Effect of Temperature on Biohydrogen and Biomethane Productions by Anaerobic Digestion of Sugar Beet by-Products

K. Aboudi, X. Gómez-Quiroga, C. J. Álvarez-Gallego, J. M. Quiroga-Alonso, and L. I. Romero-García

***Abstract*—This work analyzes the effect of temperature on the anaerobic digestion of sugar beet by-products for both biohydrogen and biomethane production. The findings demonstrate that the anaerobic process was significantly affected by the increase in temperature from mesophilic to thermophilic or hyper-thermophilic conditions. Therefore, it was found that the mesophilic temperature was more suitable for the anaerobic digestion of sugar beet by-products, using either the raw feedstock or the pretreated feedstock at higher temperatures. The specific production of biohydrogen from thermophilic acidogenic digester was 1.7 fold higher than that obtained from the hyper-thermophilic one. Moreover, when raw feedstock was used in single stage digesters, a methane production rate of 0.55 LCH4/Lr*d was obtained from the mesophilic digester, which was 45% higher than that of the thermophilic one. It has been observed that the increase in temperature led to a high accumulation of volatile and long chain fatty acids, inhibiting and slowing down the anaerobic process.**

***Index Terms*—Anaerobic digestion, inhibition, sugar beet by-products, temperature.**

I. Introduction

Anaerobic digestion (AD) is considered as an attractive technology for renewable energy production from different organic wastes [1]. Temperature plays an important role in biological processes such as the anaerobic digestion process [2]. Anaerobic digestion can be performed at psychrophilic (25 °C), mesophilic (35 °C), thermophilic (55 °C), and even higher temperatures, such as hyper-thermophilic (65 °C). In general, higher temperature conditions lead to higher metabolic rates. Thus, increasing the operating temperature increases the hydrolysis rate of complex organic waste, which can lead to a high generation of methane, since hydrolysis is commonly recognized as a limiting-step in the anaerobic process [3]. However, in some cases, working at higher temperatures can be counterproductive because increased rates of hydrolysis and acidogenesis can lead to an excessive release of inhibitory intermediates in the medium (i.e. volatile fatty acids, free ammonia, long chain fatty acids, furans [3], [4]), which negatively affects the whole process.

The use of temperature-phased (TPAD) or two-phase (TP) configurations for the AD process has shown to be efficient for some substrates, such as organic fraction of municipal solid waste [5], food waste [6] and sewage sludge [7]. The TP configuration allows the production of both biohydrogen and biomethane in two separate consecutive digesters (acidogenic and methanogenic), where the effluent from the acidogenic digester is used as feed for the methanogenic one. The TPAD configuration consists on the use of two digesters operating at different temperatures either for the production of biomethane or biohydrogen. Commonly, in the TPAD process, the first reactor operates at a higher temperature than the second one. In both configurations, the first stage can be considered as a previous pretreatment of the raw material for the second digester, which receives a suitable hydrolyzed material for the methanogenic microorganisms [8].

In this context, the aim of this study was, firstly, to compare the performance of two acidogenic digesters for biohydrogen production from SBB, operating at different temperatures (thermophilic and hyper-thermophilic). Moreover, the effluents from these acidogenic systems were used as a pretreated feedstock for two different mesophilic digesters. The results of these systems have also been compared to the single-stage AD of SBB without pretreatment, operating in both mesophilic and thermophilic conditions.

II. Material and Methods

A. Substrate

Sugar beet by-products consisted of dried pellets of exhausted pulp (85%) mixed with molasses (15%). This substrate was collected from the sugar production company "Azucarera" (AB-sugar group) located in southern of Spain. The raw SBB was used at a total solid content of 8% by rehydrating the pellets with deionized water 24 hours before of the daily feeding (once a day) [9].

B. Semi-continuous Digesters Start-up Procedure

Six semi-continuous stirred tank digesters with a 2.5 L working volume were used in this study. Digesters are made of stainless steel and are covered by a metal jacket for better heat transfer from a heating plate, located at the base of each digester, in order to maintain the temperature selected. Temperature was continuously monitored by an inner

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temperature sensor and controlled by a PID (Proportional-Integral-Derivative) control system. Each digester had an independent motor of agitation and a stirring blade [10].

