A VFA-based controller for anaerobic digestion of industrial winery wastewater

Gustavo Vargas-Morales, Rolando Chamy and Santiago García-Gen

ABSTRACT

A variable-gain controller for anaerobic digestion of industrial winery wastewater is presented. A control law using both volatile fatty acids (VFA) and methane production rate as controlled variables and organic loading rate (OLR) as manipulated variable is defined. The process state is quantitatively estimated by an empirical function comparing VFA measurements against a setpoint value; then, it is modified with a second empirical function that compares the methane flow rate with a maximum capacity reference, and finally it is adjusted with a third factor considering the actual hydraulic retention time. The variable-gain function determines the extent of the OLR change applied to the system. The controller was successfully validated in a 95 L upflow-anaerobic-sludge-blanket (UASB) reactor, treating industrial wine wastewater at OLR ranged between 2.0 and 39.2 g COD/L d for 120 days at mesophilic conditions. Higher performance was achieved contrasted with a conventional strategy carried out in a parallel UASB unit.

Key words | anaerobic digestion, biogas, control, VFA, wine wastewater

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ABBREVIATIONS

AD	Anaerobic digestion
C/N	Carbon to nitrogen ratio
COD	Chemical oxygen demand
HRT	Hydraulic retention time
OLR	Organic loading rate
PA	Partial alkalinity
TA	Total alkalinity
TS	Total solids
UASB	Upflow anaerobic sludge blanket
VFA	Volatile fatty acids
VS	Volatile solids
VSS	Volatile suspended solids

INTRODUCTION

Wine industry produces large amounts of easily biodegradable wastewater, composed of a high content of soluble organic matter, mainly ethanol and volatile fatty acids (VFA) (Bolzonella *et al.* 2010). Anaerobic digestion (AD) is an alternative to treat this type of wastewater, which has been successfully proven at laboratory (Andreottola *et al.* 2009; Ioannou *et al.* 2015) and industrial scales (Moletta 2005).

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AD is a spontaneous process found in nature that consists on the biological degradation of the organic matter, catalysed by a cluster of microorganisms in absence of an external electron acceptor. The reaction pathway includes four major stages: hydrolysis, acidogenesis, acetogenesis and methanogenesis (Gujer & Zehnder 1983); the final product is biogas, a gaseous mix of methane, carbon dioxide and other trace elements such as hydrogen sulphide and hydrogen (Appels *et al.* 2008).

Even though this process has been massively used for sludge stabilization and treatment of agro-industrial wastes, digesters are frequently operated far from their maximum capacity, at moderate organic loading rates (OLR) to prevent overloading or system failure (Nguyen *et al.* 2015). As AD is a highly non-linear process, other more complex techniques than the classical time-invariant linear controller were developed to guarantee a good performance of the process (Steyer *et al.* 2006; Olsson 2012); these include adaptive control, model-based control, fuzzy logic and neural network. A detailed description of these techniques and examples for AD are summarised by Nguyen *et al.* (2015). Adaptive control can be suitable for non-linear systems since the control parameters could be automatically adjusted to offset process disturbances. According to an objective function, the adaptive control is able to self-adapt to process changes and output the optimal manipulated input that better fits the state of the system. The optimal parameter values are estimated based on a process model, which is normally derived from mass balances of the system. The main drawback of this technique could reside in the complexity of the model that requires advanced algorithms and knowledge of process kinetics. On the other hand, in model-based control, a reliable model of the process should be available. As large uncertainties can come from model kinetics, models should not be used in a wide range of operating conditions. Fuzzy logic is a knowledge-based control scheme that has been also applied to AD control. These controllers work as decision-making algorithms that rely on the knowledge of operators rather than on precise data from the plant. In fuzzy logic, expert knowledge is converted into functions to be applied in automatic control. The process parameters are divided into subsets ranging from 0 to 1. Eventually, the manipulated input is estimated based on the empirical rules defined from the interrelationship between those subsets. The limitation of fuzzy logic control lies in the strongly dependency on the expertise of the operator. As a result, the performance of this control approach varies widely from plant to plant. An artificial neural network algorithm is based on a computational model that is loosely inspired by the human brain. Plant data are processed through a net of nodes sorted in interconnected layers, in order to generate a final output signal. The core of the algorithm consists in adjusting the weight of the different nodes that generate an output signal in agreement with the current state of the process. This requires a training algorithm, where the neural network learns a wide variety of operating conditions. This technique can be very useful for process regulation purposes; however, it requires a substantial training time and a great deal of information. In addition to all these strategies, reliable instrumentation should be available to monitor the key process variables of AD. An extensive review of instrumentation and control for AD can be found in Jimenez *et al.* (2015) and Nguyen *et al.* (2015).

