Vol. 23, No. 3(2024) Bio24313
Revista Mexicana de Ingeniería Química

Evaluation of the reaction capacity of early warning indicators to failures in biogas production systems

Evaluación de la capacidad de reacción de los indicadores de alerta temprana ante fallas en sistemas de producción de biogás

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Received: April 19, 2024; Accepted: July 11, 2024

Abstract

To avoid inhibition in biogas full-scale production processes, continuous monitoring along with control and instrumentation techniques is needed. A crucial part of the monitoring system is the use of early warning indicators, which facilitate the timely identification of suboptimal performance within the digester and allow the application of corrective measures in an acceptable time frame. This work evaluated the ability of early warning indicators to react to a failure in a biogas production process after an alteration in the substrate composition. The results show that the most used indicators in the literature failed to react satisfactorily to the scheduled failure. Statistical analysis was used to develop an indicator based on the relationship between biogas productivity and volatile fatty acid concentration, which exhibited adequate precision at the programmed failure. This research contributes to the biogas production processes by offering in-depth analysis data and by enhancing our understanding of the methods required for effective monitoring and control.

Keywords: alkalinity, anaerobic digestion, monitoring and control, methane, stability, inhibition.

Resumen

Para evitar la inhibición en los procesos de producción de biogás a escala industrial, se necesita un monitoreo continuo junto con técnicas de control e instrumentación. Una parte crucial del sistema de monitoreo es el uso de indicadores de alerta temprana, que facilitan la identificación oportuna de un desempeño subóptimo dentro del digestor y permiten la aplicación de medidas correctivas en un marco de tiempo aceptable. Este trabajo evaluó la capacidad de indicadores de alerta temprana para reaccionar a una falla en un proceso de producción de biogás luego de una alteración en la composición del sustrato. Los resultados muestran que los indicadores más utilizados en la literatura no lograron reaccionar adecuadamente a la falla programada. Se utilizó el análisis estadístico para desarrollar un indicador que se basa en la relación entre la productividad del biogás y la concentración de ácidos grasos volátiles, que exhibió una precisión adecuada a la falla. Esta investigación contribuye a los procesos de producción de biogás al ofrecer datos de análisis en profundidad y mejorar nuestra comprensión de los métodos necesarios para un seguimiento y control eficaces.

Palabras clave: alcalinidad, digestión anaerobia, monitoreo y control, metano, estabilidad, inhibición.

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https://doi.org/10.24275/rmiq/Bio24313
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Anaerobic digestion (AD) is a multi-stage biological process where various microbial consortia degrade complex organic matter into biogas (CH_4, CO_2, H_2, and H_2S) and biofertilizer. The biodegradation of organic matter consists of four main stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Gerardi, 2003). From a technical-environmental perspective, the biogas generated in this process has the potential as a clean energy source by substituting fossil fuels and fertilizers (Albalate-Ramírez *et al*., 2022).

Europe had more than 18,943 biogas plants by the year 2020 (EBA, 2020), which operate with food waste, the organic fraction of municipal solid waste, sewage sludge, agro-industrial and slaughterhouse waste, energy crops, and cattle manure as substrates (Dahlin *et al*. 2015). However, many of these wastes present a nutritional imbalance (in terms of carbohydrates, proteins, and lipids), and the AD processes turns vulnerable to different types of inhibition, which tends to aggravate the process instability (Miramontes-Martínez *et al*. 2021) and methane production (Albalate-Ramírez *et al*., 2023), which leads to economic losses for biogas plants (Liu *et al*. 2021). A study carried out by Miramontes-Martínez *et al*. (2022), which evaluated the techno-economic and environmental impacts of waste-to-energy processes from the anaerobic digestion of food waste on an industrial scale in Mexico, found that stable biogas yields, together with government subsidies for the clean energy production, play a fundamental role in the economic success of these processes. Additionally, the study found that these recovery alternatives lead to high greenhouse gas mitigations of -71. kg CO_2 eq/t of food waste, compared to 867 kg CO2 eq/t if these wastes are confined in landfills.

Process instability, in AD, is understood as the inability to buffer the effects made by external disturbances to the digester (Liu *et al*., 2017). At an industrial level, instability can be reflected in periods with low degradation of organic matter and limited biogas production. Nielsen *et al*. (2008) analyzed several digesters in operation in Denmark and found that methane production decreased by up to 70% over several months due to process instability caused by changes in feed composition. The authors conclude that the main factor that leads to the instability of the process in digesters on an industrial scale is the lack of knowledge of the composition of the substrates and the low monitoring of the production of metabolic intermediates in the process. According to Galván-Arzola *et al*. (2022), approximately 65% of AD systems in Latin America are Covered Anaerobic Lagoons (CALs). Of these, a significant 92% lack agitation systems and temperature control mechanisms, reducing the degradation of organic matter and methane production.

To mitigate the instability and increase the methane production in AD, it is crucial to know the metabolic state of the digesters. This task can be achieved through monitoring and control methods (Ren *et al*. 2018).

Developing monitoring systems requires parameters with high sensitivity and rapid response, which provide valuable information on the metabolic state of the digester. For this, early warning indicators (EW) are recommended in scientific literature (Onwosi *et al*., 2019; Li *et al*., 2014; Sun *et al*., 2019). The most widely used EW are those based on physicochemical tests, which are economical, and the technology is readily available (Wu *et al*., 2019). Biological EW are also present in the scientific literature, but their application in full-scale AD operation is not feasible due to high economic value and technical sophistication.

Physicochemical EW measures AD metabolic intermediates or end products, which can often be determined through titration or gas chromatography techniques. Although the fluctuations in the values of the physicochemical EW can be considered as the result and not the origin of the instability, monitoring these indicators is good enough to provide valuable information regarding the digester health. Threshold, values for the limit of stability of these EW have also been investigated, and diverse operating conditions have been proposed based on this information (Li *et al*., 2018).

Alkalinity indicates the capacity of the aqueous environment to react with acidic species and buffer the pH. I.e., it is the ability to prevent pH changes to transitions in acid concentrations. In AD, three types of alkalinities are usually used, depending on certain ionized species' presence. Partial alkalinity (PA) is due to the presence of species such as OH^-, NH_3, HCO_3^-, and CO_3^{2-}, while intermediate alkalinity (AI) is linked to the presence of conjugated bases of volatile fatty acids generated as metabolites intermediate in AD. The sum of these two alkalinities results in total alkalinity (TA).

The use of EW has been a subject of extensive and intricate study in the scientific literature. Despite numerous works, a unified consensus on the ideal value for detecting process instability is yet to be established, as they exhibit significant variations (Hagos *et al*. 2017). A commonly employed EW is the ratio between intermediate and partial alkalinity (IA/PA), which symbolizes two crucial phenomena in AD: the production of organic acids and the buffering capacity of the reactive medium. However, even with the discovery of many limit values in IA/PA, most studies present conflicting results. Pereira *et al*. (2013) suggested an IA/PA lower than 1.44 for digesters managing manure. In contrast, Santana *et al*. (2005) proposed values lower than 0.44 for processes using the same substrate. Escamilla-Alvarado *et al*. (2012) found no detrimental effect up to 1.22 for fermented organic waste. Ferrer *et al*. (2010) recommended a maximum of 0.9 for digesters handling residual sludge, while Fernandes *et al*. (2006) advised values below 0.23 when treating residual sludge.

