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Article

Anaerobic Treatment of Concentrated Black Water in a UASB Reactor at a Short HRT

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Abstract: This research describes the feasibility of applying a UASB reactor for the treatment of concentrated black (toilet) water at 25 °C. On average 78% of the influent load of COD at an HRT of 8.7 days was removed. Produced methane can be converted to 56 MJ/p/y as electricity and 84 MJ/p/y as heat by combined heat and power (CHP). Minimum reactor volume at full scale was calculated to be 63L per person (for black water containing 16 gCOD/L produced at 5 L/p/d) and this is more than two times smaller than other type of reactors for anaerobic treatment of concentrated black water.

Keywords: black water; anaerobic treatment; UASB reactor; sanitation; separation at source

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1. Introduction

Separation of domestic waste(water) at the source results in black water from the toilet (faeces and urine) and less polluted grey water from showers, laundry and kitchen. These source separated waste(water) streams differ in quantity and quality and should be treated separately according to their concentrations and composition. The main benefits of such an approach include the possibility of recovering energy and nutrients and the efficient removal of micro-pollutants. Grey water has a high

potential of reuse because it is the major fraction (70%) of domestic wastewater and relatively low in pollution [1]. Black water contains half the load of organic material in domestic wastewater, the major fraction of the nutrients nitrogen and phosphorus [2,3] and can be collected with a small amount of water (one liter per flush) using, for example, vacuum toilets. Black water also contains most of the pathogens, hormones and pharmaceutical residues. The volume of black water depends on the type of toilet and amount of water needed to flush.

Anaerobic treatment is regarded as the core technology for energy and nutrient recovery from source separated black water [3-5] because it converts organic matter to methane, which can be used to produce electricity and heat, while at the same time anaerobic treatment yields low amounts of excess sludge. The nutrients are largely conserved in the liquid phase and can be subsequently recovered with physical-chemical processes such as precipitation and ion-exchange or removed biologically [6,7]. Depending on the distance to agricultural fields, direct reuse of nutrient rich anaerobic effluent is possible if it is treated to remove pathogens and micro-pollutants [8,9].

With an average load of 62 gCOD/p/d and a methanisation level of 60% [10], 12.5 L CH_4/p/d can be produced from black water (0.35 L CH_4 /gCOD, (Standard temperature and pressure (STP))). When solid kitchen refuse is included ((60 gCOD/p/d), [10]) the biogas production can be doubled, resulting in 25 L CH_4/p/d, which represents 335 MJ/p/y (35.6 MJ/Nm^3 CH_4). Combined heat and power (CHP) generation systems can be used to produce heat and electricity at an efficiency of 85% (of which 40% electricity and 60% heat) [11]. This would result in a production of 32 kWh/p/y electricity (2.1% of the electricity consumption in a household (87 PJ electricity consumption in The Netherlands in 2006 [12] *i.e.*, 1487 kWh/p/y)) and 47 kWh/p/y of heat when using the methane produced from black water and solid kitchen refuse.

The use of three types of reactors for anaerobic treatment of black water collected with vacuum toilets at different temperatures is reported in literature, namely a CSTR (continuously stirred tank reactor), an Accumulation system and a UASB-septic tank (Upflow anaerobic sludge blanket).

Wendland *et al*. [13] investigated anaerobic treatment of black water from vacuum toilets in a CSTR operated at mesophilic conditions (37 °C). A removal efficiency of total COD of 61% was achieved at an HRT (Hydraulic retention time) of 20 days. Applying a CSTR for anaerobic treatment of black water (7 L/p/d) requires a volume of 140 L per person [13].

Kujawa-Roeleveld *et al*. [10] investigated anaerobic treatment of black water and kitchen refuse in an accumulation system operated at 20 °C. An accumulation system is a continuously fed reactor and combines digestion and storage in one reactor volume. Stabilization of the black water for 80% was achieved within 150 days. Due to the long storage time a relatively large volume is needed of 1.0 m^3 per person for the treatment of black water. An accumulation system therefore is only suitable for even more concentrated streams (e.g., only faeces (brown water) and kitchen waste) and less suitable for black water [10].

The second system that Kujawa-Roeleveld *et al*. [10,23] investigated was a UASB-septic tank operated at 15 and 25 °C. UASB reactors enable long sludge retention times (SRT) at relatively short hydraulic retention times (HRT), because biomass retention is accomplished by an internal gas/sludge/liquid separation system [4]. A UASB-septic tank is a continuous reactor with respect to the liquid, but accumulates the solids, combining the features of a UASB reactor and a septic tank. The UASB-septic tank removed 61% of the total COD at 15 °C and 78% of the total COD at 25 °C. For

sludge stabilization and total reduction of volatile fatty acids (VFA) at 25 °C a minimum volume of 200 L per person is needed, corresponding to an HRT of about 30 days [10].

The reactors mentioned above require relatively large volumes per person (Table 1). Unlike the UASB-septic tank, a UASB reactor without additional space for the accumulation of solids (no septic tank) would require regular sludge removal, but it will reduce the volume of the reactor [4]. This is important for application at larger scale where space might be limited.

Table 1. Reactors for anaerobic treatment of concentrated black water.

	CSTR [13]	Accumulation system [10]	UASB-septic tank [10,23]	
Temperature (°C)	37	20	15	25
Total COD removal (%)	61	80	61	78
HRT (d)	20	150	30	30
SRT (d)	20	150	>365	>365
Volume required (L/p)	140	$1.0*10^3$	n.d.	200
Methanisation* (%)	60	58	39	60

n.d. = not determined

*calculation based on obtained methane production and influent load

UASB reactors so far have not been investigated for their capability to treat concentrated wastewater streams such as black water and was only shortly discussed by Zeeman *et al*. [14]. The volume of a UASB reactor will depend on the minimum SRT required to achieve methanisation and stabilization of the sludge [4]. For the anaerobic treatment of black water hydrolysis of particulate organic substrates is the rate-limiting step [15]. With first order kinetics and a hydrolysis constant of 0.1 d^{-1} (average value at 20–30 °C [16]) it can be calculated that a high percentage of hydrolysis (between 80 and 90%) can be achieved at a SRT between 40 and 90 days. Other research showed as well that the minimum SRT was estimated to be 75 days at 25 °C to achieve methanisation and stabilization of the sludge [4,17]. Other factors that are important for the anaerobic treatment of black water are the temperature and inhibition by free ammonia [18]. Luostarinen *et al*. [19] investigated the effect of temperature on anaerobic treatment of black water in UASB-septic tanks. The temperature had no significant effect on suspended solids removal, but the removal of dissolved COD improved because sludge adapted to lower temperatures (15 °C) [19]. The black water can be produced at a temperature of about 20 °C [20]. A higher temperature could result in a shorter HRT, but this would require extra energy requirements for heating the black water. Therefore a temperature of 25 °C was selected for the treatment of black water in a UASB reactor. In concentrated black water high concentrations of ammonium (0.8–1.4 gNH_4-N/L) are present which can inhibit methanisation and therefore higher retention times could be needed to achieve a maximum production of methane [21].

This paper describes the feasibility of applying a compact UASB reactor for the treatment of concentrated black water from vacuum toilets at these conditions. Furthermore the design of the UASB reactor will be discussed, as well as the minimum volume needed at full scale.

