

THE STABILITY OF ANAEROBIC DIGESTERS OPERATING ON A FOOD-PROCESSING WASTEWATER

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ABSTRACT

The response to step overloads of laboratory-scale contact process and UASB reactors and a pilot-scale anaerobic filter is examined. The reactors were operated on ice-cream waste water and changes in operational parameters including VFA levels and COD reduction were monitored. In the laboratory-scale experiments, feed strength was increased from 4500 to 22,000 mg [O] per litre for periods of 2 to 12 hours in contact process and one and two-stage UASB reactors. UASB stability was improved by supplementing with trace metals. Two-stage UASB's gave better COD removals under shock loading than single-stage UASB's or contact process reactors. In an 8 hour step overload experiment on the pilot-scale reactor, bicarbonate alkalinity was shown to be a good indicator of instability.

KEY WORDS

Anaerobic digestion, UASB, filter, contact process, pilot scale, bicarbonate alkalinity, step overload, shock, ice-cream.

INTRODUCTION

The UK Science and Engineering Research Council's (SERC) Anaerobic Facility has been sited for the past three years at a large ice-cream factory. The facility consists of the four most common types of anaerobic digester for agro-industrial waste treatment: a completely mixed contact process (CP) reactor (5m³), an upflow anaerobic filter (5m³), an upflow anaerobic sludge blanket (UASB) reactor (5m³) and a 0.5m³ fluidized bed reactor. Results of shock experiments, tracer studies and microbiological investigations on these reactors are currently being published (Caine et al. in press, Monroy et al., submitted, Morgan et al., submitted, Smith et al. a and b, submitted) and one such step overload experiment on the anaerobic filter is reported here.

The UASB pilot scale reactor was operated for three years in an attempt to form granules but granules did not develop nor was granulation successful using ice-cream wastewater at laboratory scale (Cayless et al., 1990). The anaerobic filter pilot plant reactor performed consistently well (Monroy et al., submitted) giving a Chemical Oxygen Demand (COD) removal of up to 70% at loading rates of up to 9 kg COD/m³ day.

An extensive laboratory based research programme has accompanied the pilot-scale work, investigating scale-up, start-up, reactor stability, options for monitoring and control and the microbiology of the process. Results related to this more basic research programme are presented here and in other papers already published or submitted (Cayless et al., 1989, Cayless et al., 1990, Morgan et al., 1990 and 1991, Smith et al., submitted c and d).

The waste water from the ice-cream factory was reported to have a COD of 4,500 mg[O]/l. Because of a problem with a cleaning agent used episodically in the factory, Caine et al. (in press) report that in the pilot

scale studies the wastewater was simulated by mixing ice-cream and water-ice products with the addition of the two major detergents used by the factory, trace elements (Fe, Mn, Co, Ni, dosed at 0.02 mg/l) and N and PO₄ dosed at COD:N:PO₄ 100:2:1. Because of the constraints of digester design which limited the volume of influent fed per day, it was decided to increase the COD level of the simulated wastewater above that of the factory effluent. Monroy *et al.* (submitted) report the wastewater used in the pilot plant studies had, on average, a COD of 5800 mg[O]/l and was high in fat, containing 610 mg/l of oil and grease. The acidity of this mixture increased rapidly on storage, and the contents of the feed tank were controlled automatically to pH 6.6 - 6.8 by addition of about 0.54 kg NaOH per m³, augmenting the otherwise poor buffering capacity of the waste, (Monroy *et al.*, submitted).

This paper examines the response to step overloads of laboratory scale contact process and one and two stage UASB digesters and of the pilot scale anaerobic filter operating on simulated ice-cream waste. Because of the lack of granulation described above, commercially available granules were utilised in these laboratory-scale studies. The COD of the simulated ice-cream waste-water utilised was that of the actual factory effluent. The effect of trace metal addition on UASB stability was studied.