These discrepancies and contradictions in the research findings are of significant importance. For instance, if the substrate undergoing AD is predominantly protein material, the hydrolysis of the proteins and acidogenesis of the amino acids will lead to high concentrations of $N\text{-}NH_3$ and fatty acids such as valeric and acetic (Gerardi, 2003). Clearly, these species will influence the evaluation of the IA/PA as an early warning indicator. This EWI can vary considerably when other types of fractions dominate in the substrate, such as carbohydrates, which, during the acidogenesis process, will result in higher concentrations of acids like acetic, propionic, and butyric (Batstone *et al*., 2002). The disparities in the alert values are evident in almost every EWI. Table 1 provides a comprehensive review of the threshold values of the EWI in various AD processes, considering different types of substrates, reactor configurations, and operational conditions.

The lack of knowledge in the predictive capacity of EW is a latent issue for AD processes at industrial scale. Through an in-depth evaluation of operating digesters, this work aims to evaluate the reaction capacity of early warning indicators to a programmed alteration in the substrate composition of anaerobic digesters. The results will provide valuable information for decision-making in the operation of anaerobic digesters, directly helping their monitoring and control. Correct monitoring and control of AD is crucial to ensure its economic and operational success, a process for generating valuable products and sustainable waste management.

Table 1. Summary of early warning indicators (EWI) of anaerobic digestion experiments carried out in recent decades.

Substrate	Reactor configuration	Operative condition	EWI	Stability thresholds	Reference
Cellulose and glucose	Batch	35 ºC	VFA	<6000 mg L^{-1}	Siegert *et al*., (2005)
AW, manure, and SS	ND	ND	VFA	<4000 mg L^{-1}	Lee *et al*., (2015)
Manure	UAF	ND	VFA	<200 mg L^{-1}	Santana *et al*., (2005)
Manure	UAF	22.5 °C	VFA	<100 mg L^{-1}	Pereira *et al*., (2013)
Manure	UAF	21 °C	VFA	<150 mg L^{-1}	Pereira *et al*., (2009)
Cheese serum and manure	PFR	26 °C	VFA	<5000 mg L^{-1}	Escalante-Hernández *et al*., (2017)
AW and manure	CSTR	54 °C	VFA	<1500 mg L^{-1}	Søndergaard *et al*., (2015)
SS	CSTR	55 °C	VFA	<2500 mg L^{-1}	Ferrer *et al*., (2010)
OFMSW	CSTR	37 °C	VFA	<2500 mg L^{-1}	Martín-González *et al*., (2013)
Manure	CSTR	35 °C	VFA	<6000 mg L^{-1}	Li (2018)
OFMSW	CSTR	-	VFA/TA	0.1-0.4	Zahedi *et al*., (2019)
AW and YW	Batch	-	VFA/TA	0.4-0.6	Brown & Li, (2013)
SW, FW, and YW	CSTR	37 °C	VFA/TA	<0.5	Korai, *et al*., (2018)
AW; OFMSW	CSTR	55 °C	VFA/TA	<0.07	Ghanimeh *et al*., (2012)
Cheese serum and manure	PFR	26 °C	VFA/TA	<0.4	Escalante-Hernández *et al*., (2017)
AW	Batch	ND	VFA/TA	0.3-0.4	Kong *et al*., (2016)
Manure	CTR	23 °C	VFA/TA	<0.6	Castro *et al*., (2017)
CR	Batch	39 °C	VFA/TA	<0.63	Pilarska *et al*., (2019)
SW	Batch	35 °C	VFA/TA	<0.5	Slimane *et al*., (2014)
Glucose	CSTR	37 °C	VFA/TA	<0.15	Pontoni *et al*., (2015)
AW	Batch	ND	VFA/TA	<0.9	Wang *et al*., (2012)

SS	UASB	30–35 °C	VFA/TA	<0.3-0.4	Sánchez *et al*., (2005)
Manure, FVW, and Slurry	CSTR	35 °C	VFA/TA	<0.4	Callaghan *et al*., (2002)
Manure and FW	PFR	31 °C	VFA/TA	<0.08	Lane, (1984)
AW and OFMSW	CSTR	55 °C	HPr	<488 mg L^{-1}	Ghanimeh *et al*., (2012)
HAc, HPr, HBt, and EtOH	Batch	35 °C	HPr	<900 mg L^{-1}	Wang *et al*., (2009)
Manure	ND	26–30 °C	HPr	<5000 mg L^{-1}	Yeole *et al*., (1996)
Manure	CSTR	55 °C	HPr	<3000 mg L^{-1}	Ahring *et al*., (1995)
SS	AnMBr	55 °C	HPr	<2000 mg L^{-1}	Wijekoon *et al*., (2011)
Hac, HPr, and HBt	UAF	20 °C	HPr	<312 mg L^{-1}	Demirel *et al*., (2002)
Manure	Batch	38 °C	HPr	<1000 mg L^{-1}	Hobson & Shaw, (1976)
Glucose	CSTR	35 °C	HPr	<2750 mg L^{-1}	Pullammanappallil *et al*., (2001)
Hac, HPr, and HBt	FBR	37 °C	HAc	<200 mg L^{-1}	Gorris *et al*., (1989)
SS	CSTR	33 °C	HAc	<4700 mg L^{-1}	Kaspar & Wuhrmann, (1977)
Manure	CSTR	35 °C	HAc	<800 mg L^{-1}	Hill & Bolte, (1987)
Manure	CSTR	55 °C	HAc	<6000 mg L^{-1}	Ahring *et al*., (1995)
AW and OFMSW	CSTR	55 °C	HAc	<825 mg L^{-1}	Ghanimeh *et al*., (2012)
SS	CSTR	55 °C	HAc	<600 mg L^{-1}	Ferrer *et al*., (2010)
Glucose	CSTR	55 °C	HPr/HAc	ND	Marchaim *et al*., (1993)
Manure	CSTR	35 °C	HPr/HAc	<1.4	Hill & Bolte, (1987)
SS	CSTR	55 °C	HPr/HAc	<0.5	Ferrer *et al*., (2010)
AW	Batch	ND	HPr/HAc	<0.08	Wang *et al*., (2012)
AW and OFMSW	CSTR	55 °C	IA/PA	<0.45	Ghanimeh *et al*., (2012)
Manure	UAF	22.5 °C	IA/PA	<1.44	Pereira *et al*., (2013)
Manure	UAF	ND	IA/PA	<0.47	Santana *et al*., (2005)
SS	UAF	21 °C	IA/PA	<0.23	Fernandes *et al*., (2006)
Manure	UAF	24 °C	IA/PA	<0.41	Pererira *et al*., (2009)
Manure	Batch	35 °C	IA/PA	<0.3	Ripley *et al*., (1986)
OFMSW	CSTR	37 °C	IA/PA	<0.3	Martín-González *et al*., (2013)
Manure	UAF	21 °C	IA/PA	<0.85	Pereira *et al*., (2009)
SS	CSTR	55 °C	IA/PA	<0.9	Ferrer *et al*., (2010)
AW	UAF	30 °C	IA/PA	<0.4	Balaguer *et al*., (1992)
Manure and glycerol	CSTR	35 °C	IA/PA	<0.4	Astals et al., (2012)
VW	CSTR	55 °C	CH_4/CO_2 IA/TA	<0.2 <0.8	Li *et al*., (2018)
FVW and SS	CSTR	35 °C	IA/TA	<0.4	Arhoun *et al*., (2019)