2. Materials and Methods

2.1. Black water collection

Black water, collected in vacuum toilets, was obtained from the DESAR (Decentralized Sanitation and Reuse) demonstration site in Sneek (Friesland, NL) [22]. Every two weeks jerry cans were filled with black water from the buffer tank at the demonstration site (hydraulic retention time of 4 h, not cooled), transported to the lab and stored at 4 °C. Black water was pumped from a stirred, cooled (6 °C) influent tank into the UASB reactor with a Masterflex L/S peristaltic pump. A course filter (4–5 mm holes) in the influent tank prevented clogging of the inlet tube.

2.2. UASB reactor

A 50 L flocculent sludge UASB (Upflow Anaerobic Sludge Blanket) reactor (Figure 1) was operated for 951 days at 25 °C to produce biogas from black water. The reactor was made of a transparent Perspex/Plexiglas tube (height: 1.30 m and inner diameter 0.20 m) with a double wall for temperature control. Temperature was controlled with a thermo stated water bath (Haake DC10/K10). The top was made of non-transparent plastic (polypropylene) and served as a gas/solid/liquid separator (height 0.19 m and width 0.22 m). Five taps at different heights (0, 0.26, 0.52, 0.78 and 1.02 m) enabled sludge sampling. Liquid effluent and gas were collected at the top. Gas production was monitored with a gasflow meter (Ritter TG05/5). A magnetic stirrer (VarioMag Mobil) at the bottom of the UASB reactor provided an even distribution of the influent through the sludge bed. The magnetic stirrer only mixed the bottom section of the sludge bed. The reactor was inoculated with 20 L anaerobic sludge (1.1 gVSS/L) from a UASB-septic tank treating concentrated black water at a temperature of 25 °C and a loading rate of 0.42 kgCOD/m^3/d [23].

2.3. Design of the UASB reactor

The HRT to be applied in the UASB reactor was calculated using the following equation proposed by Zeeman and Lettinga [4]:

$$HRT = C \cdot \frac{SS}{X} \cdot R \cdot (1 - H) \cdot SRT \quad (1)$$

where C is the COD concentration in the influent (COD_{total}, in gCOD/L), SS is the fraction of suspended solids in the influent (COD_{SS}/COD_{total}), X is the sludge concentration in the reactor (in gCOD/L), R is the fraction of COD_{SS} removed and H is the level of hydrolysis of the removed solids.

Values of C, SS, X, R and H were taken from the research of Kujawa-Roeleveld [16] and this resulted in a design HRT of 6.9 days (Table 2).

Initially the reactor was operated at a longer HRT of 14 days to prevent accumulation of volatile fatty acids (VFA). The first 200 days were used as start-up period and the HRT was subsequently reduced in steps (every 5-6 weeks) when no VFA accumulation was observed. The average HRT achieved was 8.7 days, but fluctuated between 5.8 and 13 days due to silting of the influent tube. The influent tube was cleaned monthly to remove the silted solids.

Sludge was removed regularly from tap 4 at a height of 1.02 m to maintain a maximum sludge bed height of 75% of the reactor volume.

Figure 1. UASB (Upflow Anaerobic Sludge Blanket) reactor treating concentrated black water.

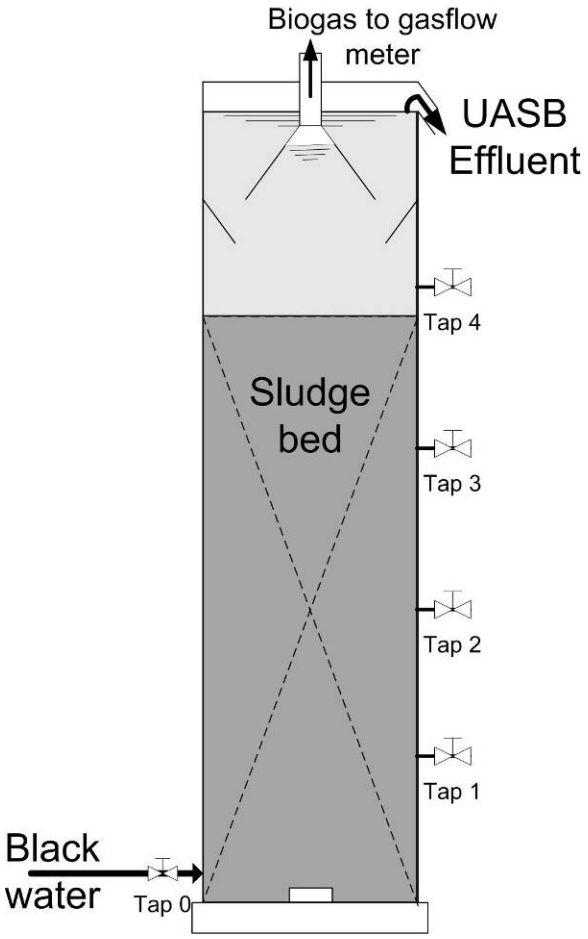


Table 2. Initial design values for the UASB reactor.

	Unit	Remarks	Initial design of UASB reactor
SRT	d	Minimum value at 25 °C	75
C	gCOD/L	COD_{total} in the influent	12
SS	-	CODss/CODt influent	0.78
X	gCOD/L	Sludge concentration in the reactor	28
R	-	Fraction of CODss removed	0.9
H	-	Level of hydrolysis	0.7
HRT	d		6.9

2.4. Analyses and measurements

Every week influent and effluent composition was analyzed (125 samples in total) immediately after sample collection. Influent was collected from a tap just before the inlet of the UASB reactor (tap 0, Figure 1) and effluent was collected during 3 hours in the morning (about 0.5–1 L for both samples). COD_{total} was determined from unfiltered samples, filtered COD (COD_f) was determined from paper filtered samples (black ribbon paper filter (Schleicher & Schuell)) and soluble COD ($COD_{soluble}$) was determined from membrane filtered samples (0.45 µm membrane filter (Cronus filter PTFE)) using DrLange kits (LCK514). Total Nitrogen (TN) en Total Phosphorus (TP) were determined from unfiltered samples using DrLange kits (LCK238 and LCK350). DrLange kits LCK302 and LCK303 were used to determine the total ammonia nitrogen concentration (NH_4-N) in paper filtered samples. Ion chromatography (Metrohm 761 Compact IC) was used to measure anions concentrations (Cl^-, NO_3^- NO_2^-, SO_4^{2-} and PO_4^{3-}) and volatile fatty acids (VFA: acetic acid, propionic acid and butyric acid) in membrane filtered samples. ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectroscopy) was used to measure concentrations of the element phosphorus in the membrane filtered sample. Inorganic carbon (IC) was determined with a Shimadzu TOC analyzer by difference from the measured total carbon (TC) and non-purgeable organic carbon (NPOC) in the paper filtered sample. Total Suspended Solids (TSS) and Volatile Suspended Solids (VSS) were determined according to standard methods using black ribbon ashless paper filter (Schleicher & Schuell) [24]. Biological oxygen demand (BOD) of the UASB effluent was determined using OxiTop heads calibrated for BOD determination. Depletion of oxygen was monitored for five days (BOD_5) (17 samples in total). Biogas composition (sample of 5 mL, 43 samples in total) was analyzed with gas chromatography (Shimadzu GC-2010 Gas Chromatograph containing GS-Q (CO_2) and HP molsieve (O_2, N_2, H_2S and CH_4) columns). Wasted sludge (51 samples in total) was analyzed for TSS and VSS and total COD using the methods indicated above. Maximum biodegradability of the black water (2 samples in total) and stability of the UASB sludge (13 samples in total) was tested in closed bottles with Oxitop pressure measuring heads by incubation at 37 °C [16]. The development of the sludge bed was analyzed by taking sludge samples from every tap (0.2 L) and these samples were analyzed for TSS and VSS and total COD (10 times 5 samples in total). The flow rate in the UASB was measured by weighing the collected effluent over a certain period of time and this was used to calculate the HRT.

