Anaerobic treatment of a synthetic dairy effluent using a hybrid digester

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Abstract

A mesophylic laboratory-scale hybrid anaerobic digester, combining an upflow sludge blanket and a fixed-bed design, was evaluated for the anaerobic treatment of a synthetic dairy effluent. In the first experimental study, the chemical oxygen demand of the dairy effluent was increased stepwise from 3 700 to 10 300 mg·ℓ^{-1}. In the second experimental study the chemical oxygen demand (COD) of the synthetic dairy effluent was kept constant at 10 000 mg·ℓ^{-1} and the hydraulic retention time (HRT) was shortened stepwise from 4.1 to 1.7 d. A COD removal of between 90 and 97% was achieved at organic loading rates of between 0.82 and 6.11 kg COD·m^{-3}·d^{-1}. At an HRT of 1.7 d, the digester achieved a methane yield of 0.354 m^3 CH_4 per kg $COD_{removed}$. The best results in terms of methane yield were achieved at an HRT of 1.9 d. The data also showed that the maximum operational potential of the digester had been reached, as indicated by the drop in methane yield observed at the end of the second experimental study. The results clearly show that this particular type of digester would be suitable for the anaerobic treatment of dairy effluents. An important consequence of the data from this study is that a two-phase set-up will be required to protect the methanogens in the digester from inhibitively low pH values and high concentrations of volatile fatty acids (VFAs) produced during the acidogenic phase. The two-phase system will allow pH control in the acidogenic phase, should it be needed at a full-scale or pilot-scale treatment plant.

Introduction

Water management in the dairy industry is well documented (Jones, 1974; Water Research Commission, 1989), but effluent production and disposal remain a problematic issue for the dairy industry. A survey in 1989 (Water Research Commission, 1989) concluded that South African dairies apply either very basic or very inefficient effluent treatment procedures. Moreover, a more recent survey of the South African dairy industry (Strydom et al., 1993) revealed that effluent disposal is currently the most important water-related factor where improvement is desirable. Dairy effluent disposal in South Africa usually results in one of two problems: firstly, high treatment levies are charged by local authorities for industrial effluents; and secondly, further pollution can be caused when untreated effluents are either discharged into the environment or used directly as irrigation water. For the year 1991 (Strydom et al., 1993), a total of R1.5 m. on effluent disposal was spent by dairies which process 70% of the milk in South Africa. The second problem of disposal was more prevalent at large dairies situated near dairy farms in rural areas, where access to adequate waste-water treatment works is not available.

To enable the dairy industry to contribute to water conservation, an efficient and cost-effective effluent treatment technology has to be developed. To this effect, anaerobic digestion offers a unique treatment option to the dairy industry. Not only does anaerobic digestion reduce the COD of an effluent, but little microbial biomass is produced. The biggest advantage is energy recovery in the form of methane and up to 95% of the organic matter in a waste stream can be converted into biogas (Weber et al., 1984).

Many high-rate digester designs are currently available, and some have successfully been used for the treatment of dairy effluents. Lettinga and Hulshoff-Pol (1991) reported that of the 205 full-scale upflow anaerobic sludge blanket digesters in use world-wide in 1991, six were used to treat dairy effluents. The fixed-bed digester is another high-rate digester that has been used for the treatment of dairy effluents (De Haast et al., 1983). A high-rate combination design, using the upflow anaerobic sludge blanket (UASB) and the fixed-bed digester types, was developed by Guiot and Van den Berg (1984). This design was successfully used to treat landfill leachate (Myburg and Britz, 1993) and baker's yeast factory effluent (Van der Merwe and Britz, 1993). Landfill leachate and yeast effluent both have high COD concentrations and both are difficult to degrade biologically. On the other hand, dairy effluents are fairly easily biodegradable, since they consist mainly of diluted dairy products. Thus, the aim of this study was to evaluate, on laboratory-scale under mesophilic conditions, the use of the hybrid digester as an option in the treatment of a synthetic dairy effluent.

