Electrical Conductivity for Monitoring the Expansion of the Support Material in an Anaerobic Biofilm Reactor

Authors:

Oscar Marín-Peña, Alejandro Alvarado-Lassman, Norma A. Vallejo-Cantú, Isaías Juárez-Barojas, José Pastor Rodríguez-Jarquín, Albino Martínez-Sibaja

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Keywords: organic solid wastes, anaerobic biofilm reactor, inverse fluidized bed reactor, electrical conductivity, anaerobic digestion

Abstract:

This article describes the use of the electrical conductivity for measuring bed expansion in a continuous anaerobic biofilm reactor in order to prevent the exit of support material from the reactor with the consequent loss of biomass. The substrate used for the tests is obtained from a two-stage anaerobic digestion (AD) process at the pilot scale that treats the liquid fraction of fruit and vegetable waste (FVW). Tests were performed with the raw substrate before anaerobic treatment (S1), the effluent from the hydrolysis reactor (S2), and the effluent from the methanogenic reactor (S3) to evaluate its effect on the electrical conductivity values and its interaction with colonized support material. The tests were carried out in a 32 L anaerobic inverse fluidized bed reactor (IFBR), which was inoculated with colonized support material and using two industrial electrodes at different column positions. The results with the previously digested samples (S2 and S3) were satisfactory to detect the presence of support material at the points where the electrodes were placed since the electrical conductivity values showed significant changes of up to 0.5 V, while with substrate S1 no significant voltage differences were appreciated. These results demonstrate that electrical conductivity can be used as an economic and simple mean for monitoring the support material expansion in order to avoid over expansion in the IFBR. It was also demonstrated that the conditions of the substrate in the methanogenic stage (pH and presence of volatile fatty acids) do not affect the operation of the electrical conductivity detection system.

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División de Estudios de Posgrado e Investigación, Tecnológico Nacional de México/Instituto Tecnológico de Orizaba, Av. Tecnológico No. 852 Col. E. Zapata, 94320 Orizaba, Veracruz, Mexico; oscar.marin.ind@gmail.com (O.M.-P.); nvallejoc@prodigy.net.mx (N.A.V.-C.); isaias_jubi@hotmail.com (I.J.-B.); jprj_02@hotmail.com (J.P.R.-J.); albino3_mx@yahoo.com (A.M.-S.)

* Correspondence: lassman@prodigy.net.mx

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Keywords: anaerobic digestion; electrical conductivity; inverse fluidized bed reactor; anaerobic biofilm reactor; organic solid wastes

1. Introduction

Anaerobic digestion (AD) is a biochemical technological process where complex organic substrates are degraded by a consortium of microorganisms through four stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis producing intermediary products, mainly volatile fatty acids (VFAs) and biogas as the final product, which consists mostly of methane (CH_4) and carbon dioxide (CO_2) [1–3]. The anaerobic process has received increasing attention in recent years and has been widely used for the treatment of organic substrates such as sewage, industrial effluents, animal manure, and organic solid substrates, both at laboratory and pilot scales [4–7]. The anaerobic reactor is the main element in which the AD process occurs. Between several configurations, the anaerobic inverse fluidized bed reactor (IFBR) has the advantage of operating in continuous with high organic load effluents, using low density support particles that are downward fluidized by the liquid flow, requiring lower

energy requirement for expanding the particles [5,8–12]. Unlike traditional fluidization, the inlet for the reactor feed is at the top, while the effluent outlet at the bottom. This system has a recirculation flow which takes advantage of the downward force exerted by the liquid by expanding the support towards the bottom of the reactor in a reverse fluidization. The use of support particles in IFBRs solves the problems that occur during conventional fluidization [13]. These particles require low flow rate for expansion, therefore, it can be easily controlled [11,14]. The maximal biofilm thickness at which no diffusional limitation is observed, is usually around 100 µm. Therefore, to operate the bioreactor efficiently, the biofilm thickness should be approximately 100 µm [15].

There are numerous investigations in the treatment of wastewater using the IFBR and studies are generally focused on achieving optimal configurations to improve removal efficiencies and increase CH_4 yield under high organic loads and short hydraulic residence times [7,11,13]. However, due to the dynamic nature of the system, variations in the density of the support material as a consequence of biomass accumulation result in over-expansion and loss of support material causing the release of the biofilm and consequently, decrease in the efficiency of the reactor with respect to the biogas production and organic matter degradation. In addition, the biogas generation as the final product of the decomposition of organic matter also contributes the bed expansion, which is a phenomenon known as pseudofluidization. This phenomenon is one of the main causes of the rapid expansion of the support material up to 100% of the working volume [16,17]. A possible solution to this problem is through the implementation of a sensor system that detects variations in electrical conductivity between the substrate and the biofilm support as this is expanded so the inlet flow can be modified and controlled to avoid the exit of support material.