MATERIALS AND METHODS

Laboratory-scale Reactors

Three UASB reactors consisting of 4.6 litre Perspex cylinders (10 cm internal diameter, height of liquid to effluent outflow tube 60 cm) were used. The reactors were heated to 35°C by a water jacket. A 45° cone was inserted in the upper part of each digester as a liquid/solid/gas separator which efficiently prevented granule loss. Five 5 litre Quickfit vessels fitted with stirrers were modified with an effluent side arm giving a liquid volume of 5.7 litre. These Contact Process (CP) reactors were operated in a thermostatted water-bath at 35°C. Later in the experimental work, a two-stage acidogenic-methanogenic process was constructed out of the reactors utilised previously, the contact process reactors described being converted to the acidogenic stage, then connected to the UASB reactors as the methanogenic stage.

Ice-cream Wastewater

The effluent from the factory was simulated by mixing 160 g of ice-cream diluted to 25 l with tap water to produce a COD of approximately 4,500 mg [O]/l. Urea and diammonium hydrogen orthophosphate were added to give a COD:N:PO₄ ratio of 100:2:1. The pH of freshly made feed was approximately 7.0, but rapidly acidified. The feed was thus adjusted to pH 10 with NaOH and stored in a tank at 10°C during use. The feed was stirred and made up freshly every 1-3 days.

Two of the UASB reactors were given a feed with a trace metal supplement according to Wiegant *et al.*, (1983). 1 ml of this supplement was added per litre of feed, containing Co, Fe, Mn, Se, Mb, B, Zn, Ni and Cu. Other reactors were not supplemented.

Analytical Methods

The COD was determined on diluted samples using COD reagent (BDH, Poole, Dorset) and heating in sealed tubes at 140°C for 2 hours in a Techne Dri Block. All effluent COD measurements were performed on samples allowed to settle for 30 minutes in a 100 ml cylinder. Biogas and individual volatile fatty acids were determined by the methods of Peck *et al.* (1985). Bicarbonate alkalinity (partial alkalinity) was determined by titration to pH 5.75, according to Ripley *et al.* (1975). Suspended solids in mixed liquor samples of contact process reactors (MLSS) and mixed liquor volatile suspended solids (MLVSS) were determined by Standard Methods (1985).

Laboratory-scale reactors: Start-up and Operation

The UASB reactors were each seeded with 1 l of granules donated by Paques, The Netherlands, originating from a plant treating dairy waste. The reactors were operated on loading rates (B_v 's) of 0.5-1.4 kg COD/m³

day, over the first 2 months. The reactors were continuously fed and operated for 13 months stably reaching average organic loading rates (B_v 's) of approximately $15 \text{ kg COD/m}^3 \text{ day}$, equivalent to an hydraulic retention time of approximately 8 hours. The granules did not break up during operation.

The contact process reactors were seeded with working digester contents from the pilot plant contact process reactor and started up on a loading rate of $0.2\text{-}0.6 \text{ kg/m}^3 \text{ day}$ over the first four months. Some problems were experienced with pH, the bicarbonate alkalinity being approximately 600 mg/l since at this stage bicarbonate was not added to the feed. Once this was corrected the reactors were operated for 2 years. The reactors were fed every two hours throughout the day and, in order to retain biomass, the stirrers were automatically switched off 30 mins prior to each feed and were not reactivated until 30 mins after feeding. The effluent from each reactor was allowed to settle and the biomass re-added daily manually to the reactor.

Towards the end of this work, three CP reactors were fed at a loading rate above that previously found to cause failure, in order to produce an acidogenic first stage. The effluent from these reactors was fed into the UASB reactors and steady state attained in 28 days. The loading rate to the system was (considering the combined volumes of the two reactors) $6.6 \text{ kg/m}^3 \text{ day}$. This corresponds to a loading rate to the UASB alone (assuming no COD was lost in the acidogenic stage) of $15 \text{ kg/m}^3 \text{ day}$. The HRT in the acidogenic reactor was 9 hours and in the methanogenic reactor was 7.2 hours.

