

# Biological sludge stabilisation

## Part 2: Influence of the composition of waste activated sludge on anaerobic stabilisation

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### Abstract

An experimental investigation was carried out to establish a relationship between the composition of the organic material of waste activated sludge and the extent of its transformation into biogas in an anaerobic digester. The volatile fraction of waste activated sludge was considered to be composed of an active fraction, formed by live material, and an inactive fraction, which does not exhibit metabolic activity in an aerobic environment. Five different sludges were generated with active fractions varying from 76 to 16% of the volatile sludge mass. The active fractions were calculated from the measured specific utilisation rates.

The results of the investigation showed that the conversion efficiency of the organic material in waste activated sludge depended strongly on the sludge composition. In an anaerobic digester operating at 25°C, 53% of the active fraction and 15% of the inactive fraction were digested. The digestion efficiency of the active sludge fraction corresponded to the value obtained for primary sludge. It was established that the release of ammonia nitrogen and alkalinity corresponded to the stoichiometric values, expected on the basis of the sludge composition.

### Introduction

The sludge produced in an activated sludge process (waste sludge) may be composed of both settleable influent material (primary sludge) and excess biological sludge from the aeration tank (secondary or waste activated sludge). Both sludges have high fractions of putrescible material and decomposition starts within hours when left unaerated, leading to serious odour problems. Anaerobic sludge digestion is a widely applied method for stabilisation of waste activated sludge. The waste sludge is introduced into the anaerobic digester in which anaerobic bacterial populations develop in the exclusion of air. These populations use the waste sludge as their substrate, transforming it into stable gaseous products and anaerobic bacterial mass. After digestion the rheological, mechanical and biological properties of the sludge improve significantly and the final disposal of the digested sludge becomes a relatively minor problem.

Anaerobic digestion is a process that develops well only at temperatures above 18 to 20°C, so that in countries with a moderate or cold climate there is a need to heat the anaerobic digester, which makes the process less attractive. However, in the tropics the environmental temperature is high enough to maintain near optimal conditions for anaerobic digestion throughout the year in unheated digesters. This increases not only its economic feasibility but also renders the process much more simple and reliable. Along with the temperature, the composition of the waste activated sludge has a strong influence on the extent of sludge digestion. Primary sludge is composed of predominantly biodegradable material and correspondingly a high proportion of the VSS can be converted into biogas: 55 to 65% in the temperature range of 25 to 35°C (O'Rourke, 1968). Waste activated sludge is less subject to decomposition, especially if the operational sludge age is long. In that case a minor proportion is composed of live material and a large fraction of the sludge is

organic material that is not or very slowly biodegradable. Therefore it is to be expected that the sludge composition has a marked influence on the biogas production per unit mass applied sludge.

