Two-phase anaerobic digestion of three different dairy effluents using a hybrid bioreactor

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Abstract

The South African dairy industry is a major water user and as a result has to reconsider current effluent treatment and disposal methods. The effluents from three dairy factories (cheese, fresh milk and milk powder/butter factories) were analysed and the chemical oxygen demand (COD), pH and effluent volumes were found to be highly variable over short time intervals during the daily production cycles. The pH was found to vary between 2.2 and 11.8 units and the COD values ranged from 800 to 15 000 mg·ℓ$^{-1}$ over a period of 2 h. The average COD of the effluents emerging from the three factories varied between 1 908 and 5 340 mg·ℓ$^{-1}$. Significant differences were also found in the composition of the effluents from the three factories.

In this study, a mesophilic laboratory-scale hybrid bioreactor was used in conjunction with a pre-acidification step to treat the three dairy factory effluents. It was clear from the data obtained on the cheese, fresh milk and milk powder/butter waste waters that dairy effluents are suitable for treatment by means of the anaerobic digestion process and the use of a hybrid anaerobic bioreactor can be seen as a viable treatment option. The COD values of the three pre-digested dairy waste waters were reduced by between 91 and 97% at organic loading rates of between 0.97 and 2.82 kgCOD·m^{-3}·d^{-1} and subsequent methane yields varied from 0.287 to 0.359 m^3 CH_4·kg^{-1}$COD_{removed}$ (73 and 91% of the theoretical maximum yield) during anaerobic digestion. The pH values of all the digester effluents were >7.5 units. The data clearly indicated that anaerobic treatment of the different dairy effluents was successful and that this particular type of bioreactor 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 bioreactor from prohibitively low pH values and high VFA concentrations produced during the acidogenic phase. The two-phase system will allow pH control in the acidogenic phase should it be needed in a full-scale or pilot-scale treatment plant.

Introduction

Water is South Africa's most limiting natural resource and the dairy industry is considered to be a major water user. The Water Research Commission (1989) estimated the total annual water usage of the South African dairy industry to be 4.5 million m^3. Generally, between 75 and 95% of this "process water" is discharged as effluent. In order to contribute to water conservation in South Africa, the dairy industry has to seriously reconsider present effluent treatment and disposal methods and an efficient and cost-effective effluent treatment technology has to be developed.

In a recent survey (Strydom et al., 1993), it was reported that South African dairies were experiencing effluent-related problems. It was also found that dairies generally dispose of their effluents either to municipal sewage treatment works, or by means of irrigation onto pastures. Thus, dairy factories either run the risk of causing surface- or groundwater pollution, or they incur high financial costs for disposing of their effluents to nearby municipal sewage treatment works.

Anaerobic digestion, as an effluent treatment option, offers several benefits to the dairy industry (Anon., 1990; Strydom et al., 1995). Furthermore, successful treatment of dairy effluents not only offers pollution control in the short term, but can also serve as the starting point for the longer term development of a total water reuse biotechnology.

Since an anaerobic digester using a hybrid design was successfully used for the treatment of a synthetic dairy effluent (Strydom et al., 1995), the aim of this study was to evaluate the hybrid digester as an option for the treatment of waste waters from a cheese, fresh milk, and a milk powder/butter factory.

Materials and methods

Dairy factories

Cheese factory

This factory handles up to 160 t of milk per day for the production of various hard cheeses, notably Gouda, Edam and Cheddar. The whey produced is evaporated and transported by tanker to other factories for spray drying. The general factory effluent consists of diluted products and wash water as well as the initial rinse water from the silos and tankers. Presently, the final effluent is irrigated onto approximately 45 ha of pasture.

Fresh milk factory

This factory produces mainly pasteurised milk, fruit juice blends, yoghurt and cottage cheese, and processes about 230 t of milk per day. The cottage cheese whey is included in the general factory effluent. The effluent passes through a fat trap before being discharged into the local municipal sewage treatment works.

