

# Lauter tun (brewery) waste in UASB systems - Feasibility, alkalinity requirements and pH control

RE Moosbrugger, MC Wentzel\*, GA Ekama and GvR Marais

Department of Civil Engineering, University of Cape Town, Rondebosch 7700, South Africa

## Abstract

Lauter tun (brewery) waste developed a pelletised sludge bed in a laboratory UASB reactor. Product formation along the line of flow in the bed was similar to that when treating a pure carbohydrate, apple juice waste. Virtually no  $\text{H}_2\text{CO}_3^*$  alkalinity was present in the waste or generated by the process; the  $\text{H}_2\text{CO}_3^*$  alkalinity required to buffer the minimum bed pH to 6,6 had to be supplied from an external source. On a flow-through UASB reactor with a base influent COD ( $\text{COD}_b$ ) = 4 000 mg/l, the  $\text{H}_2\text{CO}_3^*$  alkalinity requirement was 0,9 mg as  $\text{CaCO}_3$  per mg  $\text{COD}_b$ . By imposing a recycle from the effluent to the influent, the  $\text{H}_2\text{CO}_3^*$  alkalinity required per mg  $\text{COD}_b$  was reduced substantially.  $\text{H}_2\text{CO}_3^*$  alkalinity (in the form of NaOH) was added to the recycle stream, not to the base influent flow. When added to the base influent flow the pH increased to 11 and, apparently, trace elements precipitated leading to partial failure of the process. When added to the recycle stream (recycle ratio 15:1) the pH downstream did not rise above 8,5 and the process operated satisfactorily. In a UASB system with a recycle, the  $\text{H}_2\text{CO}_3^*$  alkalinity to be supplemented to maintain a near neutral minimum bed pH was assessed using the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity as reference parameter, not the influent  $\text{H}_2\text{CO}_3^*$  alkalinity, to take into account any internally generated  $\text{H}_2\text{CO}_3^*$  alkalinity. For a  $\text{COD}_b$  = 13 000 mg/l and a practical COD loading rate of 9 kg/(m<sup>3</sup> sludge bed-d), a recycle ratio as high as 22:1 reduced  $\text{COD}_b$  to an effective influent COD of 570 mg/l and did not significantly influence COD reduction (> 90 per cent) or effluent SCFA (about 50 mg/l as acetic acid).

## Introduction

In reviewing parameters that influence anaerobic treatment systems, Moosbrugger et al. (1993a) noted that pH is one of the principal ones: for optimal treatment the reactor pH > 6,6. In anaerobic systems, "buffering agents" resist a decline in pH. In systems operating "normally", the carbonate weak acid/base is the dominant buffering agent; its buffering ("ability" to resist pH change) at any pH can be assessed via the parameter  $\text{H}_2\text{CO}_3^*$  alkalinity (Loewenthal et al., 1989).

The  $\text{H}_2\text{CO}_3^*$  alkalinity requirements to control the minimum reactor pH in upflow anaerobic sludge bed (UASB) systems differs for different types of substrates. Substrates can be divided into two basic categories:

- substrates that do not generate internal buffer and hence depend completely on buffer from an external source to control the minimum pH in the reactor; and
- substrates that generate internal buffer from, for example, influents containing proteins (deamination), sulphates (reduction to sulphides) and/or short-chain fatty acids (acetoclastic methanogenesis). These may cause that the anaerobic process becomes partially or completely independent of buffer from an external source for pH control.

This paper presents a study of the behaviour of a waste of the first category, i.e. a waste producing very little internal buffer, brewery lauter tun waste. In a further paper the behaviour of a waste in the second category will be presented.

In a brewery plant, there are a number of different waste streams generated from the bottling hall, cleaning of fermentation tanks, filter unit, wort kettle and lauter tun. Some of these waste streams, e.g. the wash water from the bottling hall, may contain substances inhibitory or toxic to anaerobic micro-organisms. In the particular brewery from which the waste batches were

obtained, all the waste streams discharge to a central holding tank and from there, controlled discharge to a sewer. Of all the waste streams, that from the lauter tun contributes the main mass of COD generated in the brewing process. Lauter tun waste has a high carbohydrate content so that it has potential for treatment in a UASB system. Furthermore, in the particular brewery the lauter tun waste stream could be separated easily from the other waste streams, thereby avoiding the risk of interference from potentially inhibitory or toxic chemicals. Accordingly, the lauter tun waste stream was selected for study. The study was subdivided into two parts:

- Feasibility study of the treatment of this waste in a laboratory-scale UASB system, to ascertain formation of a pelletised sludge bed and to study the product formation pattern along the line of flow of the reactor.
- Investigation into the effects of recycling on system performance and mass of  $\text{H}_2\text{CO}_3^*$  alkalinity required to maintain a near neutral minimum pH in the lower part of the sludge bed.

## Feasibility study

### Experimental set-up

A laboratory-scale UASB reactor was constructed from a transparent perspex cylinder of 94 mm diameter, 900 mm high, with a conically shaped inlet at the bottom and a solid/liquid/gas separator at the top, total reactor volume ca. 6,5 l (Fig. 1). The substrate was fed by means of a constant speed multi-channel peristaltic pump, the feed rate being controlled by an on/off timer. Gas collection was by means of a hollow inverted cone: Rising gas bubbles are deflected into the cone by a deflector collar around the inside wall of the reactor below the cone; liquid effluent discharges via an annular space between the gas collection cone and the reactor wall, to enter a small liquid/solid separator (1 000 ml); clarified liquid flows over a launder into the collection vessel while solids settle out and return into the reactor by gravity. Ten sample ports were installed evenly spaced up the

\*To whom all correspondence should be addressed.

Received 5 February 1992; accepted in revised form 16 July 1992.

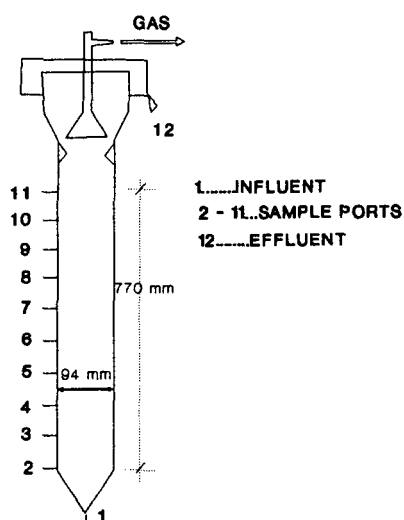


Figure 1

Schematic diagram of laboratory UASB reactor

reactor wall for sampling along the line of flow. Temperature was maintained at 30° C by a thermostat-controlled electrical heating tape wrapped around the reactor.

#### Waste-water characteristics

The influent feed stock (lauter tun waste) was collected from the brewery in batches and stored at 4°C. The waste batches contained different low concentrations of particulate material; to eliminate this variable the waste was settled and the supernatant only used as feed to the UASB reactor. The characteristics of the supernatant of the batch used in the feasibility study are shown in Table 1. The settled supernatant contained principally carbohydrates; tests for short-chain fatty acids (SCFA; also termed total volatile fatty acids, TVFA) showed that these were present only in minor concentrations, less than 100 mg/l as HAc.

Each day the feed to the reactor was made up from the settled feed stock by diluting to a COD of 4 000 mg/l using tap water. The daily feed was kept refrigerated at 4° C. The feed composition after dilution is given in Table 2.

#### Assessment of feed for treatment in a UASB system

The feed characteristics in Table 2 were assessed against the prerequisites set out by Sam-Soon et al. (1987) for suitability as a substrate to generate a pelletised sludge in the UASB system, i.e. (1) develop a high hydrogen partial pressure ( $pH_2$ ) environment; (2) cysteine deficiency; (3) excess supply of nitrogen; and (4) a near neutral minimum bed pH.

With regard to (1), the high  $pH_2$  environment, the lauter tun waste contains a high fraction of carbohydrates that should yield hydrogen under high  $pH_2$  conditions when undergoing acidogenesis in the lower part of the sludge bed.

With regard to (2), cysteine deficiency, no judgement was possible; it was presumed that there would be a cysteine deficiency.