Electrical conductivity is defined as the ability of a solution to conduct electrical current and is directly proportional to the ion concentration. Measuring the electrical conductivity is quite simple: a cell formed by two electrodes is placed in the sample and the current is measured when an alternative potential is applied across the electrodes. Furthermore, this type of sensor has a very long lifespan and does not require maintenance [18]. This parameter has been previously used for AD process monitoring. The evolution of bicarbonate concentrations and the generation of biogas were estimated in line by means of conductivity sensors to indicate the start-up period of an anaerobic fixed-bed reactor [19]. It has also been combined with pH measurement in the development of a titration system for total ammoniacal nitrogen, VFAs, and inorganic carbon [20]. In another study, the concentrations of VFAs, alkalinity, and bicarbonate were measured during the AD process in two different processes, the first in an anaerobic reactor for hydrogen production on a laboratory scale and the second in a pilot scale reactor for CH_4 production [18]. These investigations confirm the use of electrical conductivity as a reliable parameter for measuring different phenomena within the AD process. Goswami and Sarma [21] evaluated the biodegradable part of the organic solid wastes (OSW) through analysis of pH, electrical conductivity, and available concentration of organic matter as well as carbon. In this study, the pH was mainly found to be almost neutral to alkaline in the water-soluble part of the solid waste samples and the electrical conductivity value for water soluble part and the acid digested part varied widely for the same sample extract. Siddiqqui and Rameshwar [22] found that electrical conductivity is a factor that influences the decomposition reactions of municipal organic solid wastes (MOSW) composed of fruit and vegetable wastes (FVWs) and food wastes (FWs) and indicates the variability in compost material due to the activities of microorganisms. Another study showed that electrical conductivity increases during the end of the MOSW composting process. This increase is related to changes in organic matter and mineral salts [23].

To date, there are no reports of the use of electrical conductivity measurements for monitoring the support material expansion in anaerobic biofilm reactors, for this reason, the aim of this work was to find out if the electrical conductivity using an industrial sensor system can be an effective method to measure the support bed expansion analyzing the differences between the support material that has low conductivity but it is surrounded by biofilm and the wastewater that in this case is the liquid

fraction of FVW and thus be able to avoid one of the main problems of this configuration by bed over expansion and the consequent loss of the support material.

2. Materials and Methods

2.1. Laboratory-Scale IFBR Characteristic

A laboratory-scale 32 L IFBR was implemented to emulate a wastewater treatment system. In figfig:processes-677427-f001 are shown the IFBR diagram and the system used for the electrical conductivity tests.

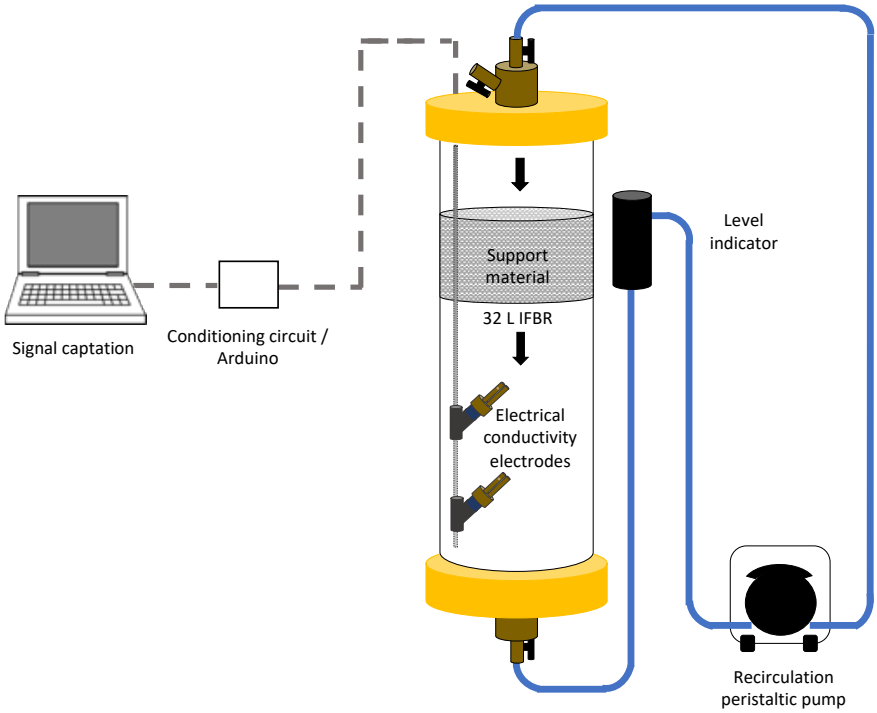

Figure 1. Inverse fluidized bed reactor (IFBR) diagram at laboratory scale and conductivity monitoring system.