2.5. Calculations

The concentration of suspended solids COD (COD_{SS}) was calculated as the difference between COD_{total} and COD_f. The concentration of colloidal COD ($COD_{colloidal}$) was calculated as the difference between COD_f and $COD_{soluble}$. The SRT in the UASB (SRT_{UASB}) was calculated using the following equation:

$$SRT_{UASB} = \frac{solids_{reactor}}{solids_{washed,out} + solids_{wasted}} \quad (2)$$

where $solids_{reactor}$ is the amount of solids in the reactor (gVSS), $solids_{washed,out}$ is the amount of solids that washed out with the effluent (gVSS/d) and $solids_{wasted}$ is the amount of solids that was wasted manually (gVSS/d).

The total amount of sludge in the reactor was calculated using the analyzed concentration and a volume of 1/6 of 50L as each tap is evenly distributed over the reactor (including effluent 'tap'):

$$solids_{\text{reactor}} = \sum_i \left(x_i \cdot \frac{1}{6} \cdot 50 \right) \tag{3}$$

where x_i is the sludge concentration in gVSS/L of each tap i and i is 0, 1, …, 5.

The COD mass balance was calculated by adding up all measured incoming COD (influent and inoculum sludge) and measured outgoing COD (produced methane, sludge in reactor, wasted sludge and effluent) over the total period of operation including the start-up period.

$$\begin{aligned} &\sum CODIN = \sum CODOUT \\ &COD_{\text{total,influent}} + COD_{\text{inoculumsludge}} = COD_{\text{methane}} + COD_{\text{sludge,reactor}} + COD_{\text{sludge,wasted}} + COD_{\text{total,effluent}} \end{aligned} \tag{4}$$

The amount of produced methane-COD was calculated from the average measured biogas composition, the average gas flow rate (L/d) and a conversion factor of 2.6 gCOD / L CH_4 at 25 °C at standard pressure.

The reported level of methanisation in the UASB reactor was calculated as the percentage of cumulative methane-COD production from the cumulative load of influent COD over the total period of operation.

The level of hydrolysis of solids was determined with the following formula [17] :

$$hydrolysis = \frac{COD_{\text{CH}_4} + COD_{\text{soluble,effl}} - COD_{\text{soluble,in}}}{COD_{\text{total,in}} - COD_{\text{soluble,in}}} \tag{5}$$

where COD_{CH4} is the methane produced, $COD_{soluble,effl}$ is the soluble COD in the effluent, $COD_{soluble,in}$ is the soluble COD in the influent and $COD_{total,in}$ is the total COD load in the influent (all in gCOD).

The rate of hydrolysis was estimated using first order kinetics: $\frac{dF_{\text{degr}}}{dt} = -k_h \cdot F_{\text{degr}}$ [15,25].

Assuming the sludge bed as a CSTR, the following equation can be derived:

$$\frac{F_{\text{degr}}}{F_{\text{degr,0}}} = \frac{1}{1 + k_h \cdot SRT} \tag{6}$$

where F_{degr} is the amount of biodegradable solids in the sludge bed, $F_{degr,0}$ is the amount of biodegradable solids in the influent and k_h is the hydrolysis constant. $1 - \frac{F_{\text{degr}}}{F_{\text{degr,0}}}$ is representative for the hydrolysis of suspended solids and therefore representative for the stabilization of the sludge.

The bicarbonate concentration was calculated from the total inorganic carbon (IC) as a function of pH and temperature [18].

3. Results

3.1. Performance of the UASB reactor

The composition of the black water influent to the UASB reactor is shown in Table 3. The black water was more diluted in the second period of operation because more flushing water was used in the vacuum toilets due to installation of noise reducers (Table 3) (resulting in a black water production of 7.8 L/p/d instead of 5 L/p/d, [26]).

The maximum biodegradability of the black water was 55% after 70 days of incubation at 37 °C. In Figure 2 the influent and effluent COD concentrations and the total COD removal are shown. The UASB reactor removed an average of 74% of the influent load of COD.

Table 3. Composition of the black water influent.

		Day 1 – 518		Day 519 – 951	
	Unit	Influent	s.d.	Influent	s.d.
pH	-	8.8	0.22	8.6	0.53
COD_{total}	[gCOD/L]	9.8	2.6	7.7	2.5
COD_{SS}	[gCOD/L]	5.1	2.7	4.9	2.0
$COD_{colloidal}$	[gCOD/L]	1.3	0.42	0.5	0.22
$COD_{soluble}$	[gCOD/L]	3.4	0.47	2.3	0.81
VFA	[gCOD/L]	1.5	0.48	1.2	0.89
HCO_3^-	[gC/L]	1.2	0.37	0.67	0.20
TN	[gN/L]	1.9	0.19	1.2	0.18
NH_4-N	[gN/L]	1.4	0.15	0.85	0.15
TP	[gP/L]	0.22	0.067	0.15	0.064
TP soluble	[gP/L]	0.090	0.0087	0.057	0.018
PO_4-P	[gP/L]	0.079	0.0085	0.054	0.027

s.d. = standard deviation

The removal efficiency varied between 42 and 94% as shown in Figure 2, and became stable at a value of about 80% from day 500 onward. On average 10 L/d of biogas was produced, consisting of 78% (s.d. 5.8%) CH_4, 22% (s.d. 5.7%) of CO_2 and traces of H_2S (<0.5%).

In Figure 3 the HRT in the UASB is plotted, together with the VFA concentration in the effluent. A few times VFA concentrations in the effluent increased, showing that anaerobic degradation was incomplete. The increase in VFA was always accompanied by a drop in COD removal, but never resulted in complete inhibition of biogas production. Furthermore a thick scum layer in the gas/solid/liquid separator was observed when the VFA concentration increased in the effluent (Figure 3). This scum layer was transferred back to the bottom of the reactor and was no longer observed afterwards.

Figure 2. Influent and effluent COD concentrations and the COD total removal of the UASB reactor.