With regard to (3), excess ammonium supply, Sam-Soon et al. (1990) suggested a minimum of 0,02 mg N/mg influent COD for

TABLE 1  
CHARACTERISTICS OF SETTLED LAUTER TUN WASTE  
(BATCH 0)

Soluble COD fraction	98%
Soluble COD concentration	12 000 mg/l
SCFA (as HAc)	< 100 mg/l
Inorg-N (as N)	≈ 0 mg/l
TKN (as N)	136 mg/l
Phosphorus (as P)	10 mg/l
pH	5,2

TABLE 2  
CHARACTERISTICS OF DILUTED SETTLED LAUTER TUN  
WASTE

Soluble COD	4 000 mg/l
SCFA (as HAc)	< 30 mg/l
Inorg-N (as N)	≈ 0 mg/l
TKN (as N)	45 mg/l
Phosphorus (as P)	3 mg/l
pH	6,0

TABLE 3  
MACRO-NUTRIENT AND TRACE ELEMENT (AFTER  
ZEHNDER AND WUHRMANN, 1977) STOCK SOLUTIONS

Macro-nutrients	Concentration g/l
NH <sub>4</sub> Cl	10,00
K <sub>2</sub> HPO	4,00
Trace elements	Concentration g/l
H <sub>3</sub> BO <sub>3</sub>	0,05
FeCl <sub>2</sub> ·2H <sub>2</sub> O	2,00
ZnCl <sub>2</sub>	0,05
MnSO <sub>4</sub>	0,5
CuCl <sub>2</sub> ·2H <sub>2</sub> O	0,03
(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O	0,05
AlCl <sub>3</sub> ·6H <sub>2</sub> O	0,05
CoCl <sub>2</sub> ·6H <sub>2</sub> O	2,00
MnCl <sub>2</sub>	0,25
MgCl <sub>2</sub>	1,00
EDTA	0,05
Ni Cl <sub>2</sub> ·6H <sub>2</sub> O	0,25
HCl	1 ml

optimum pellet production. The settled and diluted lautur tun waste contained virtually no ammonium but contained organic nitrogen of about 45 mg N/l, giving 0,011 mg N/mg influent COD. There was no certainty as to whether the organic nitrogen would be deaminated fast enough to satisfy or partially satisfy the nitrogen requirement for pelletisation in the lower part of the reactor. Besides nitrogen, the other macro-nutrient, phosphorus, also appeared to be in short supply, COD:P = 350:0,3; whereas an adequate COD:P ratio should be near to 350:1. To supplement the macro-nutrients in the influent a stock solution containing nitrogen and phosphorus was made up as shown in Table 3. Addition of 20 ml of the macro-nutrient solution per litre of diluted lautur tun waste increased the COD:P ratio to 350:1 and the TKN/COD ratio to 0,024 mg N/mg COD.

With regard to (4), a near neutral minimum bed pH, the influent waste flow had no or virtually no H<sub>2</sub>CO<sub>3</sub>\* alkalinity as indicated by the pH of 6,0, and would generate only a small

amount of  $\text{H}_2\text{CO}_3^*$  alkalinity from deamination of the organic nitrogen (abstracting  $\text{H}^+$  and generating  $\text{OH}^-$ ) - addition of  $\text{H}_2\text{CO}_3^*$  alkalinity would be required. For external alkalinity provision, NaOH was selected. In the process, the  $\text{OH}^-$  ions (of NaOH) react with the  $\text{CO}_2$  ( $\text{H}_2\text{CO}_3^*$ ) generated to form principally  $\text{HCO}_3^-$  and  $\text{CO}_3^{2-}$  species depending on the pH that develops. From preliminary tests, with NaOH dosing in the influent for an  $\text{H}_2\text{CO}_3^*$  alkalinity addition of 1,2 mg/mg influent COD (as suggested by Sam-Soon et al., 1991 for a pure carbohydrate substrate), the pH in the feed rose to > 12. There was uncertainty as to the pH level that would be established in lower part of the sludge bed with  $\text{OH}^-$  ions as sole alkalinity source because this would depend on the rate at which  $\text{OH}^-$  ions react with  $\text{CO}_2$  generated in the bed. Accordingly, for the feasibility study, NaOH was used in combination with  $\text{NaHCO}_3$  in the following proportion:  $\text{NaOH} = 875 \text{ mg/l}$  as  $\text{CaCO}_3$  and  $\text{NaHCO}_3 = 2\,680 \text{ mg/l}$  as  $\text{CaCO}_3$ , giving a total supplementation of  $\text{H}_2\text{CO}_3^*$  alkalinity =  $3\,555 \text{ mg/l}$  as  $\text{CaCO}_3$ . After alkalinity supplementation the pH in the feed stabilised at 9,1. The ratio of (influent alkalinity concentration)/(influent COD concentration) was now  $\text{Alk/COD} = 3\,555/4\,000 \approx 0,9$ . This value was lower than the minimum ratio of 1,2 suggested by Sam-Soon et al. (1991) to control the minimum bed pH to > 6,6. The reasons for choosing a lower Alk/COD ratio were twofold:

- In this study a different substrate was investigated, i.e not entirely made up of carbohydrates; and
- the COD loading rate on the sludge bed at the beginning of the feasibility study was low in which event, from the data obtained by Sam-Soon et al. (1991), the pH in the lower part of the sludge bed should not decline as much as at a higher COD loading rate.

If at any stage the alkalinity addition per COD should prove to be inadequate, it could be increased. However, when the COD loading rate was increased substantially the Alk/COD ratio of 0,9 still proved to be adequate.

#### Trace element solution

To avoid potential deficiencies of trace element supply, a trace element solution (5 m/l influent), suggested by Zehnder and Wuhrmann (1977) for enrichment cultures of methanogenic bacteria, was added to the feed. Detailed composition of this trace element solution is given in Table 3.

#### Parameters measured

The following analyses and measurements were performed on the system at 1 to 2 d intervals:

- filtered influent and effluent COD;
- filtered influent and effluent TKN and inorg-N;
- pH in settler and sample ports 2 and 3, the sample ports likely to have the lowest pH; and,
- substrate flow rate.

In addition to the monitoring programme above, profiles of pH, COD, TKN, inorg-N and the SCFA propionic (HPr) and acetic (HAc) were measured along the axis of the reactor at the highest loading rates applied, with the system still showing stable steady state response. Samples were taken at each sample port, starting at the top. Samples were filtered using ordinary filter paper,

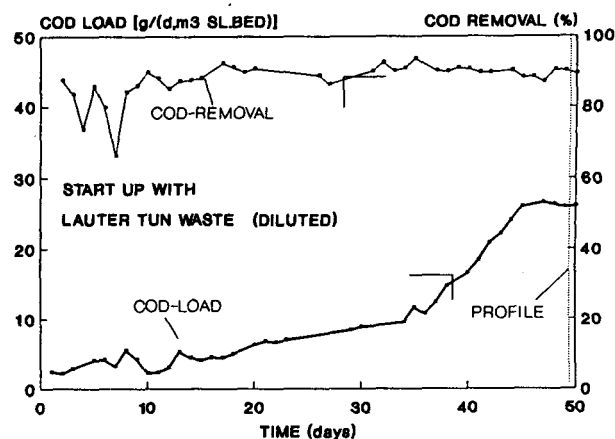


Figure 2

Start-up period of flow-through laboratory UASB reactor seeded with 4 l of pelletised sludge from a laboratory-scale UASB system treating wine distillery waste. The reactor was fed with diluted lauter tun waste (using tap water); the diluted influent COD (4 000 mg/l) was kept constant during the start-up period, i.e. the COD loading rate was increased by increasing the flow rate. The term "profile" indicates the time location for measuring various parameters along the line of flow of the reactor (see Fig. 3)

Schleicher und Schuell 595. For HAc and HPr, samples were refiltered through a 0,45  $\mu\text{m}$  filter paper (Millipore) and the concentrations measured by gas chromatography using a 60/80 Carbo pack C/0,3% Carbo wax packing (for details, see Sam-Soon et al., 1989). COD, TKN and inorg-N were measured in accordance with *Standard Methods* (1989). For the feasibility study no measurements of  $\text{H}_2\text{CO}_3^*$  alkalinity and SCFA by the 5 pH point titration method (Moosbrugger et al., 1993b) were undertaken, nor was gas production monitored.

#### Starting-up procedure

For the feasibility study, the system was operated as a flow-through system. Sam-Soon et al. (1987), operating a flow-through system found that if the influent COD concentration is in the range 1 000 to 5 000 mg/l and the load less than the maximum, the process should exhibit stable response and clearly distinguished product zones. Accordingly, for the lauter tun waste an influent COD concentration of 4 000 mg/l was selected.

The reactor was seeded with 4 l of pelletised sludge (up to Port 8) from a laboratory-scale UASB reactor treating wine distillery waste, to give a bed depth of 580 mm. The sludge bed volume was kept constant to Port 8 by draining excess sludge via sample Port 7. The influent COD concentration was kept constant throughout the starting-up period, the COD loading rate being increased by increasing the flow rate. The rate of increase in COD loading was controlled by monitoring the effluent COD to ensure that the percentage COD removal did not decline substantially when the COD loading rate was increased, and the minimum bed pH (occurring at Port 2 or 3), to ensure that this did not decline below 6,6.