In Table 1, the main features of the anaerobic reactor are specified. The anaerobic reactor consists of an acrylic column and two hermetically sealed caps. It was installed a level indicator through which the substrate was recirculated with a MasterFlex L/S 77200-52 variable speed peristaltic pump. The support material used for the test was colonized Extendosphere® since these particles have been studied in IFBRs at different operating conditions such as descending velocity, organic load rate (OLR) and type of substrate presenting stable biofilm formation, low energy requirements for fluidization and high organic matter removal [5,7,13]. Extendosphere® is mostly composed of SiO_2. The shape of the particle is perfectly spherical, the surface of the material present small crevices with an air bubble in the interior. The density of this particles is 0.69 g/mL, average diameter of 170 mm and the specific surface area is 20,000 m^2/m^3 [16,24]. In the present investigation, the colonized support material was taken from a pilot–scale IFBR that had been operated for a 1-year period with liquid fraction of FVW.

For the electrical conductivity measurement, two industrial polysulfone electrodes for water treatment were installed into the IFBR at 55% for electrode 1 (E1) and 75% for electrode 2 (E2) of the column height. These electrodes have a double cylinder structure in which its anti-filtration surface is resistant to corrosive environments. A signal conditioning and amplification circuit was used for signal collection. This circuit was connected to an Arduino UNO R3 and the software used was Arduino 1.8.8.

Table 1. Scale-laboratory IFBR characteristics.

Parameter	Value	Unit
Height	1.15	m
Diameter	0.19	m
Total volume	32	L
Of total volume		
Biogas chamber	5	L
Working volume	27	L
Of working volume		
Useful volume	19	L
Support material	8	L

2.2. Substrate Description

The IFBR was fed and recirculated with the liquid fraction of FVW that were collected from a local market located in Orizaba, México. This liquid fraction of the FVW is treated as wastewater in a two-stage AD process which is carried out in a hydrolysis reactor (HR) and a methanogenic reactor (MR), both pilot-scale. The two-stage AD process is operated at mesophilic temperature with average biogas production of 2040 L/day. The liquid fraction of FVW was obtained through a pretreatment, which include selection, trituration and centrifugation. FVWs are the main composition of MOSW, but woody waste, leaves and bones are also found (these residues are not usable to obtain a liquid fraction and, therefore, are removed). Residues with high moisture content are triturated and then filtered in an industrial centrifuge with a 3.5 mm mesh for solids retention to obtain the liquid fraction. The remaining solid fraction was withdrawn for other studies.

The tests were conducted with the substrate taken from three different places of sample with different conditions: Substrate taken from sampling place 1 (S1) corresponding to raw substrate, the sampling place 2 (S2) corresponding to HR effluent/MR influent and sampling place 3 (S3) corresponding to MR effluent. The diagram of S1, S2, and S3 obtained from the two-stage AD at pilot-scale is shown in Figure 2.

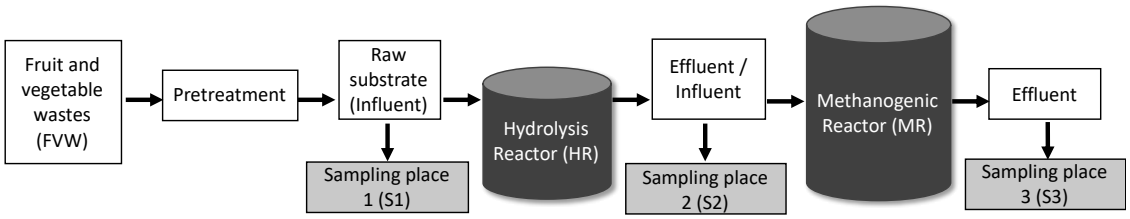

Figure 2. Substrate obtained from a two-stage Anaerobic Digestion (AD) at pilot-scale.

The tests were performed with the substrate obtained at different phases of the AD process due to it has different characteristics and therefore, different electrical conductivity. pH in S1 is acid, with average values of 4.5. pH values in S2 are between 6.8–7.2, and for S3, pH is 8.3. The physicochemical characteristics of the substrate at different conditions are shown in Table 2.