Milk powder/butter factory

The milk powder/butter factory produces up to 40 t of butter per day. Roller-dried milk powder and buttermilk are also produced

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in this factory. The effluent consists of highly diluted products and wash water without large quantities of buttermilk. After all possible butterfat has been reclaimed from the initial rinse water from the cream silos and tankers, the combined effluent is finally disposed of by irrigating onto pastures.

Effluents and sampling

The various dairy effluents were obtained by taking grab samples at half-hour intervals over the entire daily production cycle of each factory. The effluent flow rate, at each sampling interval, was determined by means of a bucket and stopwatch method. The resultant individual samples were then combined proportional to the flow rates, as measured for each respective interval. This resulted in flow-proportioned composite effluent samples. This is generally recommended as the most accurate method for sampling effluents of varying quality such as those produced by the dairy industry (Vernick, 1977).

Pre-acidification

In order to counter persistent pH instability experienced during the studies, the effluents from the various factories were pre-acidified as described by Strydom et al. (1995) using the same *Klebsiella oxytoca* strain (Strain A1) previously isolated from an anaerobic digester treating a yeast factory effluent (Van der Merwe and Britz, 1994). The pH of the three flow-proportioned effluents was adjusted to 7.0 before inoculation with the *K. oxytoca* strain and incubated at 30°C. The pH was measured at 3 h intervals, over a period of 24 h. The pre-acidified substrates were then fed to the digester.

Anaerobic digester

A 5 ℓ mesophilic hybrid bioreactor, combining an upflow anaerobic sludge blanket and a fixed-bed, was used (Myburg and Britz, 1993). The bioreactor was inoculated with sewage sludge, rumen fluid and effluent from two other mesophilic laboratory-scale bioreactors. The temperature of the bioreactor was maintained at 35°C using a heating tape and electronic control unit (Meyer et al., 1985). The volume of the biogas was determined using a manometric unit equipped with an electronically controlled counter and a gas-tight valve. The biogas volumes were corrected to standard temperature and pressure (STP). The bioreactor was, prior to this study, used to treat a pre-acidified synthetic dairy effluent (Strydom et al., 1995) consisting of diluted whey and yoghurt, at organic loading rates of up to 6 kg COD·m^{-3}.d^{-1}. The substrate was for this study changed from the synthetic effluent to the actual cheese, fresh milk and milk powder/butter factory effluents. The hydraulic retention time (HRT) of the bioreactor was reset to 1.9 d, which was found to yield the optimum results with the synthetic effluent as substrate (Strydom et al., 1995).

Experimental studies

Three separate experiments were conducted with effluent from the cheese factory being treated during the first study. After stable-state conditions had been obtained, the feeding was suspended for 14 d and the substrate feed then changed in the second study to fresh milk factory effluent, and again after reaching stable state and suspending the feed, the substrate again changed in the third study to the milk powder/butter factory effluent. Stable state is defined as a state which can be maintained indefinitely without system failure (Cobb and Hill, 1990; Hill, 1991), during which the variation in bioreactor performance parameters is less than 10%. Thus, the length of each phase was based on the stability of the bioreactor effluent pH and the COD removal.

Analytical procedures

The following parameters were monitored, using methods described in *Standard Methods* (1985): pH, total solids (TS), volatile solids (VS) and non-volatile solids (NVS). COD was determined colorimetrically on a DR 2000 spectrophotometer (Hach Co. Loveland, CO) using the method from *Standard Methods* (1985) and the appropiate chemicals from Merck Chemicals (E. Merck, Darmstadt).

Total volatile fatty acids (TVFA) and bicarbonate alkalinity were determined according to the five-point pH titration method of Moosbrugger et al. (1992). The biogas composition was determined using 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 Porapack Q

