Anaerobic Digestion of Blackwater With Various Co-Substrates in Eudiometer Scale

Ana Karenina Pahimalan–Pera
Universität Stuttgart, Germany

ABSTRACT: This paper investigates the anaerobic biodegradability of blackwater and its suitability for co-digestion in batch digesters (eudiometers) under mesophilic temperature. Readily available co-substrates such as grease trap waste, concentrated urine and high strength domestic wastewater which are high-strength organic substrates were introduced. These substrates were treated in a decentralized reactor through anaerobic digestion. The biomethane potential of these substrates at different inoculum to substrate ratios was investigated using 250 mL reactor bottles according to DIN 38414-8. Biogas production and quality was measured on a regular basis to determine the extent of the substrate degradation in terms of biogas production, volatile solids and chemical oxygen demand reductions, methane content and methanation level. The co-digestion experiments revealed that blackwater is best co-digested with grease trap waste based on the increased biogas production of 57 percent and the enhanced biogas quality. The co-digestion of blackwater with urine is only possible up to 10 percent v/v addition due to inhibition by ammonia at higher urine fractions.

KEYWORDS: Anaerobic digestion, blackwater, grease trap waste, urine, high strength domestic wastewater, energy recovery, biogas, co-digestion

Introduction

In countries where a centralized municipal wastewater (WW) treatment is too expensive to operate, the concept of resource-oriented waste management is meant to address global sanitation problems as well as the

problems recognized from centralized WW management (Wilderer 2001, 39-54). This concept focuses on the separation of WW flows and organic waste at the source, that is, household level, and then treating each WW stream accordingly in decentralized systems for subsequent reuse of water and nutrients, and recovery of energy.

Anaerobic digestion (AD) is a sustainable process which has gained popularity in today's efforts toward sustainable development and renewable bioenergy production. AD is a biochemical technology that is capable of converting a wide range of organic materials into biogas, which contains methane (CH_4), a main component of natural gas (Deublein and Steinhauser, 2011), and carbon dioxide (CO_2). It is one of the sustainable approaches that combines waste treatment and recovery of useful by-products, and is even considered as the core technology for the recovery of energy and nutrient from source separated domestic WW (Otterpohl 2002, 149-158; Kujawa-Roeleveld and Zeeman 2006, 115-139).

Although extensive studies have been made recently on the AD of blackwater (BW), most of them focused on co-digestion with kitchen waste (Gallagher 2010; Wendland 2008; Lim 2011; and Graaff 2010). Enhanced biogas production from the co-digestion of municipal sludge and fats, oils and grease (FOG), have also been reported (Kabouris et al. 2009a, 476-485; 2009b 3701-3705; Loustarinen, Luste and Sillanpaa 2009, 79-85). However, studies on the co-digestion of BW and readily available substrates such as urine (U), grease trap waste (GTW) and high strength domestic WW have not yet been conducted extensively.

This study aims to assess the suitability of BW as a substrate for AD and to focus on the recovery of energy from the AD of BW. Moreover, the potential for increasing the gas yield via co-digestion is also investigated. To achieve this, anaerobic co-digestion batch experiments of BW with co-substrates such as U, fat and grease from the grease trap, and high strength WW from a WW treatment facility at different loading rates are also conducted.

Materials and Methods

Collection and preparation of inoculum and substrates

The inoculum used in this study was the mesophilic digested sludge collected from the digester of the WW treatment facility at the Universität Stuttgart. The digested sludge was sieved four times and diluted with distilled water at a ratio of about 1:3. A fine screen sieve was used to ensure homogeneity and adequate mixing. Prior to addition of substrates, the inoculum was allowed to react with itself anaerobically at 35±2°C for 24 hours.