Table 2. Physicochemical characteristics of the substrate at different conditions and places of sample.

Parameter	Unit	Sampling Place 1 (S1)	Standard Deviation	Sampling Place 2 (S2)	Standard Deviation	Sampling Place 3 (S3)	Standard Deviation
COD	g/L	30	5.89	23.18	4.04	9.90	1.86
SCOD	g/L	26.15	4.70	16.83	4.37	8.21	1.40
TS	g/L	12.86	2.98	7.15	3.07	3.28	1.24
VS	g/L	7.82	2.66	3.79	1.93	0.90	0.83
pH	-	4.5	0.15	6.8	0.28	8.3	0.16
Voltage	V	0.85	0.04	1.9	0.02	2.0	0.03

pH in substrate S3 was increased to 9 with sodium hydroxide (NaOH) because in the period in which the electrical conductivity test was carried out average pH on MR effluent was 8.3. However, in previous operation periods, the MR effluent had almost reached a pH value of 9.

Due to this type of sudden changes that may occur in pilot-scale anaerobic reactors, acid, neutral and alkaline pH were tested to evaluate response of electrical conductivity measurement.

2.3. IFBR Operating Conditions

The IFBR operating conditions were similar to the conditions carried out at MR in two-stage AD process. 8 L of colonized support material (30% of working volume) taken from the MR were used to inoculate the IFBR. For each test, 19 L of each substrate from the different sampling places (S1, S2 and S3) were fed and then recirculated in order to expand the support material. The data acquisition initiate when the recirculation was started and finish with the compaction of the support material when the recirculation flow was stopped. Data acquisition were carried out using the signal conditioning and amplification circuit connected to an Arduino UNO R3 using Arduino 1.8.8 software. Voltage values were registered every second in order to obtain more accurate data in the variation of electrical conductivity. These voltage lectures were varying as the support material were descending and having contact with both electrodes.

2.4. Expansion Tests of Support Material to Determine the Downward Velocity

Hydrodynamic tests were performed for the colonized support material expansion (Figure 3a) with respect to time to determine the appropriate downward velocity for the electrical conductivity tests. Figure 3b shows the three different downward velocities used for these tests, 0.98 m/h, 1.30 m/h, and 1.41 m/h. The purpose of these hydrodynamic tests was to evaluate three different downward velocities, (low, medium and high), in order to analyze the behavior of the support material expansion. The three downward velocities were obtained according to the peristaltic pump flows: low (0.98 m/h), which can be used to reduce recirculation when there is an over expansion of the support material, medium (1.30 m/h), which can be used for stable recirculation within an IFBR and high (1.41 m/h), which can be used to quickly fluidize the support material. Alvarado-Lassman et al. [7] studied similar downward velocities using Extendosphere® and triturated polyethylene concluding that liquid velocity, amount, shape, and size of support material had a significant effect on support expansion.

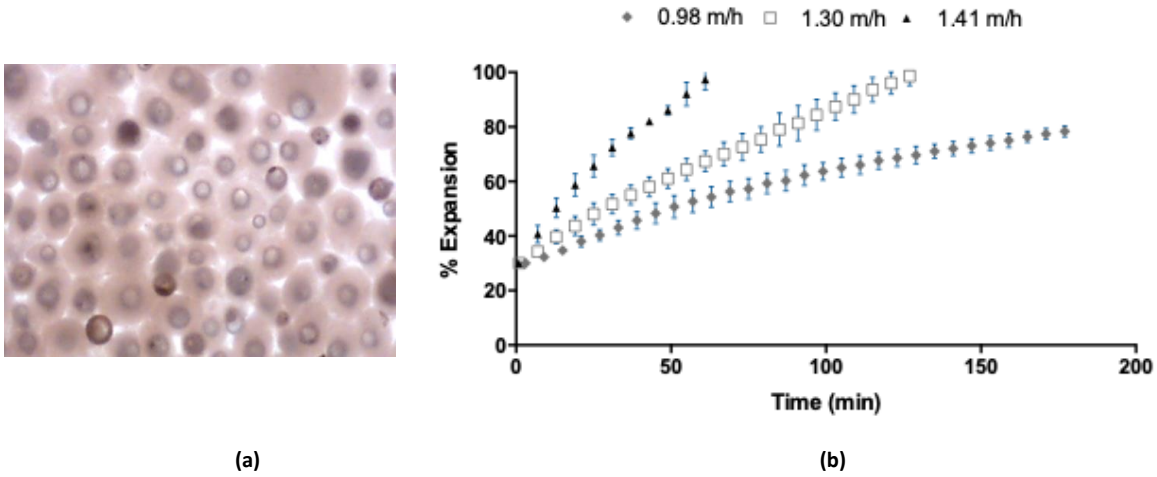

Figure 3. Hydrodynamic tests for the expansion of the support material: (**a**) Colonized Extendosphere® support micro photo used at 200× and (**b**) Downward velocities tested.