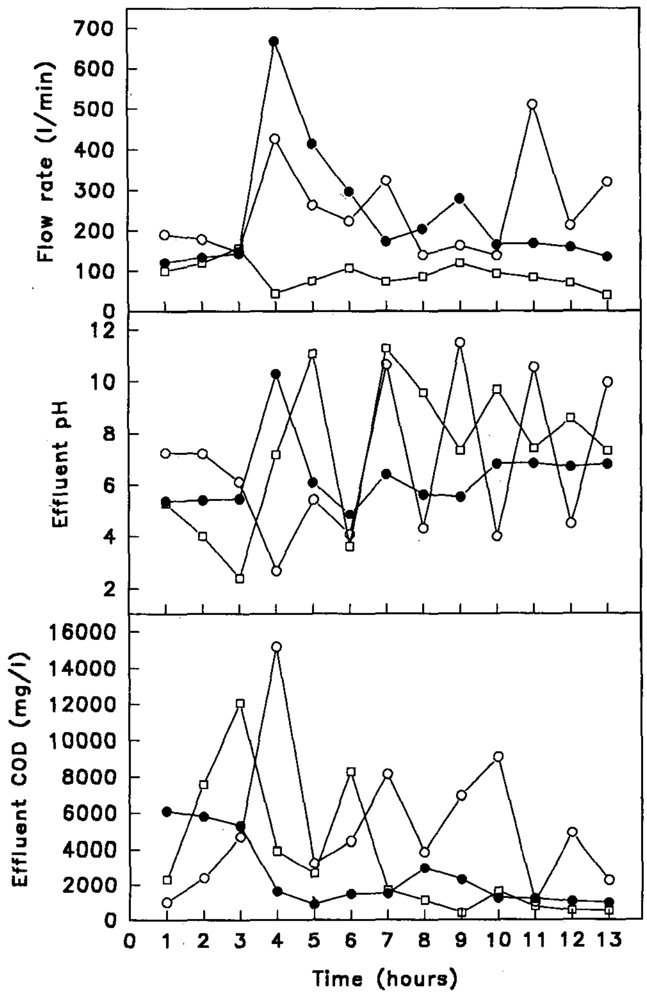

Figure 1
Effluent composition variations during the daily production cycles in terms of COD, pH and flow rate (O=cheese factory, □= fresh milk factory, ●= milk powder/butter factory).

TABLE 1 COMPARISON OF THE FLOW PROPORTIONED EFFLUENTS AND SPECIFIC WATER USE FROM THE THREE DIFFERENT DAIRY FACTORIES (ASSUMING A 22 WORKDAY MONTH)			
Parameter	**Cheese factory**	**Fresh milk factory**	**Milk powder/ Butter factory**
pH	5.22	6.92	5.80
COD concentration (mg·ℓ$^{-1}$)	5 340	4 656	1 908
Water usage (kℓ·d^{-1})	495	682	390
Milk intake (kℓ·d^{-1})	168	223	86*
Specific water usage (ℓ·ℓ$^{-1}$)	2.94	3.06	4.54

*Figures for butter factory are in tons milk and cream

TABLE 2 ACCURACY OF FLOW-PROPORTIONING OF THE THREE EFFLUENTS, IN TERMS OF COD PREDICTION			
Parameter	**Cheese factory**	**Fresh milk factory**	**Butter factory**
Predicted COD (mg·ℓ$^{-1}$)	5 390	4 735	1 872
Measured COD (mg·ℓ$^{-1}$)	5 340	4 656	1 908
Proportioning error (%)	-0.93	-1.67	+1.92

(Waters Ass. Inc, Milford, MA), 80 to 100 mesh. The oven temperature was set at 55°C and hydrogen was used as carrier gas at a flow rate of 40 mℓ·min^{-1}. The biogas volume was determined using an electronically controlled manometer and counter. Each unit on the gas counter corresponded to 13.158 ml biogas at ambient temperature and pressure. Barometer readings were also taken, and the methane yield was then calculated using the universal gas equation, to obtain methane yield values which are corrected for STP.

Results and discussion

Effluent production and composition

Effluent variability

The chemical composition and the flow rates during the daily production cycles of all three effluents were found to be highly variable over the sampling periods (Fig. 1). These variations were probably due to the different manufacturing processes conducted at different times during the various production cycles. The cleaning processes included tanker cleaning-in-place (CIP), CIP of various storage silos, pipelines, plate pasteurisers and evaporators. Cheese vats and factory floors are manually washed with hoses. In all three factories, CIP caustic and acid chemicals were recycled to a large extent while initial rinse water was discharged immediately. The exception was the butter factory, where butterfat was recovered from the initial rinse water.