Step Load Experiments on CP Reactors

Prior to shock loading the laboratory scale reactors were monitored for a 12 hour period in order to ensure steady state operation and to evaluate the degree of fluctuation expected during periods of stability. This was followed, for a preset period of time, by an increase in the feed concentration from approximately $4500 \text{ mg[O]}/1$ to $22,500 \text{ mg[O]}/1$, corresponding to a five-fold increase in organic load. The feed concentration was then returned to its initial value. Volatile Fatty Acid (VFA) and COD levels were measured every two hours until steady state levels were regained.

To compare response to step overloads at different mixed liquor suspended solids, for a period of five days before the shock the MLSS in all five of the reactors was adjusted by dividing the settled solids from the digester effluents unequally and returning different amounts of solids by hand to each reactor. During the shock, settled solids were no longer returned to the reactor to avoid variation in the liquid volume added daily to each reactor.

Step Load Experiments: Comparison of UASB Single-Stage and Two-stage Process

The single-stage UASB reactors operating at steady state on a loading rate of $15 \text{ kg/m}^3 \text{ day}$ were subject to step overloads for periods of 2, 4, 6 and 8 hours by increasing the feed strength five-fold, and effluent COD and individual VFA levels monitored. The feed concentration was then returned to its initial value. After conversion of three UASB's to two-stage reactors, and attainment of steady state over a 28 day period, these reactors were subject to an 8 hour step overload by increasing the loading rate to the system (considering the combined volumes of the two reactors) from $6.6 \text{ kg/m}^3 \text{ day}$ to $33.2 \text{ kg/m}^3 \text{ day}$ for a 8 hour period by increasing the feed strength. This corresponds to a loading rate to the UASB alone (assuming no COD was lost in the acidogenic stage) increasing from 15 to $74 \text{ kg/m}^3 \text{ day}$.

Step Load of Pilot Plant Reactor

The upflow anaerobic filter, actual liquid volume 5.28 m^3 , contained 1.64 m^3 Pall rings of specific surface area $160 \text{ m}^2 \text{ per m}^3$ rings, in the upper one-third of the reactor. The reactor had been operating successfully for three years at the time of this experiment. At the start of the 8 hour shock the feed balance tank which had been delivering feed of 4.7 kg COD/m^3 was filled with simulated ice-cream waste water of 13.6 kg COD/m^3 .

RESULTS AND DISCUSSION

Effect of Trace Metal Addition on UASB Stability

Three UASB reactors, two of which had a trace metal supplement, were operated at a B_v of 15 kg COD/m³ day to steady state, and shocked for 8 hours as described above by a five-fold increase in feed concentration. Measurement of effluent individual volatile fatty acids showed that all of the reactors had recovered to pre-shock levels in all cases 16 hours after the initiation of the shock. However, the actual maximum levels of individual volatile fatty acids, particularly propionic acid, produced in response to the shock are clearly higher in the unsupplemented reactor. Fig. 1a) and b) shows examples of the effect on individual VFA and effluent COD levels of an 8 hour shock to the supplemented and unsupplemented reactors.

