Winery effluent treatment at an anaerobic hybrid USBF pilot plant under normal and abnormal operation

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Water Science & Technology **Vol 56 No 2 pp 25–31**

Abstract A 1.1 m^3 hybrid USBF fully instrumented pilot plant has been used for the treatment of diluted wine for four years. In this work, the performance of the wastewater treatment plant (WWTP) during start up and operation (normal operation and overload experiments) is shown. A complete description of the treatment process behaviour (gas and liquid phase composition and anaerobic sludge characteristics) is given by on-line and off-line monitoring of 28 process variables.

The results presented here demonstrate the reliability of this technology for the treatment of wastewater from seasonal processes, such as winery wastewaters, during a long period of time (four years). Furthermore, the USBF reactor presented very short start up periods after short and long shut down of the WWTP and rapidly turned back to normal operation after suffering a complete destabilization due to organic overload.

Both effluent and biogas were of good quality. Dissolved organic carbon concentration in the effluent was always lower than 100 mg DOC l^{-1} under normal operation, while methane concentration in the biogas was in the range 70–74%, making it suitable for energy recovering.

Keywords Anaerobic treatment; pilot plant; USBF reactor; winery effluent

Introduction

The anaerobic digestion process is a good option for industrial wastewater treatment, as it presents a high capacity to degrade high concentrated wastewaters with a low sludge production, low energy demand and also for energy recovery (Rajeshwari *et al.*, 2000). These features make anaerobic digestion especially attractive for beverage industries wastewater treatment (beer, wine and distilleries).

Distillery effluents are highly polluted and can produce important negative effects on the environment if they are discharged without treatment. Anaerobic digestion is a well established technology for the treatment of high concentrated wastewaters as distillery effluents (Driessen *et al.*, 1994). Furthermore, anaerobic digestion has the main advantage when the effluents are seasonal, like in winery processes, where wastewater production occurs mainly during harvesting and wine making. In this situation, the anaerobic digester can start up rapidly after a shut down (15 days in general required for restarting period; Moletta, 2005).

UASB technology has been applied to treatment of wastewaters from breweries, wineries and distilleries of different raw materials, such as sugar cane juice, sugar cane molasses, grape wine, and sugar beet molasses (Driessen *et al.*, 1994; Wolmarans and de Villiers, 2002; Moletta, 2005). Other anaerobic technologies such as anaerobic fluidized

doi: 10.2166/wst.2007.468

bed and down flow anaerobic filter were also successfully applied for distillery wastewater treatment (Speece, 1996; García-Calderón *et al.*, 1998).

Hybrid USBF reactor configuration is an interesting technology since it combines the main advantages of the up flow anaerobic filter (UAF) and UASB reactors for biomass retention. In this way, it is possible to minimize clogging or biomass flotation problems (Fernandez *et al.*, 2001). Hybrid USBF is especially adapted for treating wastewaters that do not produce granular sludge (Soroa *et al.*, 2006).

The aim of this work was to investigate the performance of a hybrid USBF pilot plant applied to the treatment of winery wastewaters in a long-term operation. The reactor behaviour in start-up after short and long stop periods, and also during overload events, was studied.

Materials and methods

Experiments were conducted at 37 $\pm$ 2 °C in a hybrid USBF pilot plant. The reactor was continuously fed with diluted wine, nutrients (nitrogen and phosphorus) and alkalinity, with a COD/bicarbonate/N/P ratio of 1000/400/7/1 (Speece, 1996). After a start-up period, the feed flow rate was $30\,l\,h^{-1}$ and the average COD concentration was $8\,g\,l^{-1}$. High recycling flow rate ($200\,l\,h^{-1}$) guaranteed mixing and allowed a superficial velocity (V_s) of $0.5\,m\,h^{-1}$ to be maintained.