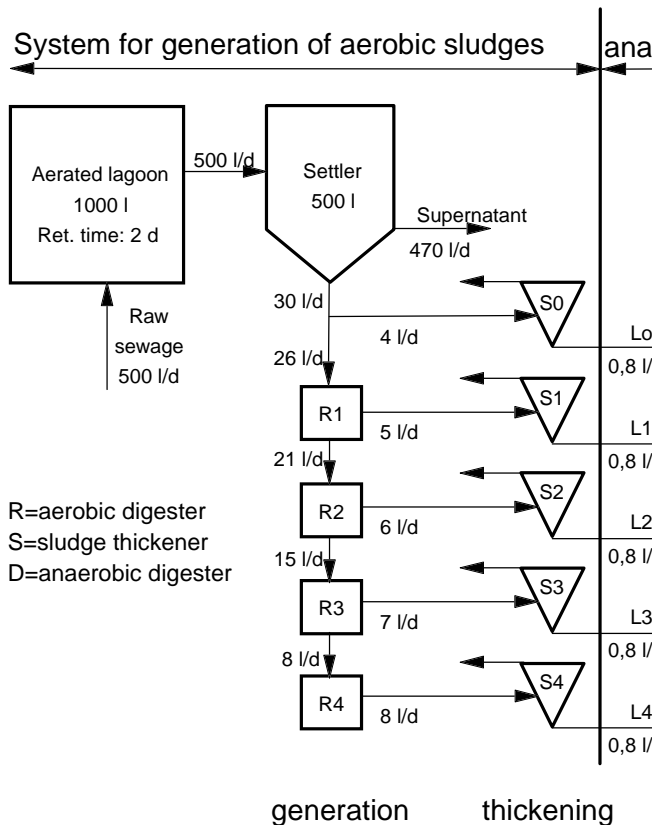
In the present paper an experimental investigation is described to evaluate the influence of the composition of waste activated sludge on the extent of anaerobic digestion in a completely mixed digester. Five different sludges were generated at different sludge ages, having active (live) sludge fractions varying from 16 to 76% of the volatile sludge concentration as calculated from the measured specific oxygen utilisation rates (SOUR) for the sludges ( $\text{mgO}\cdot\text{g}^{-1}\text{VSS}\cdot\text{d}^{-1}$ , using the steady state activated sludge model by Marais and Ekama, 1976). Municipal sewage was used to generate the sludges. Pilot-scale completely mixed anaerobic sludge digesters were operated at 25°C while a retention time of 20 d was maintained. The digestion efficiency was 53% for the active sludge and only 15% for the inactive sludge. The extent of anaerobic digestion of the inactive sludge is almost equal to the value found by O'Rourke for primary sludge. The mineralisation of the sludge led to release of ammonia and alkalinity to the water phase. It was established that the values were approximately equal to the stoichiometric values that could be expected on the basis of sludge composition.

### Experimental investigation and results

The experimental investigation was carried out at pilot scale. The experimental unit was composed of two parts (Fig. 1): an aerobic part for the generation of the sludges and a second for anaerobic digestion of the generated sludges. In the sludge generation part, five different sludges were produced. This part comprised a completely mixed aerated lagoon, a settler and four aerobic digesters in series. The lagoon, with a volume of 1 000 l, had a retention time of 2 d and was fed with municipal sewage from the city of Campina Grande, Brazil. Daily batches of 500 l of mixed liquor were withdrawn from the lagoon and after solid-liquid separation in the settler, 470 l of supernatant were discarded as effluent, leaving a volume of 30 l containing the excess sludge

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**Figure 1**

Schematic representation of the systems for generation and anaerobic digestion of sludges during the experimental investigation

TABLE 1 MAIN CHARACTERISTICS OF THE FIVE GENERATED AEROBIC SLUDGES, WHICH WERE FED TO FIVE CORRESPONDING ANAEROBIC DIGESTERS					
Parameter	Sludge				
	L <sub>0</sub>	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>	L <sub>4</sub>
Total sludge conc. (gTSS·ℓ <sup>-1</sup> )	4.20	3.74	2.91	2.51	2.11
Volat. sludge conc.(gVSS·ℓ <sup>-1</sup> )	3.01	2.52	1.89	1.57	1.26
Flow (ℓ·d <sup>-1</sup> )	4	5	6	7	8
Daily mass (gTSS·d <sup>-1</sup> )	16.9	18.7	17.5	17.6	16.9
OUR (mgO·ℓ <sup>-1</sup> ·h <sup>-1</sup> )	44	29	16	8	4
Active sludge conc.(gVSS·ℓ <sup>-1</sup> )	2.29	1.51	0.83	0.42	0.21
Volatile fraction	0.71	0.67	0.64	0.62	0.60
Active fraction	0.76	0.60	0.44	0.26	0.16
Equivalent sludge age (d)	2	5	10	23	43

from the lagoon. A portion (4 ℓ) of the settled excess sludge was used to feed an anaerobic digester (D<sub>0</sub>) and the rest was fed to the first of a series of four aerobic digesters.