In Fig. 2 the COD loading rate is plotted versus time, together with the percentage COD removal. The start-up period can be divided into two, an initial period of adaptation of the pelletised

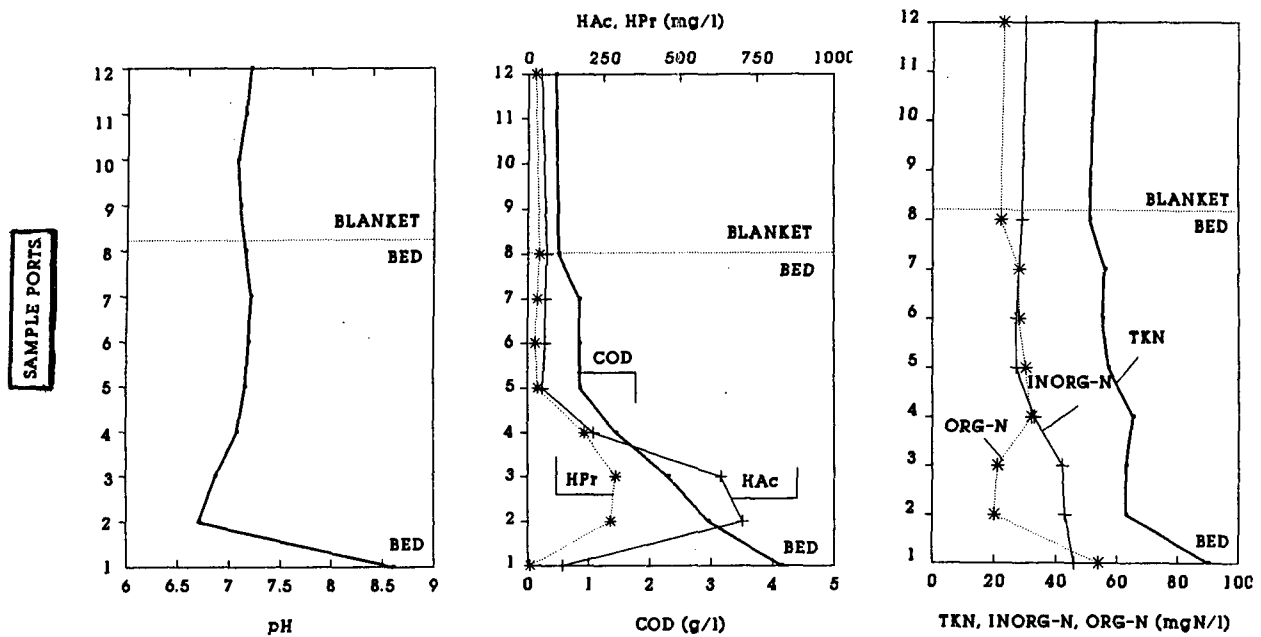


Figure 3

Flow-through laboratory UASB reactor treating diluted lauter tun waste: Profiles of pH, COD, HAC, HPr, TKN, inorg-N and org-N along the line of flow. Flow rate: 25  $\text{d/d}$ , influent COD concentration: 4 000  $\text{mg/l}$ , COD loading rate: 25  $\text{kgCOD}/(\text{m}^3 \text{ sludge bed-d})$

sludge to the new substrate with a slow sequential increase in COD loading rate (about 35 d), and a second period of rapid COD loading increase (about 10 d).

During the first 10 d (Fig. 2), caution had to be exercised in increasing the COD loading rate. The COD loading rate had to be kept below 5  $\text{kg COD}/(\text{m}^3 \text{ sludge bed-d})$  otherwise the minimum pH in the bed fell sharply. Thereafter, the loading rate could be gradually increased, to about 10  $\text{kg COD}/(\text{m}^3 \text{ sludge bed-d})$  by day 35; concomitantly the stability of the process improved. After day 35, over a period of 10 d, a rapid increase in COD loading rate from 10 to 25  $\text{kg COD}/(\text{m}^3 \text{ sludge bed-d})$  was readily accepted by the system, the COD removal remaining greater than 90 per cent indicating that the sludge bed had adapted to the new substrate. A COD loading rate > 25  $\text{kg COD}/\text{m}^3 \text{ sludge bed-d}$  was not envisaged - it was noted that at this loading rate, on occasion gas collected at random levels in the sludge bed to lift the sludge mass above up into the settler; the pellets appeared to be relatively lighter than those obtained by Sam-Soon et al. (1987) when treating apple juice at about the same COD loading rate. This physical behaviour forced the decision not to impose higher loading rates (If a larger diameter reactor had been used, very likely the collection of gas in the sludge would not have occurred).

### Steady state behaviour

After operating the system, apparently stable, at its maximum imposed loading rate of 25  $\text{kg}/(\text{m}^3 \text{ sludge bed-d})$  for 4 d, on day 49 profiles of pH, COD, TKN, inorg-N, HAC and HPr were measured (Fig. 3). Assessment of the profiles is facilitated by comparison with the findings of Sam-Soon et al. (1987) on product profiles in a UASB system treating a carbohydrate waste:

Sam-Soon et al. (1987) in their studies of flow-through UASB systems suggested measurement of profiles of pH, COD, TKN, inorg-N, HAC and HPr along the line of flow of the reactor, to identify biochemical processes taking place at different levels in

the sludge bed. The profiles of the various products, in particular TKN, inorg-N, HAC and HPr provided information from which Sam-Soon et al. (1987) developed their hypothesis on pelletisation. They identified three zones of product formation in the bed along the reactor, designated as (1) lower active, (2) upper active, and (3) an upper inactive zones. The lower active zone was characterised by a significant decrease in pH, an unexpectedly high uptake of inorg-N, accumulation of HAC and HPr and rapid decrease in soluble COD. The upper limit of the lower active zone was taken where the HPr attained its maximum value. In the upper active zone the pH increased, the concentration of HAC and HPr decreased to near zero and the COD decreased further to a stable minimum value. The upper limit of this zone was taken where the rate of change in SCFA concentration decreased to near zero. In the upper inactive zone virtually no changes in product concentrations took place.

An analysis of the product formation in these zones led to the following: In the lower active zone the accumulation of HPr indicates a high  $\text{pH}_2$  environment and from the uptake of inorg-N (and information in the literature), Sam-Soon et al. (1987) hypothesised that the high  $\text{pH}_2$  induces the production of amino acids in cells of *Methanobrevibacter arboriphilicus* but protein formation is limited by the external supply of the amino acid cysteine. The excess amino acids produced are extruded as a biopolymer (polypeptide). This biopolymer forms the basic matrix for pellet formation capturing both *Methanobrevibacter arboriphilicus* and other anaerobic micro-organisms.

A comparison of the profiles on the lauter tun waste, with those observed by Sam-Soon et al. (1987), indicated the following:

**HAc and HPr profiles:** The HAC and HPr concentrations reached their maxima between Ports 2 and 3 defining the upper limit of the lower active zone. HPr and HAC thereafter declined rapidly up to Port 5 defining the limit of the upper active zone. Above Port 5, HAC and HPr remained virtually constant up to the top of the bed at Port 8, defining the inactive zone. According to Sam-Soon et al. (1987), HPr accumulation would occur only in

a high  $pH_2$  environment. Thus the observed accumulation of HPr indicates that the prerequisite of high  $pH_2$  was satisfied when treating lauter tun waste. Both the HAC and HPr profiles reached stable minimum values well within the sludge bed indicating that the further increase in COD load was possible.

**pH profile:** Within the lower active zone the pH declined from 8,6 to about 6,7; thereafter the pH increased to a stable value of 7,2 in the upper active and inactive zones, sludge blanket and settler. The decline in pH coincided with the increases in HAC and HPr. Comparing the pH profile with those by Sam-Soon et al. (1987), a similar pattern is apparent.

**COD profile:** The COD decreased at a high rate from 4 100 mg/l to 900 mg/l up to sample Port 5. Above this level the rate of COD removal reduced and the COD concentration stabilised at about 400 mg/l at the top of the sludge bed and remained unchanged in the effluent. The more rapid rate of COD removal in the lower part of the sludge bed is similar to that observed by Sam-Soon et al. (1987).

**TKN, inorg-N and org-N profiles:** The org-N profile showed a decline of org-N in the lower part of the sludge bed, due to deamination, followed by a slight increase in the upper part of the sludge bed probably due to pellet break-up. With regard to the inorg-N profile, Sam-Soon et al. (1987) observed a marked decrease of inorg-N in the lower part of sludge bed. In the diluted lauter tun waste profile, however, inorg-N profile showed little change; this may be attributed to the simultaneous generation of inorg-N (due to deamination of the org-N present in the influent) and uptake of the generated inorg-N due to pelletisation. The uptake of nitrogen is more effectively demonstrated in the TKN profile - a significant decrease of 28 mg N/l (from 90 to 62 mg N/l) occurred below sample Port 2. In this part of the sludge bed about 1 200 mg COD/l were removed, giving a (N removed)/(COD removed) ratio of 0,023. Under "normal" anaerobic fermentation conditions, nitrogen requirements would be approximately 0,004 mg N/mg COD for protoplasm synthesis. Thus, the observed nitrogen removal was about six times higher than that normally expected, supporting the hypothesis that pellet formation was taking place.

**Pellet bed growth:** The pelletised sludge bed increased in volume and the excess pelletised sludge was removed; however, quantitative measurements were not undertaken as the objective in the feasibility study was limited to a qualitative assessment of pelletisation only.