Substrates. OFMSW=organic fraction of municipal solid waste; AW=agroindustrial waste; YW=yard waste; VW=vegetable waste; FW=fruit waste; FVW=fruit and vegetable waste; SS= sewage sludge; SW= slaughterhouse waste; CR= confectionery residues; HPr=propionic acid; HAc=acetic acid; HBt=butyric acid; EtOH= ethanol.

Reactor configurations. CSTR=continuous stirred tank reactor; CTR= continuous tubular reactor; AnMBR= anaerobic membrane bioreactor; UAF=upflow anaerobic filters; FBR=fluidized bed reactor; PFR=plug-flow reactor; UASB= upflow anaerobic sludge blanket.

EWI. VFA=volatile fatty acids; IA=intermediate alkalinity; PA=partial alkalinity; TA=total alkalinity; HPr=propionic acid; HAc=acetic acid.

2 Materials and methods

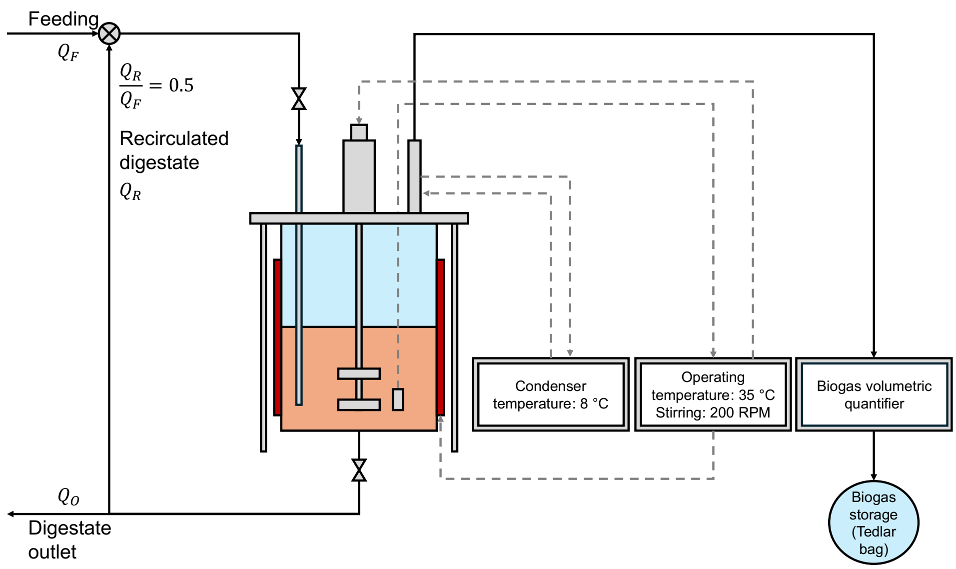

Fig. 1. Design of the reactor used. QF is the feed flow rate, QR is the recirculated digestate flow rate and QO is the outlet digestate flow rate.

Table 2. Inoculum characterization results.

Parameter	Units	R1	R2	Quality criteria*
Methanogenic activity	mL CH_4 g VS^{-1}	325.9 ± 74.4	325.9 ± 74.4	340 a 395
TS	g L^{-1}	68.86	58.70	
VS	g L^{-1}	15.68	15.78	
Ash	g L^{-1}	53.18	42.92	
TA	mg $CaCO_3$ L^{-1}	8090 ± 659.62	7806.67 ± 115.04	> 3000
IA	mg $CaCO_3$ L^{-1}	2483.33 ± 417.41	2426.67 ± 70.24	
PA	mg $CaCO_3$ L^{-1}	5606.67 ± 307.46	5380 ± 104.40	
IA/PA		0.44 ± 0.06	0.45 ± 0.05	
VFA	mg L^{-1}	544.40 ± 184.39	498.84 ± 27.61	< 1000
VFA/TA		0.067 ± 0.017	0.064 ± 0.003	
pH		7.76	7.62	7.0 an 8.5

TS: total solids. VS: volatile solids. TA: total alkalinity. IA: intermediate alkalinity.
PA: partial alkalinity. VFA: volatile fatty acids.
*Based on Holliger *et al*. (2016); Holliger *et al*. (2020).

2.1 *Experimental tests of programmed failures in anaerobic digestion processes*

2.1.1 *System reaction*

The AD process was carried out in two Applikon® borosilicate glass reactors, R1 and R2. Shown in Fig. 1. Each reactor had a stainless-steel flange at the top, sealed with a rubber gasket, which houses the feeding ports, liquid effluent outlet, and gas outlet. To minimize moisture loss within the reactor, the gas outlet is equipped with a counterflow condenser cooled with water at 8 °C. The cooling water for the condenser was provided by a Scorpion® temperature bath equipped with a recirculation pump. The biogas flowed through a Prendo® volumetric flow sensor connected to a Tedlar® bag. The volumetric flow was used to quantify biogas productivity (m^3 m^{-3} biogas d^{-1}). The biogas was accumulated in the bag and replaced daily by an empty bag. The biogas collected daily was stored for subsequent quantification by gas chromatography.

Table 3. Nutrient concentration of the mineral solution added to the substrate feed.

Nutrient	Concentration (mg L^{-1})
NH_4Cl	1500
KH_2PO_4	300
$FeCl_3 c6H_2O$	5
$NiSO_4 \cdot 6H_2O$	0.447
$(NH_4)6Mo_7O_{24} \cdot 4H_2O$	0.037
$CoCl_2 \cdot 6H_2O$	0.4

Each reactor was connected to a control interface for temperature and stirring and a pH sensor. Temperature control was achieved by maintaining an isothermal regime at 35 °C using an Applikon® heating blanket. At the same time, powered stirring was facilitated at the top of the reactor using 60 mm diameter Rushton propellers.