The sensitivity of the different process indicators can vary significantly under the same disturbance (Boe *et al.* 2010). A good number of studies found the VFA intermediates as the main variables to detect organic overloading (Björnsson *et al.* 2000; Nielsen *et al.* 2007; Boe *et al.* 2010; Adam *et al.* 2015); others, instead, have used the accumulation of the gas-phase hydrogen intermediate as the key control variable (Rodríguez *et al.* 2006; García-Diéguez *et al.* 2010; Giovannini *et al.* 2016). In addition, it is worth noting that some pre-treatment processes can increase the presence of these intermediate compounds before entering the digester. In general, pre-treatments aim at increasing solubilisation, enhance biodegradability, reduce particle-size, form refractory compounds and lose organic material by means of biological, thermal, mechanical or chemical processes (Carrère *et al.* 2010). For instance, VFA can be accumulated after a chemical pre-treatment with peroxides (Wang *et al.* 2018) to such an extent that affects the operation of the subsequent anaerobic digester. Finally, according to Jimenez *et al.* (2015), feed flow rate is the most commonly used manipulated variable for AD systems. However, this might not be appropriate for wastewater having a highly fluctuating organic content.

The aim of this work is to develop a variable-gain control strategy based on the VFA measurement (control variable) and OLR (manipulated variable) for the treatment of a real vineyard wastewater (having a variable chemical oxygen demand (COD) content), and validate it *in situ* in an upflow-anaerobic-sludge-blanket (UASB) pilot reactor. The performance of this controller will be compared against a conventional control approach (applied to a parallel UASB reactor) where the OLR changes of the entire operation are programmed from the beginning.

METHODS

Control strategy

A variable-gain controller was developed for the continuous operation of an AD process. The control law (Equation (1)) sets that the change in the OLR (manipulated variable) depends on the current OLR value and a variable gain K_C (Equation (2)). The gain expression, adapted from García-Diéguez *et al.* (2010), is a function of three variable factors (K, f_{CH4}, f_{VFA}), related to hydraulic retention time (HRT), methane production rate and VFA concentration (controlled variable), respectively.

$$\frac{dOLR}{dt} = OLR \cdot K_C \tag{1}$$

$$K_C = K \cdot f_{CH4} \cdot f_{VFA} \tag{2}$$

This study uses the discrete form of the control law, where for each control cycle n, the OLR is given by Equation (3).

$$OLR_{n+1} = OLR_n + OLR_n \cdot \Delta t \cdot K \cdot f_{CH4} \cdot f_{VFA} \tag{3}$$

where OLR_{n+1} is the OLR (kg COD m^{-3}d^{-1} units) that will be applied in the next cycle $n+1$; OLR_n is the average OLR applied during the current cycle n; Δt (d) stands for the time elapsed during a control cycle; K (d^{-1}) is the gain factor defined by Equation (4); f_{CH4} and f_{VFA} are defined as methane production factor and VFA factor, respectively. These two indicators are expressed through two empirical formulas (Equations (5) and (6)), similar to those developed by Rodríguez *et al.* (2006).

$$K = \frac{1}{3 \cdot HRT_n} \tag{4}$$

$$f_{VFA} = \left(1 - \frac{[VFA]}{[VFA^*]}\right)^m \qquad si\ [VFA] \leq [VFA^*] \tag{5.a}$$

$$f_{VFA} = \left(\frac{[VFA^*]}{[VFA]}\right)^n - 1 \qquad si\ \ [VFA] > [VFA^*] \tag{5.b}$$

$$f_{CH_4} = \frac{\alpha Q^*_{CH_4}}{Q_{CH_4} + \alpha Q^*_{CH_4}} \tag{6}$$

K is calculated at each control cycle from the current HRT value. As the controller is executed for a time gap $\Delta t = 3 \cdot HRT$ (steady state condition), then, the variable gain of the discrete form only depends on the term $f_{CH4} \cdot f_{VFA}$. If the controller is executed for a lapse $\Delta t \geq 3 \cdot HRT$ (beyond steady state condition), the gain will be higher or equal to the product $f_{CH4} \cdot f_{VFA}$; otherwise (before achieving steady state), the gain value of the discrete form will be lower than $f_{CH4} \cdot f_{VFA}$.