The aerobic digesters (R<sub>1</sub> to R<sub>4</sub>, Fig. 1) had volumes of 45 ℓ each and were operated in series, according to the following procedures: A daily volume of 8 ℓ was withdrawn from the fourth and last aerobic digester (R<sub>4</sub>) and left to settle out. The withdrawn volume was substituted with sludge from the third aerobic digester (R<sub>3</sub>) from which another 7 ℓ·d<sup>-1</sup> were taken and set aside for settling. The withdrawn volume of the third digester, being 8+7 = 15 ℓ was substituted by sludge from the second aerobic

digester (R<sub>2</sub>) and an additional 6 ℓ were taken from R<sub>2</sub> and set aside to settle out. The total withdrawn value of 21 ℓ from R<sub>2</sub> was substituted with sludge from R<sub>1</sub> and from this reactor were taken another 5 ℓ of sludge. The total volume of 26 ℓ withdrawn from R<sub>1</sub> was then substituted by the settled sludge from the aerated lagoon. During the entire investigation period the temperature of the sludges was 25 ± 2°C, the room temperature in the laboratory.

The above procedure was followed to obtain five different sludges (L<sub>0</sub> to L<sub>4</sub> in Fig. 1) for subsequent use in five identical anaerobic digesters D<sub>0</sub> to D<sub>4</sub>. The volumes of the aerobic digesters and the volumes of the sludges withdrawn from these were chosen with the intention to produce equal daily masses of the five different sludges. The Marais and Ekama (1976) model was used to make the necessary calculations before the experimental investigation was started.

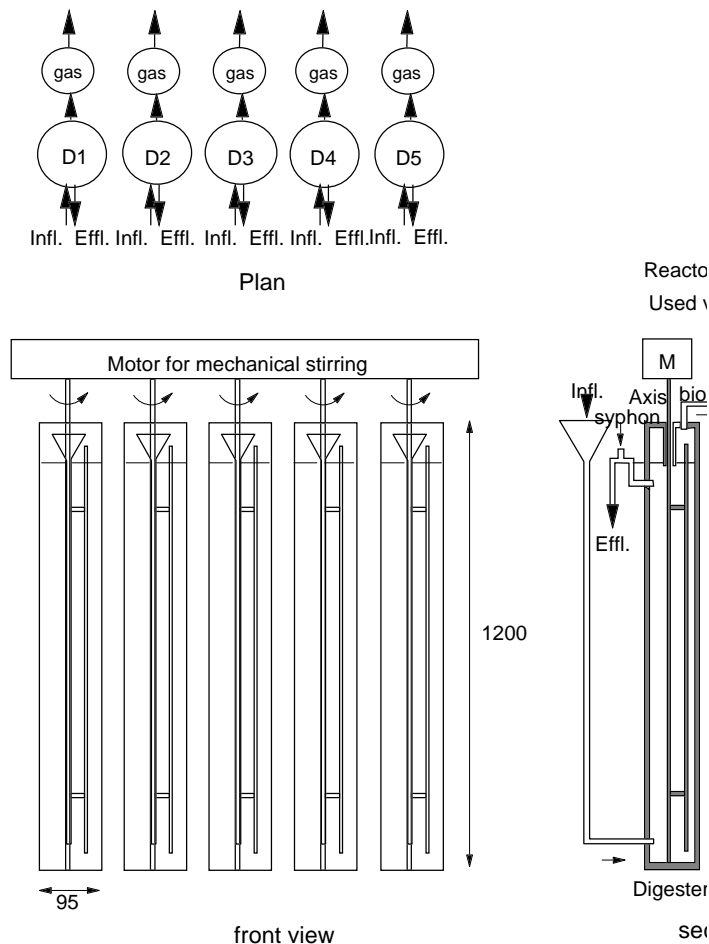
The mass volatile suspended solids (VSS) and total suspended solids (TSS) of the five different sludges (daily production of 4 ℓ from the settler and 5, 6, 7 and 8 ℓ from the aerobic digesters R<sub>1</sub> to R<sub>4</sub> respectively) was determined gravimetrically. The composition of the sludges in terms of the concentrations of active (live) and inactive sludge fractions was evaluated from oxygen uptake rate (OUR) tests, using the aerobic digestion model presented by Marais and Ekama (1976), where aerobic digestion is considered as a first order decay process for the active sludge with a decay constant of b<sub>p</sub> = 0.29 d<sup>-1</sup> at 25°C. The calculation procedure to establish the active sludge concentration from OUR tests is described in **Appendix I**.