2.1.2 Substrates and inoculum selection

The sludge from a CSTR-type anaerobic digester that manages wastewater from a chocolate industry in the south of Nuevo Leon State in Mexico (25°11'13.99" N, 99°49'36.01" W) was employed as inoculum. Experimental tests were conducted to quantify the methane production capacity of the microorganisms in the inoculum through the methodology described by Hussain *et al*. (2017). A known concentration of glucose and inoculum is added to serological bottles with an operating volume of 60 mL. The tests were carried out in triplicate, at an operating temperature of 35 °C and with manual stirring twice a day until the methane production did not exceed a 5% difference compared to the previous day. The inoculum was characterized utilizing physicochemical tests via Mexican standards (Table 2), including a solids profile analysis (NMX-AA-034-SCFI-2001), total nitrogen by the Kjeldahl method (NMX-F-068-1980), volatile fatty acids (VFA), and alkalinity measurement (NMX-AA-036-SCFI-2001).

A challenge associated with utilizing organic waste in AD processes is the limited reproducibility of results, primarily stemming from the physical and bromatological variations within the waste. Factors such as geographical location and source of collection (Albalate-Ramírez *et al*. 2024), seasonal variations (Fisgativa *et al*. 2016; Alibardi and Cossu 2015), collection methods, and consumption patterns (Alibardi and Cossu 2015) significantly contribute to this variability. To increase reproducibility and decrease the influence of the variability of organic residues, we adopted a synthetic substrate proposed by Astals *et al*. 2014. The substrate consists of Sigma-Aldrich® cellulose, analytical grade Bacto® casein, and commercial olive oil (Great-Value®) as sources of carbohydrates, proteins, and lipids, respectively, with the specified proportions detailed below. A mineral solution was added as a nutrient source (Table 3) in the feed (Demirel *et al.* , 2002).

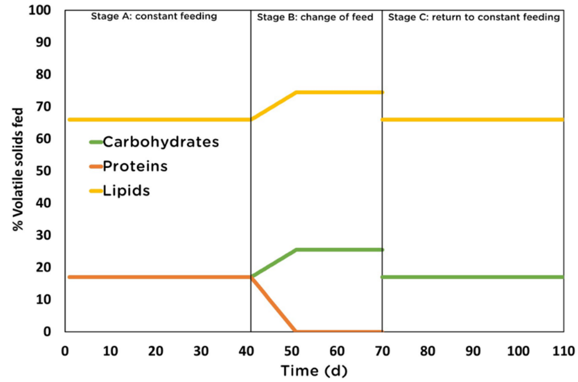

Fig. 2. Feeding scheme of the anaerobic digestion process.

2.1.3 Feeding strategy

The feeding scheme consisted of four stages, as shown in Fig. 2, excluding the incubation stage, which was not monitored. Feed preparation was carried out daily, and the hydraulic retention time (HRT) and organic load rate (OLR) for both reactors were set at 21 days and 3 g VS L^{-1} d^{-1}, respectively. The selected HRT is suitable for effective methanation in laboratory-scale organic waste anaerobic digestion processes (Miramontes-Martínez *et al*. 2021, Albalate-Ramírez *et al*. 2023).

Incubation stage. During this stage, the reactor loading process was carried out to 60% of its total volume with inoculum. Industrial-grade nitrogen was introduced for five minutes to promote anaerobic conditions. While biogas generated during this stage was stored, its volume and concentration were not quantified.

Stage A: constant feeding. Both reactors were subjected to semi-continuous feeding, divided into two parts by volume. The first part consisted of the synthetic substrate, with a dry basis proportion of 17% carbohydrates, 17% proteins, and 66% lipids. The second part was recirculated digestate, as specified in Fig. 1. These feeding conditions remained constant until a daily biogas production with no more than a 10% variation in the relative standard deviation (RSD, equation 1) compared to the previous day was achieved for at least 6 consecutive days, indicating a steady state (De Francisci *et al*. 2015). In the above equation, $\bar{x}$ is the average of the values at day i and i-1.

$$RSD = \frac{\sqrt{\sum_{i=1}^{2}(x_i - \bar{x})^2}}{\bar{x}}(100\%) \tag{1}$$

Stage B: change of feed. Following the steady state, a feeding disturbance was initiated by gradually removing the nitrogen source from the substrate. The protein content in the substrate decreased by 10% per day, compensated by a proportional increase in carbohydrate and lipid content until reaching a dry basis proportion of 25.5% carbohydrates and 74.5% lipids. These feeding conditions were maintained until daily biogas production was considered negligible. To determine the day of process failure, a pH value lower than 6.4 was used as a criterion, which is below the optimal pH range of methanogenic archaea (Liu *et al*. 2008). pH is one of the most intuitive and easy to quantify indicators, but insensitive to the destabilization of the process; however, in previous studies, it is recommended to analyze its changes as a quick measure to detect a failure in the process (Chen et al. 2008; Wu *et al*. 2019).

Stage C: return to constant feeding. Finally, the feeding conditions of stage A were reinstated to analyze the digester's recovery capacity and assess the predictive capacity of the EW.

Throughout all stages, the digestate was stored at 4 °C in sealed PET containers and characterized daily following Mexican standards for solids profile (NMX-AA-034-SCFI-2001), Kjeldahl total nitrogen (NMX-F-068-1980), volatile fatty acids (VFA), and alkalinity (NMX-AA-036-SCFI-2001). Methane content in the biogas was determined daily using a Shimadzu® gas chromatograph equipped with a flame ionization detector. The chromatographic program was initiated with an oven temperature of 40 °C for 1.5 minutes, followed by a ramp of 40 °C per minute up to 200 °C. Nitrogen (N_2) was utilized as the carrier gas, and a mixture of hydrogen (H_2) and air was employed for ignition. Detector and injector temperatures were set at 280 °C and 250 °C, respectively. The gas sample in the inlet chamber was split at a 100:1 ratio. Methane determination in the biogas was performed using a 5-point calibration curve with an R_2 value of 0.999.

2.1.4 *Selection of early warning indicators subject to study*

The EW analyzed were pH, volatile fatty acids (VFA), total (TA), partial (PA) and intermediate (IA) alkalinity, the methane content in the biogas, and the ratios of VFA/TA, IA/PA, and CH_4/ CO_2. The ability to react to the appearance of a programmed failure of the EW was analyzed according to the statistical process control theory via the use of Shewhart control charts (Miramontes-Martinez *et al*. 2020). A control chart for the EW of interest required means and standard deviations. These were calculated using Equation 2 and 3, using data from a period where the process displayed the desired behavior. The time range used in the study spanned from the beginning of stationary state (determined in section 2.1.3) to the end of the experiment.

$$\mu_j = n^{-1} \sum_{i=1}^{n} x_{i,j} \ \forall j \tag{2}$$

$$\sigma_j = \sqrt{n^{-1} \sum_{i=1}^{n} \left(x_{i,j} - \mu_j\right)^2} \ \forall j \tag{3}$$

where j is the process variable (EW of interest), i is a single data point in the sample, μ_j is the sample mean; σ_j is the standard deviation of the jth variable; n the number of data points; and $x_{i,j}$ is the *i-th* data point of the j-th variable in the sample.