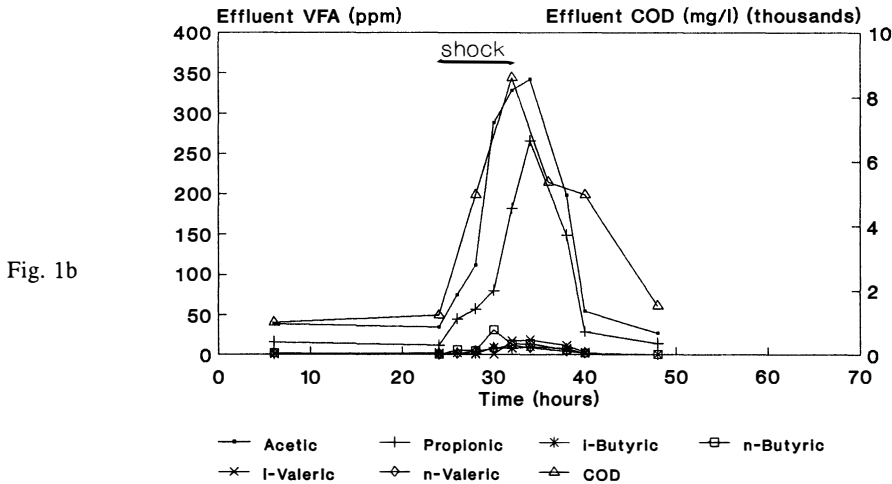
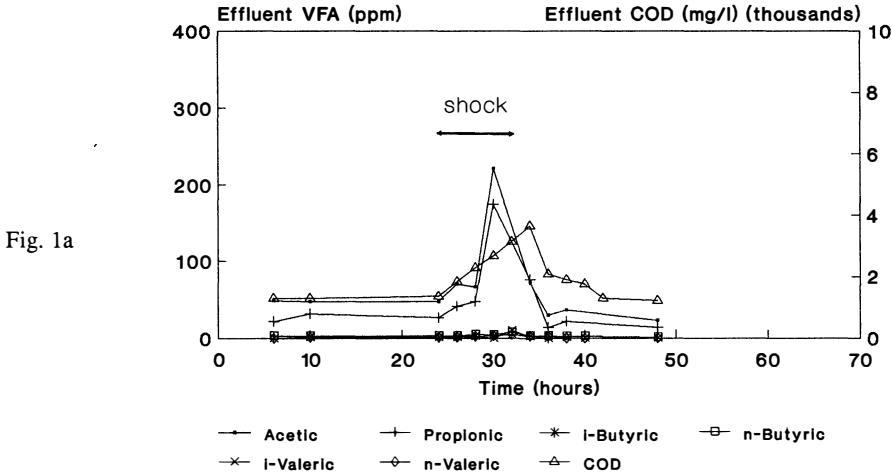


Fig. 1 a) and b). Effect of an 8-hour step overload on a) two trace-metal-supplemented UASB's (average results) b) one non-metal-supplemented UASB reactor.

Fig. 2 shows a further difference between the supplemented and unsupplemented reactors. The maximum effluent COD levels resulting from overloads increase linearly with increasing shock duration in the supplemented reactors while the unsupplemented reactor shows a greater sensitivity to increasing overloads. This more sensitive response of the unsupplemented digester to an 8 hour shock can be observed in the maximum total VFA measurements (Fig. 3). These results suggest that metal-supplemented UASB reactors may be more able to support a shock of this type, although under steady state operation there is apparently no effect of metal supplementation on performance.

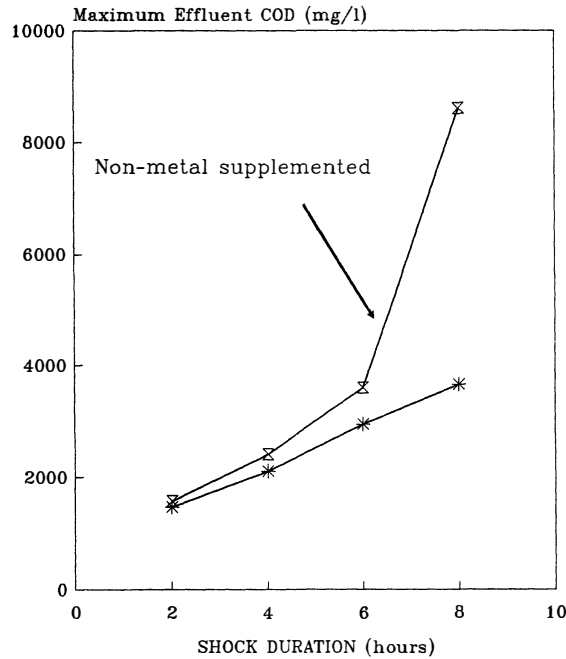


Fig. 2. Peak effluent COD levels obtained during 2, 4, 6 and 8 hour step overloads to two metal-supplemented (average results) and a non-metal-supplemented UASB reactors.