In a 180 min period, the support material only expanded up to 80% of the IFBR column with 0.98 m/h. These conditions are very unlikely to occur in a full load IFBR due to the ascending generation of biogas that expands the support material without the use of recirculation [16,17]. For

both conditions of descending velocity: 1.30 m/h and 1.41 m/h, the support material reached 100% expansion of the column in 130 and 63 min respectively. However, due to the presence of biogas and unpredictable expansions of the support material in full load IFBRs, the descending velocity selected for the electrical conductivity tests was 1.41 m/h, thus also adjusting the substrate recirculation for the electrical conductivity tests to 1-h period.

3. Results

3.1. Colonized Extendosphere® Expansion

Electrical conductivity tests were performed with the substrate at different conditions in combination with the support material already colonized. The results obtained from each expansion test were expressed in voltage units and are shown in Figure 4.

Figure 4a shows the results of S1 substrate recirculation at pH 4.5 corresponding to raw substrate. In this condition, the substrate, which had not been received the two-stage AD treatment yet, presents a high amount of organic matter that is easily degraded and acidified [25–27]. The E1 had contact with the support material after 660 s and the E2 after 1140 s. However, there was not any significant variation in voltage during contact between the support material and the substrate. Recirculation was stopped at 3600 s for compaction of the support material.

The results of the test with the S2 substrate are shown in Figure 4b. This condition corresponds to HR effluent and MR influent with pH adjusted to 7 with the purpose of emulate the neutral conditions of the methanogenic stage of the two-stage AD process. pH values in a range of 7.0–7.5 favor the methanogenic stage of the anaerobic digestion process since this factor increases biogas production and the specific CH_4 yield [28]. Under these conditions, the bed expanded faster than in tests with the S1 substrate. E1 had contact with the support material approximately at 480 s and E2 at 840 s. A favorable voltage variation is observed at the moment of contact between the support material and both sensors, with a decrease of 2.01 to 1.61 V for E1 and 1.96 to 1.45 V for E2. After expansion of support material, recirculation was stopped and the voltage slowly returned to its initial values due to bed compaction.

Figure 4c corresponds to the test performed with S3 substrate. Substrate condition S3 was recirculated and E1 had contact with the support material after 540 s and E2 after 900 s. The recirculation was stopped at 3600 s for bed compaction. This test also shows a favorable voltage difference between the compacted and expanded support material. The voltage variation is even greater than the variations found with the S2 substrate, since in the case of E1, the voltage decreased from 1.92 to 1.58 V in its maximum and minimum values. For E2, the voltage difference was even greater, decreasing from 1.95 V in its maximum values to 1.38 V in its minimum values. Voltage difference was between 0.4–0.5 V for E1 and E2, respectively. As shown in the graphs, for the case of substrate S2 and S3, even with the biofilm present in the support material, the difference in voltage between the compacted bed and the expanded bed is significant.