Materials and methods

Digester design

A laboratory-scale hybrid anaerobic digester (Myburg and Britz, 1993) was used. The digester had an operational volume of 5.0 ℓ and combined a UASB and a fixed-bed digester design with a gas/solids separator at the top of the digester. This high-rate hybrid digester successfully combines advantages of both systems while avoiding their drawbacks. It also facilitates an important phenomenon of microbial community acclimation to the elevated VFA concentrations. The gas exited through the top, while the substrate was introduced into the digester at the base. The overflow of the digester emptied through a U-shaped tube to prevent atmospheric oxygen from entering the system. The temperature of the digester was maintained at 35°C using a heating tape and an electronic control unit (Meyer et al., 1985) and the digester was insulated. The volume of the biogas was determined using a

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manometric unit equipped with an electronically controlled counter and a gas-tight valve. The biogas volumes were corrected to standard temperature and pressure (STP).

Substrate

The synthetic dairy effluent consisted of a mixture of 20 $g \cdot \ell^{-1}$ plain yoghurt and 75 $m\ell \cdot \ell^{-1}$ cottage cheese whey. This was diluted to the required COD concentration. Initially, the substrate was supplemented with 100 $mg \cdot \ell^{-1}$ urea and 100 $mg \cdot \ell^{-1}$ K_2HPO_4 to prevent any nitrogen and phosphorus limitation during the start-up period. The substrate was also supplemented with 1.0 $m\ell$ trace element solution, as described by Nel et al. (1985).

Digester start-up

The digester was originally seeded with a mixture of sewage sludge obtained from a municipal digester, as well as rumen fluid and digester effluents from other mesophilic digesters. This was done in order to supply the digester with a diverse mesophilic microbial community. The digester was then allowed to stabilise for 48 h in order to allow the bacterial community to acclimatise and attach to the polyethylene fixed-bed. After the stabilisation period, feeding was commenced with a diluted substrate (COD = 3 700 $mg \cdot \ell^{-1}$). The substrate was semi-continuously fed to the digester by means of a peristaltic pump (Watson-Marlow 500) controlled by an electronic timer. After the start-up period, the HRT was set at 4.5 d.

Pre-acidification step

In order to counter persistent pH instability experienced during start-up, the feed was pre-acidified using plain yoghurt as inoculum and incubated at 30°C for 24 h. The resultant acids were neutralised and the pH adjusted to 9.0 units with 6.0 N sodium hydroxide. This pre-acidified substrate was then fed to the digester. The use of yoghurt as inoculum was later discontinued and replaced with a *Klebsiella oxytoca* (strain A1) previously isolated from a digester treating a yeast factory effluent (Van der Merwe and Britz, 1994).

Analytical procedures

The following parameters were analysed according to *Standard Methods* (1985): pH, alkalinity, total Kjeldahl nitrogen (TKN), total solids (TS), volatile solids (VS) and total non-volatile solids (TNVS). COD and orthophosphate phosphorus (PO_4-P) were determined colorimetrically using a DR 2000 spectrophotometer (Hach Co. Loveland, CO) and standardised procedures (*Standard Methods*, 1985).

VFAs were determined using a Hewlett Packard (Avondale, PA) gas chromatograph, equipped with a flame ionisation detector and a 30 m x 0.75 mm i.d. Nukol (Supelco, Inc., Avondale, PA) capillary column. The column temperature was initially held at 120°C, then increased at a rate of $6°C \cdot min^{-1}$ to 185°C. The detector and the inlet temperatures were set at 250°C and 160°C respectively and nitrogen was used as carrier gas at a flow rate of 5 $m\ell \cdot min^{-1}$.

The biogas composition was determined on a Varian 3300 gas chromatograph (Varian Ass., Walnut Creek, CA) equipped with a thermal conductivity detector and column (2.0 m x 0.3 mm i.d.) packed with Porapak Q (Waters Ass. Inc, Milford, MA), 80-100 mesh. The oven temperature was set at 55°C and hydrogen was used as carrier gas at a flow rate of 40 $m\ell \cdot min^{-1}$.

Experimental studies

The study comprised two experimental studies (I and II). In the first study (I), the synthetic dairy substrate COD concentration was increased stepwise from 3 700 to 10 300 $mg \cdot \ell^{-1}$ in five phases. In the second experimental study (II), the COD concentration was kept constant at 10 000 $mg \cdot \ell^{-1}$, while the HRT was reduced stepwise from 4.1 to 1.7 d in nine phases. In both studies, the digester was allowed to reach stable-state conditions before each HRT reduction. Stable state is defined as a state which can be maintained indefinitely without system failure (Cobb and Hill, 1990), during which the variation in digester performance parameters is less than 10%. Thus, the length of each phase was based on the stability of the digester effluent pH and the COD removal.