To facilitate the discussion of results, Table I shows the nomenclature of each digester according to system configuration and temperature conditions. Fig. 1 depicts a schematic illustration of the experimental design for this study. Moreover, in Table II the physical-chemical characteristics of the raw SBB are given.

TABLE I: NOMENCLATURE OF THE DIGESTERS USED IN THIS STUDY

Digesters	Temperature and Stage
Ac-55 ℃	Thermophilic Acidogenic
Ac-65 ℃	Hyper-thermophilic Acidogenic
SS-55 ℃	Thermophilic Methanogenic Single Stage
SS-35 ℃	Mesophilic Methanogenic Single Stage
TP-55 ℃-35 ℃	Thermophilic Acidogenic - Mesophilic Methanogenic
TP-65 ℃-35 ℃	Hyper-thermophilic Acidogenic - Mesophilic Methanogenic

TABLE II: CHARACTERISTICS OF SBB USED IN THIS STUDY

Items	Values
pH	5.84 ± 0.47
Total Solids (%)	82.91 ± 0.24
Volatile Solids (%TS)	91.76 ± 2.48
Total - Chemical Oxygen Demand (g O_2/kg) *	154.84 ± 13.57
Soluble - Chemical Oxygen Demand (g O_2/kg) *	64.52 ± 2.48
Dissolved Organic Carbon (g C/kg) *	41.82 ± 6.08
Total Volatile Fatty Acidity (g HAc/kg) *	5.27 ± 0.75
Ammonia - Nitrogen (g N/kg) *	0.33 ± 0.06
Total Nitrogen (g N/kg) *	8.09 ± 1.54
Alkalinity (g$CaCO_3$/kg) *	0.11 ± 0.01

*g/kg on wet basis.

C. Analytical Parameters

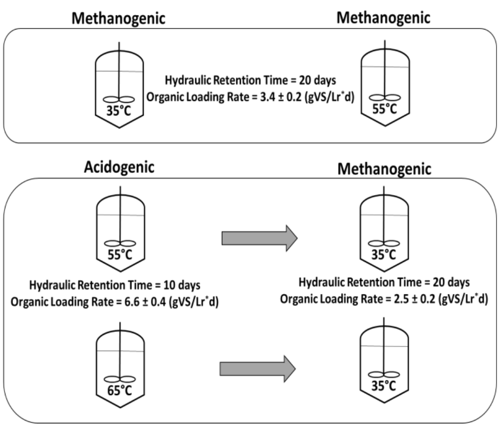

Fig. 1. Schematic illustration of the experimental design.

Standard Methods for the Examination of Water and Wastewater [11] were used to analyse the samples and effluents of all reactors. Samples of VFAs were analysed by using a gas chromatograph (Shimadzu® GC-2010) equipped with a flame ionization detector (FID) and a capillary column filled with Nukol® (diameter of 0.25 μm and 30 m length). Biogas generated during the assays was collected in a 10 L Tedlar® gas bag (SKC) and its volume was measured daily using a high precision drum-type gas meter (Ritter® TG5). Composition of biogas (H_2, CH_4, CO_2) was determined by using a gas chromatograph (Shimadzu® GC-2010) with a stainless steel column packed with Carbosieve® SII (diameter of 3.2 mm and 3.0 m length) and a thermal conductivity detector (TCD).

III. RESULTS AND DISCUSSION

A. Performance of Acidogenic Digesters

The specific hydrogen productions obtained from thermophilic and hyper-thermophilic acidogenic digesters are shown in the Fig. 2. Both digesters started with similar hydrogen production rates. However, after around 20 days of operation, productions from the thermophilic digester significantly exceeds those of the hyper-thermophilic one, reaching a final production 1.7 fold higher.