f_{CH4} and f_{VFA} are empirical functions where [VFA^*] is the setpoint value of VFA concentration (expressed in mg L^{-1}); [VFA] is the average VFA concentration in the reactor during a control cycle Δt; $Q^*_{CH_4}$ is the setpoint value of the methane productivity (expressed in m^3 CH_4 m^{-3} d^{-1}); and Q_{CH_4} is the average methane productivity during a control cycle. Parameters m, n and α shape both f_{CH4} and f_{VFA} functions (Figure 1); they can be calibrated for each specific case depending on the substrate to be treated or the dynamics of the operation. In this study, the same values that García-Diéguez *et al.* (2010) were used (m = 0.5, $n = 10$, $\alpha = 0.1$). [VFA^*] was set to 500 mg L^{-1} based on references (Batstone & Steyer 2007) and previous experience (see Supplementary Material, available with the online version of this paper). The value of $Q^*_{CH_4}$ (12.7 m^3CH_4 m^{-3} d^{-1}) was derived from the maximum theoretical OLR (around 35 kgCOD $m^{-3}d^{-1}$) that the system can stand without reaching hydraulic overload, assuming that the maximum upflow velocity in the reactor is 0.5 m h^{-1}, a typical value for digester design (Chernicharo *et al.* 2015).

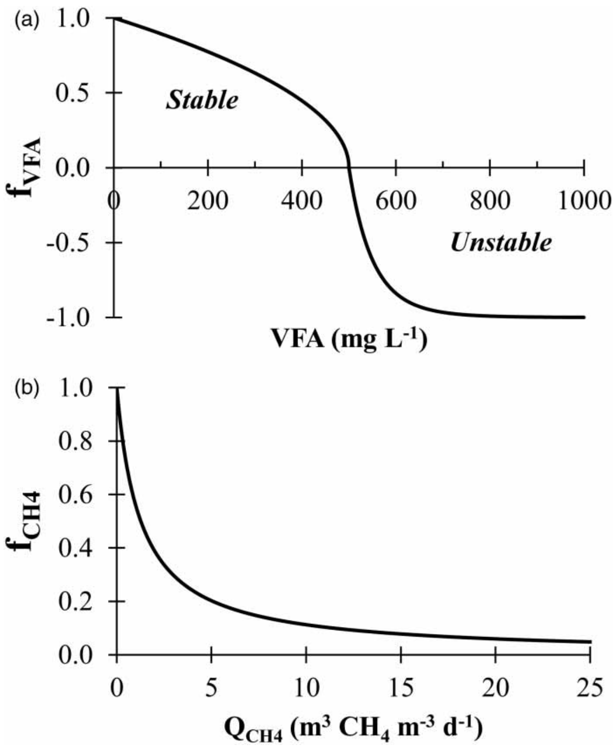

Figure 1 | (a) Empirical function of VFA factor, f_{VFA}. [VFA^*] = 500 mg L^{-1}, m = 0.5, $n = 10$. (b) Empirical function of methane production factor, f_{CH4}. Q^*_{CH4} = 12.7 m^3CH_4 $m^{-3}d^{-1}$, $\alpha = 0.1$.

The VFA factor informs of the stability in terms of acidification or organic overload. The setpoint [VFA^*] depicts the stability threshold; above this point, the system is considered acidified; otherwise, the operation remains stable. In order to quantify a stability level, f_{VFA} takes values in the interval [−1, 1]. Positive values indicate stable operation and negative values indicate acidification of the system.