Table 1 shows the experimental values of the daily masses of produced sludges and their OUR rates as well as the calculated values of the active sludge fractions. It can be noted that, in effect, the sludge mass produced was almost the same for the

different sludges (as was the intention of the operational procedure), but the OUR values and the corresponding active sludge fractions varied considerably: the active fraction ranged between a maximum of 76% of the VSS in sludge from the lagoon and only 16% in the sludge from the last aerobic digester. The former value corresponds to sludge from a high-rate activated sludge process, whereas the latter value is that of a well stabilised aerobic sludge. For comparison, in Table 1 the required sludge ages to produce the compositions of the five sludges in a conventional activated sludge system are also indicated (last line).

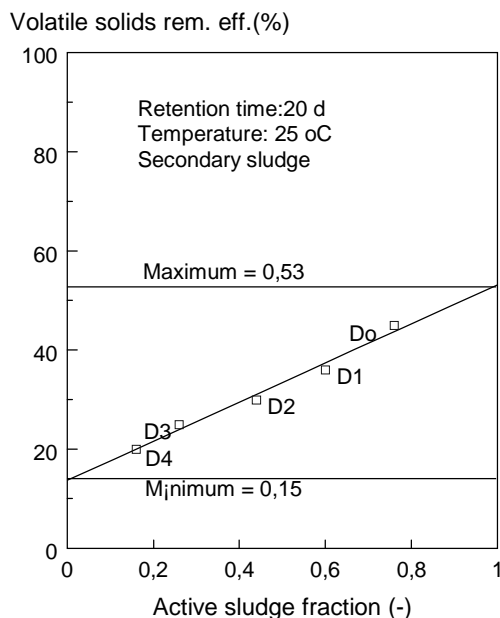
Before using the sludges in the anaerobic digesters, they were all thickened in batch settlers to a volume of 0.8 l. On alternating days the sludges were used as feed for the anaerobic digesters D<sub>0</sub> to D<sub>4</sub>. Each of these digesters had a volume of 8 l so that the nominal retention time was 20 d. The retention time of 20 d was chosen, because it has been shown that this period is sufficient to obtain the highest possible digestion at 25°C (O'Rourke, 1968). Figure 2 shows some details of the anaerobic digestion system used during the investigation. All digesters had identical stirring mechanisms to maintain the sludge in suspension.

After an introduction period of three months to build up the bacterial populations in the anaerobic digesters, they were then operated for four months to obtain the experimental data. The main parameters to characterise the performance of the anaerobic digesters were observed during this period: suspended solids concentration (total, TSS, and volatile, VSS), ammonia, COD in the liquid phase, volatile fatty acids (VFA) concentration, alkalinity, pH and methane production. The last parameter was measured by means of Mariotti flasks and using NaOH in the flasks to absorb CO<sub>2</sub> as indicated in Fig 2. All tests were carried out in accordance with the procedures in *Standard Methods* (1995) except for VFA