## Conclusions

The information gathered during the feasibility study can be summarised as follows:

- The pH, COD, HAC and HPr profiles were similar to those observed on a carbohydrate substrate.
- The pelletised bed mass increased.
- The rate of nitrogen uptake by the pelletised sludge was about six times that expected in a "normal" anaerobic system.

These observations were similar to those observed by Sam-Soon et al. (1987) for a carbohydrate substrate. It was concluded that lauter tun waste has characteristics that makes it suitable for treatment in a pelletised UASB system.

## Recycling and alkalinity requirements

The feasibility study indicated that lauter tun waste in a flow-through UASB system (no recycle) would produce a pelletised sludge bed and that, at a COD loading rate of 25 kg/(m<sup>3</sup> sludge bed·d), an influent H<sub>2</sub>CO<sub>3</sub>\* alkalinity/COD of 0,9 would be adequate to sustain a minimum bed pH above 6,7. However, in a full-scale UASB plant addition of H<sub>2</sub>CO<sub>3</sub>\* alkalinity of this magnitude would make the treatment of lauter tun waste, or similar wastes, too expensive. Sam-Soon et al. (1991), when treating apple juice wastes in a UASB system, also found uneconomically high alkalinity requirements of approximately 1,2 mg of alkalinity (as CaCO<sub>3</sub>)/mg feed influent COD. Accordingly, they introduced a recycle from the effluent to the influent, to reduce the H<sub>2</sub>CO<sub>3</sub>\* alkalinity requirements by recovery of the H<sub>2</sub>CO<sub>3</sub>\* alkalinity from the reactor effluent. With the recycle they found that the base H<sub>2</sub>CO<sub>3</sub>\* alkalinity requirements (i.e. alkalinity requirements without recycle) could be reduced significantly. Sam-Soon et al. (1991) formulated the reduced H<sub>2</sub>CO<sub>3</sub>\* alkalinity requirements per litre base influent flow, when imposing a recycle, as follows:

$$\text{Alk/l influent} = C \cdot \text{COD}_b \cdot \frac{Q}{Q+Q_r} = C \cdot \text{COD}_b \cdot \frac{1}{1+r} \quad (1)$$

where:

- Alk/l influent = H<sub>2</sub>CO<sub>3</sub>\* alkalinity required in base influent flow
- C = mg H<sub>2</sub>CO<sub>3</sub>\* alkalinity required per mg base influent COD without recycle
- Q = base influent flow, l/d
- Q<sub>r</sub> = recycle flow, l/d
- r = recycle ratio, Q<sub>r</sub>/Q
- COD<sub>b</sub> = COD concentration in undiluted influent flow, mg/l

Sam-Soon et al. (1991) assumed that C would remain substantially constant, independent of the recycle ratio r, at C ~ 1,2. The factor C was established for base influent COD concentrations ranging from 2 500 to 8 500 mg/l and recycle ratios up to 3:1. In selecting the magnitude of the recycle ratio, Sam-Soon et al. (1991) recommended that a ratio be used that dilutes the base influent COD concentration into a range of 1 000 to 5 000 mg/l because they found that the process exhibited improved stability within this influent concentration range.

The influent COD of the undiluted lauter tun waste ranged from 9 000 to 13 000 mg/l, significantly higher than the concentrations examined by Sam-Soon et al. (1991). With the higher influent COD, a recycle was introduced to dilute the influent into the COD range suggested by Sam-Soon et al. (1991), and reduce the alkalinity requirements. In practice, it may arise that in order to reduce the alkalinity requirements to an economically acceptable range, a recycle ratio of such magnitude would be needed that the influent COD would be diluted below the lower limit of the range 1 000 to 5 000 mg/l suggested by Sam-Soon et al. (1991). It was of interest therefore, also to enquire if stable operation could be achieved with influent COD diluted by means of a recycle to less than 1 000 mg/l.

In formulating an approach for the investigation, the following aspects required consideration:

- In UASB systems operating at their maximum COD loading

rates the systems' behaviour can be expected to deviate from that under lower COD loading rates, particularly so with regard to  $H_2CO_3^*$  alkalinity requirements. Of practical importance would be the  $H_2CO_3^*$  alkalinity requirements under stable operating conditions, i.e. lower (practical) COD loading rates. From the feasibility study, the maximum COD load under which the process did not exhibit overloaded conditions explicitly, was 25 kg/(m<sup>3</sup> sludge bed-d); a practical loading rate would range between a third and half of the maximum, from 9 to 12 kg COD/(m<sup>3</sup> sludge bed-d).

- In the feasibility study, the lauter tun base COD ( $COD_b$ ) was reduced to 4 000 mg COD/ℓ by prior dilution (no recycle); in the present study the base COD was 9 000 to 13 000 mg/ℓ, but dilution to any selected effective influent COD would be achieved by a recycle. The effective influent COD concentration is defined as follows:

$$\text{Effective influent COD } (COD_e) = \frac{COD_b}{(1+r)} \quad (\text{mg}/\ell) \quad (2)$$

That is, the effective influent COD is the influent COD after dilution by the recycle neglecting the COD in the recycle stream.

- In the feasibility study the  $H_2CO_3^*$  alkalinity requirements were obtained for a minimum pH of 6,7 in the lower part of the sludge bed and the pH buffer was a combination of  $NaHCO_3$  and NaOH. From a practical point of view, a minimum bed pH higher than 6,7 should be aimed at, to guard against pH changes due to shock loads etc; a minimum sludge bed pH of say 7. The buffer agent used should be NaOH;  $Ca(OH)_2$  should not be used because there is substantive evidence that under the high  $CO_2$  partial pressures  $Ca(OH)_2$  addition is only partially effective as a high fraction of the  $Ca(OH)_2$  added is precipitated as  $CaCO_3$  even at pH < 7 (Capri and Marais, 1975). In a UASB system, it is not yet known to what degree this precipitation may affect pellet formation and pellet performance. To eliminate this uncertainty it was decided to use NaOH to control alkalinity and pH.

The study, when instituting a recycle, is reported in two parts; start-up and alkalinity requirements.

## Start-up

### First start-up with recycle

The experimental set-up remained the same as that in the feasibility study, except for the following modifications: a wet gas meter (model No. DM3A, Alexander Wright, London) was installed to measure the volume of gas produced in the reactor; the multi-channel peristaltic pump, formerly used as feed pump was installed as recycle pump; and a laboratory positive displacement pump was installed as feed pump. To control the flows both pumps operated on an on/off cycle, switched on and off simultaneously. The recycle stream was taken from the settled effluent (Port 12, Fig. 1) so that no pelletised sludge was recycled. The recycle stream joined the influent feed flow at a point just upstream of the point of discharge to the reactor (Port 1, Fig. 1).

For the starting-up period a batch of lauter tun waste was collected and stored at 4° C. The waste settled in the storage drum and the supernatant only served as feed to the UASB

**TABLE 4: CHARACTERISTICS OF SETTLED LAUTER TUN WASTE (BATCH 1 AND 2)**

<b>Batch 1:</b>	
Soluble COD fraction	98 %
Soluble COD concentration	9 000 mg/ℓ
Inorg-N	≈ 0 mg N/ℓ
TKN	77 mg N/ℓ
Phosphorus	8 mg P/ℓ
pH	5,6
<b>Batch 2:</b>	
Soluble COD fraction	98 %
Soluble COD concentration	13 000 mg/ℓ
Inorg-N	≈ 0 mg N/ℓ
TKN	144 mg N/ℓ
Phosphorus	11 mg P/ℓ
pH	5,3

reactor. The characteristics of the batch are listed in Table 4, batch 1.

Due to difficulties observed under high COD loading rates when gas accumulated in the bed, mentioned in the feasibility study, it was decided to reduce the bed volume to 3 ℓ, i.e. the sludge bed extended up to sample Port 6 (Fig. 1) giving a bed depth of 400 mm. The reactor was seeded with 3 ℓ of pelletised sludge which had been generated during the feasibility study. The influent feed was from batch 1 (COD = 9 000 mg/ℓ). The COD loading rate was 12 kg COD/(m<sup>3</sup> sludge bed-d), about half the maximum rate in the feasibility study. The waste needed to be supplemented with a buffering agent to maintain a near neutral minimum pH in the lower part of the bed. The source of  $H_2CO_3^*$  alkalinity was changed from (NaOH +  $NaHCO_3$ ) in the feasibility study to NaOH only. From Sam-Soon et al. (1991), alkalinity supplementation would be 1,2 mg  $H_2CO_3^*$  alkalinity (as  $CaCO_3$ )/(mg effective COD), added to the feed bucket in the form of NaOH. The recycle ratio initially applied was 7:1 to reduce the base influent COD to an effective influent COD of  $9\,000/(1+7) = 1\,125$  mg/ℓ. This recycle ratio gave alkalinity supplementation of  $1,2/(1+7) = 0,15$  mg  $H_2CO_3^*$  alkalinity (as  $CaCO_3$ )/(mg base influent COD).