The process data were plotted alongside a upper control (UCL_j), and lower control lines (LCL_j), which were calculated according to Equation 4 and 5. In these equations, α is a positive constant that sets the limits in which the process variable can oscillate, where lower values can be used to achieve tighter process control. In this case, a value of α= 3 was used, as this is the standard practice (Montgomery and Runger, 2002).

$$UCL_j = \mu_j + \alpha\sigma_j \ \forall_j \tag{4}$$

$$LCL_J = \mu_j - \alpha\sigma_j \ \forall_j \tag{5}$$

The Pearson correlation coefficient was used as a statistical measure to quantify the linear relationship between the continuous variables used in the EW and propose a new indicator. The Kolmogorov-Smirnov (K-S) test was used to evaluate the variation in the distribution of the derivative of each EW during the stable period (with beginning determined by the RSD of biogas productivity and ending with a decrease in pH to less than 6.4) and compare it with the derivative of the same indicator in the instability period (determined from the decrease in pH to 6.4 until the beginning of feeding stage C). To determine statistical significance, a level of 0.05 was chosen.

3 Results and discussion

3.1 *Performance of the biogas production process*

The pH of reactor 1 (R1, in red) and reactor 2 (R2, in blue) are shown in Fig. 3. The process failure days corresponded to the 57th for R1 and 55th for R2. Li *et al*. (2017) mention that the pH variations usually are associated with VFA accumulation. The pH gradually decreased during the Stage B operation, mainly due to the loss of protein in the feed (Fig. 2) rather than to the accumulation of VFA (Fig.6). The decrease in protein and its subsequent absence in the diet limits the fermentation of amino acids and the release of NH_3, which affects its balance with the NH_4^+ ion in aqueous solution and decreases the buffering capacity

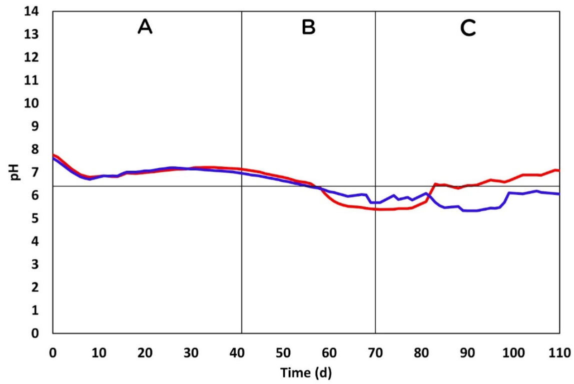

Fig. 3. pH of the reactors: Reactor R1 in red and R2 in blue.

of the reagent medium and its stability (Kumar *et al*. 2010; Zhang *et al*. 2013; Miramontes-Martínez *et al*. 2021). After the failure day, a sharp drop in pH is observed, mainly caused by the variation of the VFA of the system (Fig. 6).

The results of biogas productivity are shown in Fig. 4. R1 presents a stable state (RSD of productivity less than 10%, Fig. S1) during days 25 to 47 and R2 on days 22 to 44. During this time, the average productivity was 2.27 ± 0.14 and 2.63 ± 0.16 m^3 m^3 d^{-1}, and the average methane content was 72.85 ± 4.65% and 72.38 ± 4.27%, respectively, for R1 and R2. Ren *et al*. (2022) obtained comparable results in a lipid co-digestion process with food waste in thermophilic regime. They determined productivities of 2.36 – 3.42 m^3 m^3 d^{-1} with methane contents of 59.7 – 67.7% when lipids predominated in the feed. It has been shown that an adequate increase in the proportion of lipids in the substrate tends to increase methane production due to the high efficiency of conversion of lipids to methane (Jeganathan *et al*. 2006). However, an excessive increase in lipids can cause system failure, due to inhibition by long-chain fatty acids (LCFA) (Astals *et al*. 2021). This inhibition was not detected in this work, which could be due to the use of an inoculum acclimated to substrates rich in lipids, since it comes from an anaerobic digester that manages waste from a chocolate-making industry.

During Stage C, a high variation in the RSD of biogas productivity can be observed in both reactors (Fig. S1), which shows that these processes fail to return to a stable state quickly. This phenomenon may indicate that returning to the conditions present in the stable period through a sudden change in the feed does not guarantee the stability condition before the failure. Zou *et al*. (2022) analyzed an industrial-scale thermophilic AD process. They observed that it is difficult to recover the digester quickly when digesters decrease biogas production due to a critical failure. The results provided in this work may indicate a remnant of intermediate metabolites in the reaction medium from the feeding of Stage B, which alter the microbiota of the reaction medium. According to heuristics, at least three HRTs are required to remove a particle from the reaction medium through digestate from its appearance.

According to the literature, it is suggested to monitor the methane content in the biogas; nevertheless, this can become expensive due to the need for specialized equipment and trained personnel (Wu et. al 2019). Kainthola *et al*. (2020) recommend values greater than 50% methane in the biogas as a stability criterion. It can be seen in Fig. 4 that the methane content was unable to detect system failure (days 57 for R1 and 55 for R2), revealing its limited effectiveness as an EW.

Another indicator related to biogas composition is the CH_4/CO_2 ratio, shown in Fig. 5. In biogas plants, CH_4 and CO_2 concentrations are frequently monitored by automatic biogas analyzers (Zou *et al*. 2022) and gas chromatography at a laboratory scale (Wu et al. 2019). The literature suggests a minimum ratio of 1.2 as an alert criterion (Li *et al*. 2017). In this work, the CH_4/CO_2 ratio could not detect the day of failure in both reactors (day 57 for R1 and 55 for R2). In AD processes with carbohydrates increase in the substrate, the CO_2 content in the biogas and the VFA in the reactive medium will increase because they are generated in the acetogenesis and acidogenesis of the intermediate metabolites of carbohydrate degradation. The gradual increase in lipids in the feed also increases the VFA concentration but does not directly influence the CO_2 content in the biogas. At the same time, the generation of methane in methanogenesis will decrease due to the adverse conditions caused by the increase in VFA.

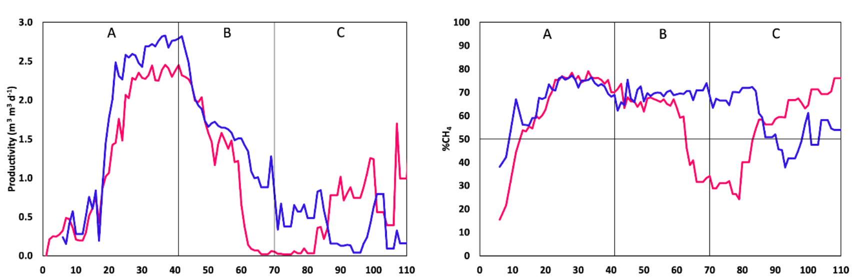

Fig. 4. Biogas productivity and methane content of the anaerobic systems. Reactor R1 in red, and reactor R2 in blue.