The term f_{CH4} concerns the methane production rate and, therefore, it is also related to the OLR applied. It accepts values in the range [0,1] and it is introduced to adjust the stability level determined by f_{VFA} depending on the current OLR. At low OLR, the effect of f_{CH4} is small (it adopts values around 1); however, at high OLR, the methane production factor significantly reduces the stability value calculated by f_{VFA}.

This variable-gain controller can be easily implemented in a real plant as a closed-loop scheme (Figure 2). A moderate investment should be made to acquire on-line analyser of COD, VFA, and biogas composition. The controller receives two inputs: methane flow rate factor (f_{CH4}) and VFA factor (f_{VFA}), which both calculated in a diagnosis procedure with Equations (5) and (6). Diagnosis block is fed with setpoint

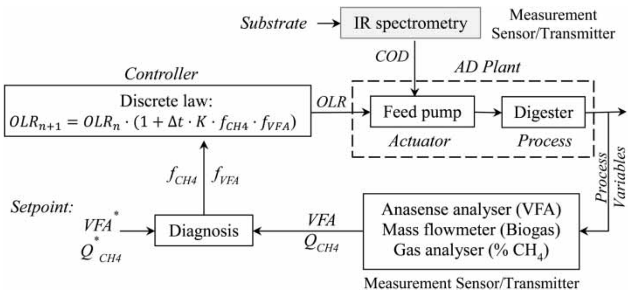

Figure 2 | Architecture of the closed-loop controller for real implementation. IR spectrometry (highlighted in grey) is an optional solution to monitor COD on-line. It was not available in this study, where COD were measured off-line.

values of methane flow rate and on-line measurements of VFA, methane flow rate and biogas composition. The discrete control law is applied and the OLR is calculated as controller output. Whenever COD content of the substrate is variable, COD should be monitored on-line; for instance, by infra-red spectrometry. This technique was validated to measure COD on-line for industrial wine distillery wastewater (Steyer *et al.* 2002). In this study, COD was measured off-line. Finally, the feed flow to digester is calculated according to OLR and COD values.

The performance of the proposed controller was compared with a conventional strategy carried out in a second unit, where the OLR increased at a rate of 25% weekly. In this case, as the HRT is lower or equal to 2 days, a gap of one week implies a lapse higher than three times the HRT.

Experimental setup for continuous experiments

The experiments were carried out at pilot scale, in a portable facility consisted of two UASB reactors of 95 L connected in parallel (Figure 3). The pilot plant is located in the wastewater treatment plant of Chimbarongo (Chile), and receives wastewater from the wine industry.

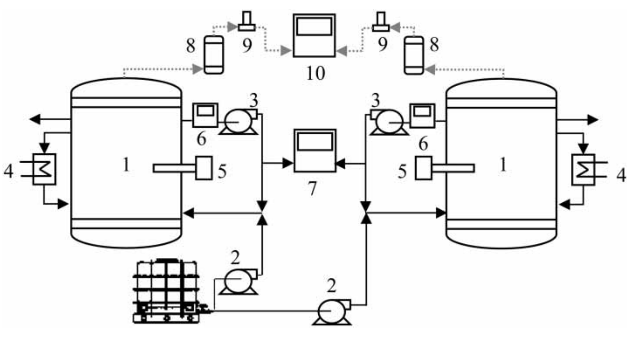

Figure 3 | Scheme of the pilot plant, which consists of: (1) 95-litre UASB digesters, (2) feeding pumps, (3) recirculation pumps, (4) thermal circulators, (5) temperature probes, (6) pH sensors, (7) PA, TA and VFA analyser, (8) water trap condensers, (9) biogas flow meters, and (10) analyser of gas composition.

The feed flows into the digesters through an analogue control peristaltic pump (Watson-Marlow, 520 Du). By means of a three-phase pump (Bredel, SPX40), controlled by a variable frequency drive (Emerson, Comander SK), a stream from the reactor is recirculated to homogenise the medium in the interior. The reactors count on thermal immersion circulators (PolyScience, MX-CA12E) to regulate the temperature and in-line instruments to measure pH (HANNA transmitter HI8614 L), temperature (INGECO, Pt100) and biogas flow rate (Bronkhorst, F-111B-NNN-BBD-2-V). An AnaSense® (AppliTek) analyser (Bernard *et al.* 2005) is connected on-line to measure and register partial alkalinity (PA), total alkalinity (TA) and VFA values. Finally, a gas analyser (MCA 100 BIO) measures the gas composition (CH_4, CO_2, H_2 y H_2S) on-line (*Note*: the analyser broke down during the initial steps of the experiment; a steady composition of 70% CH_4 was assumed based on the off-line measures of gas samples taken along the operation). All signals from sensors and analysers are monitored and recorded in the ODIN (INRIA) software, connected through National Instruments data acquisition modules.