The installed on-line measurement devices included feeding and recycling flow-meters (ABB, COPA-XE and Siemens, 7ME2531), input and output reactor pH (Cole Parmer) and temperature (Pt 100); biogas flow-meter (Brooks, 3240); infrared gas analyser (Siemens, Ultramat 22P) for measurement of CH_4 and CO concentrations in the gas phase; electrochemical hydrogen gas analyser (Sensotrans, Sensotox 420); on-line Total Organic and Inorganic Carbon (TOC/TIC) in the influent and effluent were determined by catalyst combustion oxidation and non dispersive infrared (NDIR) CO_2 detection (Shimadzu, 4100). Figure 1 shows a flow-sheet of the pilot plant.

A new titrimetric analyzer called AnaSense® (Ruiz *et al.*, 2005) was used for the on-line Volatile Fatty Acids (VFA) and alkalinity monitoring. AnaSense® uses the recorded titration curves for the calculation of four parameters: VFA, bicarbonate, partial and total alkalinity concentrations (Molina *et al.*, submitted).

Sensor signals are recorded every 5 seconds. A moving average window of 15 minutes was used for filtering on-line signals, taking into account the high stability of the signal and the timescale of the process (hydraulic retention times HRT between 10 and 20 hours). A complete description of the pilot plant and monitoring system is given by Ruiz (2005).

Changes in morphology of sludge granules were determined by image analysis (Jeison and Chamy, 1998) using the software Image ProPlus. Images of granular sludge were taken with a digital camera (Coolsnap, Roper Scientific Photometrics) combined with a stereomicroscope (Stemi 2000-C, Zeiss).

The profile of the sludge bed characteristics was determined several times during the reactor operation. With this aim, sludge samples were taken through the five sampling ports localized at different levels: 0, 0.50, 0.84, 1.20 and 1.52 m from the bottom of the reactor. Afterwards a number of sludge parameters were determined.

Specific Methanogenic Activity (SMA) was determined using the methodology proposed by Angelidaki and Sanders (2004). This test in run by triplicate at 37 °C, the accumulated methane composition in the headspace was determined by GC (FID Detection). Specific Acidogenic Activity (SAA) was determined using the same SMA medium. SAA assays were performed by monitoring glucose depletion using a colorimetric method.