Effect of Shocks on Contact Process

Five contact process reactors were operated in parallel to steady state at an organic loading rate of 3.32 kg/m³ day, corresponding to a retention time of 33 hours and giving a COD removal of 85%. As described under METHODS, the MLSS of each reactor was adjusted, giving reactors with MLSS concentrations on the day of the shock of 3.5 g/l, 4.26 g/l, 6.96 g/l, 8.78 g/l and 9.35 g/l. The feed strength was then increased five-fold for 12 hours, giving a temporary loading rate of 16.6 kg/m³ day. During recovery the feed strength was returned to its previous value. For the reactors which failed the feed was stopped until recovery.

In the case of the reactors with MLSS 6.96-9.35 g/l, the effluent COD values followed a similar pattern, rising to approximately 1500 mg(O)/l towards the end of the overload, and returning to preshock levels approximately 14 hours after the step load had ended. In the case of the reactors containing 3.5 g/l and 4.26 g/l MLSS, the effluent COD and VFA reached much higher levels and did not return to preshock levels, even after a period of three weeks with no further feeding. These findings confirm the importance of efficient biomass recycle devices in full scale reactors. In the case of the SERC Anaerobic Facility contact process reactor, problems with the recycle mechanism limited mixed liquor SS to an average of 3.0 g/l (2.3 g/l mixed liquor volatile suspended solids) rising to a maximum of 3.7 g/l SS as these problems were overcome.

However, the contact process pilot scale reactor performed consistently well, though at a low average loading rate of 1.1 kg COD/m³ day, at a 5 day HRT, giving a mean COD removal of 83%.

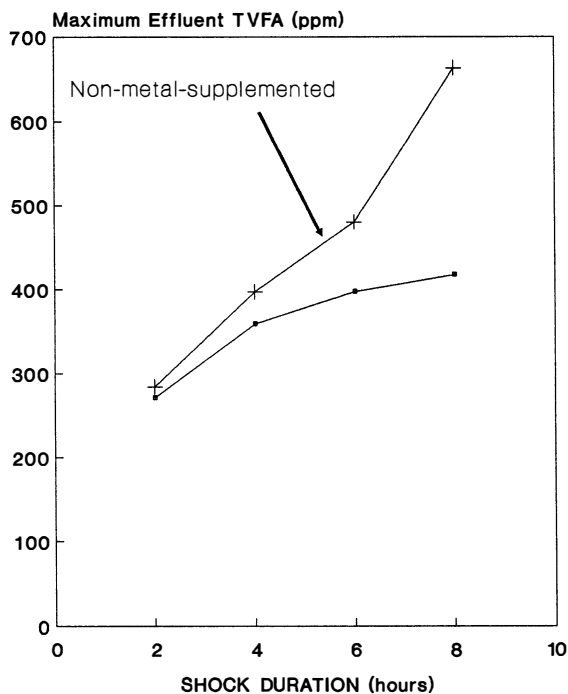


Fig. 3. Peak total Volatile Fatty Acid concentrations obtained during 2, 4, 6 and 8 hour step overloads to two metal-supplemented (average results) and a non-metal-supplemented UASB reactors.