From the data it was also clear that there was no correlation between COD, flow rate and pH (Fig. 1). This was probably a direct result of the four-step CIP process (Bogh-Sorenson, 1992; Romney, 1990). With this process in mind, COD peaks could be expected to coincide with the higher pH values (up to 11.8 units) obtained after the caustic wash, since the alkaline detergent is designed to remove the bulk of the milk-soil in the equipment. The COD peaks (up to 15 000 mg·ℓ$^{-1}$) which were found to coincide with low pH values (Fig. 1), were probably due to either the simultaneous release of an acid-cycle rinse elsewhere in the factory, or to the initial rinse of equipment which was used to manufacture yoghurt, bulk starter or other types of low pH, fermented milk products.

Effluent composition

Results from the analyses of the three flow-proportioned effluents are summarised in Table 1. The three effluents were found to vary considerably in character. Bearing in mind that between 75 and 95% of a dairy factory's initial water use (Table 1) ends up in the effluent (Strydom et al., 1993; Water Research Commission, 1989), water usage volumes will give a reliable indication of potential effluent volume trends. A notable feature is the correlation between the specific water consumption and the average effluent COD values. The data obtained also show that the milk powder/butter factory produced a fairly diluted effluent due to the much higher specific water consumption. It can be predicted that, should this factory's management establish tighter water-use controls, the effluent COD value would probably approach those of the other two factories.

The predicted COD values, as given in Table 2, were determined by multiplying each individual sample's COD value with its corresponding flow-proportioning factor, and then adding up all the COD values of the individual samples. The proportioning errors, as given in Table 2, show the experimental accuracy during the mixing of individual samples.

Effluent treatment

The overall results for the combined pre-acidification and anaerobic digestion treatment of all three effluents are presented in Tables 3 to 6. The "raw effluent" columns contain results of chemical analyses on the flow-proportioned composite effluents.

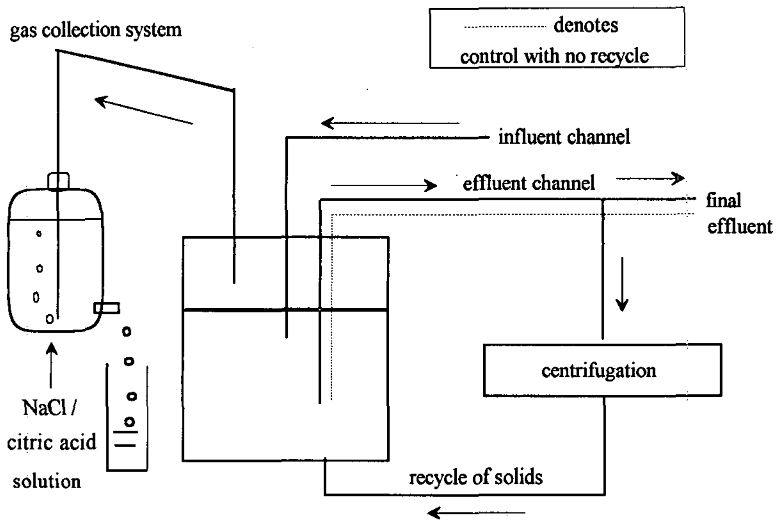

Figure 1
Diagrammatic representation of semi-continuous digester with and without recycle

a concentrated slurry (Bindoff et al., 1988).

Thus, CFMF, with a flexible woven hose, has been suggested as a process which may be used in conjunction with an anaerobic digester for the side-stream concentration and recycle of digester solids (Treffry-Goatley et al., 1986). Ouyang and Lin (1992) for example, reported that digesters operating with a recyle ratio of 0.5, and thus a recycle sludge concentration of about 28 g·ℓ^{-1} had higher dehydrogenase concentrations and methane yields. Parkin and Owen (1986) also recorded higher overall methane yields per unit organic matter destroyed but these necessitated protracted solids retention times equivalent to a working volume increase of 570%.