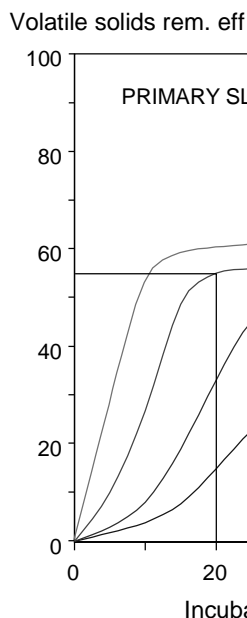


**Figure 2**  
Schematic representation of the anaerobic digesters

TABLE 2 EXPERIMENTAL RESULTS OF THE DIGESTION OF THE FIVE SLUDGE CHARACTERISED IN TABLE 1 IN COMPLETELY MIXED DIGESTERS AT A RETENTION TIME OF 20 DAYS AND AN OPERATIONAL TEMPERATURE OF 25°C										
Parameter	D <sub>0</sub>		D <sub>1</sub>		D <sub>2</sub>		D <sub>3</sub>		D <sub>4</sub>	
	Infl	Effl	Infl	Effl	Infl	Effl	Infl	Effl	Infl	Effl
TSS mass (g/2d)	16.8	11.6	18.7	12.9	17.5	12.8	17.6	13.5	16.9	13.6
VSS mass (g/2d)	12.1	6.7	12.6	8.1	11.4	8.0	11.0	8.2	10.1	8.1
NH <sub>4</sub> <sup>+</sup> (gN.t <sup>-1</sup> )	0.03	0.60	0.01	0.46	0.01	0.36	0.01	0.31	0.01	0.20
Alk (gCaCO <sub>3</sub> .t <sup>-1</sup> )	0.43	1.95	0.32	1.44	0.22	1.20	0.15	1.05	0.10	0.56
VFA (mmol/l)	-	0.24	-	0.30	-	0.25	-	0.19	-	0.28
COD (g.t <sup>-1</sup> )	0.14	0.59	0.15	0.77	0.16	0.70	0.19	0.56	0.13	0.71
pH (-)	7.88	7.89	7.73	7.88	7.51	7.85	7.20	7.81	7.23	7.86
Methane production (l/2d)	2.31		2.05		1.49		1.13		0.59	
COD mass balance	0.98		1.02		1.03		0.99		0.97	



**Figure 3**  
Percentage of digested VSS as a function of the active sludge fraction



**Figure 4**  
Digestion efficiency for primary sludge batches as a function of the incubation time for different temperatures (O'Rourke, 1968)

concentration, which was determined titrimetrically. Table 2 summarises the experimental data from the five anaerobic digesters fed with the five aerobic sludges.

## Discussion

The most important result revealed by the data in Table 2 is that the extent of anaerobic digestion of activated sludge depends on its composition. This relationship is shown in Fig. 3, where the percentage of digested VSS in activated sludge is plotted as a function of the active fraction before digestion. It can be noted that the experimental data suggest a linear relationship that can be written as:

$$\%_{VSS} = 15 + 38f_a \quad (1)$$

where:

$$\begin{aligned} \%_{VSS} &= \text{percentage of solids converted into biogas in the digester} \\ f_a &= \text{active fraction in the sludge to be digested} \end{aligned}$$

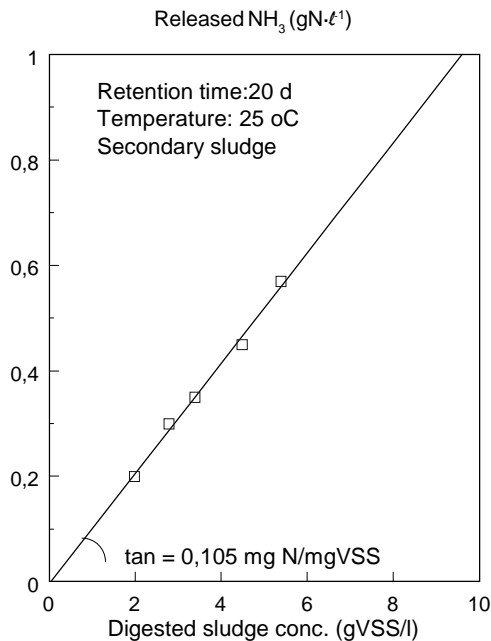
Equation (1) shows that the maximum and minimum percentages that are converted in the digester are 53% for  $f_a = 1$  (i.e. for a 100% active sludge) and 15% for  $f_a = 0$  (i.e. for inactive sludge). By comparison it has been established that 80% of the active sludge is subject to digestion in an aerobic environment, while the inactive fraction is not affected (Marais and Ekama, 1976; McCarty and Brodersen, 1962; Van Haandel et al., 1986). The relatively low conversion efficiency of active sludge to biogas may be explained by recognising that in the anaerobic process new populations grow, so that part of the aerobic bacterial mass is converted into anaerobic bacterial mass rather than into biogas. The inactive sludge, which is not degraded to a measurable extent in an aerobic environment, is partially digested in the anaerobic digester: 15% is converted into biogas.