Average daily hydrogen production rates (HPR) of acidogenic digesters were 0.45 ± 0.15 LH_2/L_r^*d and 0.32 ± 0.10 LH_2/L_r^*d for thermophilic and hyper-thermophilic conditions, respectively. These results were comparable with other findings in the literature. In [12], authors obtained a similar HPR from the dark fermentation of organic fraction of municipal solid wastes (0.43 LH_2/L_r^*d at HRT of 3 days and OLR 16 gVS/Lr*d). Nevertheless, the specific hydrogen production (SHP) was lower than that observed in the present research at similar temperature (55 ℃).

The reference [13] compared thermophilic and hyper-thermophilic AD of food wastes for bio-hydrogen production at HRT of 15, 10, 5 and 3 days. Authors reported that the thermophilic temperature was more advantageous than the hyper-thermophilic one. Stable operations were observed at thermophilic conditions operating at the HRT of 10 days. However, the higher SHP of 70.7 ml H_2/gVS_{fed} and hydrogen content in the biogas (58.6 %) were obtained at the HRT of 5 days.

The organic matter degradation trough hydrolysis and acidogenesis steps produces, together with hydrogen, intermediates compounds mainly as volatile fatty acids (VFAs). Moreover, acidification is strictly related to the substrate characteristics. Hence, in [14] it was studied the suitability of the TPAD process for different kind of substrates, including sugar beet (SB). Authors found that SB had a high sugar content and led to a higher acidification (pH drop) and hence, a high hydrogen production. Similarly, [15] studied the effect of operational parameters (HRT, OLR, pH) on the enhancement of acidification from sugar beet wastes (wastewater and pulp) and reported that the increase of sugar beet pulp in the mixture with lower HRT operation conditions led to a high acidification of the digester, as a result of the increase in VFAs generation from this substrate.

The total volatile fatty acidity (TVFA) and the different VFAs generated in acidogenic digesters are shown in Fig. 3 (a) and (b), respectively for thermophilic and hyper-thermophilic digesters.

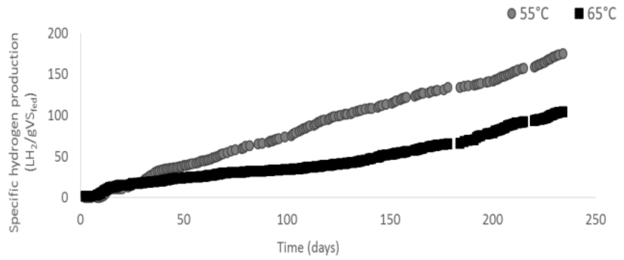

Fig. 2. Specific hydrogen productions from acidogenic digesters.

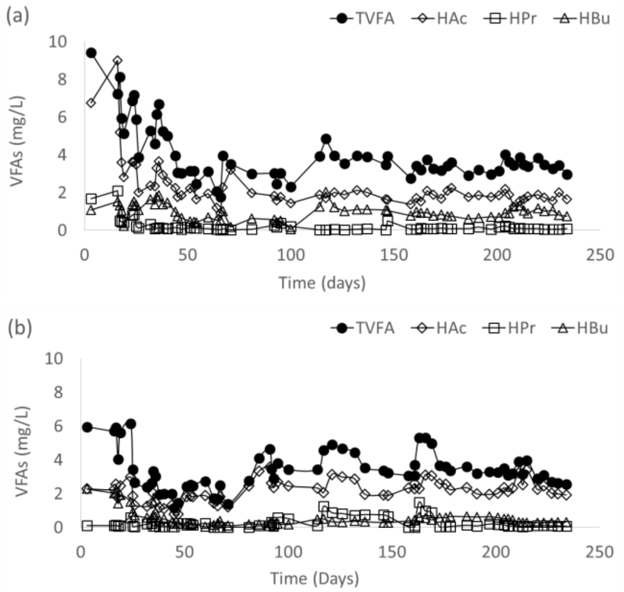

Fig. 3. Evolution of TVFA and the main VFAs in acidogenic digesters; (a) thermophilic and (b) hyper-thermophilic.