By increasing the mean solids retention time by sludge concentration or recycle, temperature and inefficient mixing perturbations should be reduced. Also, the optimum SRT may facilitate acclimation or metabolism of potential toxicants. Finally, organic material removal efficiency should also increase (Parkin and Owen, 1986). This study was, therefore, made to determine if the efficacy of anaerobic digestion of primary sludge could be improved in the presence of increased solids loadings effected by CFMF.

Experimental procedure

Digester configuration

As experimental tools, semi-continuous systems have the advantage of utilising actively growing and metabolising microbial cells to biodegrade substrates. These systems aim to operate within the exponential phase of bacterial growth thus avoiding the lag, stationary and autolytic phases. Semi-continuous digesters were, therefore, established and were operated simultaneously to determine the efficacy of digestion at different solids concentrations. The total volume of each digester was 2 ℓ, with a working or available volume of 1.5 ℓ, thus giving a headspace volume of about 500 mℓ. These digesters had no mechanical mixing device and were operated with a residence time of 30 d. The digesters were shaken daily during sampling and maintained in a waterbath at a constant temperature of 35°C. Each digester (Fig. 1) was connected to the gas collection system by silicone tubing. An effluent and influent port allowed for waste from the system to be removed and for the substrate (primary sludge) to be added daily, respectively.

Digester operation

Conventional digesters usually operate with solids concentrations of about 2 to 3% (m/v) TS, with 3% TS (m/v) the maximum solids concentration attainable. The four digesters were operated with 2% (Digester 1), 3% (Digester 2), 3.8% (Digester 3) and 4.7 % (m/v) (Digester 4) TS. Digester 1 was the control with no recycle of solids. To maintain concentrations of total solids greater than 2% (m/v), the effluent wasted per day was centrifuged and the solids recycled to the digester. The sludge was concentrated in a Beckman centrifuge at 10 000 r·min^{-1} for 25 min and the concentrate was used to prepare solutions of 3, 3.8 and 4.7% (m/v) TS.

Approximately 50 mℓ of sludge from Digester 1 were removed daily and replaced by 50 ml of primary sludge which contained 5% (m/v) total solids and 78 to 80% volatile solids. Care was taken to maintain a constant concentration. Approximately 150 mℓ of digested sludge were removed daily from Digesters 2, 3 and 4. A volume of 20 to 50 mℓ (depending on type of analysis) digested sludge was stored for analysis while the remainder was centrifuged. A total volume of 150 mℓ was added to the digester i.e., 50 mℓ substrate and 100 mℓ concentrated recycled sludge. Since Digesters 2, 3 and 4 were initially loaded with solids concentrations of 2% (m/v) TS, this procedure of removal and recycling was continued until the solids concentrations within the digesters reached a stable state of operation. Error bars are not drawn since the experimental error during sampling fell within the area of the markers used in Figs. 2 to 5.

Subsequently, the wasted sludge was subjected to various analyses such as volatile solids and total solids concentrations, volatile acid concentration, alkalinity and pH. Gas generated from waste stabilisation was bubbled through a vessel (2 ℓ) which contained a NaCl/citric acid solution (20% w/v NaCl acidified with 0.5% citric acid). This solution prevents CO_2 solubilisation and thus facilitates accurate measurement of the gas generated. The gas produced during anaerobic digestion was measured by displacement of the liquid solution in the gas collection vessel. The displaced liquid was transferred to a 100 mℓ measuring cyclinder and measured to the nearest 1 mℓ. With the exception of volatile acids and alkalinity, all of these analyses were conducted according to *Standard Methods* (1985).

Alkalinity

The digested sludge sample was centrifuged with a Beckman centrifuge at 5 000 r·min^{-1} for about 5 min. A 50 mℓ volume of supernatant was transferred to a beaker and the pH was determined with an Orion pH meter. The sample was then titrated to pH 4.0 with 0.1N H_2SO_4. The volume of acid titrated x100 gave the total alkalinity which was reported as mg $CaCO_3$·ℓ^{-1}. When the volume of supernatant was insufficient a smaller volume was diluted to give a 50 mℓ sample.