It is interesting to compare the digestion data for the secondary sludge with those for primary sludge. In Fig. 4 the results of the classical work by O'Rourke (1968) are shown: The percentage of VSS converted into biogas is plotted as a function of the incubation time for batches of primary sludges at different temperatures. It can be noted that the primary sludge digestion efficiency at 25°C (the temperature used during our experimental investigation) was 55%, almost identical to the observed efficiency for active sludge in the investigated secondary sludges. This clearly indicates that the degradation efficiency of primary sludge and the active fraction of secondary sludge are equal and much higher than the efficiency of inactive secondary sludge.

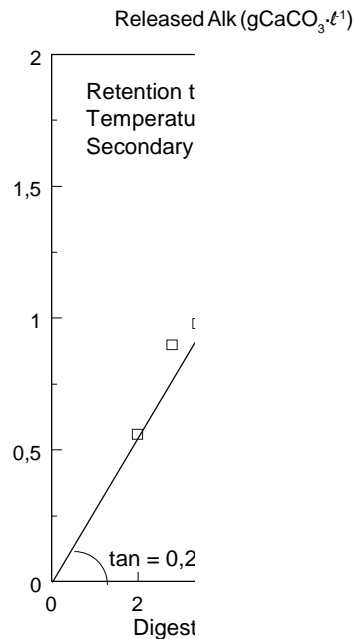
The mass balance for organic material (COD) in the anaerobic digesters is almost 100%, i.e. the flux of organic material entering the digester can be accounted for in the different fluxes of organic material leaving the digesters. For mass balance calculations the organic material fluxes must first be written in the form of measurable parameters. Using COD as a parameter for organic material measurement, the conversion of VSS is easy: in good approximation the ratio of COD/VSS is a constant given by  $f_{cv} = 1.5 \text{ mgCOD}\cdot\text{mg}^{-1}\text{VSS}$ . To convert methane into COD mass, it is considered that there is a stoichiometric relationship of  $4 \text{ mgCOD}\cdot\text{mg}^{-1}\text{CH}_4$ . Hence 1 mol of methane, with a volume of about 25 l at 25°C and atmospheric pressure, has a mass of 16 gCH<sub>4</sub> of 64 gCOD. Therefore 1 l CH<sub>4</sub> represents a COD mass of  $64/25 = 2.56 \text{ g}$ . Now the COD mass balance in the first digester can be expressed as:

$$\begin{aligned} MS_{ii} &= Q_i(\text{COD}_i + f_{cv} \cdot \text{VSS}_i) = 0.4 \cdot (0.14 + 1.5 \cdot 12.08) \\ &= 7.32 \text{ gCOD}\cdot\text{d}^{-1} \\ MS_{ie} &= Q_e(\text{COD}_e + f_{cv} \cdot \text{VSS}_e) = 0.4 \cdot (0.59 + 1.5 \cdot 6.69) \\ &= 4.25 \text{ gCOD}\cdot\text{d}^{-1} \\ MS_{\text{CH}_4} &= 2.56 \cdot Q_{\text{CH}_4} = 2.56 \cdot 2.31/2 = 2.96 \text{ gCOD}\cdot\text{d}^{-1} \end{aligned}$$

Hence the fraction of recovered organic material in digester B<sub>0</sub> is:



**Figure 5**  
Relationship between released ammonia and digested VSS in the anaerobic digesters



**Figure 6**  
Relationship between the increase of alkalinity and the concentration of released ammonia in the digesters

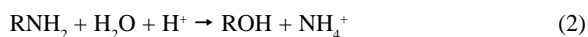
$$B_o = (MS_{te} + MS_{CH_4}) / MS_{V_i} * 100$$

$$= (4.25 + 2.96) / 7.32 * 100 = 98\%$$

The mass balance shows that there is a difference of only 2% between the fluxes of organic material entering the digester and those leaving it. Considering that the mass balance calculations are based on experimental determinations and therefore subject to errors, this result is quite satisfactory and confers a high degree of reliability to the experimental data. Similar calculations carried out for the other four digesters show near closing mass balances. The results are in Table 2 (last line).