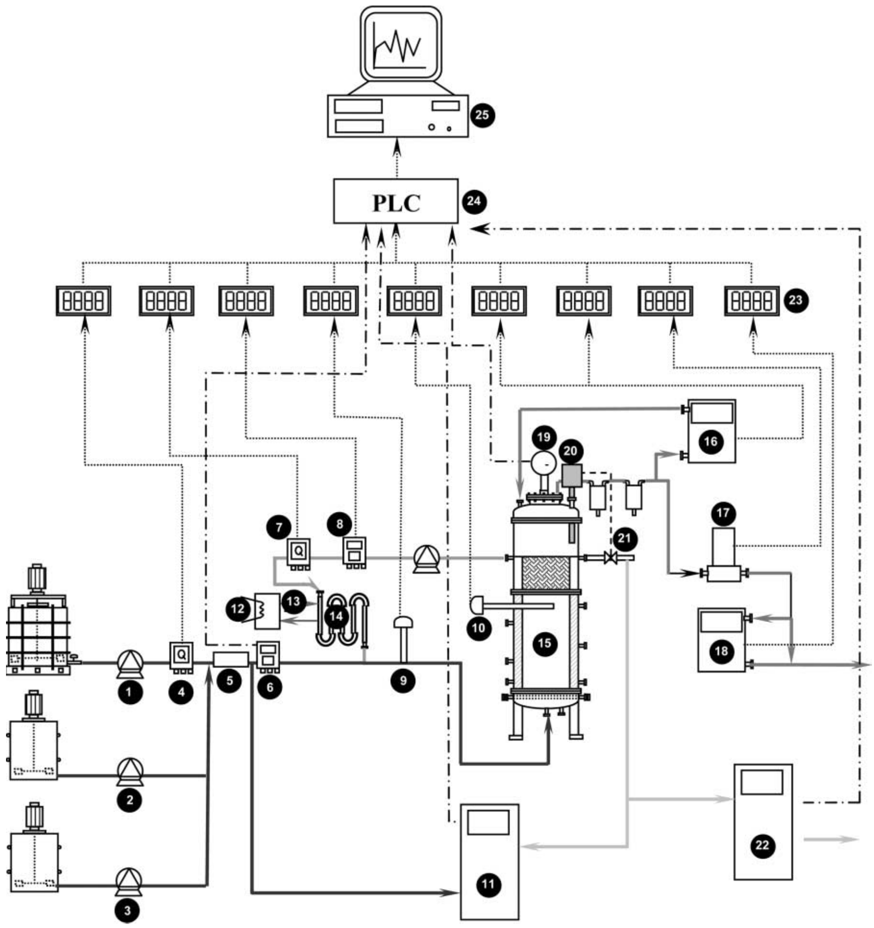

Figure 1 Schematic layout of the pilot WWTP: 1. Dilution pump; 2. Nutrient pump; 3. Wine feed pump; 4. Dilution water flow meter; 5. Static mixer; 6. Input pH meter; 7. Recycling flow meter; 8. Output pH meter; 9. Input temperature probe; 10. Reactor temperature probe; 11. TOC on-line analyzer; 12. Water heater; 13. Heat water pump; 14. Heat exchanger; 15. Hybrid reactor; 16. CH_4 and CO analyzer; 17. Gas flow meter; 18. Hydrogen analyzer; 19. Pressure probe; 20. Level sensor; 21. Output valve; 22. Alkalinity on-line sensor Anasense®; 23. Display panel; 24. PLC; 25. PC, Water and gas lines (continuous line) and Data flow (dotted line)

Sludge Volumetric Index, Sludge Settling Velocity and Volatile Suspended Solid concentration were determined according to the procedure specified in *Standard Methods for the Examination of Water and Wastewater* (1998).

Results

The inoculum consisted of a mixture of sludge collected from two anaerobic digesters treating wastewater from a fibre-board production process and from a sugar factory. The pilot plant was started up with an organic loading rate (OLR) of 0.5 kg COD m^{3} d^{-1}. Once the process was stable and the effluent COD concentration was lower than 500 mg l^{-1}, the OLR was gradually increased. An OLR of 5 kg COD m^{-3} d^{-1}, with a COD removal of 98% and a negligible accumulation of intermediates (such as VFA), was obtained at the end of this stage (after 60 days).

Afterwards, short (several days) and long (several weeks) stops of the plant were programmed for the simulation of these usual seasonal events in industrial scale winery and brewery processes (Rodriguez *et al.*, 2006). Consequently, start up experiments were conducted with successful results and a short duration of the start up (2 and 5 days, respectively). Figure 2 illustrates, as an example, the simulation of a typical start up in seasonal activity of winery, carried out after a 45 days shutdown of the WWTP. As it can be seen, this start up showed two different steps. The first step lasted only five days and the OLR increased to 4 kg COD m^{-3} d^{-1}. In a second step, OLR was slowly increased to 6.5 kg COD m^{-3} d^{-1}; effluent dissolved organic carbon (DOC) was maintained below 100 mg l^{-1} and pH was stable, (between 7.2 and 6.9).

On the other hand, a quite common event in the brewery industry is a short stop for equipment maintenance and cleaning or during weekends. Figure 3 shows the results of a start-up experiment simulating this situation (a short stop of four days). OLR was increased to 6 kg COD m^{-3} d^{-1} in only 40 hours, while DOC values were maintained at less than 50 mg l^{-1} and pH remained almost constant near a value of 7.

A maximum OLR of 20 kg COD m^{-3} d^{-1} at a HRT of 20 hours could be applied with a stable performance of the pilot plant and keeping a good effluent quality (COD < 1 gl^{-1}). For the same HRT, the process failed when the OLR was increased to 25 kg COD m^{-3} d^{-1}, as the reactor suffered an organic overload destabilization, the pH decreased to 5 and the methanogenic population was inhibited. Figure 4 illustrates this event. Once OLR was decreased to a normal value, the reactor needed a period of five days for a complete recuperation.