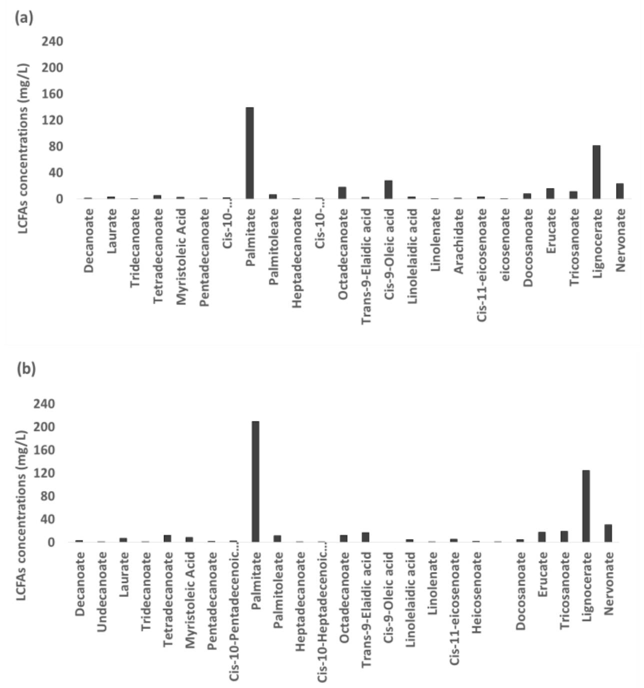

Fig. 4. Long chain fatty acids in acidogenic digesters; (a) thermophilic and (b) hyper-thermophilic.

In both digesters, the predominant VFAs were acetic acid (HAc), butyric acid (HBu) and propionic acid (HPr). The TVFA evolution started with high values and fluctuations due to microorganisms adaptation to the SBB substrate. After few days of operation, TVFA reached stable values around 4 g/L at both temperature conditions, which is similar to the levels observed by others authors in an acidogenic digester for the treatment of vegetable wastes [16]. Based on individual organic acids profiles, their accumulations can be given mainly as acetic followed by butyric and propionic acids. These results agree with other investigations in acidogenic systems [16]. The highest HAc concentrations were observed at the higher temperature of 65 ℃. In both digesters and due to acidification, pH correction with an alkali (potassium carbonate) has been necessary when the pH dropped below 5.5. Among the intermediates compounds of the anaerobic digestion process, long chain fatty acids (LCFAs) could be found. It should be noted that LCFAs present an inhibitory effect on the process, particularly for methanogens and acetogens [17], [18]. Fig. 4 shows the main LCFAs found in effluents from thermophilic (a) and hyper-thermophilic (b) acidogenic digesters. The results indicate that higher concentrations were observed at higher temperature conditions (65 ℃). In both digesters, palmitate and lignocerate were predominants.

B. Performance of Methanogenic Digesters

The specific methane productions from single stage reactors and combined TPAD and TP processes are depicted in the Fig. 5.

Thermophilic single stage digester shows the lowest methane productions. In addition, a very slow starting-up of the process was observed for thermophilic reactor. The single stage mesophilic digester showed the highest final SMP, followed by TPAD55℃-35℃ and TPAD65℃-35℃. It has been observed that mesophilic temperature is more suitable for methane generation from SBB, regardless the system configuration. Therefore, despite of the high acetate content of effluents from acidogenic digesters and the presence of LCFAs, which are known to inhibit acetogenic methanogens, mesophilic digesters in the combined configurations were able to convert VFAs into methane. The lower methane generation in SS-55℃ was related to further accumulation of acetate as shown in Fig. 6. The TVFA observed in the SS-55℃ was even higher than the observed previously in acidogenic thermophilic digester, with also very high acetic acid accumulation. Moreover, propionic acid was also found at higher concentrations. Propionic acid has been largely reported to inhibit methanogens [4]. Both acetate and propionate accumulations could explain the lower methane generation at single stage thermophilic digester. Therefore, mesophilic temperature conditions showed better process performance in separate digesters or by using single stage configuration.