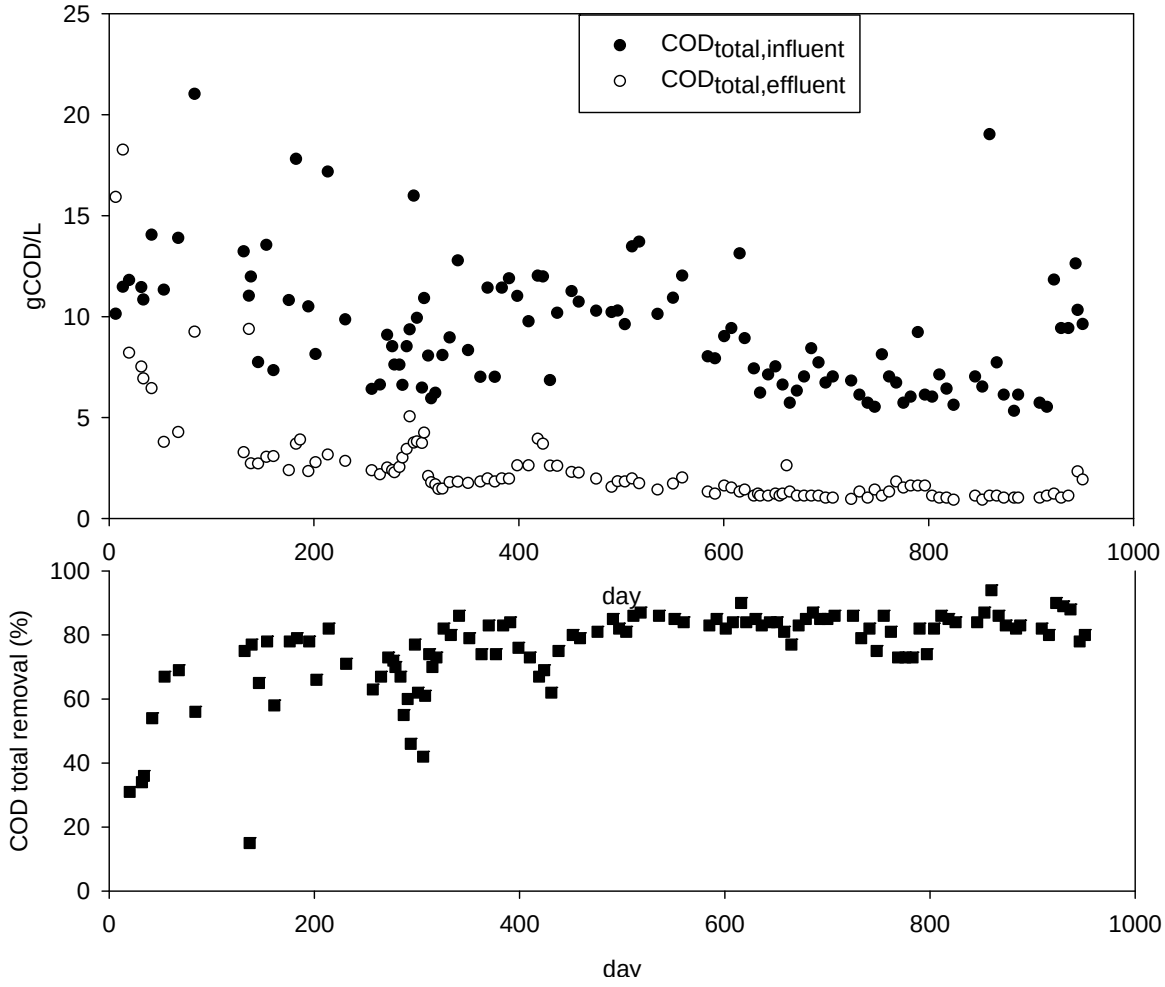
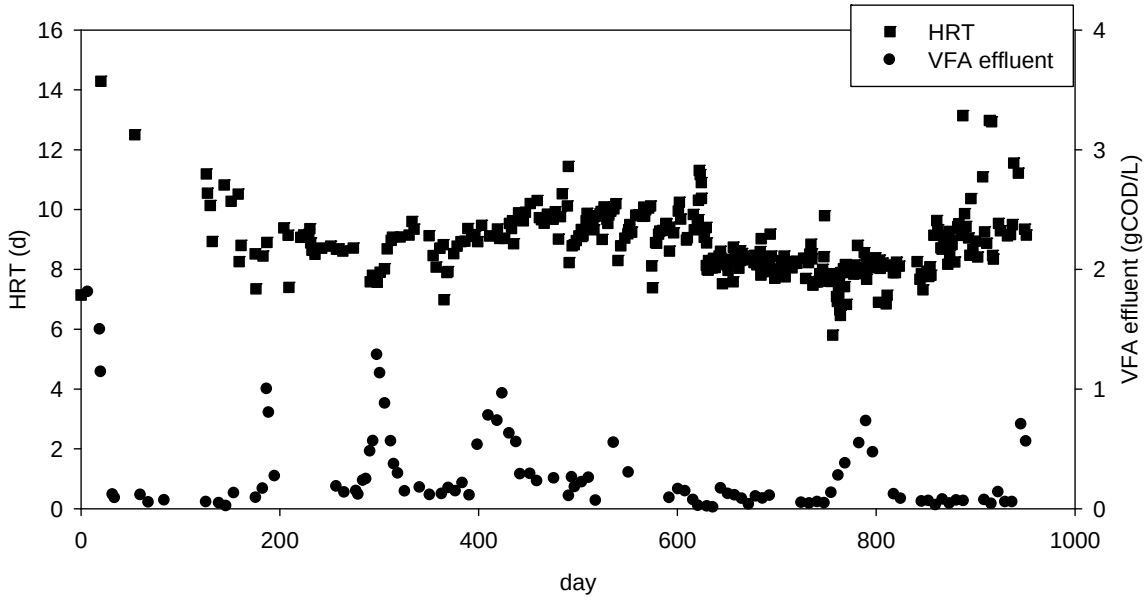


Figure 3. HRT and VFA in UASB effluent.



3.2. Sludge bed development

Figure 4 shows that the sludge bed developed to a compact sludge bed with concentrations of up to 45 g VSS/kg sludge. In the first few weeks, part of the inoculum sludge was washed out with the effluent, which explained the high effluent COD concentrations of 16-18 gCOD/L (Figure 2). Gradually the sludge adapted to a higher upflow velocity and the sludge bed increased in volume and concentration. After day 800 sludge had to be wasted less frequently because the sludge concentration increased. The ratio of VSS/TSS of the wasted sludge decreased after day 800 from 0.80 to 0.68 (Figure 4).

The SRT was estimated at 254 days and on average the reactor contained 19 $gVSS/L_{reactor}$(s.d. 4.0) and 34 $gCOD/L_{reactor}$ (s.d. 8.0). The wasted sludge from the UASB reactor showed a stability of 91% in 106 days at 37 °C, which means that only 9% of the COD in the UASB sludge could still be converted to methane. The percentage of influent suspended solids that were hydrolyzed and converted to methane was 53%.

The structure and the colour of the sludge changed from black, fine flocculant sludge in the inoculum to a brown, compact muddy sludge. This change in colour was probably due to leaching of iron, because the inoculum sludge originated from a UASB-septic tank where iron was added to precipitate phosphate (data not shown).

Figure 4. Sludge bed development (upper graph), the amount of sludge wasted (lower graph) and VSS/TSS ratio of the wasted sludge (lower graph).