For further experiments, the applied OLR was set to a value of 12 kg COD m^{-3} d^{-1} in order to work with a high COD removal (96%). A good biogas quality has been obtained

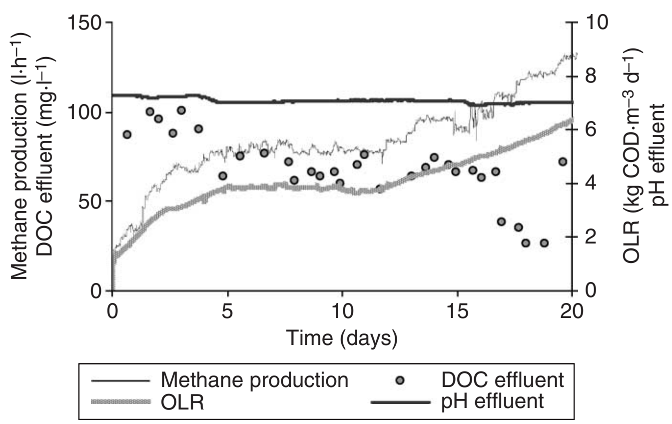

Figure 2 Start-up of the WWTP after a long stop (45 days shutdown)

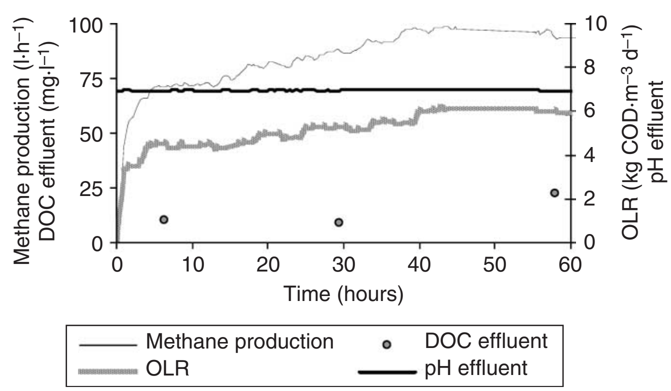

Figure 3 Start-up experiment after a short stop (4 days shutdown) of the WWTP

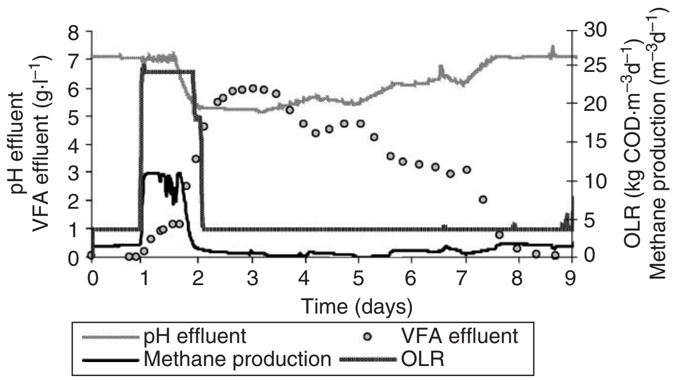

Figure 4 Destabilization and recovery of the anaerobic USBF reactor caused by an organic overload

with 70–74% of methane concentration and a methane production yield of $332\,l\,kg^{-1}$ of COD removed, which makes it suitable for energy recovery. After that, the reactor was shut down for 2 days and restarted. Figure 5 shows the start-up experiment carried out afterwards, OLR was increased to $13\,kg\ COD\,m^{-3}\,d^{-1}$ in 4.5 days while effluent quality was kept at adequate DOC values oscillating around $200\,mg\,l^{-1}$.

The good performance of the anaerobic USBF reactor can be explained by the good quality of the developed sludge. The average granule diameter was approximately 1.5 mm; settling velocity of sludge was $26\,m\,h^{-1}$ and its sludge volumetric index (SVI) was $19\,ml\,g^{-1}$ TSS. A typical granule size distribution and granule morphology obtained during the operation are shown in Figure 6.