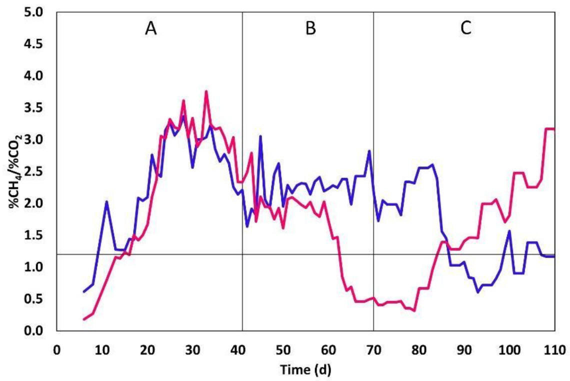

Fig. 5. Early warning indicator $\%CH_4/\%CO_2$. Reactor R1 in red, and reactor R2 in blue.

The total alkalinity (TA) concentration profile is shown in Fig. 6. TA provides stability to the reaction medium against sudden changes in pH through the balance between CO_2, bicarbonates, NH_4^+, and VFA ionized (Ward *et al*. 2008; Miramontes-Martínez *et al*. 2021). The buffer capacity of the system is proportional to the bicarbonate concentration and can be indicative of the performance of the process (Rocamora *et al*. 2020). In this work, a constant decrease in RT was observed before the day of failure (day 57 for R1 and 55 for R2), which decreases the buffer capacity of the reactive medium. Li *et al*. (2017) and Miramontes-Martínez *et al*. (2020) found a similar behavior, ascribing it to the lack of effluent recirculation in the reactive system, which causes a gradual loss of ammonia due to the dilution of the system since the feed has a low concentration of proteins, precursors of such metabolite. This work used a recirculation ratio 0.5 between the feed and the recirculated digestate (Fig. 1). However, it seems insufficient to reduce the loss of TA from the system in conditions before the failure. After day 60 for R1 and 67 for R2, a sharp increase in the TA values was observed, which may be due to the increase in soluble monosaccharides (from cellulose hydrolysis), which in the stage of acidogenesis and acetogenesis generate VFA and CO_2. The ammonia (from casein breakdown) in the solution could have reacted with the soluble CO_2 in an irreversible reaction, creating ammonium carbonate (NH_4HCO_3), which in turn reacted with the VFAs to produce ammonium salts, as described in Equations 6 and 7.

$$NH_3 + CO_2 + H_2O \rightarrow NH_4HCO_3 \quad (6)$$

$$NH_4HCO_3 + RCOOH \rightarrow NRCOONH_4 + H^+ + HCO_3^- \quad (7)$$

Fig. 6 also shows the concentration profile of the VFAs. The literature recommends a maximum of 4000 mg L^{-1} as an alert criterion (Galván-Arzola *et al*. 2022). It is observed that before the day of the failure (day 57 for R1 and 55 for R2), the VFAs did not exceed the alert condition. The ineffectiveness of VFAs as EW may be due to their accumulation is generally the result rather than the cause of instability (Angelidaki *et al*. 2002).

One of the most investigated indicators is the VFA/TA ratio. Table 4 presents various recommended values for this EW showing that there needs to be more consensus in the literature on stable values. Some authors suggest a maximum allowed value, and others recommend a range of values. Ayodele *et al*. (2021) recommend values of VFA/TA between 0.23 and 0.3, Callaghan *et al*. (2002) recommend less than 0.4 for a stable digester, 0.4 to 0.8 as a precautionary range for signs of instability, and values greater than 0.8 for noteworthy instability. Veluchamy *et al*. (2019) report a stability range of 0.3 to 0.4 and values greater than 0.4 for possible risks. The literature elucidates that the operating conditions, type of reactor, and substrate influence the threshold values for this EW. However, this indicator can provide us with relevant information about the behavior of intermediate metabolites in the process. High values of this indicator are related to an overload of the system and the generation of CO_2 and H_2. In contrast, low values are related to a low concentration of organic matter in the feed and limited biogas production.

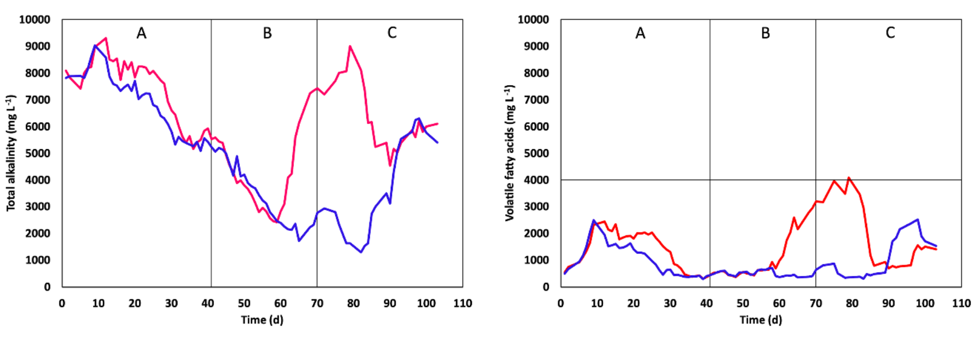

Fig. 6. Total alkalinity (TA) and volatile fatty acids (VFA) of the AD process. R1 in red and R2 in blue.

Table 4. Threshold values of the early warning indicator VFA/TA reported in the literature.

Value	Substrate	Reference
< 0.7	Fruit and vegetable waste	(Lane 1984)
< 0.5	Fruit and vegetable waste	(Korai *et al.* 2018)
< 0.4 (stable); 0.4 to 0.8 (inhibition risk); > 0.8 (inhibition)		(Callaghan *et al.* 2002)
< 0.4	Fruit and vegetable waste	(Bouallagui *et al.* 2009)
< 0.4	Coffee mucilage and swine manure	(Hernández *et al.* 2014)
< 0.4	Apple waste and swine manure	(Kafle *et al.* 2013)
< 0.35	Food waste and fruit and vegetable waste	(Jukuri *et al.* 2021)
< 0.35		(Rasapoor *et al.* 2020)
< 0.35		(Li *et al.* 2014)
0.3 to 0.4 (stable); > 0.4 (inhibition)		(Veluchamy et al. 2019)
0.23 to 0.3		(Ayodele *et al.* 2021)
< 0.25 ± 0.03	Cellulose, casein, and olive oil	This study
< 0.18	Vegetable waste	(Li *et al.* 2017)
< 0.1		(Evans *et al.* 2016)

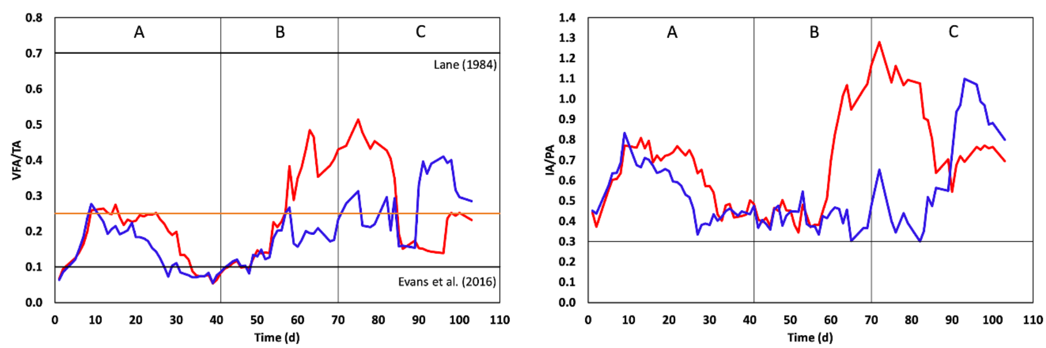

Fig. 7. Ratio (VFA/TA) and their relationship (IA/PA) in the anaerobic systems. R1 in red, and R2 in blue.