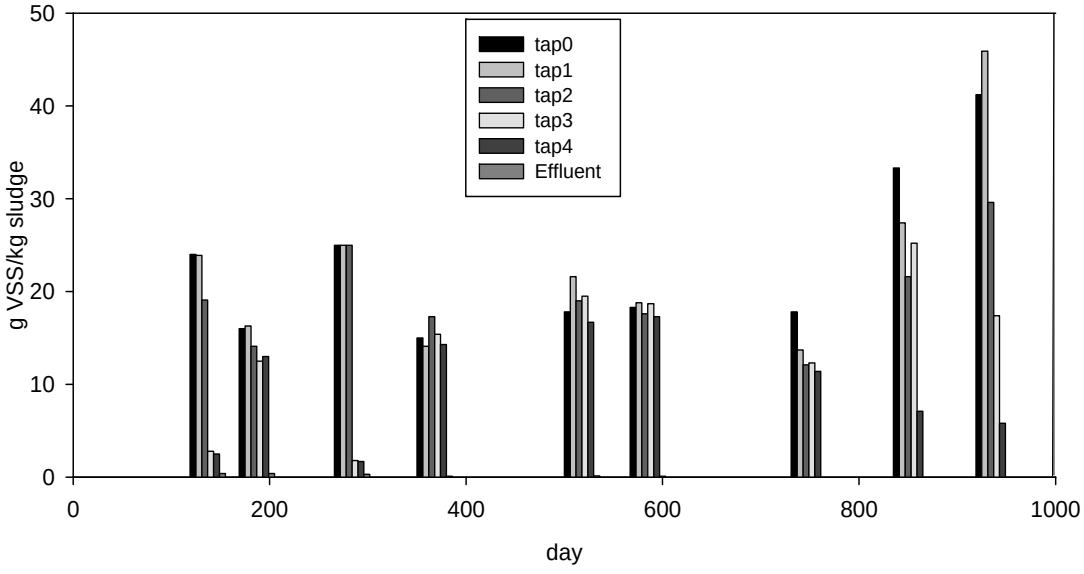
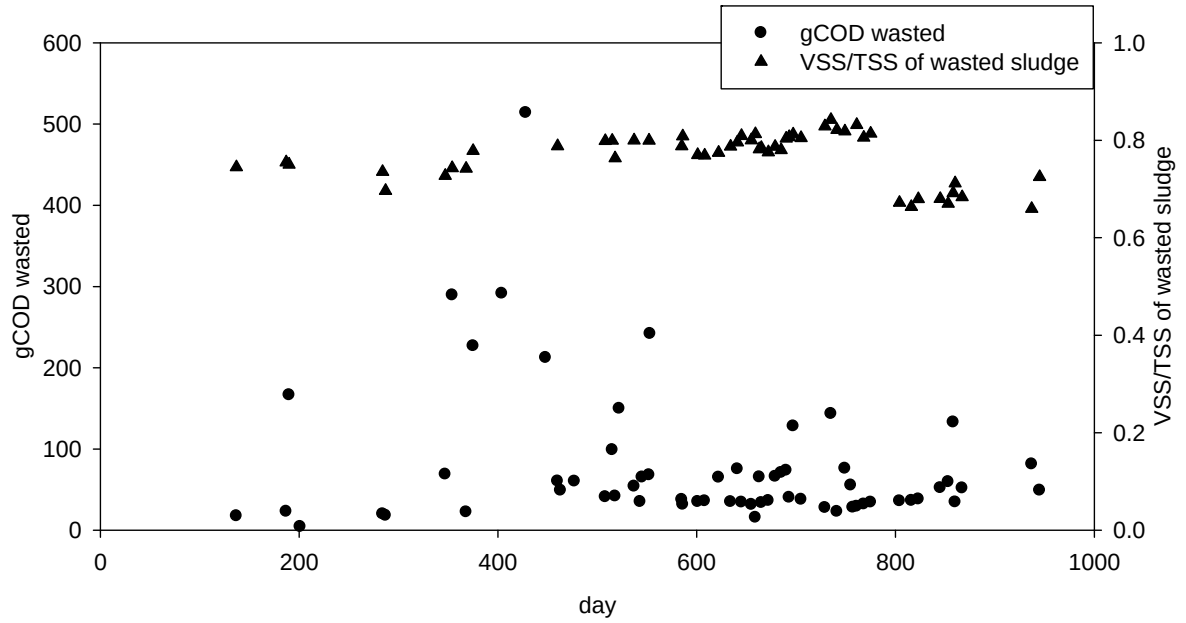


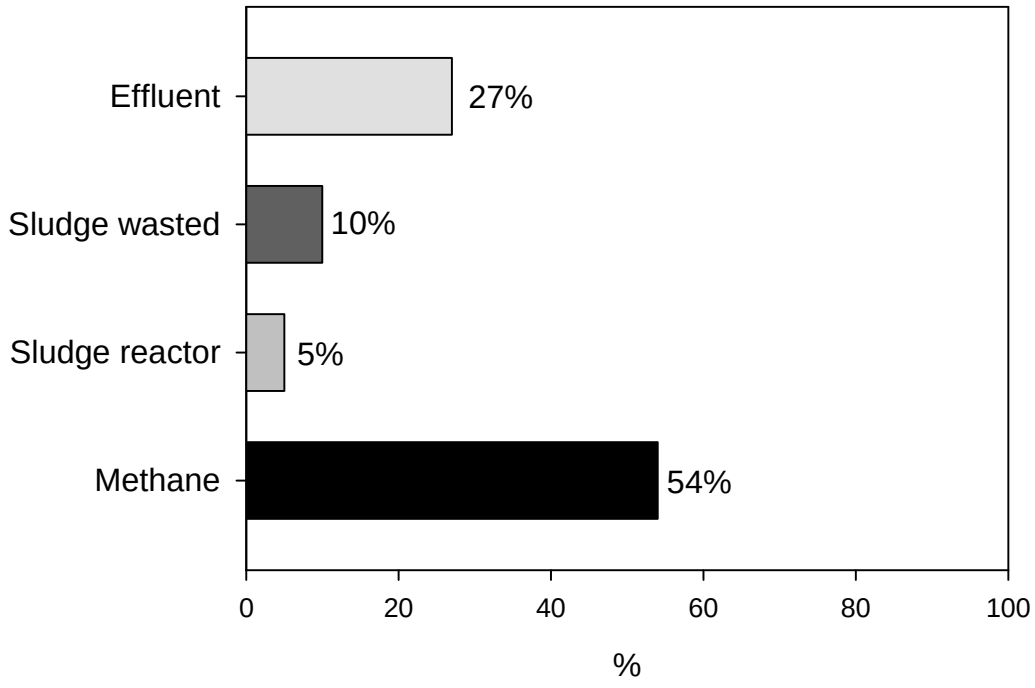
Figure 4. *Cont.*



3.3. COD mass balance

The COD mass balance over the total period of operation (951 days) showed only a small difference between total incoming COD and total outgoing COD (4%) (Figure 5), which probably can be explained by errors introduced by sampling and analyses. Maximum 1% of total incoming COD left the reactor with effluent as dissolved methane (using a maximum solubility of 22 mg CH_4/L, [27]).

Figure 5. COD mass balance over the total period of operation (951 days). The total amount of COD fed to the reactor during this period was 49 kgCOD, of which 0.1% was inoculum sludge.



3.4. Effluent composition

Table 4 shows the effluent composition of the UASB reactor. The pH dropped from 8.8 / 8.6 (Table 3) in the influent to 7.6 / 7.4 in the effluent of the UASB during the anaerobic treatment.

A fraction of the COD in the effluent was still biodegradable, because the effluent contained a BOD_5 concentration of 0.87 g/L in the first period and 0.48 g/L in the second period.

Nitrogen was conserved for more than 91% in the liquid effluent, mainly as ammonium, and the effluent contained 1.8 gTN/L (of which 1.5 gNH_4-N/L) in the first period and 1.2 gTN/L (of which 1.0 gNH_4-N/L) in the second period. Phosphorus was conserved for 61% in the effluent, mainly as phosphate, and the effluent contained 0.13 gTP/L (of which 0.092 gPO_4-P/L) in the first period and 0.094 gTP/L (of which 0.069 gPO_4-P/L) in the second period.