Effect of Shock Loads on One- and Two-Stage UASB's

The stability of the single-stage UASB reactors was compared with that of two-stage UASB operation as described under METHODS, with an acidogenic first-stage contact process reactor and an acetogenic-methanogenic population in the UASB. Although steady state VFA levels in the effluent from both reactors were low, on shocking for 8 hours the maximum effluent VFA from the two-stage process was double that of the single stage reactor and baseline levels were re-established more quickly in the single-stage process, 8 hours after the shock (Fig. 4a and b). For comparison, the acetic and propionic acid levels achieved in the acidogenic stage of the two-stage process during the same shock are also shown (Fig. 5).

From the above results it would appear that the methanogenic stage of the two-stage process was under more stress than the single-stage UASB reactor, as the volatile fatty acids reached much higher levels and required more time to return to normal in the former. However the effluent COD levels show the converse to be true. The COD levels in the single-stage reactor peak at 8600 mg[O]/l, corresponding to 62% COD removal efficiency. In the case of the two-stage reactor the effluent COD levels in the methanogenic stage reached a maximum of 3690 mg[O]/l, corresponding to a removal efficiency of 82%.

Thus the two stage UASB reactor is more able to retain a good COD removal efficiency than a single-stage UASB under these organic overload conditions. It should be noted however that the granules when developing an acetogenic population and adapting to high feed VFA levels appeared to be starting to break up, and changed colour from black to brown. Thus UASB granules operating in a two-stage process on this waste may begin to lose their good settling properties. The 2-stage process was operated for two months.

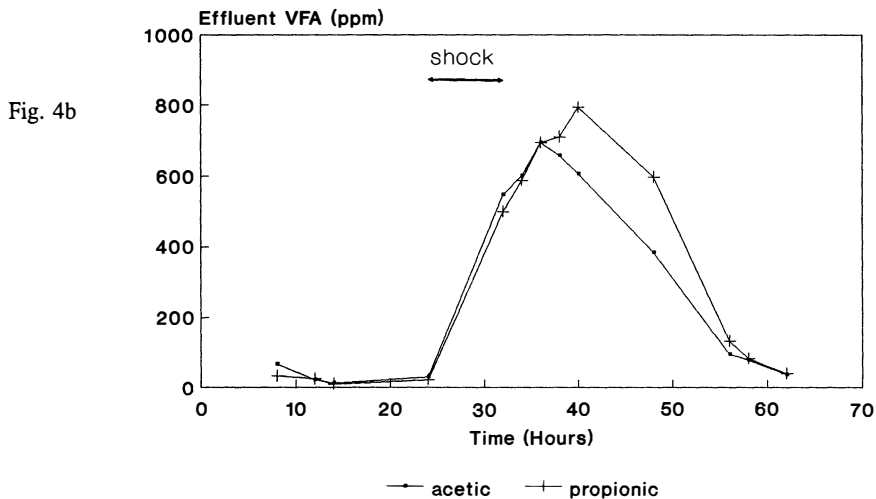
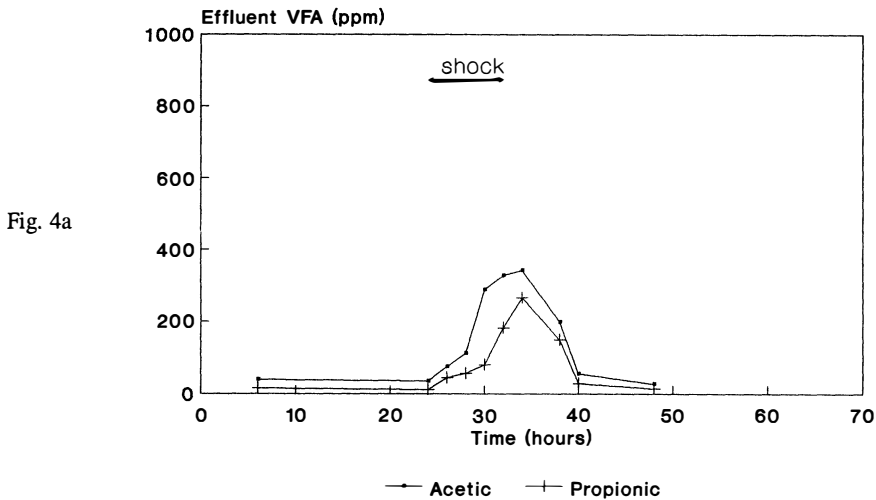


Fig. 4 a) and b). Effect of a five-fold step load on effluent acetate and propionate levels for a) three single-stage UASB's (average results) and b) two 2-stage UASB's (average results).