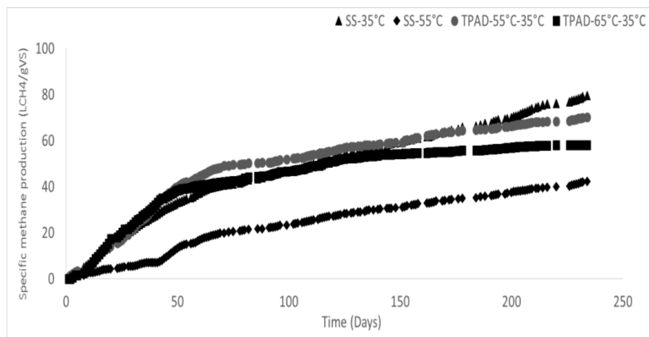

Fig. 5. Specific methane productions in methanogenic digesters.

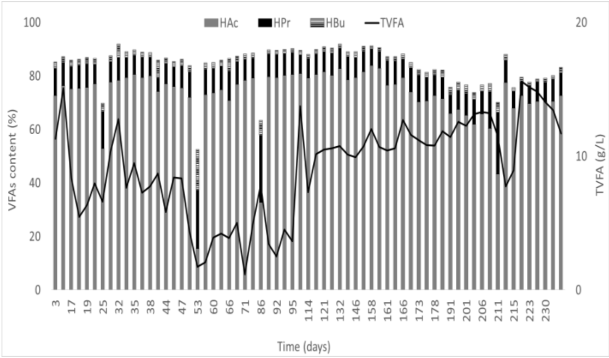

Fig. 6. TVFA and individual VFAs contents in SS thermophilic digester.

Table III summarizes the final performance of the methanogenic digesters. The higher volatile solids removal was observed in SS-35 ℃, as a consequence of the best performance for the organic matter degradation and conversion into methane. Otherwise, the total solids destructions were higher in TP-55 ℃-35 ℃ and TP-65 ℃-35 ℃ due to that the feedstock was already hydrolysed in the first acidogenic digesters.

TABLE III: FINAL PERFORMANCE AND EFFLUENT QUALITY OF ALL METHANOGENIC DIGESTERS FOR THE STEADY STATE PERIOD

Parameters	SS 35 ℃	SS 55 ℃	TP 55 ℃-35 ℃	TP 65 ℃-35 ℃
pH	7 - 7.8	7.5 - 8	7.5 - 8	7.5 - 8.5
TVFA (g/L)	8.9 ± 1.4	11.7 ± 1.7	9.7 ± 1.5	28.5 ± 6.3
HPr/HAc	0.3 ± 0.2	0.2 ± 0.1	0.3 ± 0.1	0.3 ± 0.1
HBu/HAC	0.2 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.1 ± 0.1
TVFA/alkalinity	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.2 ± 0.1
SMP ($mLCH_4/gVS_{fed}$)	166.6 ± 39.1	90.3 ± 17.1	97.7 ±15.5	41.7 ±12.1
MPR (LCH_4/L_r^*d)	0.5 ± 0,3	0.3 ± 0.1	0.2 ± 0.2	-
CH_4 (%)	56.7 ± 10.1	68.2 ± 12.8	55.9 ± 9.8	59.4 ± 3.6
CO_2 (%)	43.1 ± 10.5	31.8 ± 12.1	44.1 ± 7.7	40.6 ± 5.1
VS removal (%)	83.1 ± 9.6	65.8 ± 7.5	69.7 ± 11.5	72.1 ± 7.6
TS removal (%)	50.8 ± 5.7	51.2 ± 4.3	58.5 ± 7.4	53.9 ± 5.3

IV. CONCLUSION

The temperature increase has a detrimental effect on the anaerobic digestion of sugar beet by-products for both biohydrogen and biomethane production. The findings of this study demonstrate that the operation at high temperature conditions led to volatile and long chain fatty acids accumulation, thus inhibiting and slowing down the anaerobic process. From the obtained results, it could be concluded that the use of separated phases for the anaerobic digestion of sugar beet by-products was not necessary either.

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Prof. Romero-García has been involved in more than 50 Research Projects, being responsible for 14 of them. He has frequently acted as reviewer for more than 17 high impact factor journals. In addition, he is a regular evaluator of research projects for different national and international agencies and he has been a member of the National Project Evaluation Commission of the Spanish Plan for Research, Development and Innovation.