Volatile acid concentration

Once the alkalinity had been determined the pH of the solution was reduced to 3.5 with sulphuric acid (0.1N). The supernatant effluent.

The methane yield was 0.327 m^3 CH_4.$kg^{-1}COD_{removed}$ or 82.8% of the theoretical maximum for glucose, with a biogas methane content of 69.3%.

Powder milk/butter factory effluent treatment

The results of the combined effluent treatment for this factory's effluent are summarised in Tables 5 and 6. The pre-acidification of the milk powder/butter effluent, yielded an increase in TVFAs of 102%. However, the initial TVFA concentration of this effluent was so low (Table 5) that the pH was lowered to only 6.45 after 24 h (Fig. 2). Thus, the pre-acidification of this type of effluent is regarded as probably unnecessary, considering the results from the pre-acidification of the other effluents. The digestion of the pre-fermented effluent was successful, with the TVFA concentration reduced to zero.

The COD removal was 91% with an average final COD of 166 mg·ℓ^{-1}. This is coincidentally at the same level as that found for the digested cheese effluent.

The bicarbonate alkalinity of 553 mg·ℓ^{-1} (as $CaCO_3$) of this effluent was virtually unchanged by the digester treatment. The final pH of the effluent was 7.89, which was the highest final pH obtained during this study.

The biogas volume, and consequently the methane yield, was found to be low. The methane yield was 0.287 m^3 CH_4·$kg^{-1}COD_{removed}$. However, the biogas was of high quality, with a methane concentration in the biogas of 80.7%.

TABLE 5
PRE-ACIDIFICATION AND DIGESTER EFFICIENCY OBTAINED DURING THE TWO-STAGE ANAEROBIC DIGESTION OF THE BUTTER FACTORY EFFLUENT

Parameters	Raw effluent*	Pre-acidified effluent	Digested effluent
pH	5.80	9.00	7.89
COD (mg·ℓ^{-1})	1 908	1 836	166
Total solids (mg·ℓ^{-1})	1 720	2 060	1 450
Total volatile solids (mg·ℓ^{-1})	860	830	300
Non-volatile solids (mg·ℓ^{-1})	860	1 230	1 150
Bicarbonate alkalinity (mg·ℓ^{-1})	532	239	553
Total volatile fatty acids (mg·ℓ^{-1})**	137	278	0
Carbon dioxide (in biogas)	NA	ND	19.3%
Methane (in biogas)	NA	ND	80.7%
Methane yield (m^3·$kg^{-1}COD_{removed}$)	NA	ND	0.287

*Raw effluent = flow proportioned composite sample
**As acetic acid
ND = not determined
NA = not applicable

TABLE 6
COMPARISON OF RESULTS IN TERMS OF COD REMOVAL AND METHANE YIELD

Parameter	Cheese factory	Fresh milk factory	Powder milk/ Butter factory
OLR (kgCOD·m^{-3}·d^{-1})	2.82	2.44	0.97
COD removal (%)	97	94	91
Y_{CH4} (% of theoretical max.)	90.9	82.8	72.7

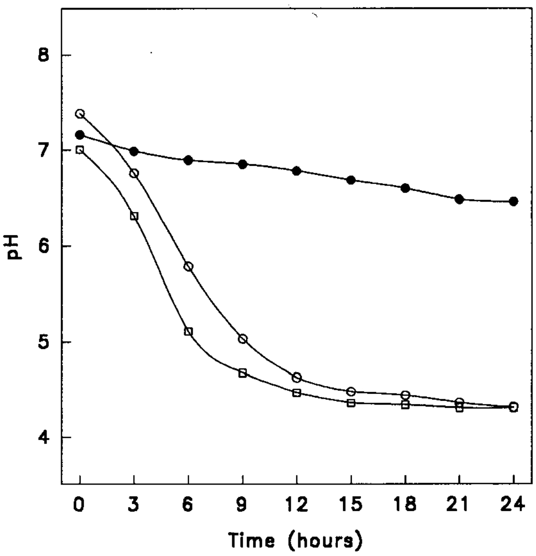

Figure 2
pH changes during the pre-acidification of the three raw effluents using the Klebsiella oxytoca *strain A1. Data are averages of duplicate runs (O = cheese factory, □= fresh milk factory, ●= milk powder/butter factory).*