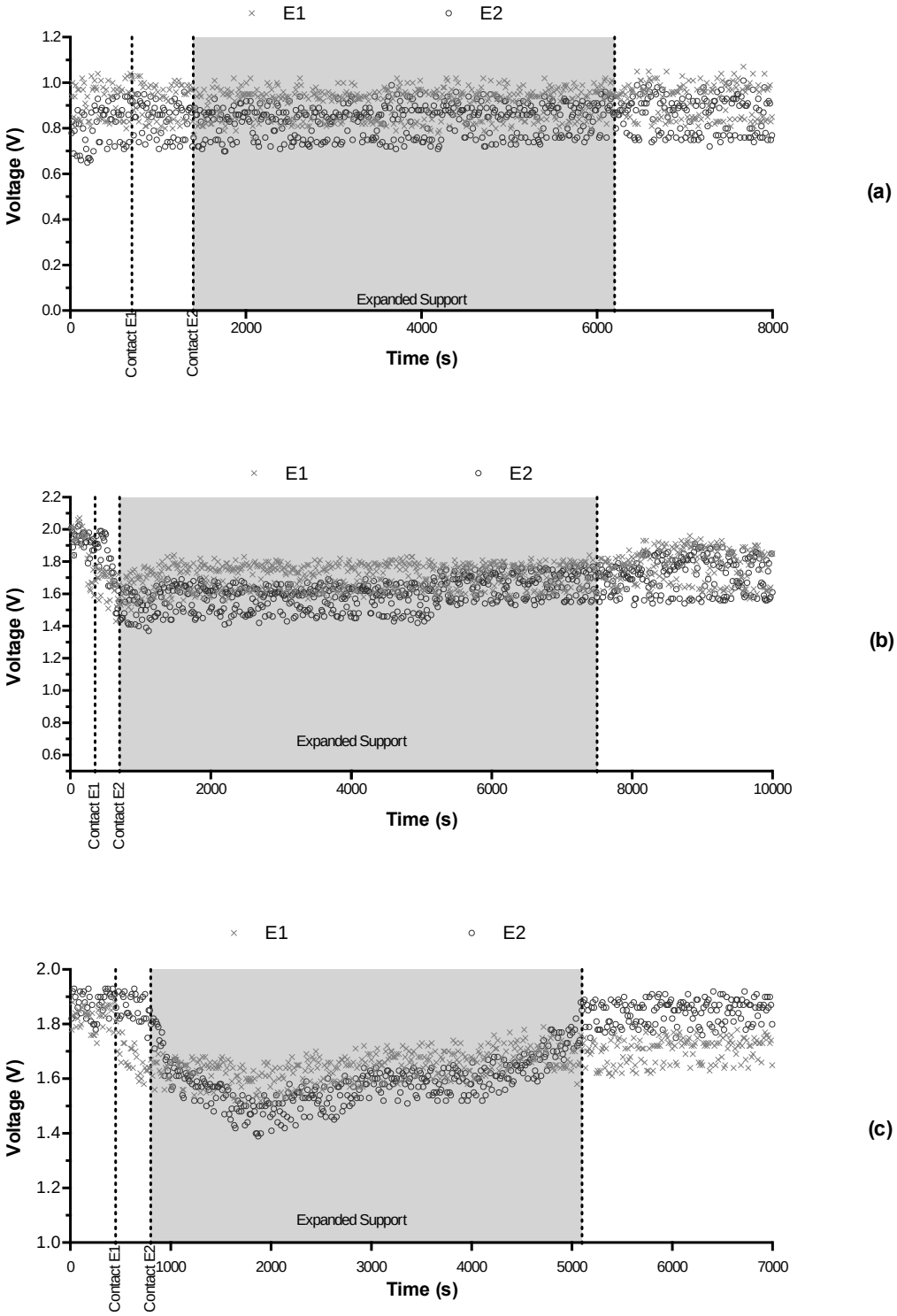

Figure 4. Electrical conductivity registered by electrode 1 (E1) and electrode 2 (E2) in the presence of colonized support material and substrate at different sampling places: (**a**) Sample 1 (S1) at pH 4.5; (**b**) Sample 2 (S2) with average pH of 6.8–7.2; and (**c**) Sample 3 (S3) at pH 9.

3.2. *Analysis of Electrical Conductivity Variation*

There are previous studies where electrical conductivity has been used to measure parameters in AD process such as VFAs, alkalinity [18], bicarbonate concentration, methane production [19], inorganic carbon, and total ammonia nitrogen [20] with different types of substrates such as molasses,

vinasses, and winery wastewater. However, the present study focused in VFA and pH analysis since FVWs are carbohydrate-rich residues which cause rapid acidification, pH decreasing, and high VFA production. The VFA production also increases with OLR variation. Studies on anaerobic digestion of FVWs have demonstrated that problems in methanogenic activity in anaerobic digesters occur with accumulation of VFAs [29–31]. In the acid stage of the two-stage AD, VFAs are produced and these intermediary products increase depending on the composition of the residue and the OLR [32]. VFAs are present throughout the AD process, in the first phase is where the formation of VFA occurs. In the second phase, methanogenic microorganisms convert VFAs to CH_4 and CO_2 [28].

There was no appreciable variation in S1 because the substrate in this place of sample had not been degraded by microorganisms and it is well known that in AD process, the ionic concentration depends mainly on the presence of VFAs and carbonates/bicarbonates and hence the increase in electrical conductivity is related to the concentrations of these elements [33]. Therefore, the conditions of the substrate S2 and S3 are the main interest in this work, since they are the conditions that may be present in a methanogenic pilot-scale IFBR.