pH increased in all five digesters. This can be explained by considering that several processes occur in the digester, affecting the alkalinity and hence pH. The first process is the anaerobic digestion of VSS: this process develops basically in two sequential steps: acid fermentation and methanogenic fermentation. In the first step complex organic material is broken down to VFA, especially acetic acid, which in turn is converted into biogas in the second step. The experimental data indicate that the anaerobic digestion itself (i.e. the conversion of organic solids into biogas) did not have important implications on alkalinity or pH: the concentration of VFA was low in the influent sludges (<0.1 mmol·l<sup>-1</sup>) as well as in the digesters (0.2 to 0.3 mmol·l<sup>-1</sup>). Hence there was a small net VFA production and consequentially a small alkalinity consumption.

However, the alkalinity in all digesters increased significantly. This can be attributed to a second process in the digesters: the generation of alkalinity by the mineralisation of organic nitrogen that accompanies the digestion of VSS. In the ammonification process there is a production of 3.57 mgCaCO<sub>3</sub>·mg<sup>-1</sup>N as can be calculated from the reaction equation:



In Eq. (2) the ammonification of 1 “mol” of organic nitrogen consumes 1 mol of H<sup>+</sup> or, equivalently, there is a production of 50 g CaCO<sub>3</sub> of alkalinity. Hence, alkalinity increases by 50/14 = 3.57 mgCaCO<sub>3</sub>·mg<sup>-1</sup>N. The data clearly show that the released alkalinity is more than sufficient to keep the pH in the appropriate range near its neutral value.

Table 2 also shows that there is a relationship between the concentration of released ammonia and the concentration of digested VSS. This relationship is shown graphically in Fig. 5. It can be seen that there is a production of 0.105 gN per digested VSS. This is in accordance with the fact that the mass fraction of nitrogen in activated sludge is about 10% (Marais and Ekama, 1976). Similarly, Fig. 6 shows the relationship between the alkalinity increase and the digested VSS concentration: The linear relationship has a proportionality constant of 0.28 mgCaCO<sub>3</sub>·mgVSS<sup>-1</sup>. The experimental alkalinity production can be calculated at 2.7 mgCaCO<sub>3</sub>·mg<sup>-1</sup>N, which is less than the stoichiometric value of 3.57 mgCaCO<sub>3</sub>·mg<sup>-1</sup>N. In part this may be explained by the fact that the dissociation equation (Eq. (2)) is incomplete (some ammonia remains as NH<sub>3</sub>). Also other acid base systems (VFA, phosphate, sulphide) may have influence. Since not all these were measured it was not possible to make corrections.

## Conclusions

- The percentage of VSS reduction during anaerobic digestion of waste activated sludge and conversion into biogas depends on its composition. In a digester operated with a retention time of 20 d and a temperature of 25°C the removal efficiency of VSS was 53% of the active sludge fraction in waste activated sludge and 15% for the inactive fraction. The digestion efficiency of the active fraction corresponds to the value already established for primary sludge.

- During the anaerobic digestion process mineralisation of organic nitrogen takes place causing an increase of alkalinity in the digester. A release of 0.105 gN·g<sup>-1</sup>VSS (converted) as ammonia and an alkalinity production of 0.28 mgCaCO<sub>3</sub>·g<sup>-1</sup>VSS (converted) were observed, independent of the sludge composition. The alkalinity production is more than sufficient to maintain a stable pH near the neutral value in the digester without the addition of external alkalinity.