Step Loading of Pilot-scale Anaerobic Filter

The anaerobic filter was operated at steady state for five weeks on an average volumetric loading rate (B_v) of $6.8 \text{ kg/m}^3 \text{ day}$. Mean values for some parameters analysed in this period were: effluent bicarbonate alkalinity $1660 \text{ mg CaCO}_3/\text{l}$ (std dev 380, number of readings (n)=12), pH 7.04 (std dev 0.17, n =36), $\% \text{CH}_4$ 73% (std dev 4, n =12), acetic acid 96 mg/l (std dev 127, n =12) and propionic acid 118 mg/l (std dev 110, n =12). It can be seen that the VFA levels measured during this period were very variable.

The filter was subject to a step load in which the B_v was tripled for 8 hours. The start of the shock corresponds to time zero on Figs. 6 and 7. As can be seen from Fig. 6, the bicarbonate alkalinity measured by titration responded immediately, mirroring an increase in acetic and propionic acid concentrations. This change

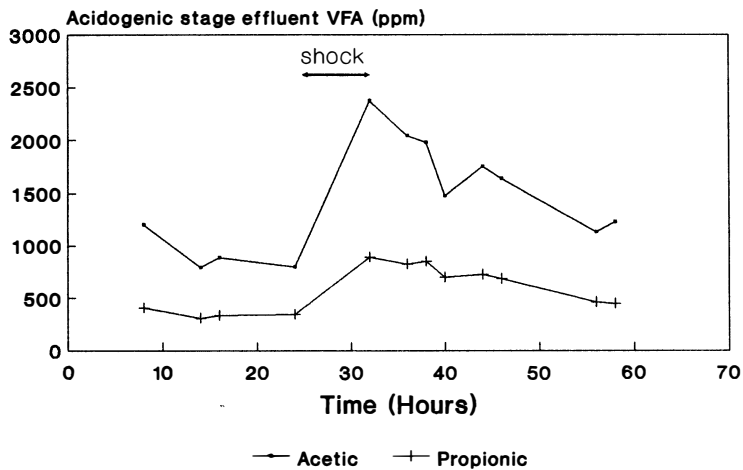


Fig. 5. Effect of 8 hour step overload on the acetic and propionic acid concentrations in the effluent from the acidogenic phase of the three two-stage reactors (average results).

of bicarbonate alkalinity is within the range distinguishable by a bicarbonate monitoring device being developed by the authors (Hawkes *et al.*, in press) which does not utilise a pH probe and is therefore unaffected by fouling. While VFAs are difficult and expensive to monitor on-line, it is anticipated that bicarbonate alkalinity could be followed on-line using the proposed device, thus signaling the existence of a problem within two hours of the onset of unstable conditions.

Changes in pH and %CH₄ in response to the shock are seen in Fig 7. The pH was not measured on-line in this experiment because of problems with probe fouling and drifting; however the pH measurements still fluctuate and do not appear to give such a clear indication of instability as the bicarbonate alkalinity. The response time to changes in methane composition will depend on the size of the head space and the rate of production of biogas.

Bicarbonate alkalinity is an important parameter in assessing digester stability (Rozzi *et al.*, 1985, Pohland and Engstrom, 1964). McCarty (1964) suggests that bicarbonate alkalinity should not drop below 1000 mg CaCO₃ /l or the digester pH will drop to undesirably low levels. During the three years of operation the mean bicarbonate alkalinity measured in the effluent from the anaerobic filter was 1430 mg CaCO₃ /l. It is probable that a control strategy based on on-line bicarbonate alkalinity measurements may improve the performance of reactors operating on similar food-processing waste-waters.