Results and discussion

Substrate

The average composition of the basic synthetic dairy effluent, after pre-acidification, as it was used as substrate for the digester, is shown in Table 1. The substrate for experimental study (I) was a dilution of the substrate given in Table 1. The COD concentration of the substrate used in the different phases of the two experimental studies is given in Tables 2 and 3. The characteristics of the basic substrate used in the study were similar to values reported by other workers (Water Research Commission, 1989) for dairy effluents. The absence of VFA is also characteristic of dairy effluents. However, once microbial degradation of the effluent starts, the VFA concentrations will rapidly increase.

COD removal

Figure 1 depicts the percentage COD removal and COD removal rate (R) plotted as a function of the organic loading rate (OLR) for both experimental studies (I and II). From Fig. 1 it is evident that

TABLE 1
AVERAGE COMPOSITION OF THE SYNTHETIC DAIRY EFFLUENT SUBSTRATE AFTER PRE-ACIDIFICATION

Parameter	Average	±s.d.
COD ($mg \cdot \ell^{-1}$)	10 486	±237
TOC ($mg \cdot \ell^{-1}$)	2 090	±49
pH	9.0	
TS ($mg \cdot \ell^{-1}$)	7 240	±400
TVS ($mg \cdot \ell^{-1}$)	5 200	±510
TNVS ($mg \cdot \ell^{-1}$)	2 040	±340
VFA ($mg \cdot \ell^{-1}$)	212	
Acetic acid ($mg \cdot \ell^{-1}$)	212	
Propionic acid ($mg \cdot \ell^{-1}$)	0	
iso-Butyric acid ($mg \cdot \ell^{-1}$)	0	
n-Butyric acid ($mg \cdot \ell^{-1}$)	0	
iso-Valeric acid ($mg \cdot \ell^{-1}$)	0	
n-Valeric acid ($mg \cdot \ell^{-1}$)	0	
Caproic acid	0	
PO_4* (as phosphorus, $mg \cdot \ell^{-1}$)	60.6	
TKN* ($mg \cdot \ell^{-1}$)	205.1	
Alkalinity (as $mg \cdot \ell^{-1}$ $CaCO_3$)	612	±86

*Mean values for 3 determinations

TABLE 2
OPERATING CONDITIONS AND DIGESTER PERFORMANCE DURING EXPERIMENTAL STUDY (I) WHERE THE COD OF THE SUBSTRATE WAS INCREASED*

Parameter	Phase				
	1	2	3	4	5
Substrate COD ($mg \cdot \ell^{-1}$)	3 700	5 095	6 165	9 200	10 300
COD removal (%)	90	93	95	95	96
HRT (d)	4.5	4.5	4.5	4.5	4.5
OLR ($kgCOD \cdot m^{-3} \cdot d^{-1}$)	0.82	1.13	1.37	2.04	2.29
pH (digester effluent)	6.85	7.30	7.34	7.78	7.32
Biogas ($\ell \cdot d^{-1}$)	n.d.	n.d.	3.32	4.25	4.71
Methane content (%)	n.d.	71	68	63	64
Y_{CH4} ($m^3 \cdot kg^{-1}$ $COD_{removed}$)	n.d.	n.d.	0.314	0.249	0.253
TS-in ($mg \cdot \ell^{-1}$)	n.d.	3 340	4 390	5 820	7 110
TS removal (%)	n.d.	57	63	67	68
VS-in ($mg \cdot \ell^{-1}$)	n.d.	2 200	2 710	3 980	5 660
VS removal (%)	n.d.	86	88	90	91
TNVS-in ($mg \cdot \ell^{-1}$)	n.d.	1 140	1 680	1 840	1 450
TNVS removal (%)	n.d.	1.8	23	16	-22
Alkalinity-in ($mg \cdot \ell^{-1}$ $CaCO_3$)	n.d.	n.d.	n.d.	653	528
Alkalinity-out ($mg \cdot \ell^{-1}$ $CaCO_3$)	n.d.	n.d.	n.d.	1 770	1 793

* Data taken after stable state had been reached and mean values for 3 determinations
n.d. = not determined

