presented. In the latter are developed models to predict ammonia levels in system using the measure of Electrical Conductivity, Volatile Fatty Acids and Alkalinity.

During steady state conditions, managed by a variable recirculation flow, the system produced a mixture of gas that met the standards for the biohythane mix with an average composition range of 7%  $\rm H_2$ , 58%  $\rm CH_4$  and 35%  $\rm CO_2$ . The overall average specific gas production (SGP) reached 0.69  $\rm m^3_{\rm Biogas}/kgTVS$  and gas production rate (GPR) of 2.78  $\rm m^3/m^3_{\rm rd}$ .

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Abbreviations: AD, anaerobic digestion; ALK tot, total alkalinity; BHy, biohythane; COD, chemical oxygen demand; COND, conductivity; CSTR, continuous stirred tank reactor; DF, dark fermentation; DW, dry weight; GP, gas production; GPR, gas production rate; HPR, hydrogen production rate; HRT, hydraulic retention time; MPR, methane production rate; OLR, organic loading rate; Ptot, total phosphorus; SDEC, standard deviation error in calculation; SDEP, standard deviation of prediction errors; SGP, specific gas production; SHP, specific hydrogen production; SMP, specific methane production; SSC, steady state conditions; TKN, total kjiendhal nitrogen; TS, total solids; TVS, total volatile solids; VFAs, volatile fatty acids; WW, wet weight; WWTP, waste water treatment plant.

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#### Introduction

The use of anaerobic digestion (AD) for treatment of biowaste and other organic waste/residues has been growing consistently for the last 30 years in Europe. A step forward for the common anaerobic digestion process of biowaste, which has gained interest among the researchers, is the two-stage approach finalized to the production of hydrogen in the first phase reactor and methane in the second one [1].

Today the hydrogen production by fermentative processes of carbohydrate-rich substrates (like biowaste, food waste and similar), named Dark Fermentation (DF), is one of the most promising technologies for high yield hydrogen production. Several studies showed that DF could be coupled with AD in order to obtain a mixture of gases to be used separately or mixed together: the typical average composition for commercial porpoises is 10% H<sub>2</sub>, 30% CO<sub>2</sub> and 60% of CH<sub>4</sub>, to achieve a second generation biofuel that can be of great interest for combined heat and power (CHP), cogenerator motors or the automotive industry [2], as a result of the upgrade for the elimination of CO<sub>2</sub>.

In DF processes the activity of enzyme hydrogenase is strongly influenced by environmental factors, such as pH and temperature, whose optimal values for maximum activity were identified to be 5.5 and 55 °C, respectively [3,4]. The pH range affects hydrogen production greatly. Maintaining pH in a given range of values for a prolonged exercise is often a hitch without an external chemical control, because the high loads applied to this type of processes involve an accumulation of VFA, resulting in an increase of acidity. Moving to values below 5, the process is controlled by fermentative metabolism, giving typical products of solventogenesis (alcohols and lactic acid) [5].

If we take a look at the literature on this topic, there are several cases in which an external control of pH is provided. Talking about this, in recent years a perceptive practice has been developing as a less expensive alternative to the use of chemicals for external control of the pH in the phase of Dark Fermentation to maintain the pH within the optimal range (5–6) for the hydrogenase catalyzed reactions [5]. It consists in applying a recirculation to the head to the process from the stage of methanogenesis in order to exploit the residual buffer capacity (ammonia and bicarbonate) of this substrate [6]. Moreover, the application of a recirculation flow allows to balance the nutrients intake and helps dilute the feedstock [7,8].

Therefore, also from an economical point of view, it is convenient to develop a pH control system which allows to manage and optimize the process in a sustainable approach, because neither chemical addition nor high costs devices would have to be used to reach the target. Therefore, this research deals with the optimization of a two-phase anaerobic digestion process that treats food waste for bio-hythane production without additional external chemicals.

Considering a long term management of the process, the main problem that can occur in a two-phase system with recirculation flow relates to the accumulation of ammonia: in thermophilic condition, free ammonia leads to inhibition of methane production in concentration exceeding 700 mg/l [9].

It is also important to point out that the rate of hydrogen production can be inhibited by the presence of ammonia at high concentrations [10,11].

According to this strategy the decisive step was to affirm the possibility to set the stability parameters to maintain the process strong and durable, performing a control system (possibly an automatic device) which allows to maintain the right amount of recycle according to the change of the stability parameters of the reactors in real time.

The main problem linked to this approach regards the choice of control parameters to be measured on line. In fact, ammonia concentration probes in such heterogeneous media could be difficult to use and, on the long-run, may prove to be not reliable. Thus, the approach used here considers to use an indirect measure of the main control parameter, using simpler 'predictors'. These indirect predictors can be also measured in easier way using on line probes.

Therefore, this paper is dedicated to the definition of the best control parameters for process control (see Fig. 1).

#### Material and methods

#### Experimental set-up

Two stainless steel CSTR reactors (AISI 304) were used for biohythane production. The first reactor (F1), dedicated to the fermentative step, had a 200 l working volume, while the second reactor (F2), dedicated to the methanogenic step, had a 380 l working volume. Both reactors were heated by a hot water recirculation system and maintained at 55 °C using electrical heater controlled by a PT100-based thermostatic probe. The feeding system was semi-continuous, arranged once per day. The organic waste was reduced in size using a grinder, mixed with tap water and liquid fraction of sludge recirculation from the methanogenic reactor and then fed to the first reactor.

The process was maintained in operation for 310 days. The operational conditions we applied were for the DF phase a HRT of 3.3 days and a OLR of 18.4 kgVS/m³d; for the Methanogenic phase a HRT of 12.6 days and a OLR of 4.8 kgVS/m³d. Regarding the recirculation ratio, the entire experimental test has been divided in three periods: during the first and second working periods the recirculation ratio was maintained steady (0.5 and 0.25 respectively), during the third working period the recirculation ratio was variable according to the ammonia concentration in the system.

#### Substrate and inoculum

The first reactor, devoted to hydrogen production, was not inoculated with an active biomass but it was filled up with a mixture of organic waste coming from the municipality of Treviso and tap water, in order to obtain a total solids content of about 8% [10]. Biowaste has a high carbohydrate content that can be converted into hydrogen and organic acids through the action of fermentative bacteria [6]. The use of biowaste generates an inoculum capable of producing hydrogen in short times, for the presence of indigenous bacterial communities. Afterwards, the first reactor was daily fed

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