Continuous experiments were performed in both digesters for 120 days under mesophilic conditions (35 °C). The initial conditions of both digesters were: OLR of 2.0 $kgCOD\ m^{-3}\ d^{-1}$, HRT of 2 days and inoculum of $10\ g\ L^{-1}$ (this having an activity of $0.39\ gCOD\text{-}CH_4\ gVSS^{-1}\ d^{-1}$). The control strategies started to be applied from day 9 on. Table 1 shows the average characterisation and range of values of the vineyard wastewater. Due to its low alkalinity, the feed was previously conditioned in a tank with $NaHCO_3$ solution until reaching 2.0 g $CaCO_3$/L before heading into the digester.

Analytical methods

Parameters such as COD, pH, PA, TA, VFA, total solids (TS) and volatile solids (VS) were measured off-line for both influent and effluent of the reactor following *Standard Methods for the Examination of Water and Wastewater* (2012). COD and $N\text{-}NH_3$ were determined with a multiparameter bench photometer (Hanna Instrument, HI 83214); pH was determined with a portable multi-parameter meter (HACH, HQ40D). CH_4 and CO_2 were analysed using a gas chromatograph (PerkinElmer, Clarus® 500), equipped with a thermal conductivity detector (TCD). The samples were manually injected on a Teflon column (dimensions L × OD × ID, 10 ft × 1/8 in × 2.1 mm) packed with HayeSep

Table 1 | Characterisation of the vineyard wastewater used as feeding of the AD experiments

Parameter	Average value	Range
pH	7.2	6.6–7.8
COD (mg L^{-1})	2,867	1,515–4,515
TS (g L^{-1})	6.44	3.67–8.76
VS (g L^{-1})	1.55	0.11–2.60
VFA (mg L^{-1})	690	120–1,928
PA (meq L^{-1})	15.4	7–28
TA (meq L^{-1})	21.3	11.5–38.5
N-NH_3 (mg L^{-1})	57	19–127

Q, 80/100 mesh, (Supelco, Bellafonte, USA). The column was heated at 70 °C and maintained for 4 min. Helium was used as carrier gas, flowing at 25 mL/min. The temperature of the injector and detector were set to 80 °C and 120 °C, respectively.

Total VFA was measured with Anasense® analyser, using a titrimetric method developed by AppliTek (AppliTek NV, Belgium). A detailed description and validation of Anasense® for wastewater treatment plants can be found in Molina *et al.* (2009). The method was developed to provide an accurate and easy measurement of low VFA concentrations by air stripping the sample, where bicarbonate and hydrogen sulphide can be stripped at pH >5. Then, total VFA can be directly measured by titration from pH 5 to 4. This approach avoids the interference of bicarbonate in VFA determination.

RESULTS AND DISCUSSION

Control strategy results

The proposed control strategy was validated at pilot scale for 120 days. The ultimate aims of the controller were fulfilled: (1) increase the treatment capacity of the digester and, hence, augment the methane production when the system is stable and (2) recover the system from acidification when the system goes through transient instability periods. Along the operation, the OLR increased from the initial 2.0 kg COD $m^{-3}d^{-1}$ up to 39.2 kg COD $m^{-3}d^{-1}$ by the end of the experiment, OLR varying up and down according to the controller response. The control algorithm was executed 45 times at control cycles ($\Delta t = 3 \cdot HRT$) ranged between 0.8 and 7 days and an average COD removal of 81% was achieved. Figure 4(a) shows all the operation conditions applied (OLR and HRT) and the percentage of COD removal (measured from the inlet and outlet COD of the digester).