Fig. 7 shows the VFA/TA ratio of both reactors. The alert time of this indicator depends on the selected threshold value. Lane (1984) recommends a maximum value of 0.7, the highest encountered in the literature (Table 4); however, as shown in Fig. 7, for detecting the programmed failure (day 57 for R1 and 55 for R2), it was insufficient. Evans *et al.* (2016) suggest a maximum value of 0.1, a value surpassed on day 42 for both reactors, which gives an alert time of 15 and 13 days, respectively. Regardless, this value is relatively low and can generate "false positives" as in days 45 to 47, which decreased below 0.1. Considering the day of failure of both reactors (pH less than 6.4), the threshold value of VFA/TA for this study is 0.25 ± 0.03 (Table 4, orange line in Fig. 7).

The relationship between the intermediate (IA) and partial (PA) alkalinity is shown in Fig. 7. The IA results from the difference between the TA and the PA and is related to the alkalinity of the VFA. The PA is related only to the alkalinity of bicarbonate (Ripley *et al.* 1986). In the literature, values lower than 0.3 are recommended to avoid risks of instability (Martín-González *et al.* 2013; Jukuri *et al.* 2021). In this work, VFA/TA and IA/PA are influenced by the gradual loss of alkalinity of the digesters. The longer the operation time, the more these indicators will become sensitive to increases in VFA concentration.

3.2 *Stability analysis and proposal of new indicators*

The EW ability to react to a programmed failure was evaluated over two periods, from days 25 - 47, covering the process stability stage, and the second period covers the instability stage, starting on day 47 and ending on day 70 of operation. The mean value and standard deviation of the EW derivative, along with the parameters used to construct the Shewhart control charts, are presented in Table S1. Shewhart control graphs for R1 are shown in Figure 8 (Control graphs for R2 are shown in Figure 2S). When the values fall outside the limits set by UCL and LCL (black dotted lines) indicates a potential problem in the system related to inhibitory effects. The variability of the data is also of interest, since a good indicator

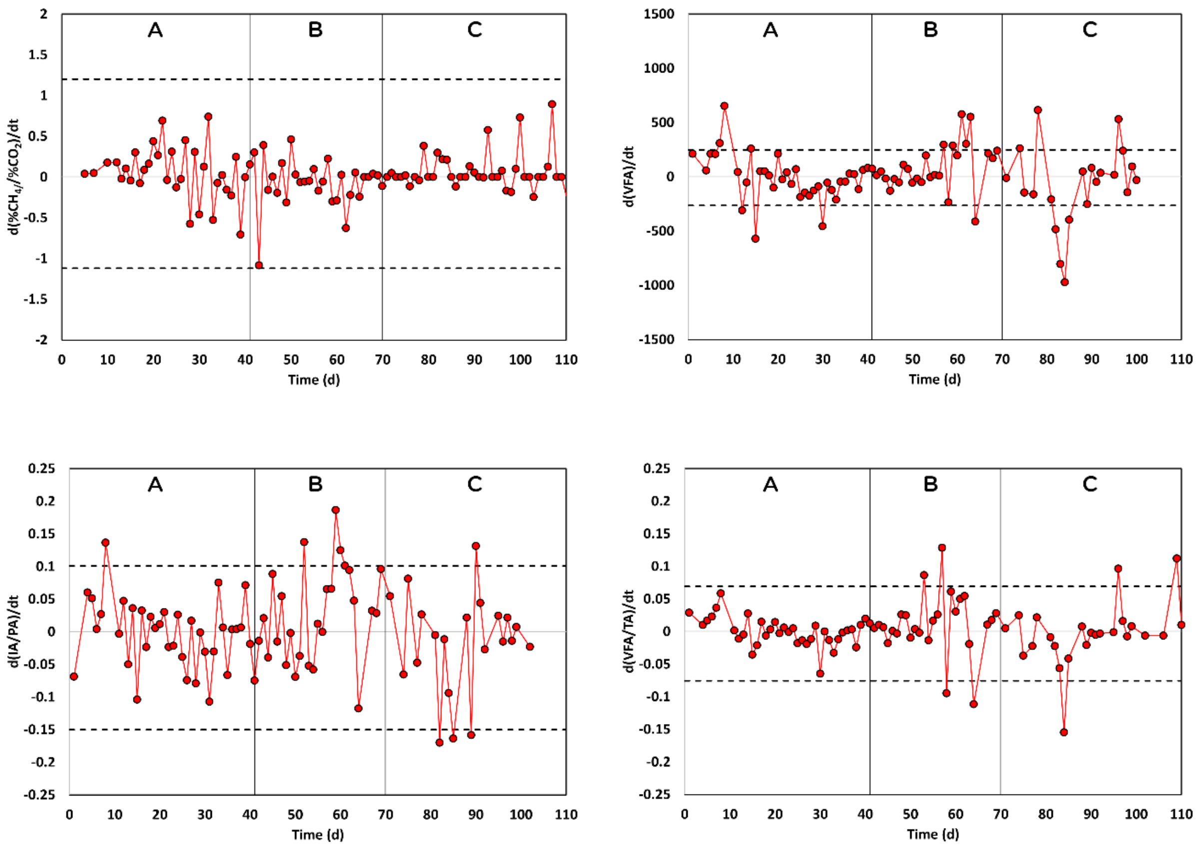

Fig 8. Shewhart control charts for the EW of R1.

Table 5. Parameter correlation for determining EW indicators.