Table 4. UASB effluent composition.

		Day 1 – 518		Day 519 – 951	
	Unit	UASB effluent	s.d.	UASB effluent	s.d.
pH	-	7.6	0.13	7.4	0.17
COD_{total}	[gCOD/L]	2.4	0.84	1.2	0.34
COD_{SS}	[gCOD/L]	0.43	0.43	0.10	0.08
$COD_{colloidal}$	[gCOD/L]	0.53	0.27	0.21	0.07
$COD_{soluble}$	[gCOD/L]	1.5	0.39	0.90	0.21
VFA	[gCOD/L]	0.36	0.30	0.14	0.18
BOD_5	[g/L]	0.87	0.24	0.48	0.06
HCO_3^-	[gC/L]	1.4	0.14	0.87	0.10
TN	[gN/L]	1.8	0.22	1.2	0.12
NH_4-N	[gN/L]	1.5	0.19	1.0	0.18
TP	[gP/L]	0.13	0.015	0.094	0.018
TP soluble	[gP/L]	0.103	0.010	0.070	0.011
PO_4-P	[gP/L]	0.092	0.011	0.069	0.013

s.d. = standard deviation

4. Discussion

4.1. Removal efficiency of the UASB reactor

This research shows that anaerobic treatment of concentrated black water can successfully be achieved in a UASB reactor. Generally the reactor exhibited a stable operation and removed more than 78% of the incoming COD. On three occasions VFA accumulated in the effluent and this was probably due to a sudden increase in load due to higher influent concentrations. In those periods scum formation was observed, which was probably caused by the reduced conversion efficiency of the anaerobic treatment process that led to a temporary accumulation of VFA [17]. In the concentrated black water the free ammonia concentration was as high as 485 $mgNH_3$-N/L (25 °C, pH 8.8, 1.4 gNH_4-N/L) and reduced in the effluent to 35 $mgNH_3$-N/L (25 °C, pH 7.5, 1.5 gNH_4-N/L). It is likely that inside the

UASB reactor a pH gradient exists and shock loads may have caused a temporary inhibition of the methanogenesis by free ammonia [21], resulting in a reduced efficiency. The decrease in pH in the UASB reactor was probably caused by the production of CO_2 which dissolves in the liquid phase. The concentrated black water, however, contains enough alkalinity and pH control is therefore not necessary [18].

Almost all suspended solids were retained in the reactor (93%, Table 5) and high sludge bed concentrations were obtained. After day 800 the sludge contained more inorganic material than before because the ratio of VSS/TSS of the wasted sludge decreased from 0.80 to 0.68 and the sludge bed concentrations increased from about 20 gVSS/kg sludge to 30–45 gVSS/kg sludge (Figure 4). This was probably because the concentrated black water contained more calcium than before (80 mg/L instead of 40 mg/L) and this caused more inorganic precipitation with carbonate and/or phosphate than before [25].

Table 5. Operational characteristics of anaerobic treatment of concentrated black water at 25 °C; (standard deviation).

	Unit	UASB reactor This research	UASB-septic tank [10,23]	Pilot plant UASB-septic tank [20]
Influent	-	Black water, vacuum toilets, DESAR pilot plant Sneek, Filtered with a coarse filter	Black water, vacuum toilets, Wageningen University	Black water, vacuum toilets, DESAR pilot plant Sneek
Reactor volume	L	50	200	7400
Up flow velocity	Cm/h	0.76	0.23	0.42
Loading rate	kgCOD/m^3/d	1.0	0.42	0.36
HRT	Days	8.7 (0.96)	29	30
SRT	Days	254	>365	>365
COD_{total} removal	%	78 (9%)	78	87
COD_{SS} removal	%	93 (11%)	94	95
Methane production	L CH_4/p/d	10	14	13
	m^3 CH_4/m^3 BW	1.8	2.0	2.1

BW = black water

In Table 5 the operational characteristics of the UASB reactor are compared with the operation in a UASB-septic tank. In comparison with a lab scale UASB-septic tank a similar removal efficiency (78%, Table 5) and effluent quality (not shown) were obtained, but the applied loading rate in the UASB reactor was much higher (1.0 kgCOD/m^3/d) than in the UASB-septic tank (0.42 kgCOD/m^3/d). Recently the anaerobic treatment of concentrated black water was also investigated at pilot scale in a

UASB-septic tank by Meulman *et al*. [20], but the applied loading rate was much lower (0.36 kgCOD/m^3/d). The removal efficiency in this UASB-septic tank was higher (87%, Table 5), because the concentrated black water did not pass a coarse filter as within this study and therefore contained a higher amount of suspended solids [20].

4.2. Design of the UASB reactor

Design values from Lettinga and Hulshoff Pol [28] show that treatment in a UASB is not recommended for wastewaters with high amount of suspended solids and high concentrations of organic material. These design values do not take into account that hydrolysis is the rate-limiting process and therefore a long SRT is needed for stabilization of the solids and sludge inside the UASB. Based on the fact that the hydrolysis is the rate limiting step, Zeeman and Lettinga [4] derived the formula for calculation of the HRT (equation 1).

An unexpected long SRT of 254 days was achieved in the present research and a large amount of sludge could be retained in the reactor (average of 19 gVSS/$L_{reactor}$, 34 gCOD/L). Because of this long SRT, the hydrolysis of influent suspended solids (53%) is close to the maximum biodegradability of the suspended solids in the black water (55%). Assuming that the same amount of sludge can be retained in the reactor, the design of the UASB reactor can be optimized. A shorter SRT will result in a lower percentage of hydrolysis and therefore less stabilized sludge, which is illustrated hypothetically in Figure 6. A shorter SRT also means a shorter HRT and therefore the reactor volume can be further reduced at the expense of less stabilized sludge and less biogas production. This can be illustrated using equations 1 and 6. For example a reduction of the volume by reducing the SRT to 75 days will result in 8.2% reduction of the hydrolysis (1-F/F0 reduces from 96.4% to 88.2%), resulting in a hydrolysis of 49%. This reduction in hydrolysis means that 4% of the suspended solids are not hydrolysed and converted to methane. On the total methane production this is a reduction of 5%. As was described in the introduction, the minimum SRT was estimated to be 75 days at 25 °C to achieve methanisation and sufficient stabilization of the sludge [4,17].

A large reduction in volume can be achieved at the expense of a small reduction in hydrolysis of the suspended solids. Using equation 1 the minimum reactor volume for application at full scale can be estimated. Values for X, SS, R and H were adjusted using the results of this research and the conditions at full scale (Table 6). The concentrated black water composition reported by Meulman *et al*. [20] was used, because in this research it appeared to be impossible to feed all the suspended solids of the black water to a lab scale reactor. Furthermore Meulman *et al*. [20] showed that the production of concentrated black water is only 5 L/p/d instead of the expected 7 L/p/d. Using adjusted values for the parameters of equation 1 the minimum reactor volume for application at full scale would be 63 L/p (based on 5 L/p/d) corresponding to an organic loading rate of 1.3 kgCOD/m^3/d (Table 6). This is a significantly lower reactor volume and higher load than when a CSTR or UASB-septic tank is applied for the anaerobic treatment of concentrated black water (Table 1). Application in practice is needed to confirm these estimations. Currently this is tested at the DESAR demonstration site in Sneek (the Netherlands). The non-hydrolyzed suspended solids left in the wasted sludge could be used for aerobic composting, including garden waste, to remove pathogens and to produce a soil conditioner [29] provided that it is safe with respect to heavy metals and micro-pollutants [30].