Conclusions

The data from this study clearly show that dairy effluents are highly variable in two aspects. Firstly, average effluents from different types of factories differ with respect to the effluent chemical/physical analyses (Tables 1 and 2) and the effects the pre-acidification had on each effluent (Tables 3-5). Secondly, individual factories produce effluents which display wide fluctuations in pH, flow rate and COD over very short time intervals. These variations are unpredictable at any given large factory, due to the complexities of the CIP processes in the factory. Considering the high variability of dairy effluent quality over such short time intervals, balancing tanks might be necessary to ensure a stable substrate feed to any form of biological treatment system.

It is clear from the data obtained on the cheese, fresh milk and milk powder/butter waste waters that dairy effluents are suitable for treatment by means of the anaerobic digestion process and the use of a hybrid anaerobic bioreactor was seen to be a viable treatment option. The COD values of the three anaerobically digested dairy waste waters were reduced by between 91 and 97% at organic loading rates of between 0.97 and 2.82 kgCOD·m^{-3}·d^{-1}

and subsequent methane yields from 0.287 to 0.359 m^3 $CH_4.kg^{-1}$ $COD_{removed}$ were achieved. The pH values of the digested effluents were >7.5 units, making them acceptable as sources 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 dairy effluents makes them easily fermentable to organic acids with the subsequent drastic drop in pH and failure of the digestion process. Thus in the cases of dairy effluents, pre-acidification is necessary for the successful high-rate anaerobic treatment of these types of effluents. In this study the pre-acidification step of the effluents from the cheese and the fresh milk factories, using the particular strain of *Klebsiella oxytoca* to convert the lactose in the effluents to organic acids, was successful. However, the pre-acidification step of the milk powder/butter factory effluent in terms of the production of precursors for the methanogens, was not as successful. This is attributed to the lack of fermentable sugars in the milk powder/butter factory effluent. The presence of small amounts of whey and milk in the effluents explains the success of the pre-acidification step in the case of the cheese and the fresh milk factories. This also underlines the fact that each type of dairy factory produces a relatively unique effluent. An important consequence of these data is that a two-phase set-up is required to protect the methanogenic population in the digester system from too low pH values and high concentrations of the volatile fatty acids produced during the fermentation of the easily degradable organic compounds in the raw effluent. 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-acidification step will have to be reduced. The effect of this reduction on the alkalinity and pH stability of the bioreactor must still be evaluated. Another option is to make use of recirculation of the bioreactor effluent, in order to utilise the high alkalinity in the bioreactor effluent.

The implications from the findings in this study are that although required for cheese/milk factory effluents, the pre-acidification step might be unnecessary for the milk powder/butter factory effluent. Where a full-scale treatment facility is concerned, this may result in substantial saving in capital expenditure. Further research will have to include assessing the necessity of pre-acidification for each type of effluent, while it was seen that some factories have an excessively high specific water use. Proper water management in a dairy factory may reduce the water use, and under certain circumstances this may lead to an increase in the effluent COD concentration. This must be taken into consideration when a factory contemplates the installation of an effluent treatment facility. A higher COD concentration will lead to an increase in the organic loading rate of the bioreactor and thus, in the biogas production. The feasibility of the anaerobic treatment of dairy effluents is positively influenced by increased biogas yield and simultaneous reduction in COD concentration. The biogas can be utilised to supplement the use of coal as energy source for the generation of steam. Furthermore, the larger reduction in total COD concentration may result in a reduction in effluent disposal expenditure when the COD concentration and pH value of the bioreactor effluent is used as basis for the calculation of trade effluent tariffs. Therefore, laboratory-scale evaluation and subsequent optimisation of the specific water use and total effluent situation must be conducted, before any dairy industry contemplates the treatment of its effluent.

Acknowledgements

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