R1	$\%CH_4$	$\%CO_2$	Productivity	VFA	TA	pH	IA	PA
$\%CH_4$								
$\%CO_2$	-0.9985							
Productivity	0.6828	-0.6828						
VFA	0.0144	-0.0144	-0.4546					
TA	-0.0449	0.0449	-0.2957	0.8158				
pH	0.2559	-0.2559	0.6288	-0.1098	0.3262			
IA	-0.0459	0.0459	-0.4457	0.9186	0.9520	0.0595		
PA	-0.0396	0.0396	-0.1162	0.6334	0.9514	0.5629	0.8116	
R2	$\%CH_4$	$\%CO_2$	Productivity	pH	TA	VFA	IA	PA
$\%CH_4$								
$\%CO_2$	-0.9855							
Productivity	0.5540	-0.5540						
pH	0.0233	-0.0233	0.3215					
TA	-0.2762	0.2762	-0.5478	0.4383				
VFA	-0.1618	0.1618	-0.7267	-0.0994	0.8105			
IA	-0.2647	0.2647	-0.6692	0.2021	0.9550	0.9174		

should present sudden changes in the presence of disturbances in the supply to the reactor and little or no variability during a continuous and stable supply. It is noted that these indicators show variability throughout the AD process. CH_4/CO_2 shows variation during the stable stage of the process (days 25 to 47) and the data did not fall outside UCL and LCL throughout the process. The other indicators exceeded these limits in point data, but quickly returned within them, not representing the correct continuation of the failure within the system.

To evaluate the EW a Pearson correlation table was made (Table 5), where those with values higher than 0.7 and lower than -0.7 (Saccenti *et al.* 2020) are considered as EW with high response sensitivity. Productivity shows a somewhat positive connection with both pH (0.6288) and VFA (0.6828), indicating that higher quantities of volatile fatty acids and elevated pH levels are associated with higher productivity. Remarkably, there are robust positive correlations between IA and PA and VFA, TA, and pH, suggesting complex interdependencies between these variables in affecting reactor dynamics. A comparable high negative correlation of -0.9855 between $\%CH_4$ and $\%CO_2$ is seen in R2. Furthermore, productivity has lesser connections with other factors and modest positive correlations with pH (0.3215). Additionally, there are strong positive associations between IA and PA and several measures, especially VFA and TA.

Table 6 shows the variations in the distribution of evaluated K-S test values between the steady state and the failure state for the EWs. Greater KS values indicate significant distributional differences when compared to other indicators, such as 0.224 for d(VFA/productivity)/dt in EW R1. This implies that, in contrast to other indicators, changes in this ratio are happening at a different rate or with a different distribution. When compared to other indicators, lower KS values, such as 0.036 for d(VFA/AI)/dt, indicate more consistent or similar changes. Spatial variability analysis is further made possible by comparisons between the identical derivatives in two different reactors (R1 and R2). For example, both R1 and R2 show KS values for d(VFA/productivity)/dt and d(AI/AT)/dt, providing information about how these indicators vary throughout experimental runs; this suggests that these specific indicators are sensitive to operational or environmental variations within the reactor setup.

Based on the statistical results, the indicator that relates the concentration of VFA and biogas productivity is proposed. A sudden change in the values of the indicator indicates a possible imbalance between the stages of acidogenesis and methanogenesis of the process, which in prolonged times of operation can represent a phenomenon of inhibition.

Table 6. K-S Test values.

EW R1	KS	EW R2	KS
d(VFA/productivity)/dt	0.224	d(VFA/productivity)/dt	0.201
d(AI/AP)/dt	0.184	d(VFA/AI)/dt	0.125
d(AI/AT)/dt	0.152	d(AI/AT)/dt	0.119
d(VFA/AT)/dt	0.125	d(AP/AT)/dt	0.119
d(TA/PA)dt	0.111	d($\%CH_4/\%CO_2$)/dt	0.111
d($\%CH_4/\%CO_2$)/dt	0.106	d(AI/AP)/dt	0.104
d(VFA/AI)/dt	0.036	d(VFA/AT)/dt	0.079

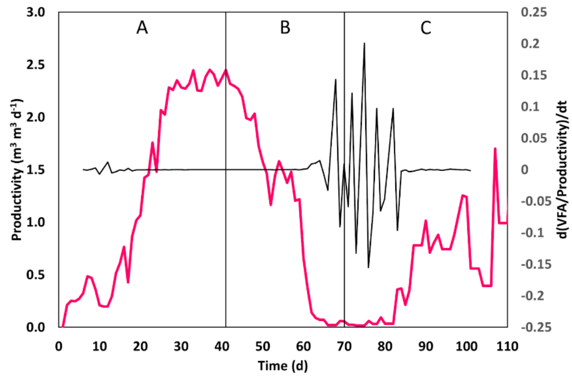

Fig. 9. Behavior of the relationship between VFA and biogas productivity proposed as EW for R1.

Figure 9 presents the suggested EW and its relationship to productivity, the values of UCL and LCL for R1 are -2.25x10^{-4} and 1.65x10^{-4}, respectively. These values are drastically exceeded from day 56, one day before the pH decreased below 6.4 (Figure 3), which could indicate the start of an unstable period and corresponds to the increase in the concentration of VFA and TA, and decreased biogas productivity and methane content. As of day 99, EW data returns within the range of UCL and LCL, which corresponds to the end of the period of instability. For R2 the EW determined a period of instability in days 92 to 102, Figure S3. The authors attribute this difference to two main reasons: *i)* Difference in the head of the liquid phase, which may cause mass transfer phenomena between the liquid and gaseous phase to have different transfer rates, which could cause a difference in association-dissociation reactions derived from the presence of carbonates in the reactive medium (Muntau *et al.*, 2021), and *ii)* differences in agitation shear effort. An increase in the volume of the reactor, maintaining the same angular speed of agitation, generates a greater cutting effort (Li *et al.*, 2023). This could result in R2 concentration profiles having a smaller volume and thus less agitation effort per unit volume as opposed to R1. This indicator presents a high sensitivity to the changes occurred in the power in the AD process compared to the indicators implemented in literature, so it proposes as a reliable indicator for the rapid and correct detection of failures in anaerobic digestion systems.

Conclusion

This work evaluates the predictive capacity of early warning indicators (EW) used for monitoring the stability of anaerobic digestion processes. Early warning (EW) indicators recommended in the literature were used as criteria to determine their reaction capacity to a programmed failure using their threshold values and Shewhart control charts. The results show that EWs have a limited ability to react to a failure. VFA/Productivity is proposed as a new EW, which was developed through a K-S statistical analysis. VFA/Productivity demonstrated an adequate sensitivity and capacity to respond to the programmed failure. However, more experiments under different operating conditions are necessary for the complete validation of this EW. The results of this work provide valuable information for decision-making in the operation of anaerobic digesters, helping to monitor and control them. Correct monitoring and control of AD is crucial to ensure its economic and operational success, a process of generating valuable products, and sustainable waste management.

Acknowledgements

The authors acknowledge the funding received from the *Programa de apoyo a la investigación científica y tecnológica-UANL (PAICYT)* (Grant numbers 591-IT-2022 and 573-IT-2022).

Nomenclature

x	average of the values at day i and i-1.
μ_j	is the sample mean
σ_j	is the standard deviation of the jth variable
n	the number of data points
$x_{i,j}$	is the *i-th* data point of the j-th variable in the sample
UCL_j	upper control
LCL_j	lower control lines
α	positive constant

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