Nutrients and trace elements were added to the feed bucket in approximately the same quantities per mg base influent COD as during the feasibility study, by adding 50 ml of the nutrient solution (giving a total of 0,023 mg N/COD) and 10 ml trace element solution per litre of undiluted feed.

The system was operated under these conditions for about 45 d. During this period the minimum sludge bed pH did not decline below 6,8. Pellet synthesis was taking place causing the sludge bed to increase above Port 7 and needed to be wasted on occasion to maintain a 3 ℓ bed volume. However, the system showed a slow but steady decline in performance; the gas production steadily declined (Fig. 4). Furthermore, the settleability of the pellets deteriorated progressively showing an increasing tendency to lift to the top of the reactor and some were lost in the effluent. The pellets appeared less compact, lighter in colour and surrounded by a gelatinous sheath.

The observations above pointed towards a possible deficiency in trace element supply. Trace elements had been supplied in the same concentration ratios as during the feasibility study. However, the pH in the feed bucket was measured to be > 11,

substantially higher than that in the feasibility study (pH ~ 9,1); possibly at the high pH the added trace elements precipitated out and either settled in the unstirred feed vessel and hence did not reach the sludge bed or caused the trace elements to be unavailable to the active mass. Accordingly, it was decided to restart the system, but to apply measures that would enhance the likelihood of stable operation and would eliminate the risk of precipitation of the trace elements.

### Second start-up with recycle

To restart the system, with recycle, the following changes were made:

- The sludge bed was drained from the reactor and replaced with 3 ℓ of new pelletised seed sludge obtained from a laboratory UASB system fed with glucose substrate.
- The feed was changed from batch 1 to batch 2, see Table 4. The COD of the second batch was 13 000 mg/ℓ; to restart the system the recycle rate was increased from 7:1 to 15:1 to dilute the base influent COD ( $COD_b$ ) to an effective influent COD of  $13\,000/(15 + 1) = 813\text{ mg/ℓ}$ .
- To ensure that any process decline would not be due to trace element precipitation, it was decided that, during the initial part of the experiment, alkalinity addition by NaOH be abandoned and replaced by  $NaHCO_3$ , added to the undiluted feed at 1 785 mg  $H_2CO_3^*$  alkalinity (as  $CaCO_3$ ) per litre of base influent. The change to  $HCO_3^-$  (as  $H_2CO_3^*$  alkalinity source) maintained the pH in the feed bucket at about 8,3 so that precipitation was unlikely.
- Addition of nutrient and trace element solutions was increased proportionally to the base influent COD. Nutrient stock solution from 50 to 65 mℓ/ℓ influent, to provide a 0,024 mg N/mg undiluted influent COD and a COD/P ratio of 350:1,6; trace element stock solution from 10 to 15 mℓ/ℓ influent.
- The point of addition of the trace element solution was changed from the influent stream to the recycle stream.
- To ensure as far as possible that failure would not be due to a lack of essential nutrients (Speece and Parkin, 1986), two further solutions were made up to enhance the concentrations of  $Fe^{++}$ ,  $Ca^{++}$  and  $Mg^{++}$ ; one stock solution containing 10 g  $FeSO_4 \cdot 7 H_2O/ℓ$  and another containing 10 g  $CaCl_2/ℓ$  and 10 g  $MgCl_2/ℓ$ . For these two solutions, 10 and 20 mℓ/ℓ undiluted feed respectively were added to the recycle stream.

As the sludge was derived from a UASB system treating glucose the sludge had to be adapted to the lauter tun waste. Accordingly the system was restarted at a low loading rate of 3,5 kgCOD/(m<sup>3</sup> sludge bed-d). This rate was kept constant for 20 d. Over this period the percentage COD removal increased steadily and stabilised at 95 %. The loading rate was then increased incrementally over a period of 45 d to 9 kg COD/(m<sup>3</sup> sludge bed-d). The COD removal remained above 95%, gas production increased proportionally with the COD loading rate applied (Fig. 5), the pelletised sludge became darker and compact with no evidence of gelatinous sheathing. Clearly the process was operating satisfactorily.

Having identified the apparent cause of failure in the first start-up experiment to be due to precipitation of essential trace elements, consideration was given to means whereby  $OH^-$  could be employed as buffer supplement instead of  $HCO_3^-$ . Clearly the NaOH could not be added to the undiluted influent. Instead it was proposed to dose the NaOH into the recycle stream so that

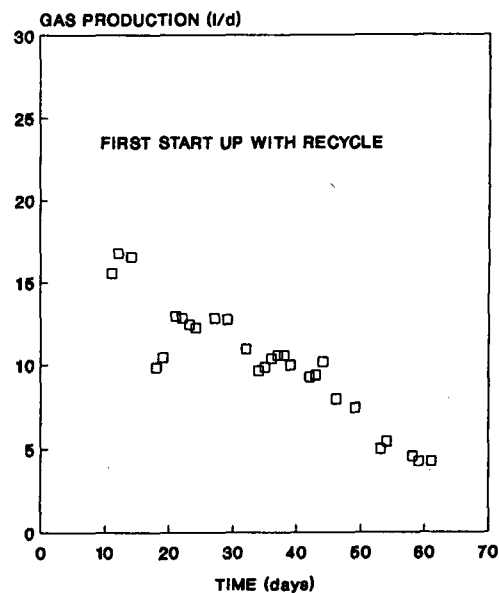


Figure 4  
First start-up period with recycle showing gradual decline in gas production: The laboratory UASB reactor was fed with undiluted lauter tun waste ( $COD_b = 9\,000\text{ mg/ℓ}$ ) and the reactor effluent recycled to the influent at a recycle ratio of 7:1; COD loading rate: 12 kg/(m<sup>3</sup> sludge bed-d)

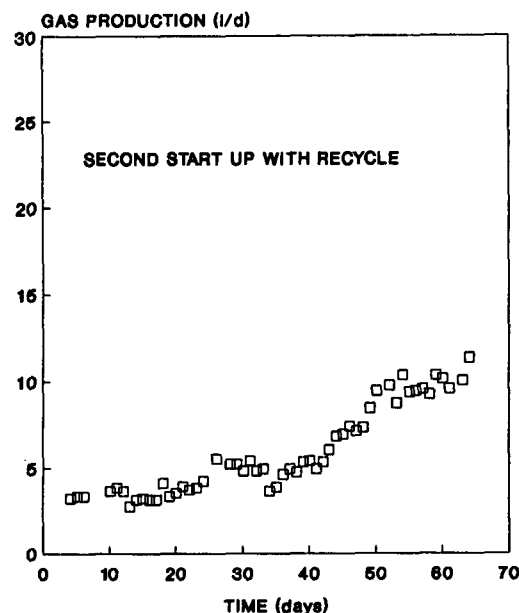


Figure 5  
Second start-up period with recycle: The laboratory UASB reactor was fed with undiluted lauter tun waste ( $COD_b = 13\,000\text{ mg/ℓ}$ ) and the reactor effluent recycled to the influent at a recycle ratio of 15:1; COD loading rate at the beginning, 3,5 kgCOD/(m<sup>3</sup> sludge bed-d) was increased incrementally to 9 kg/(m<sup>3</sup> sludge bed-d) towards the end of the second start-up period

the OH<sup>-</sup> would react with the dissolved CO<sub>2</sub> in the large recycle flow to form HCO<sub>3</sub><sup>-</sup> and limit the rise in pH downstream. Accordingly, the H<sub>2</sub>CO<sub>3</sub>\* alkalinity addition as NaHCO<sub>3</sub> to the base influent was withdrawn and the same mass of H<sub>2</sub>CO<sub>3</sub>\* alkalinity in the form of NaOH was added at a constant rate to the recycle. At the recycle ratio of 15:1, then still in operation, the pH downstream of the point of addition was measured at 8.6. Under such low pH, precipitation of trace elements in the recycle was unlikely.

### Conclusions

From the first and second start-up experiments the following conclusions were drawn:

- In the feasibility study the process had operated satisfactorily at higher COD loading rates than in the second start-up period, even though some of the trace element supplements present in the second start-up were absent in the feasibility study. It would seem that the failure in the first start-up was due to the high pH in the feed causing some of the essential trace elements to become unavailable to the micro-organisms, possibly by precipitation.
- The UASB process could be started up and operated with a high base influent COD concentration, provided the base COD was suitably diluted by a recycle. An effective influent COD of about 800 mg/l (base influent COD 13 000 mg/l with 15:1 recycle ratio) proved to be satisfactory in this study.
- In the start-up using pelletised sludge from a different source, the initial loading rate should always be low and the minimum bed pH maintained at about 7, to assist adaptation of the sludge to the new substrate.
- Buffer in the form of NaOH should be added to recycle stream.