TABLE 3
OPERATING CONDITIONS AND DIGESTER PERFORMANCE DURING EXPERIMENTAL STUDY (II) WHERE THE HRT WAS SHORTENED*

Parameter	Phase								
	1	2	3	4	5	6	7	8	9
Substrate COD ($mg \cdot \ell^{-1}$)	10 360	10 300	10 800	10 800	10 485	10 765	10 220	10 345	10 386
COD removal (%)	96	95	97	96	94	95	95	93	92
HRT (d)	4.10	3.72	3.25	3.05	2.65	2.52	2.33	1.90	1.70
OLR ($kgCOD \cdot m^{-3} \cdot d^{-1}$)	2.53	2.77	3.32	3.54	3.96	4.27	4.39	5.44	6.11
pH (substrate)	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
pH (digester effluent)	7.54	7.56	7.70	7.25	7.55	7.70	7.54	7.12	7.56
Biogas ($\ell \cdot d^{-1}$)	5.44	5.46	7.67	9.03	12.52	13.12	13.42	19.08	17.79
Biogas CH_4 content (%)	64	55	62	61	60	61	64	57	56
Y_{CH4} ($m^3 \cdot kg$ $COD_{removed}$)	0.264	0.206	0.278	0.299	0.357	0.356	0.372	0.372	0.354
Substrate TS ($mg \cdot \ell^{-1}$)	7 020	6 460	7 560	6 920	7 250	7 630	7 540	7 670	8 570
TS removal (%)	67	70	68	73	70	69	70	68	76
Substrate VS ($mg \cdot \ell^{-1}$)	5 040	4 640	5 510	4 440	4 670	5 750	5 440	5 690	6 780
VS removal (%)	91	90	91	92	91	91	91	90	94
Substrate TNVS ($mg \cdot \ell^{-1}$)	1 980	1 820	2 050	2 480	2 580	1 880	2 100	1 980	1 800
TNVS removal (%)	6	19	5	35	33	-1	16	3	4
Substrate alkalinity ($mg \cdot \ell^{-1}$)	562	543	640	778	560	725	583	593	633
Alkalinity-out ($mg \cdot \ell^{-1}$)	1 843	1 718	2 138	1 778	2 018	2 213	2 380	2 178	2 232

*data taken after stable state had been reached and mean for 3 determinations
n.d. = not determined

the digester was operating efficiently in terms of COD removal, with the COD removal never below 90%. The best COD removal of 97% was achieved in Phase 3 of the second experimental study (Table 3). The COD removal rate (R) for both experimental studies is also plotted in Fig. 1, as a function of the OLR. The linearity of the removal rate (Fig. 1), is a clear indication that the digester had not yet reached its maximum operational limit even though the final percentage COD removals show a decrease. When this limit is approached, the COD removal rate (R) normally reaches a plateau and will then start decreasing (Van der Merwe and Britz, 1993). This is then indicative of insufficient digester capacity, or insufficient microbial biomass accumulation to handle the increased organic load.

The data also indicate that the digester effluent COD was not reduced sufficiently (Fig. 2) to allow direct disposal. In South Africa, the maximum COD concentration that is allowed for effluents which are discharged directly into rivers, is 75 mg·ℓ^{-1} (Republic of South Africa, 1962). With a COD concentration of 810 mg·ℓ^{-1} remaining in the digester effluent in the final phase of the second study, it will be necessary to use a final polishing step (Ross, 1991), to reduce the final effluent COD to an acceptable level. The lowest COD concentration achieved in the effluent, was 287 mg·ℓ^{-1} during Phase 3 of the first study. However, it must be stated that the COD values given are for total COD and not soluble COD. Thus, if a filtration or centrifugation step were to be added to the process, the microbial biomass or sludge would be removed, thereby further reducing the final effluent COD values.

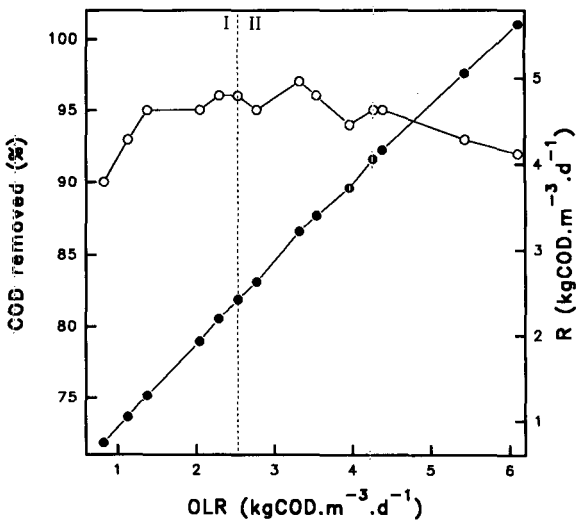

Figure 1
Percentage COD removal (○) and COD removal rate (R) (●) as a function of the OLR as found during experimental studies (I) and (II)