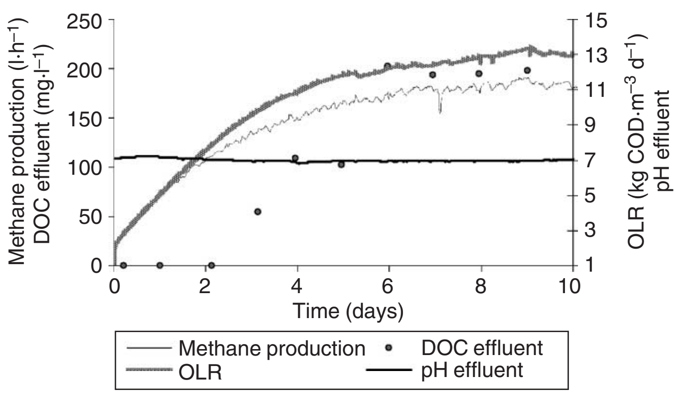

Figure 5 Start-up experiment carried out after short stop of WWTP

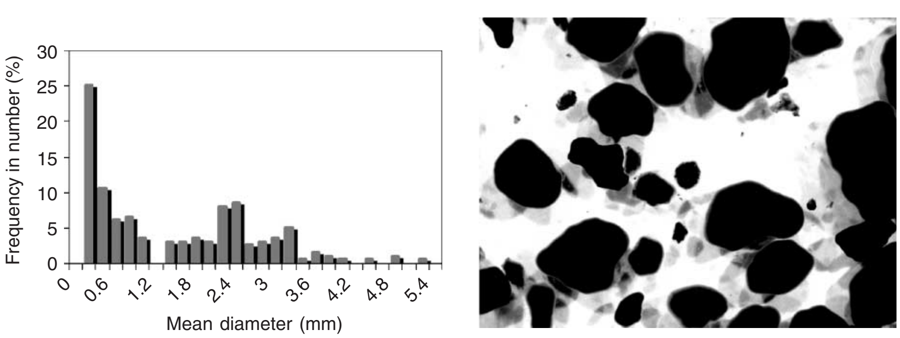

Figure 6 Typical granule size distribution and granule morphology of the sludge sampled from the USBF reactor

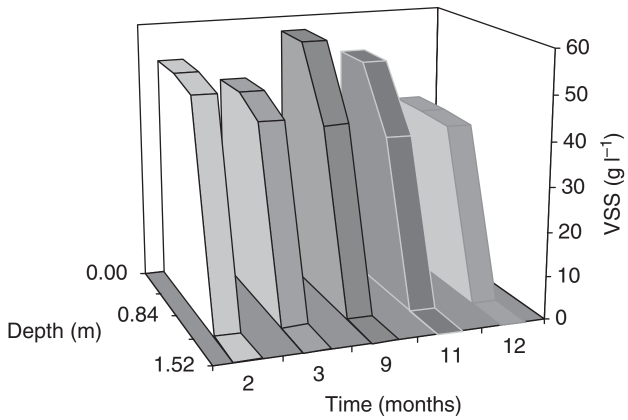

Figure 7 Sludge bed profile evolution along one year of WWTP operation

The aggregated biomass had a specific methanogenic activity of 0.66 ± 0.18 kg COD kg VSS^{-1} d^{-1} and a specific acidogenic activity of 15.9 ± 2.3 kg COD kg VSS^{-1} d^{-1}.

Average VSS concentration in the sludge blanket was 44 ± 6 g l^{-1} and the average total biomass observed in the reactor was 18.6 ± 1.5 kg VSS, meaning an average VSS concentration in the reactor of 16.9 ± 1.4 g l^{-1}. The sludge is accumulated in the first meter of reactor depth, being stable during the operation, as no expansion or flotation of the bed was observed in the upper part of the USBF reactor. Figure 7 shows the sludge bed profile evolution during the last year of WWTP operation.

Conclusions

The results presented in this work show the reliability of the anaerobic digestion process for the treatment of winery effluents. Useful data for fault detection, diagnosis and model calibration are shown in this paper.

The WWTP start up after short and long shut down periods (simulating typical start up in seasonal industrial processes) were quite fast, thus making this technology really valuable for winery wastewater treatment as well as for other seasonal process industries with similar wastewater characteristics.