Figure 6. Stabilisation of the sludge represented by 1-F/F0 as a function of the SRT (equation 6, hypothetical, k_h is 0.1 d^{-1}).

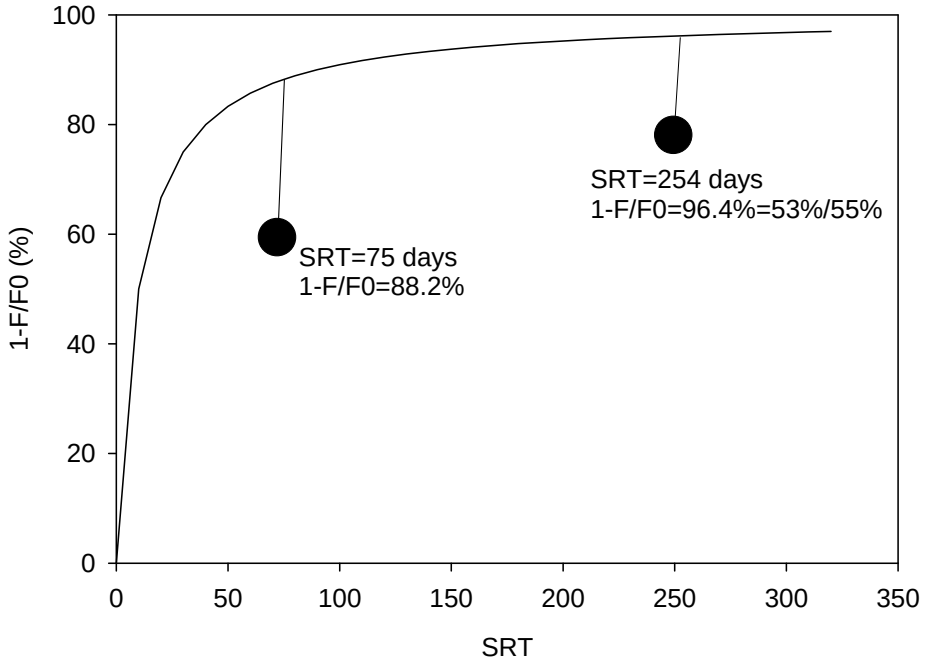


Table 6. Adjusted design values for the UASB reactor.

		UASB reactor full scale
SRT	(d)	75
C	COD concentration in the influent (gCOD/L)	16.1[a]
SS	CODss/CODt influent (-)	0.75[a]
X	sludge concentration in the reactor (gCOD/L)	34.2
R	fraction of CODss removed (-)	0.93
H	fraction of removed solids which is hydrolyzed (-)	0.49
HRT	(d)	12.6
V	(L/p)	63
Loading rate	($kgCOD/m^3/d$)	1.3

[a] using data from Meulman *et al*. [20]

4.3. Fate of nutrients and further treatment

When direct reuse in agriculture of the anaerobic effluent is not feasible because of long distances between cities and agriculture, treatment of the anaerobic effluent is required before the wastewater can be discharged to surface waters. The effluent of the UASB reactor still contains 1.2–2.4 gCOD/L, of which part could be removed by aerobic treatment. Furthermore this stream still contains pathogens and hormones and pharmaceutical residues.

Nitrogen was for more than 91% conserved in the effluent, mainly in the form of ammonium. Because nitrogen is not a limiting compound the choice to recover or remove nitrogen from wastewater will depend on the energy requirements. For the nitrogen concentration in black water (about 1.5 kgN/m^3) biological removal is preferred [31]. Techniques such as a one or two reactor nitritation-anammox process are available to remove ammonium from wastewater streams with a low COD/N ratio [32] and results show that a one or two reactor nitritation-anammox process is suitable to treat the effluent from the UASB reactor [33,34].

For phosphorus a conservation of 60% was observed. The removal of 40% is mainly due to the removal of suspended solids. Probably part of the released phosphate during degradation of the solids directly precipitated in the UASB reactor [35]. Detailed analysis of the sludge fraction is needed to close the phosphorus balance. Similar phosphorus conservation in the effluent was found by Kujawa-Roeleveld *et al*. [23] in the UASB-septic tank. The degree of phosphorus conservation is most likely related to the pH and degree of dilution of the black water. In a UASB reactor on more diluted black water a much higher phosphorus conservation in the liquid effluent stream of 95% was observed (toilets used 5L of water per flush) [36]. Phosphorus is a limiting resource and the available phosphorus resources become increasingly scarce [6,37]. Depending on the degree of phosphorus conservation during the anaerobic treatment, phosphorus can be recovered from the sludge or from the effluent in the form of struvite ($MgNH_4PO_4.6H_2O$) [38]. Preliminary results show that struvite precipitation in the UASB effluent occurred at relatively low pH of 8 by addition of magnesium.

Further research will focus on the subsequent removal and recovery of the nutrients nitrogen and phosphorus and on the removal of the remaining micro-pollutants such as hormones and pharmaceutical residues.

5. Conclusions

Anaerobic treatment of concentrated black water in a UASB reactor was successfully achieved at a HRT of 8.7 days and a load of 1.0 $kgCOD/m^3/d$. Due to high sludge concentrations (19 $gVSS/L_{reactor}$) a long SRT of 254 days was achieved and 53% of the suspended solids were hydrolyzed to methane. The effluent of the UASB reactor needs further treatment to remove remaining COD (2.4 gCOD/L, BOD_5 is 0.87 g/L) and to remove and/or recover nutrients nitrogen and phosphorus.

On the expense of a lower hydrolysis of 49% and some less stabilized sludge, the design of the UASB reactor was optimized and the minimum reactor volume at full scale was calculated to be 63 L/p for black water containing 16 gCOD/L produced at 5 L/p/d.