pH and alkalinity

Initially, at the start of the study, the digester showed signs of pH instability, with a tendency towards pH values below 6.8 units, leading to the continuous failure of the anaerobic digestion system. As a result, a pre-fermentation step was introduced. In the first pre-fermentation step, a plain yoghurt culture was used to acidify the dairy substrate, and lactic acid was found to be the most prominent fermentation product. Since acetic acid is more desirable as a direct precursor for methanogenesis (Weber et al., 1984), the inoculum for the synthetic dairy substrate used in the pre-fermentation was changed. With the use of the *Klebsiella oxytoca* (Strain A1), acetic acid was produced as main fermentation product during the pre-fermentation. The pH of the substrate was then adjusted to 9.0 units after a 24 h pre-fermentation period. Although acetic acid was the main fermentation product of the *K. oxytoca* strain, the levels were comparatively low (Table 1). In spite of this, the pre-fermentation was still effective, reducing the pH of the effluent within 24 h at 30°C (Fig. 3), from 7.3 units to between 4.0 and 4.2 units (Fig. 3). However, as expected, the pre-fermentation continued after adjustment to pH 9.0, since the substrate was kept at room temperature while feeding. Thus, the pH of the remaining substrate was invariably much lower than 9.0 by the time it was replaced with new substrate. This was as a result of the lactose remaining in the substrate after pre-fermentation. At the end of the pre-fermentation period, only 22% of the original 1 370 mg·ℓ^{-1} lactose was fermented by the *K. oxytoca* strain. To minimise the effect of this variability, the substrate was prepared daily. However, complete conversion of a substrate during acidogenesis in a two-phase digester is not desirable (Lettinga and Hulshoff-Pol, 1991), and a maximum substrate utilisation of only 20% is generally recommended.

The rapid fermentation of the effluent components necessitated the pre-fermentation step, since the methanogenic population was not adapted to the high flux of organic acids at the beginning

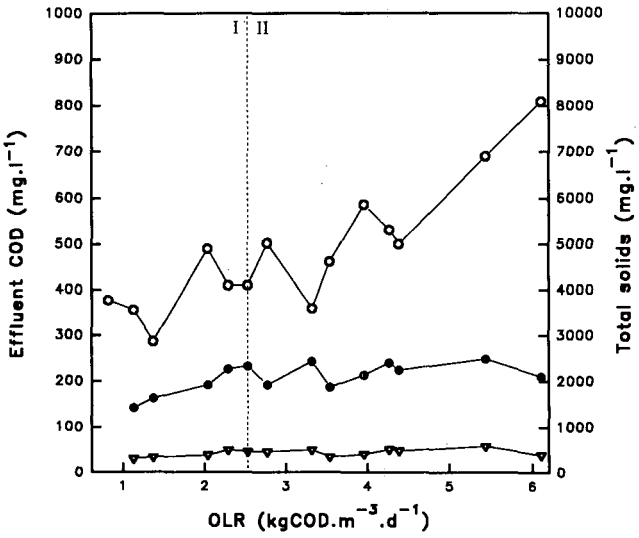

Figure 2
Digester effluent COD (○), total solids (●) and volatile solids concentrations (▽) as a function of the OLR as found during experimental studies (I) and (II)

of this study. After the pre-acidification step was introduced, the internal pH of the digester stabilised above pH 7.0 and the COD concentration could successfully be raised to 5 095 mg·ℓ^{-1} (Phase 2, experimental study (I)). During the remainder of the study, the pH of the digester effluent remained above 7.0 units, with one exception

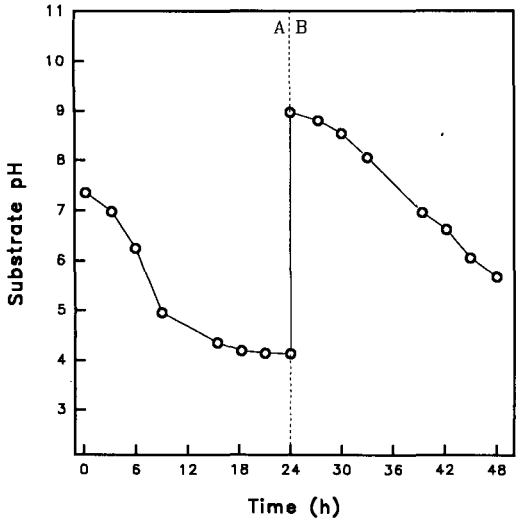

Figure 3
pH of the dairy substrate during the pre-fermentation period (A) and the subsequent period after neutralisation (B) to pH 9.0, at room temperature