The experimental results of the electrical conductivity variation with respect to the concentration of VFAs in the substrate are shown in Figure 5.

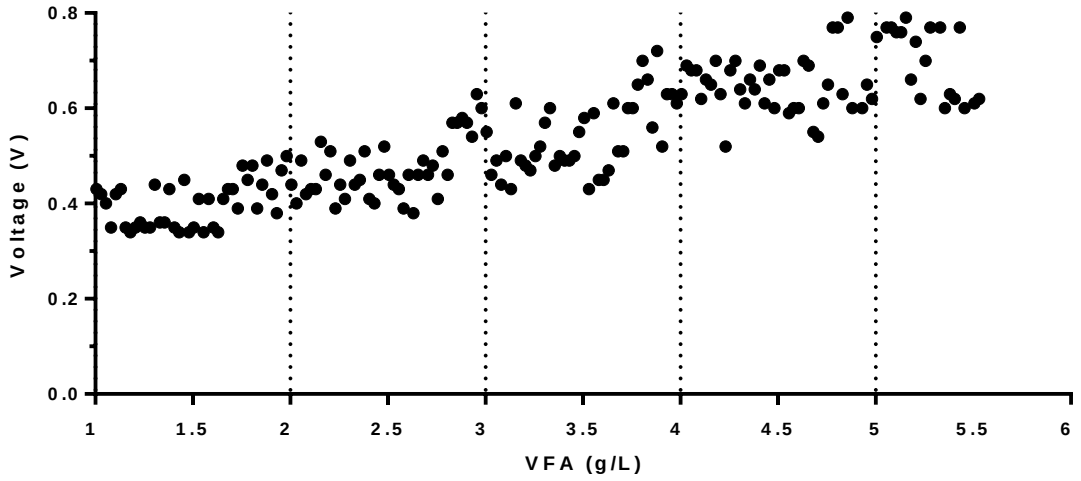

Figure 5. Electrical conductivity versus volatile fatty acid (VFA) concentrations in the liquid fraction of fruit and vegetable wastes (FVWs).

For this test, the IFBR was recirculated using liquid fraction of FVW at pH 7 and acetic acid (CH_3COOH) since this compound is present in greater quantity within the VFA produced in AD [28,32]. Different concentrations of CH_3COOH (1–5 g/L) with substrate were prepared. These concentrations were gradually added into the reactor and the electrical conductivity variation was detected with the electrodes as the amount of CH_3COOH increased. VFA measurements were performed using the alkalimetric titration method proposed by Ripley et al. [34]. The amount of VFA produced in previous studies conducted in the two-stage AD process at pilot scale from which the substrate was obtained has been less than 5 g/L. There are studies of anaerobic digestion of FVWs where the range of VFA produced in AD process is between 1 and 5 g/L [18,29,35,36]. There are studies where the VFA production in AD of FVW exceeded 5 g/L [32,37]. However, methanogenic activity is drastically reduced when VFA production excedeed 5 g/L [38,39]. For these reasons, VFA tests with electrical conductivity electrodes were performed in a range of 1 to 5 g/L for this study.

These results obtained in Figure 5 are similar to those predicted and evaluated by Aceves-Lara et al. [18]. It is observed that with the increase in VFA concentration from 1 to 5 g/L, the electrical conductivity in the medium increases from 0.3 to 0.79 V. These results are consistent with the tests in substrates S2 and S3, since there is a large voltage difference when the support material is compacted and when it is expanded, because in the first case, the electrodes detected the electrical conductivity of

the substrate that has been hydrolyzed and in the second case, the electrodes detected a decrease in the electrical conductivity due to the insulating characteristics of the support material with biofilm.

3.3. Effect of VFA Generation on the Detection of Electrical Conductivity with Expanded Support Material

VFAs have proven to be toxic inhibitory compounds within AD if they are generated in an uncontrolled manner. For this reason, previous studies suggest a two-stage AD process for FVW treatment [25,40,41]. In order to evaluate possible effects that may cause the generation of VFAs in anaerobic digestion to the electrical conductivity system by electrodes, 25 L of 5 g/L CH_3COOH concentration were injected into the IFBR in a 1-h period in continuous mode operation, using the S2 substrate and colonized support material. The results of this test are shown in Figure 6.