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References

1. Hernandez Leal, L.; Zeeman, G.; Temmink, H.; Buisman, C. Characterisation and biological treatment of greywater. *Water Sci. Technol.* **2007**, *56*, 193-200.
2. Otterpohl, R.; Albold, A.; Oldenburg, M. Source control in urban sanitation and waste management: ten systems with reuse of resources. *Water Sci. Technol.* **1999**, *39*, 153-160.
3. Kujawa-Roeleveld, K.; Zeeman, G. Anaerobic treatment in decentralised and source-separation-based sanitation concepts. *Rev. Environ. Sci. Bio/Technol.* **2006**, *5*, 115-139.
4. Zeeman, G.; Lettinga, G. The role of anaerobic digestion of domestic sewage in closing the water and nutrient cycle at community level. *Water Sci. Technol.* **1999**, *39*, 187-194.
5. Verstraete, W.; Morgan-Sagastume, F.; Aiyuk, S.; Waweru, M.; Rabaey, K.; Lissens, G. Anaerobic digestion as a core technology in sustainable management of organic matter. *Water Sci. Technol.* **2005**, *52*, 59-66.
6. Driver, J.; Lijmbach, D.; Steen, I. Why recover phosphorus for recycling, and how? *Environ. Technol.* **1999**, *20*, 651-662.
7. Maurer, M.; Pronk, W.; Larsen, T.A. Treatment processes for source-separated urine. *Water Res.* **2006**, *40*, 3151-3166.
8. Halalsheh, M.; Abu Ghunmi, L.; Al-Alami, N.; Fayyad, M. Fate of pathogens in tomato plants and soil irrigated with secondary treated wastewater. In Proceedings of the Sanitation Challenge, Wageningen, The Netherlands, May 2008, 2008.
9. Huibers, F.P.; van Lier, J.B. Use of wastewater in agriculture: the water chain approach. *Irrig. Drain.* **2005**, *42*, S3-S9.
10. Kujawa-Roeleveld, K.; Elmitwalli, T.; Zeeman, G. Enhanced primary treatment of concentrated black water and kitchen residues within DESAR concept using two types of anaerobic digesters. *Water Sci. Technol.* **2006**, *53*, 159-168.
11. van den Berg, W.J. KNN Advies, Efficiency of combined heat and power. Personal communication, 2008.
12. CBS-Statline. *Energy balance, energy consumption of households*, 2006. http://statline.cbs.nl/StatWeb/publication/?VW=T&DM=SLNL&PA=70846ned&D1=0-1,3-4,30-33&D2=30&D3=5&D4=a&HD=080606-2108&HDR=T&STB=G3:.(accessed on 6 June 2008).
13. Wendland, C.; Deegener, S.; Behrendt, J.; Toshev, P.; Otterpohl, R. Anaerobic digestion of blackwater from vacuum toilets and kitchen refuse in a continuous stirred tank reactor (CSTR). *Water Sci. Technol.* **2007**, *55*, 187-194.
14. Zeeman, G.; Kujawa, K.; De Mes, T.; Hernandez, L.; de Graaff, M.S.; Mels, A.; Meulman, B.; Temmink, H.; Buisman, C.; van Lier, J.; Lettinga, G. Anaerobic treatment as a core technology for energy, nutrients and water recovery from source separated domestic waste(water). *Water Sci. Technol.* **2008**, *57*, 1207-1212.
15. Zeeman, G.; Sanders, W. Potential of anaerobic digestion of complex waste(water). *Water Sci. Technol.* **2001**, *44*, 115-122.

16. Kujawa-Roeleveld, K. *Anaerobic treatment of concentrated wastewater in DESAR concepts*; 2005-14; Stowa: Utrecht, The Netherlands, 2005.
17. Halalsheh, M.; Koppes, J.; den Elzen, J.; Zeeman, G.; Fayyad, M.; Lettinga, G. Effect of SRT and temperature on biological conversions and the related scum-forming potential. *Water Res.* **2005**, *39*, 2475-2482.
18. Tchobanoglous, G.; Burton, F.L.; Stensel, H.D. *Wastewater Engineering - Treatment and Reuse*. 4th ed.; McGraw-Hill: New York, NY, USA, 2003.
19. Luostarinen, S.; Sanders, W.; Kujawa-Roeleveld, K.; Zeeman, G. Effect of temperature on anaerobic treatment of black water in UASB-septic tank systems. *Bioresour. Technol.* **2007**, *98*, 980-986.
20. Meulman, B.; Zeeman, G.; Buisman, C.J.N. Treatment of concentrated black water on pilot scale: options and challenges. In Proceedings of the Sanitation Challenge; Wageningen, The Netherlands, 19-21 May 2008, 2008.
21. Koster, I.W.; Koomen, E. Ammonia inhibition of maximum growth rate of hydrogenotrophic methanogens at various pH-levels and temperatures. *Appl. Microbiol. Biotechnol.* **1988**, *28*, 500-505.
22. Zeeman, G.; Kujawa, K.; Meulman, B.; Kwant, F. Full scale demonstration of vacuum collection, transport & treatment of black water. Poster presentation at the Advanced Sanitation Conference, Aachen, Germany, March 2007.
23. Kujawa-Roeleveld, K.; Fernandes, T.; Wiryawan, Y.; Tawfik, A.; Visser, M.; Zeeman, G. Performance of UASB septic tank for treatment of concentrated black water within DESAR concept. *Water Sci. Technol.* **2005**, *52*, 307-313.
24. APHA, *Standard methods for the examination of water and wastewater*. American Public Health Association: Washington, DC, USA, 1998.
25. Batstone, D.J. Mathematical modelling of anaerobic reactors treating domestic wastewater: Rational criteria for model use. *Rev. Environ. Sci. Bio/Technol.* **2006**, *5*, 57-71.
26. Gorter, K. Black water production at the Decentralized Sanitation and Reuse demonstration project in Sneek, the Netherlands, personal communication, 17 November 2009.
27. Lide, D.R. *CRC Handbook of Chemistry and Physics*. 85th ed.; CRC Press: Boca Raton, FL, USA, 2004.
28. Lettinga, G.; Hulshoff Pol, L.W. UASB-process design for various types of wastewaters. *Water Sci. Technol.* **1991**, *24*, 87-107.
29. Vinnerås, B.; Björklund, A.; Jönsson, H. Thermal composting of faecal matter as treatment and possible disinfection method - laboratory-scale and pilot-scale studies. *Bioresour. Technol.* **2003**, *88*, 47-54.
30. Winker, M.; Vinnerås, B.; Muskolus, A.; Arnold, U.; Clemens, J. Fertiliser products from new sanitation systems: Their potential values and risks. *Bioresour. Technol.* **2009**, *100*.
31. Mulder, A. The quest for sustainable nitrogen removal technologies. *Water Sci. Technol.* **2003**, *48*, 67-75.
32. van der Star, W.R.L.; Abma, W.R.; Blommers, D.; Mulder, J.W.; Tokutomi, T.; Strous, M.; Picioreanu, C.; van Loosdrecht, M.C.M. Startup of reactors for anoxic ammonium oxidation:

Experiences from the first full-scale anammox reactor in Rotterdam. *Water Res.* **2007**, *41*, 4149-4163.

33. de Graaff, M.S.; Zeeman, G.; Temmink, H.; Van Loosdrecht, M.C.M.; Buisman, C.J.N. Combined anaerobic treatment and autotrophic nitrogen removal from black water. In *IWA 2nd Specialized Conference Nutrient Management in Wastewater Treatment Processes*, Krakow, Poland, September 2009.
34. Vlaeminck, S.E.; Terada, A.; Smets, B.F.; van der Linden, D.; Boon, N.; Verstraete, W.; Carballa, M. Nitrogen removal from digested black water by one-stage partial nitritation and anammox. *Environ. Sci. Technol.* **2009**, *43*, 5035-5041.
35. Ohlinger, K.N.; Young, T.M.; Schroeder, E.D. Predicting struvite formation in digestion. *Water Res.* **1998**, *32*, 3607-3614.
36. van Voorthuizen, E.; Zwijnenburg, A.; van der Meer, W.; Temmink, H. Biological black water treatment combined with membrane separation. *Water Res.* **2008**, *42*, 4334-4340.
37. Cordell, D.; Drangert, J.-O.; White, S. The story of phosphorus: Global food security and food for thought. *Global Environ. Change* **2009**, *19*, 292-305.
38. Ueno, Y.; Fujii, M. Three years experience of operating and selling recovered struvite from full-scale plant. *Environ. Technol.* **2001**, *22*, 1373-1381.