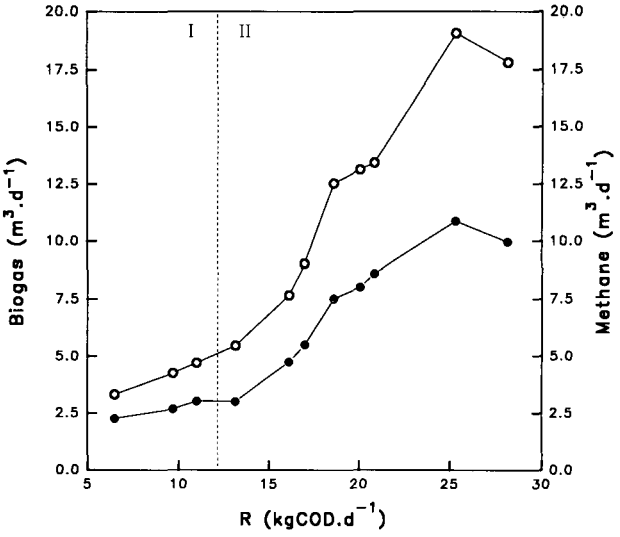

Figure 5
Biogas (○) and methane (●) production rate, as a function of COD removal rate (R) during experimental studies (I)and (II)

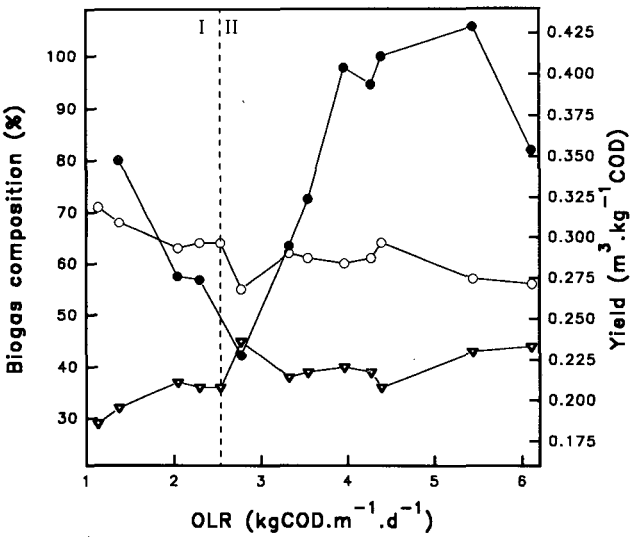

Figure 4
Biogas composition (○ = CH_4; ▽ = CO_2) and methane yield per kg of COD removed (●), as a function of the OLR as found during experimental studies I and II.

- at the start of the eighth phase of the second experimental study (Table 3) the pH of the digester effluent dropped to a low of 6.7 units, but the digester recovered and the pH stabilised again after four days at 7.12 units. Thus, whenever changes are made to the environmental parameters of the digester, such as an increased OLR, the bacterial populations need time to adapt to the new conditions. During this time, the temporarily reduced pH is indicative of a less than optimal digester performance.

The alkalinity of the digester effluent throughout both experimental studies (Tables 2 and 3) showed that there was substantial buffering capacity available. This alkalinity, however, might have been due, in part, to the neutralisation of the acids after the pre-fermentation step. Thus, the alkalinity might possibly be negatively affected if the amount of sodium hydroxide is reduced later on. The reduction of the amount of the neutralising agent after the pre-fermentation is important in order to minimise the costs involved in the process.

Total solids, volatile solids and non-volatile solids

In Tables 2 and 3 the digester efficiency, in terms of the TS, VS and TNVS percentage removal values, is given. In Fig. 2 the effluent COD and the TS concentrations during both experimental studies are shown. The percentage VS removal shows a good correlation to the COD removal, with a correlation coefficient of 0.94 ± 0.004.