The OLR fluctuated based on the values of VFA concentration (Figure 4(b)). When the average VFA exceeded the stability threshold of 500 mg L^{-1}, the controller decreased the OLR applied to the next period. Figure 4(b) shows that VFA was highly sensitive to OLR changes. In general, VFA tended to increase when the OLR increased and to decrease as OLR did. This effect was attenuated as the inoculum adapted to the new operating conditions. The VFA concentration turned out to be an adequate indicator to estimate the stability of the process. In this study, VFA remained around 300–600 mg L^{-1}, a typical range for this type of waste (Batstone & Steyer 2007).

Methane production increased as OLR stepped up. Assuming that there is a maximum OLR that can be employed to avoid overloads (Latif *et al.* 2011), the methane factor f_{CH4} (Figure 4(c)) served to adjust the level of stability calculated by f_{VFA}, according to the actual OLR. At high rates, f_{CH4} moderated the changes of OLR more significantly than at low rates.

The length of the control cycle (Δt) varied during the entire operation. The first 30 days, the product $K \cdot \Delta t$ was around 1 (as $\Delta t \approx 3 \cdot HRT$), so that the gain was only conditioned by the f_{VFA} and f_{CH4} factors. As HRT decreased far below 1 day, it was no longer practicable to execute the controller 3 times the HRT (no automated system to regulate the feed pump based on methane production, VFA and COD measures was available). Then, a longer cycle time was selected, involving a product $K \cdot \Delta t > 1$. From day 60 on, due to the high values of $K \cdot \Delta t$ achieved, this product was limited to 1.25 in order to keep the significance of f_{VFA} and f_{CH4}, the real indicators of the process state.

Finally, the proposed control law became a useful tool to control a vineyard wastewater having a variable COD content (see Table 1). Unlike other algorithms using dilution rate or feed flow rate as a manipulated variable, the choice of OLR as manipulated variable seems to be more convenient when the input COD fluctuates significantly.

Control strategy versus conventional operation

The conventional approach led to a stable operation for all conditions applied. The OLR increased from initial 2 kg COD $m^{-3}d^{-1}$ up to 36.8 kg COD $m^{-3}d^{-1}$, and an average COD removal of 82% was reached (Figure 4(d)). The weekly rise of the actual OLR by 25% became adequate to