### Alkalinity requirements with recycle

The minimum bed pH serves as an indicator whether alkalinity dosage needs to be changed. However, on its own the minimum pH is not adequate because it does not permit changes in dosing to be determined readily; other parameters, related to the minimum bed pH, had to be sought as basis for dosing estimates.

Sam-Soon et al. (1991) selected two parameters, the H<sub>2</sub>CO<sub>3</sub>\* alkalinity and COD in the base influent stream to estimate alkalinity dosage. They argued that a certain mass of H<sub>2</sub>CO<sub>3</sub>\* alkalinity would be required per mass of base influent COD to maintain a minimum bed pH. Hence, they linked the H<sub>2</sub>CO<sub>3</sub>\* alkalinity requirement in the base flow to the base influent COD concentration (Eq. 1). From the measurement of the base influent COD and H<sub>2</sub>CO<sub>3</sub>\* alkalinity, H<sub>2</sub>CO<sub>3</sub>\* alkalinity is supplemented to the influent to meet the requirements given by Eq. (1). However, there are arguments that the H<sub>2</sub>CO<sub>3</sub>\* alkalinity measurement in the effluent is to be preferred to that in the influent:

In an anaerobic system treating a pure carbohydrate waste, under stable operating conditions there is virtually no difference in H<sub>2</sub>CO<sub>3</sub>\* alkalinity between the influent and effluent. However, with some wastes there is internal generation of H<sub>2</sub>CO<sub>3</sub>\* alkalinity which will take place if the influent contains, for example, proteins and/or SCFA. In the UASB system, deamination of the proteins is rapidly completed in the lower part of the bed, yielding H<sub>2</sub>CO<sub>3</sub>\* alkalinity. Conversion of influent SCFA to methane and carbon dioxide thereby generating H<sub>2</sub>CO<sub>3</sub>\*

alkalinity, can take place throughout the bed; in a stable system usually virtually all the influent SCFA will be converted in the sludge bed. In both cases the internally generated H<sub>2</sub>CO<sub>3</sub>\* alkalinity contributes to control of pH in the system, either directly or via the recycle from the effluent to influent. If the H<sub>2</sub>CO<sub>3</sub>\* alkalinity is measured in the base influent feed flow the internally generated alkalinity is not included in the measurement. Control of the minimum pH should, therefore, incorporate the effluent H<sub>2</sub>CO<sub>3</sub>\* alkalinity as a parameter.

To incorporate the alkalinity generated in the process in Eq. (1) the term H<sub>2</sub>CO<sub>3</sub>\* alkalinity in the base influent was replaced by the term H<sub>2</sub>CO<sub>3</sub>\* alkalinity in the effluent, i.e.:

$$\text{H}_2\text{CO}_3^* \text{ alkalinity/l effluent} = \frac{1}{1+r} \cdot C \cdot \text{COD}_b \quad (3)$$

where:

$$\text{H}_2\text{CO}_3^* \text{ alkalinity/l effluent} = \text{H}_2\text{CO}_3^* \text{ alkalinity in the effluent flow associated with a near neutral minimum sludge bed pH.}$$

The intent of the experiment would be to establish the factor C for lauter tun waste.

### Experimental procedure

At a COD load of 9 kg/(m<sup>3</sup> sludge bed·d) and a base influent COD concentration (COD<sub>b</sub>) of 13 000 mg/l, different recycle ratios were tested. With each recycle the mass of NaOH added to the recycle stream was adjusted until the minimum bed pH was ~ 7. The minimum bed pH was measured daily in the lower part of the sludge bed at sample Port 2, and at intervals at all sampling ports located in the bed, to verify that the minimum bed pH was best reflected at sample Port 2 and to check the bed pH profile. The H<sub>2</sub>CO<sub>3</sub>\* alkalinity and SCFA concentrations were measured daily in the reactor effluent using the 5 pH point titration method (Moosbrugger et al., 1993b), together with the filtered effluent COD. The sludge bed volume was kept constant at sample Port 7 by wasting excess sludge and maintaining a sludge bed volume of 3 l. The average sludge bed density was approximately 36 kg VSS/(m<sup>3</sup> sludge bed). No data were collected on sludge production due to pressure of time (an unfortunate omission in retrospect) - reliable estimates require a long period of data accumulation under steady state conditions without disturbance of the bed, by for example, sampling for pH measurement. The study centered on the effect of the recycle ratio on process performance; the period required to adapt and attain stability after change of recycle was 15 to 20 d.

## Results and discussion

**Recycle and process performance:** The base influent COD (13 000 mg/l) was diluted by the recycles to an effective COD ranging from 1 860 to 580 mg/l under the respective recycle ratios ranging from 6 to 22:1. For each period of operation at a particular recycle ratio, in Fig. 6 the average filtered effluent COD (COD<sub>eff</sub>) and the effective influent COD (COD<sub>e</sub>), and in Fig. 7 the average effluent SCFA are all plotted versus the recycle ratio. Noting the low effluent SCFA and COD throughout the experiment, the magnitude of the recycle ratios had virtually no effect on these effluent values. Thus it would



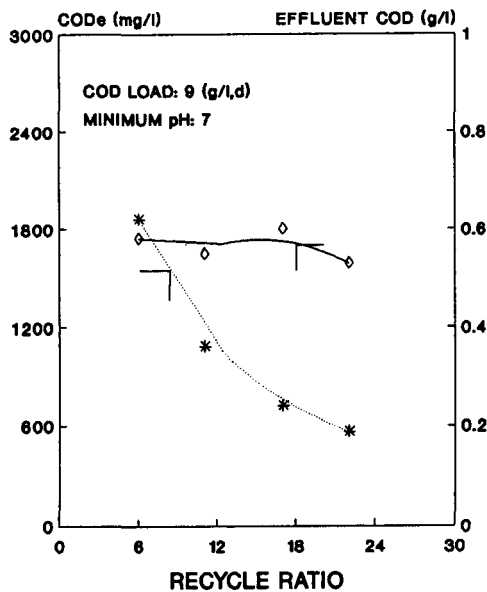


Figure 6  
Effect of recycle ratio or, equivalently, effect of effective influent COD ( $COD_e$ ) on the effluent COD concentration. COD loading rate  $9 \text{ kg}/(\text{m}^3 \text{ sludge bed}\cdot\text{d})$ ;  $COD_b = 13\,000 \text{ mg}/\text{t}$ ;  $COD_e = COD_b \cdot 1/(1+r)$

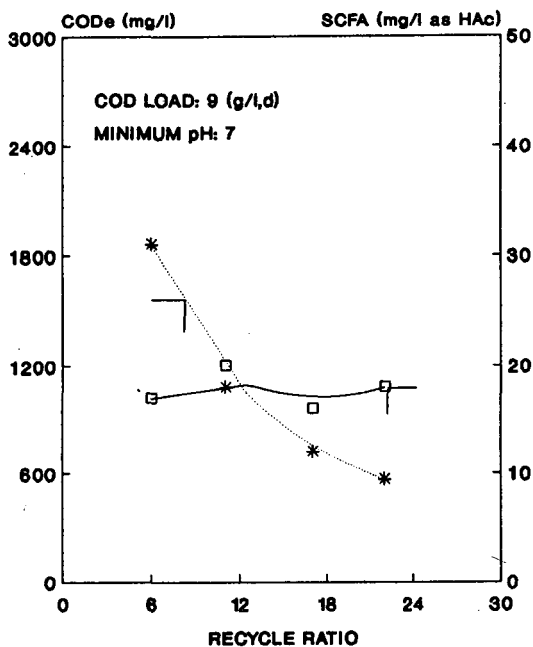


Figure 7  
Effect of recycle ratio or, equivalently, effect of effective influent COD ( $COD_e$ ) on the effluent SCFA concentration. COD loading rate  $9 \text{ kg}/(\text{m}^3 \text{ sludge bed}\cdot\text{d})$ ;  $COD_b = 13\,000 \text{ mg}/\text{t}$ ;  $COD_e = COD_b \cdot 1/(1+r)$

seem that at a COD loading rate of  $9 \text{ kg}/(\text{m}^3 \text{ sludge bed}\cdot\text{d})$ , for the minimum bed pH of  $\sim 7$  the magnitude of the recycle ratio between 6 and 22:1 had no noticeable adverse effect on the COD removal even though  $COD_e$  was reduced from 1 860 to 580  $\text{mg}/\text{l}$ .