The TNVS concentration was reduced during the first three phases of experimental study (I), but thereafter it was found to be substantially higher. This is not surprising, since the TNVS represent mainly minerals, which are utilised only to a very limited extent by the bacterial community in any biological system. These minerals may also have been precipitated in the digester and washed out at a later stage, thus explaining the removal efficiency of -22% which was found in Phase 5 of the first study (Table 2).

Volatile fatty acids

Throughout the study, the concentration of all the important fatty acids (acetic, propionic, *i*- and *n*-butyric, *i*- and *n*-valeric, and caproic acids) in the digester effluent at stable state, was below normal detection limits. This is indicative of a high methanogenic activity, and it is possible that the OLR for each phase could have been raised far more quickly without seriously affecting the operational efficiency of the digester.

Carbon:nitrogen:phosphorus ratio

The carbon:nitrogen:phosphorus (C:N:P) ratio has an important influence on digester stability and performance (Lettinga and Hulshoff-Pol, 1991). Since the optimal C:N:P ratio for anaerobic digesters is around 100:10:1, this particular effluent is very well suited to anaerobic digestion, with a C:N:P ratio of 100:9.8:2.89. This C:N:P ratio is for the substrate used in the second experimental study, without any urea or phosphate additions. During the first study the effluent was enriched with K_2HPO_4 and urea only to prevent nutrient limitations during start-up.

Biogas production and methane yield

Figure 4 illustrates the digester efficiency in terms of the biogas composition, as a function of the OLR. An overall decrease in the methane content of the biogas is evident for both experimental studies (Fig. 4). During experimental study (I), the methane yield ($Y_{CH4/CODremoved}$) showed a sharp decrease (Fig. 4). This was attributed to possible "dead space" in the digester, which was overcome during experimental study (II) where the HRT was continually shortened, resulting in a higher upflow velocity. The higher upflow velocity probably improved the mixing in the digester, thus reducing dead spaces. Thus, although the methane content of the biogas showed an overall decrease, the methane yield improved. In fact, the methane yield of the digester was slightly higher than the theoretical maximum yield that can be obtained when glucose is used as carbon source (0.35 m^3 $CH_4 \cdot kg^{-1}COD_{removed}$).

Figure 5 illustrates the rate of both biogas ($m^3 \cdot d^{-1}$) and methane ($m^3 \cdot d^{-1}$) produced, plotted against the rate of COD removal (kg $COD_{removed} \cdot d^{-1}$). The non-linearity of these plots is a clear indication that the digester microbial biomass was continuously adapting to the changing environmental conditions (increase in organic loading rate). However, the sudden downward trend at an OLR of 6.11 kg $COD \cdot m^3 \cdot d^{-1}$ (Phase 9 - Table 3) is indicative that the digester was probably reaching its maximum operational potential.

Conclusions

In conclusion, it is clear from the data obtained in this study that dairy effluents are suitable for treatment by means of the anaerobic digestion process and specifically with the use of a hybrid anaerobic digester. The anaerobically digested synthetic dairy effluent had a greatly reduced COD concentration (>90%) and a pH value around 7.2 units, making it more acceptable as a source of irrigation water or to a local water authority. Successful anaerobic digestion of dairy effluents will enable dairy companies to make a contribution to the conservation of South Africa's water resources.

The high biodegradability of the dairy effluent causes it to be easily hydrolysed and fermented to organic acids. Thus, the rate-limiting step of the process is methane generation. The most important consequence of the data from this study is that a two-phase set-up will be required to protect the methanogens in the digester from inhibitively low pH values and high concentrations of VFAs produced during the acidogenic phase. The two-phase system will allow pH control in the acidogenic phase, should it be needed at a full-scale or pilot-scale treatment plant. Bearing in mind the extreme and often hourly fluctuation of dairy effluent quality, this is an important advantage. However, the acid neutralisation requirement of the pre-fermentation step will have to be reduced. The effect of this reduction on the alkalinity and pH stability of the digester must still be evaluated. Another option is to make use of recirculation of the digester effluent, in order to utilise the high alkalinity in the digester effluent.

The economic feasibility of anaerobic treatment of dairy effluents using the hybrid digester is positively influenced by the high methane yield and the reduction in COD concentrations. Methane can possibly be utilised at dairy factories to supplement the use of coal as energy source for the generation of steam. Furthermore, the large reduction in total COD concentration should result in a reduction in effluent disposal expenditure, when using COD concentration and pH value of the digester effluent as basis for the calculation of trade effluent tariffs.

Acknowledgements

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