Normal operation of WWTP at OLR of 12 kg COD m^{-3} d^{-1} with a high COD removal was carried out. Organic loading rates, as high as 20 kg COD m^{-3} d^{-1}, were also applied and a quite stable WWTP behaviour was achieved. This USBF reactor performance is mainly due to the development of granular biomass with suitable specific methanogenic activity and very good settling characteristics. An adequate biogas quality was also obtained (70–74% CH_4) thus enabling an interesting energy recovery.

Acknowledgements

The authors acknowledge the Spanish National R&D Program and European Regional Development Fund (ERDF) for the CICyT project ANACOM CTQ2004-07811-C02-01.

References

Angelidaki, I. and Sanders, W. (2004). Assessment of the anaerobic biodegradability of macropollutants. *Rev. Environ. Sci. Biotechnol.*, **3**, 117–129.

Driessen, W.J.B.M., Tielbaard, M.H. and Vereijken, T.L.F.M. (1994). Experience on anaerobic treatment of distillery effluent with the UASB process. *Water Sci. Technol.*, **30**(12), 193–201.

Fernandez, J.M., Omil, F., Mendez, R. and Lema, J.M. (2001). Anaerobic treatment of fibreboard manufacturing wastewaters in a pilot scale hybrid USBF reactor. *Water Res.*, **35**(17), 4150–4158.

García-Calderón, D., Buffiere, P., Moletta, R. and Elmaleh, S. (1998). Anaerobic digestion of wine distillery wastewater in down-flow fluidized bed. *Water Res.*, **32**(12), 3593–3600.

Jeison, D. and Chamy, R. (1998). Novel technique for measuring the size distribution of granules from anaerobic reactors for waste-water treatment. *Biotechnol. Tech.*, **12**(9), 659–662.

Moletta, R. (2005). Winery and distillery wastewater treatment by anaerobic digestion. *Water Sci. Technol.*, **51**(1), 137–144.

Molina, F., Ruiz, G., Roca, E. and Lema, J.M. Validation at pilot scale of a new sensor for on-line analysis of volatile fatty acids and alkalinity in anaerobic biological waste water treatment. *Biochem. Eng. J.* (submitted).

Rajeshwari, K.V., Balakrishnan, M., Kansal, A., Lata, K. and Kishore, V.V.N. (2000). State-of-the-art of anaerobic digestion technology for industrial wastewater treatment. *Renew. Sust. Energ. Rev.*, **4**(2), 135–156.

Rodríguez, J., Ruiz, G., Molina, F., Roca, E. and Lema, J.M. (2006). A hydrogen-based variable-gain controller for anaerobic digestion processes. *Water Sci. Technol.*, **54**(2), 57–62.

Ruiz, G (2005). Monitoring and Advanced Control of Anaerobic Reactors, PhD thesis, Group of Environmental Biotechnology and Engineering, University of Santiago de Compostela Spain (in Spanish).

Ruiz, G., Molina, F., Steyer, J.-P., Vanrolleghem, P., Zaher U., Roca, E. and Lema, J.M. (2005). Industrial scale validation of a new titrimetric sensor for anaerobic digestion processes: comparison of methodologies. In: *Proceedings 2nd IWA Conference on Instrumentation Control and Automation*, Busan, Korea.

Soroa, S., Gomez, J., Ayesa, E. and Garcia-Heras, J.L. (2006). Mathematical modelling of the anaerobic hybrid reactor. *Water Sci. Technol.*, **54**(2), 63–71.

Speece, R.E. (1996). *Anaerobic Biotechnology for Industrial Wastewaters*, Archae Press, Nashville, TN.

Standard Methods for the Examination of Water and Wastewater (1998). 20th edn. American Public Health Association/American Water Works Association/Water Environment Federation, Washington DC

Wolmarans, B. and de Villiers, G.H. (2002). Start-up of a UASB effluent treatment plant on distillery wastewater. *Water SA*, **28**(1), 63–68.