**TKN uptake:** In their study on nitrogen limitation on pelletisation in a UASB system fed with glucose, Sam-Soon et al. (1990) found that an influent TKN/COD ratio of 0,02 ( $\text{mg N}/\text{mg COD}$ ) was adequate for unimpeded pellet formation (cf TKN/COD  $\sim 0,004$  for anaerobic fermentation systems without pelletisation). In this study the waste contained only 0,011  $\text{mg N}/\text{mg COD}$  (144  $\text{mg N}/\text{l}$  influent, see Table 4, batch 2) and hence was supplemented with  $\text{NH}_4\text{Cl}$  to increase this ratio to a total of 0,024  $\text{mg N}/\text{mg COD}$ . From the daily TKN measurements in the influent and effluent the average TKN uptake was 190  $\text{mg N}/\text{l}$  giving a TKN uptake/influent COD of 0,015. Thus the TKN/COD uptake in this study was lower than that proposed by Sam-Soon et al. (1990), probably because the fraction of carbohydrate in the influent COD of the lauter tun waste was lower than that of glucose substrate and hence the mass generation of pellets per influent COD was lower. Even though the TKN/COD demand (equal to 0,015) was lower, it still was four times higher than that in a "normal" anaerobic system - the TKN/COD must be taken into account in assessing the nitrogen requirements for lauter tun waste.

**Bed pH profile:** The minimum recycle ratio employed was 6:1, and the maximum 22:1. In Fig. 8 the bed pH profiles are shown for these two recycles. At recycle ratio = 6:1 ( $COD_e = 1860 \text{ mg}/\text{l}$ ) the minimum pH was only slightly lower than the pH in upper part of the bed; at a recycle ratio = 22:1 ( $COD_e = 580 \text{ mg}/\text{l}$ ) the

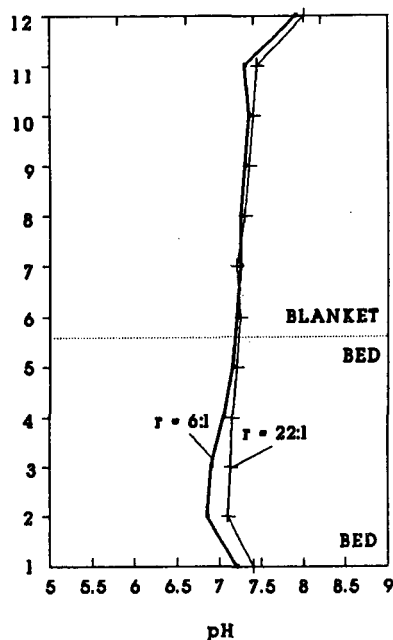


Figure 8  
pH profiles along the line of flow of the laboratory UASB reactor at two different recycle ratios (6:1 and 22:1);  $COD_b = 13\,000 \text{ mg}/\text{t}$  and COD loading rate =  $9 \text{ kg}/(\text{m}^3 \text{ sludge bed}\cdot\text{d})$ . Note that the influent pH (at sample Port 1) is the pH of the combined base influent and recycle stream

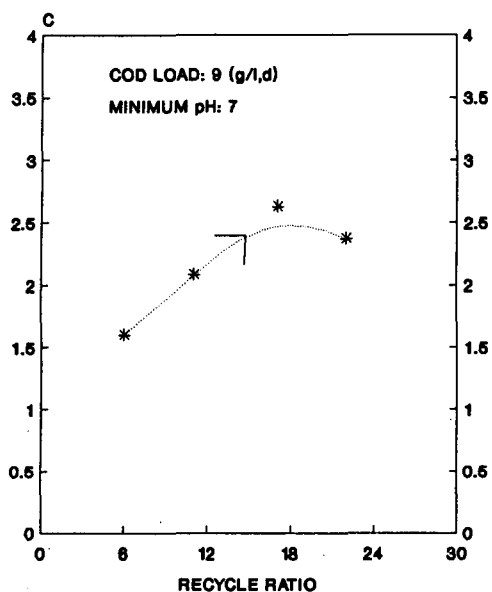


Figure 9

Relationship between recycle ratio and the factor  $C$ , see Eq. (1) to maintain a near neutral minimum bed pH treating undiluted lauter tun waste ( $COD_b = 13\ 000\ \text{mg/l}$ ) when applying different recycle ratios at a constant COD loading rate of  $9\ \text{kg COD}/(\text{m}^3\ \text{sludge bed}\cdot\text{d})$  ( $C$  is expressed in  $\text{mg H}_2\text{CO}_3^*$  alkalinity required per  $\text{mg}$  base influent COD without recycle)

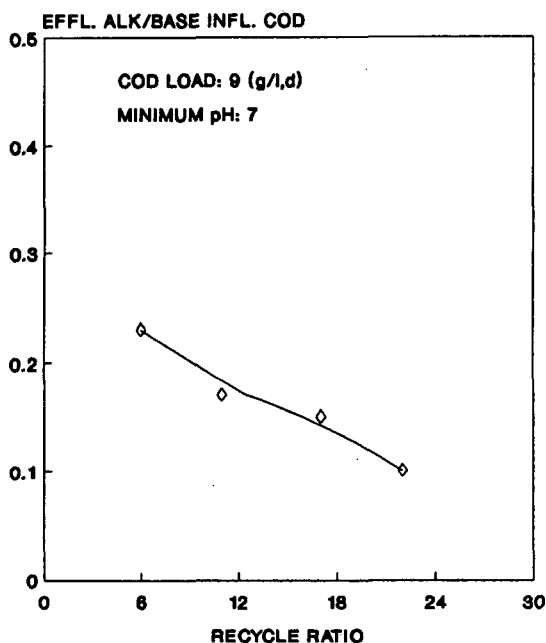


Figure 10

Relationship between effluent  $\text{H}_2\text{CO}_3^*$  alkalinity requirements per unit mass of base influent COD (to maintain a near neutral minimum bed pH) and recycle ratio; treatment of undiluted lauter tun waste ( $COD_b = 13\ 000\ \text{mg/l}$ ) at a constant COD loading rate of  $9\ \text{kg}/(\text{m}^3\ \text{sludge bed}\cdot\text{d})$

pH was virtually constant throughout the bed. Without a detailed evaluation of the bed behaviour it is not possible to give a definitive explanation for the equalizing tendency in the pH profile when the recycle ratio increased. Very likely this arose from the dynamic situation in the lower part of the sludge bed, through interaction between flow rate, rate of SCFA generation by acidogens, SCFA uptake by acetoclastic methanogens, deamination,  $\text{CO}_2$  production by acidogens and acetoclastic methanogens,  $\text{CO}_2$  uptake by hydrogenotrophic methanogens, alkalinity in the influent and recycles, and others.

**Behaviour of  $C$ :** From the measured data, the value of  $C$  was derived via Eq. (3), and is shown plotted versus the recycle ratio in Fig. 9. Evidently  $C$  did not remain constant as hypothesised by Sam-Soon et al. (1991), but increased with increasing in recycle ratio; that is, the observed reduction in the alkalinity requirements with increasing recycle ratio would not be as large as with a constant  $C$ . Also, the value of  $C$  at a specific loading rate would be influenced by the selected minimum bed pH and therefore could be expected to be higher for a minimum pH  $\approx 7$  than for one of pH  $\approx 6,7$ . Furthermore, from the discussion earlier, the pH established at a certain point in the bed would be a function also of the dynamic situation in the bed. Thus, a constant  $C$  at a specific loading rate for different recycle ratios is likely to be the exception rather than the rule. Consequently, the determination of  $C$  from a flow-through system using a diluted base flow would provide, at best, an approximate lower limit estimate.

Despite the fact that  $C$  increases with increasing recycle ratios, the recycle ratio still exerts a dominating effect in reducing the  $\text{H}_2\text{CO}_3^*$  alkalinity requirements per litre of effluent. This is illustrated in Fig. 10: the mass of effluent  $\text{H}_2\text{CO}_3^*$  alkalinity required per mass of base influent COD is significantly reduced as the recycle increases, i.e. the imposition of a recycle remains an effective means to reduce  $\text{H}_2\text{CO}_3^*$  alkalinity requirements in a UASB system. Since very little  $\text{H}_2\text{CO}_3^*$  alkalinity is generated in the sludge bed, the  $\text{H}_2\text{CO}_3^*$  alkalinity requirements will have to be met by supplementation.

#### Practical alkalinity and pH control

Accepting that  $C$  declines as the recycle ratio increases, from a practical point of view, to determine the alkalinity dosing requirements to control the minimum pH in the bed, the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity should be related to the effective influent COD concentration ( $COD_e$ ). In Fig. 11, for the COD loading rate of  $9\ \text{kg}/(\text{m}^3\ \text{sludge bed}\cdot\text{d})$  and a minimum bed pH of 7, the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity (as  $\text{CaCO}_3$ ) is plotted versus  $COD_e$  and clearly illustrates the reduced effluent  $\text{H}_2\text{CO}_3^*$  alkalinity requirements at lower  $COD_e$  resulting from higher recycle ratios.