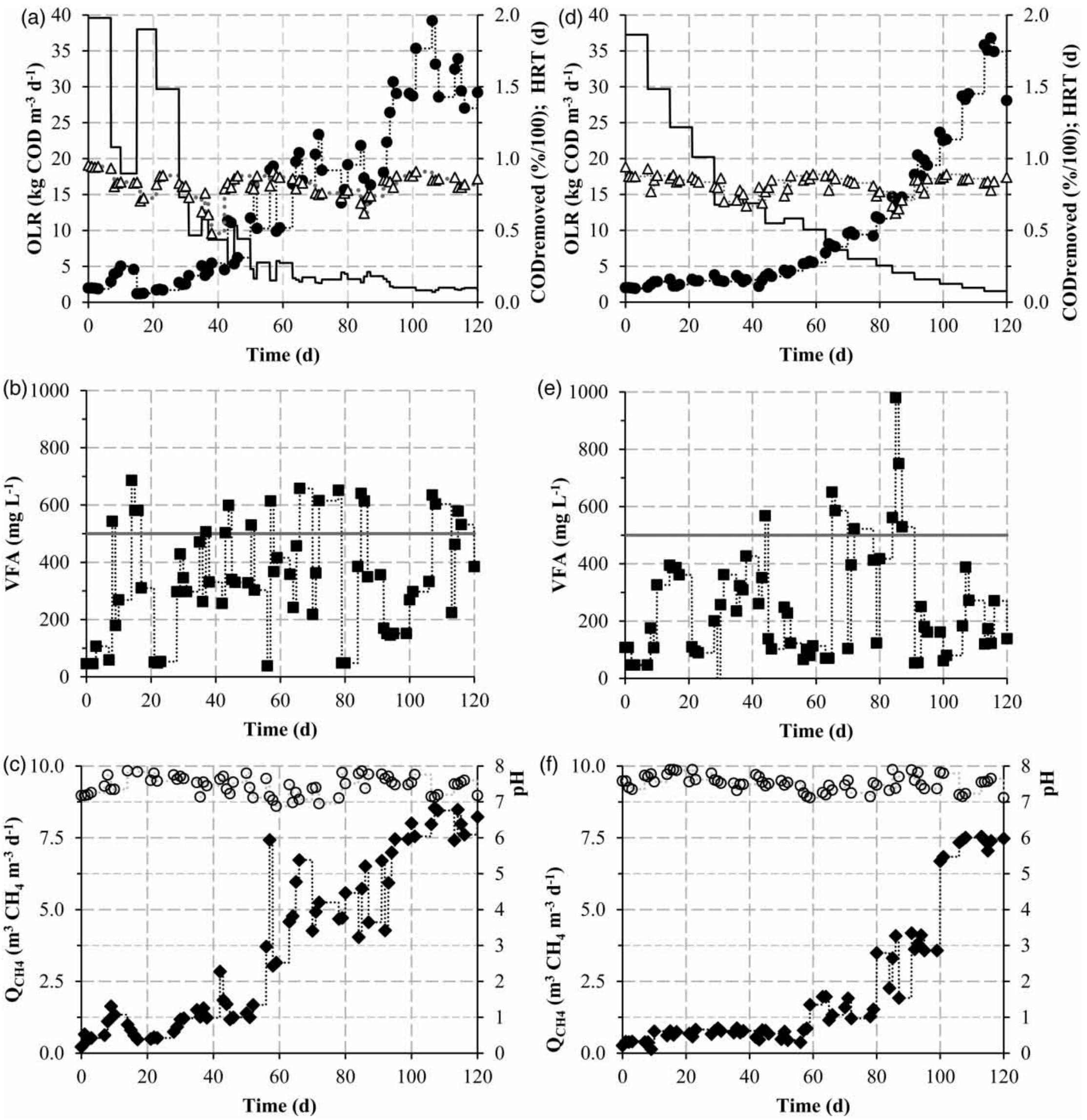

Figure 4 | Experimental results of the proposed control strategy (a–c) and conventional operation (d–f), treating wine wastewater in UASB reactors. (-) HRT, (●) OLR, (Δ) COD removed, (■) [VFA], (-) [VFA*], (○) pH and (◇) Q_{CH4}.

maintain the stability of the operation. Unlike the experiment carried out under the control law (Figure 4(a)), the OLR increased steadily (Figure 4(d)) and, overall, the effluent VFA (Figure 4(e)) remained at lower values. Only an important transient acidification (VFA reached 1,000 mg L^{-1}) occurred within the period 80–85 d, eventually reverted in the coming days. Notwithstanding the conventional mode worked satisfactorily, however, the proposed controller processed and removed 32% more COD and produced 28% more methane (both calculated from the areas under the curves of OLR and methane productivity (Figure 4(f)), respectively). Therefore, the proposed control algorithm contributed to enhance the digester capacity far beyond the conventional operation; particularly, it showed a better performance during the start-up phase.

Both the proposed and conventional strategies covered a broad range of OLR. They both reached very high values (36–39 kg COD $m^{-3}d^{-1}$), close to the maximum capacity for UASB reactors (Latif *et al.* 2011; Nnaji 2014). They also worked close to the hydraulic overload, up to upflow velocities of 0.7–0.8 m h^{-1}, maximum limits for the design of UASB reactors (Chernicharo *et al.* 2015). In both cases, pH remained between 7 and 8 throughout the operation (Figure 4(c) and 4(f)), ammonia nitrogen at 52 mg L^{-1} and

C/N ratio kept around 14. Finally, at the end of the operation, the solids concentration at the bottom of the reactor was 18 g VS L^{-1} and 2–3 g VS L^{-1} in the middle to top.

CONCLUSIONS

The proposed variable-gain controller was validated in a pilot-scale UASB reactor treating industrial wine wastewater continuously, this containing a variable COD content. The algorithm successfully assessed the process state over a wide range of operating conditions, using VFA and methane production rate as key process indicators. Based on the diagnosis outcome, the control law was able to both increase methane productivities and recover the system from acidification by adequately changing the OLR of the process. A better performance was achieved compared to a conventional operating strategy carried out simultaneously in a parallel UASB unit, treating the same wastewater.

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