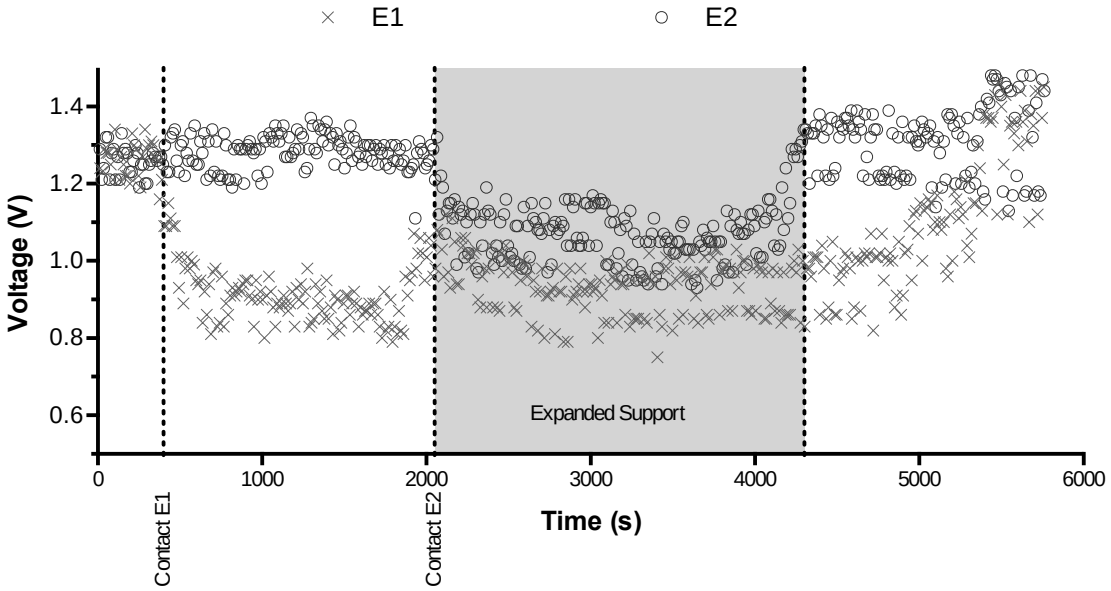

Figure 6. VFA effect in electrical conductivity detection.

The downward velocity was lower (0.98 m/h) with respect to the previous tests with the purpose to avoid the rapid expansion of the support material, and thus detect a possible increase in conductivity due to the passage of the substrate with CH_3COOH. The contact between E1 and the support material occurred at 450 s. The electrical conductivity decreased significantly. E2 had contact until 2100 s, and it was also observed a considerable decrease in electrical conductivity. The substrate recirculation was stopped after 3600 s and then, as the bed compacted, the voltage increased again. Slight variations in electrical conductivity can be observed for E2 between the start of the test until contact with the support material. These variations are attributed to the pass of CH_3COOH over the electrodes before bed expansion. However, it was found that regardless of the concentrations up to 5 g/L of VFA generated in the AD process, the electrical conductivity decreases considerably when there is contact between the support material and the electrodes.

4. Conclusions

From the analysis of the experimental results obtained in this study, the following conclusions are obtained: the variations in electrical conductivity were analyzed with the expansion of the support material using three substrate conditions, which represent three points in the two-stage AD process: raw substrate (S1), HR effluent/MR influent (S2), and MR effluent (S3). A significant difference in the electrical conductivity was observed in the tests with the substrates S2 and S3, concluding that the generation of VFAs, the increase in pH, as well as the presence of biofilm, favor the detection of the support material when expanding inside the IFBR column. The support material expansion detection system by electrical conductivity sensors has better performance in the methanogenic stage

of the two-stage AD process. The presence of VFAs of up to 5 g/L in the methanogenic stage does not affect the detection of the expansion of the support material by means of electrical conductivity. FVWs are carbohydrate-rich substrates which acidify and degrade rapidly causing inhibition of methanogenic activity. For these reasons, this study was conducted to evaluate pH and VFA production in electrical conductivity measurement. The electrical conductivity can be interesting to monitor ionic concentrations within the medium by means of industrial conductivity electrodes, which are characterized by being reliable, robust, and easy to maintain. This monitoring parameter allows one to identify the moment in which the support material reaches the desired height in the column of an IFBR. Once the electrical conductivity electrodes are installed, they could also be used to monitor parameters such as pH, alkalinity, VFA, organic matter concentration, start-up period reactors, sulfates and sulfides, ammonium nitrogen and ammonia, and carbonates among other parameters. The results obtained in this study will serve as a basis for further studies in the implementation of an automatic control system in a pilot-scale methanogenic IFBR to monitor, detect, and control the expansion of the support material and, thus, avoid the loss of biomass attached to the support material and the consequent decrease in the anaerobic reactor efficiency.

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