In treating a specific waste, to make use of  $COD_e$  and the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity to control the selected minimum bed pH, the relationship between  $COD_e$  and effluent  $\text{H}_2\text{CO}_3^*$  alkalinity (Fig. 11) should be established experimentally under design conditions, using the procedures developed above. The control strategy then is as follows:

Effective influent COD ( $COD_e$ ) is determined from measurement of the base influent COD ( $COD_b$ ) and the recycle ratio. From the experimental relationship between the  $COD_e$  and the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity, the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity required to maintain a near neutral minimum bed pH is determined. This is then compared to the effluent  $\text{H}_2\text{CO}_3^*$  alkalinity measured for the operating system (the 5 pH point

titration method can be used, Moosbrugger et al., 1993b). From the difference between measured and required effluent  $H_2CO_3^*$  alkalinity the pH buffer dosing requirements can be estimated, and the dose added to the recycle stream. Should the loading rate change significantly this procedure will need to be repeated at the new loading rate. If the system is not yet in operation, a preliminary estimate of the dosing requirements can be obtained by setting the dose equal to the required effluent  $H_2CO_3^*$  alkalinity. Once the system is in operation, the dosing requirements should be adjusted using the procedure above.

## Conclusions

- The study of lauter tun waste in a laboratory-scale UASB system has shown that treatment of this substrate is feasible in a UASB system and that the substrate develops a granular sludge bed. The pattern of product formation along the line of flow of the reactor is very similar to that observed under similar conditions when treating a pure carbohydrate type substrate, e.g. glucose or apple juice concentrate.
- The TKN/COD ratio of the lauter tun waste was 0,011 mg N/mg COD. For unimpeded pelletisation when treating glucose in a UASB system Sam-Soon et al. (1990) suggested an influent TKN/COD ratio of 0,02 mg N/mg COD. In this study the feed was supplemented with  $NH_4Cl$  to give an influent TKN/COD ratio of 0,024; the observed TKN uptake was 0,015 mg N/mg undiluted influent COD. Thus it appears that lauter tun waste needs to be supplied with nitrogen when treated in UASB systems to achieve unimpeded pelletisation. The TKN/COD ratio is less than that required for the treatment of glucose substrate probably because the lauter tun has a smaller fraction of carbohydrates per unit influent COD than glucose substrate.
- Lauter tun waste generates only insignificant, internal pH buffer; buffer needs to be supplied from an external source to control the minimum pH in the reactor to acceptable levels. When supplying  $H_2CO_3^*$  alkalinity via a strong base (e.g. NaOH) to the base feed flow, the pH may increase to such high levels that some of the trace elements may precipitate and become unavailable to the micro-organisms giving rise to partial or complete failure of the process. Hence, the dosing point needs to be selected such that a drastic pH increase at the dosing point is avoided. In this study an appropriate dosing point was found to be the recycle stream. The presence of dissolved  $CO_2$  and  $H_2CO_3^*$  alkalinity in the recycle stream buffered the pH downstream of the point of NaOH addition to a pH < 8,5, instead of a pH of > 11 when NaOH was added directly to the influent flow.
- The alkalinity requirement (mg  $H_2CO_3^*$  alkalinity/l base influent) to maintain a selected minimum pH in the bed, can be reduced by imposing a recycle. Sam-Soon et al. (1991) formulated the alkalinity requirement/l influent as  $C \cdot (\text{base influent COD}) / (1 + \text{recycle ratio})$ , and accepted that C is constant, at 1,2 mg  $H_2CO_3^*$  alkalinity (as  $CaCO_3$ )/(mg effective influent COD). This study showed that for a specific COD loading rate C is not constant but increases as the recycle ratio (r) increases, from  $C = 1,5$  (r = 6) to  $C = 2,4$  (r = 22) for the selected COD loading rate of 9 kg/(m<sup>3</sup> sludge bed-d) and the selected minimum pH  $\approx 7$ . Despite this increase, the recycle factor  $1/(1 + r)$  still causes a significant reduction in alkalinity requirements per litre base influent flow.
- With the target minimum bed pH of  $\approx 7$  the pH profile in the bed exhibited only a slight depression at the minimum value

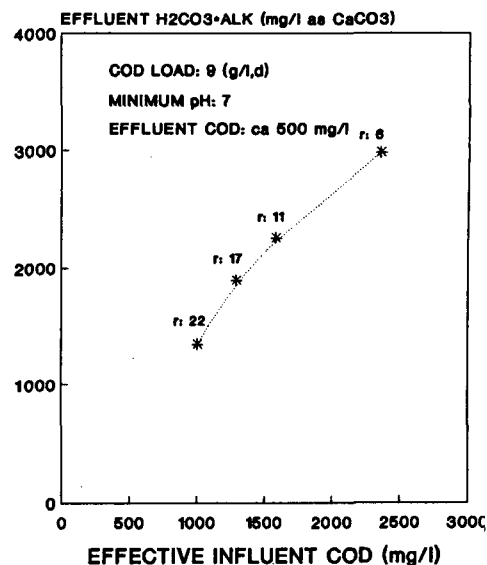


Figure 11

Relationship between effective influent COD ( $COD_e = COD_b / (1+r)$ ) and effluent  $H_2CO_3^*$  alkalinity requirements (to maintain a near natural minimum bed pH): treatment of undiluted lauter tun waste ( $COD_b = 13\ 000$  mg/l) applying different recycle ratios at a constant COD loading rate of 9 kg/(m<sup>3</sup> sludge bed-d)

for a recycle ratio of 6:1 (base influent COD diluted by the recycle to 1 860 mg/l and no significant depression at higher recycle ratios. This tendency to smooth out the "dip" in the pH profile when the effective influent COD is reduced, conforms with the observations of Sam-Soon et al. (1991).

- Dilution of the base influent COD from 13 000 to an effective influent COD of 570 mg/l, by applying a recycle ratio of 22:1, appeared to have no adverse effect on the process performance. Thus it would seem that the lower limit of the effective influent COD of 1 000 mg/l suggested by Sam-Soon et al. (1991), can be substantially lowered.
- To maintain a minimum sludge bed pH  $\approx 7$  at a COD loading rate of 9 kg/(m<sup>3</sup> sludge bed-d), a base influent COD concentration of 13 000 mg/l and a recycle ratio of 22:1, the pH buffer requirements, expressed as  $H_2CO_3^*$  alkalinity, were 1 350 mg/l (as  $CaCO_3$ ) per litre of base influent flow and with the recycle ratio changed to 6:1, 2 980 mg/l (as  $CaCO_3$ ). Virtually all of the pH buffer needed to be supplied from an external source.
- Trace elements were added throughout the investigation, consequently, no pronouncement as to whether the UASB system would operate effectively without trace element supplementation can be made.

## Acknowledgement

This research was supported jointly by the Foundation for Research Development and the Water Research Commission of South Africa and this paper is published with their permission.

## References

- CAPRI, MG and MARAIS, GvR (1975) pH adjustment in anaerobic digestion. *Water Research* **9** 307-314.
- LOEWENTHAL, RE, EKAMA, GA and MARAIS, GvR (1989) Mixed weak acid/base systems Part I - Mixture characterisation. *Water SA* **15**(1) 3-24.
- MOOSBRUGGER, RE, WENTZEL, MC, EKAMA, GA and MARAIS, GvR (1993a) Weak acid bases and pH control in anaerobic systems - A review. *Water SA* **19**(1) 1-10.
- MOOSBRUGGER, RE, WENTZEL, MC, LOEWENTHAL, RE, EKAMA, GA and MARAIS, GvR (1993b) Alkalinity measurement: Part 3 - A 5 pH point titration method to determine the carbonate and SCFA weak acid/bases in aqueous solution containing also known concentrations of other weak acid/bases. *Water SA* **19**(1)29-40.
- SAM-SOON, PALNS, LOEWENTHAL, RE, DOLD, PL and MARAIS, GvR (1987) Hypothesis for pelletisation in the upflow anaerobic sludge bed reactor. *Water SA* **13**(2) 69-80.
- SAM-SOON, PALNS, LOEWENTHAL, RE, WENTZEL, MC and MARAIS, GvR (1989) Pelletisation in the Upflow Anaerobic Sludge Bed (UASB) Reactor. Research Report W 72, Dept. Civil Eng., Univ. of Cape Town, Rondebosch 7700, South Africa.
- SAM-SOON, PALNS, LOEWENTHAL, RE, WENTZEL, MC and MARAIS, GvR (1990) Effect of nitrogen limitation on pelletisation in upflow anaerobic sludge bed (UASB) systems. *Water SA* **16**(3) 165-170.
- SAM-SOON, PALNS, LOEWENTHAL, RE, WENTZEL, MC, MOOSBRUGGER, RE and MARAIS, GvR (1991). Effects of a recycle in the upflow anaerobic sludge bed (UASB) systems. *Water SA* **17**(1) 37-46.
- SPEECE, RE and PARKIN, GF (1986) Anaerobic treatment - Nutrient stimulation and toxicity response. *Proc. of the 1st Anaerobic Dig. Symp.*, Bloemfontein, South Africa.
- STANDARD METHODS (1989) *Standard Methods for the Examination of Water and Wastewater*. American Public Health Association, American Water Works Association and Water Pollution Control Federation, Washington DC.
- ZEHNDER, AJB and WUHRMANN, K (1977). Physiology of *Methanobacterium* strain AZ. *Arch. Microbiol.* **111** 199-205.
-