CONCLUSIONS

Although granulation on ice-cream wastewater was not achieved, commercially available UASB granules obtained from a full-sized dairy waste reactor operated stably for over a year on this waste at laboratory scale.

At laboratory scale, the stability of UASB reactors operating on ice-cream wastewater is enhanced by supplementation with trace metals, although there is no apparent effect on steady-state operation.

At equivalent COD removal efficiencies, a two-stage UASB is more able to retain a good COD removal efficiency than a single-stage UASB. Given an equivalent shock, contact process reactors with levels of SS at least equal to that obtained in the pilot plant, failed.

Bicarbonate alkalinity was shown to be a rapidly responding indicator of instability during a step overload of a pilot plant anaerobic filter, and the change observed was in the range which could be followed by a novel bicarbonate alkalinity monitor being developed by the authors.

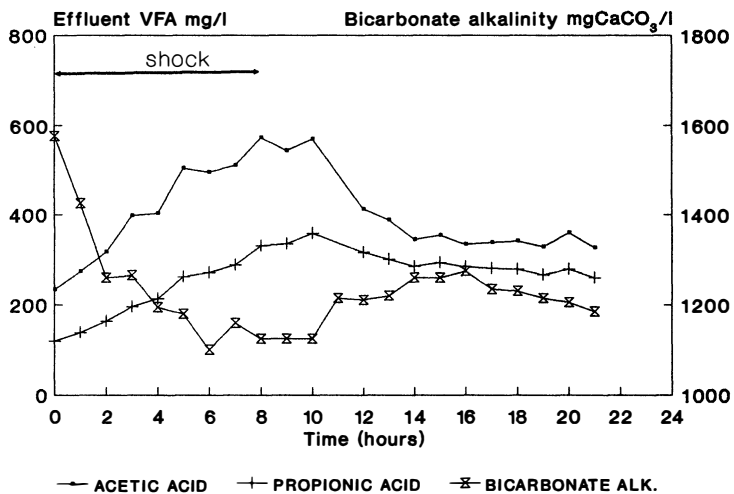


Fig. 6. Effect on effluent acetate, propionate and bicarbonate alkalinity levels of an 8 hour step overload to the pilot scale anaerobic filter. B_v increased 3-fold to 21 kg COD/m³ day at time zero.

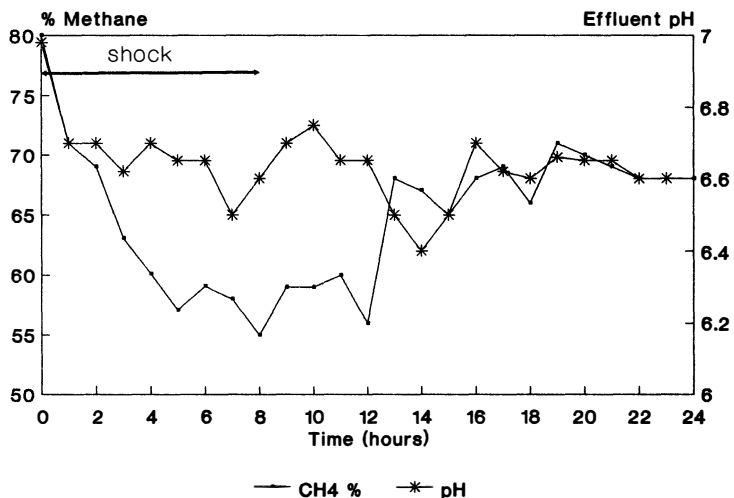


Fig. 7. Effect on % methane and effluent pH of an 8 hour step overload to the pilot scale anaerobic filter. B_v increased 3-fold to 21 kg COD/m³ day at time zero.

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