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## Appendix 1 Determination of the active sludge concentration from oxygen uptake rate measurements

Marais and Ekama (1976) developed a model for aerobic sludge digestion in which the waste activated sludge is divided in two fractions: an active fraction composed of live, oxygen consuming organisms; and an inert fraction that has no metabolic activity and therefore does not consume oxygen. The active sludge concentration can be determined from the OUR in sludge samples, when these are aerated without feeding, so that the oxygen consumption is due to endogenous respiration. The fundamental points of the aerobic digestion model are: the active sludge decay is a first order process with respect to the active sludge concentration; and a constant proportion  $f$  of the decayed material is not oxidised in the endogenous respiration process, but instead it accumulates as an inactive solids fraction - the endogenous residue. Hence the oxidation rate of active sludge can be expressed as:

$$r_{ox} = (1-f)b_h X_a \quad (A1)$$

where:

- $r_{ox}$  = sludge oxidation rate (mgVSS·t<sup>-1</sup>·d<sup>-1</sup>)
- $b_h$  = decay constant for active sludge  
= 0.24\*(1.04)<sup>t-20</sup> d<sup>-1</sup> (t em °C)  
= 0.29 d<sup>-1</sup> for the operational temperature of 25 °C.
- $f$  = endogenous fraction remaining upon decay of active sludge  
= 0.2
- $X_a$  = active sludge concentration (mgVSS·t<sup>-1</sup>)

Knowing that there is a proportionality between the mass of volatile sludge and the COD of that mass ( $f_{cv} = 1.5$  gCOD/gVSS, Marais and Ekama, 1976) and that, by definition, there is a consumption of 1 of oxygen for the oxidation of 1 g COD, it is calculated that the OUR due to oxidation of the organic material during aerobic sludge digestion can be expressed as:

$$OUR_c = f_{cv} \cdot r_{ox} = f_{cv} (1-f)b_h X_a \quad (A2)$$

However, it is known that a part of the digested sludge mass is composed of nitrogen which is transformed into ammonia upon mineralisation of the sludge. In the mixed liquor this ammonia can be oxidised to nitrate and thus exert an additional oxygen demand. It has been established that about 10% of the volatile sludge mass is composed of nitrogen. Knowing that the oxygen demand for ammonia oxidation to nitrate is 4.57 mgO·mg<sup>-1</sup>N, one has:

$$OUR_n = 4.57f_n r_{ox} \quad (A3)$$

Hence the total OUR (that can be measured experimentally) can be written as:

$$OUR = OUR_c + OUR_n \quad (A4)$$

$$= (p+4.57f_n)(1-f)b_h X_a$$

Equation (A4) opens the possibility to determine the active sludge concentration from OUR measurements of a sludge sample:

$$X_a = OUR / [(f_{cv} + 4.57f_n)(1-f)b_h] \quad (A5)$$

The active sludge fraction is now calculated as the ratio of the active sludge concentration (from Eq. (A5)) and the measured VSS concentration:

$$f_{av} = X_a / X_v = (OUR / X_v) / [(f_{cv} + 4.57f_n)(1-f)b_h]$$

$$= (SOUR) / [(f_{cv} + 4.57f_n)(1-f)b_h]$$

where:

$$SOUR = \text{specific oxygen uptake rate (mgO} \cdot \text{mg}^{-1} \text{VSS} \cdot \text{d}^{-1})$$

The following experimental values have been measured:

- $f_n$  = 0.1 (Marais and Ekama, 1976) to 0.105 (this paper)
- $b_h$  = 0.24\*1.029<sup>(t-20)</sup> (Marais and Ekama, 1976) (t < 20°C)  
= 0.24\*1.04<sup>(t-20)</sup> (Van Haandel et al. 1986) (t > 20°C)
- $f_{cv}$  = 1.48 mgCOD·mg<sup>-1</sup>VSS (Marais and Ekama, 1976) or  
= 1.50 mgCOD·mg<sup>-1</sup>VSS (this paper)

Using these values and the experimental data in Table 1, for the sludge in aerobic digester R<sub>4</sub> the following composition is calculated:

$$X_a = OUR / [(f_{cv} + 4.57f_n)(1-f)b_h]$$

$$= 4 \cdot 24 / (1.5 + 4.57 \cdot 0.1)(1 - 0.2) \cdot 0.29] = 210 \text{ mg} \cdot \text{t}^{-1}$$

$$= 0.21 \text{ mg} \cdot \text{t}^{-1}$$

$$X_v = 1.26 \text{ g} \cdot \text{t}^{-1}$$

$$\text{Hence: } f_{av} = X_a / X_v = 0.21 / 1.26 = 0.16$$