The substrates used in this study include BW, U, GTW, and high strength WW. All these substrates were taken from the facilities installed at the Institute of Sanitary Engineering, Water Quality and Solid Waste Management (ISWA). The BW, the main substrate, was taken from two water saving toilets, with a flush volume of 0.5 – 2.5 L/flush. The U samples were collected via grab sampling from a waterless urinal. The GTW were the fat and grease taken from the surface of the sand trap of the WW treatment plant. The WW was taken from the inflow of the WW treatment facility with inflow originating from the nearby residential area of Büsnau. Both BW and GTW were sieved. However, the latter was further heated to 70°C to melt and for better mixing and then diluted to achieve a 10 g organic dry matter (oDM) per kg. Prior to use, the substrates were stored at 4°C. A summary of the characteristics of the inoculum and substrates is listed in Table 1.

TABLE 1: Characterization of the inoculum and substrates

Parameter	Unit	Inoc	BW	U	GTW	WW
COD	mg/L	13917-14453	8150-8570	6064-9460	-	990-1460
TS	g/kg	15.63-15.83	6.0-8.0	20.7-29.0	9.53-11.2	-
VS	%	62-64	61-68	45-48	93-96	-
TKN	mg/L	-	1060-1970	9180-10600	-	-

Experimental Set-Up and Design

The batch digestion tests were carried out based on the German standard DIN 38414-8 using 250mL reactors at mesophilic temperature (35°C) for a period of twenty-eight days. The tests were conducted in Selutec Eudiometers, which are 250 mL flat bottom reaction vessels, with a 200 mL working volume. A schematic diagram of the experimental setup is shown in Figure 1.

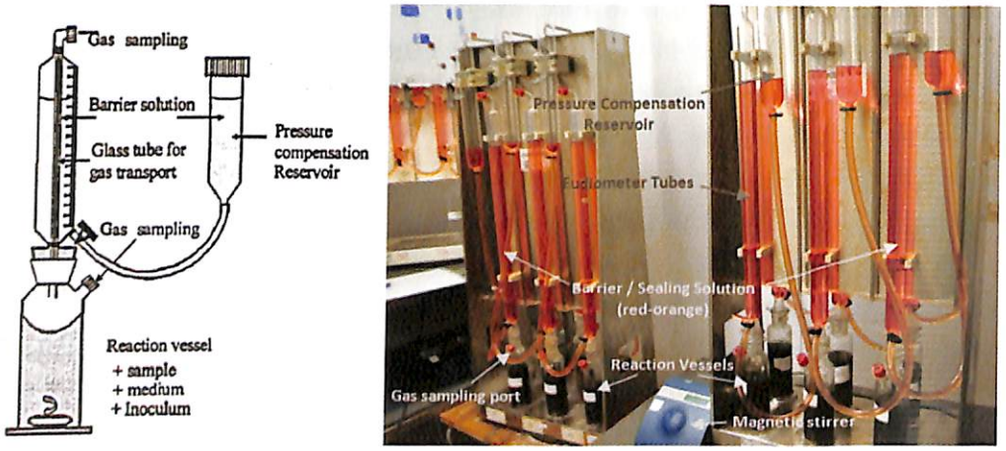

FIGURE 1: The experimental setup: (A) schematic diagram of a eudiometer unit based on DIN 38414-8 and (B) the actual experimental setup at a temperature-controlled room.

In each reactor, varying inoculum to substrate ratios (ISRs) were prepared. To create anaerobic conditions, the headspace of all the reactors was purged with nitrogen gas for thirty seconds before tightly closing them with rubber septa and screw caps. Blank reactors containing only the inoculums were also set up to monitor the biogas production from the inoculum alone. This is important for the correction of the biogas produced from the substrates under investigation. Even mixing was ensured using magnetic stirrers (Thermoscientific Stirrer, Thermo Electron LED GmbH) and were set at 100 percent stirring power and speed of 525 RPM (revolutions per minute). The batch tests using the different substrates were conducted in triplicates, while the digested sludge and reference substrate were conducted in duplicates.

Room temperature and pressures were monitored daily for the duration of the batch experiments to help calculate the biogas production. Final pH and temperature of the substrate mixtures were also measured to determine the presence of possible inhibitions.

Physical and Analytical Methods

Chemical analyses

Chemical analyses were carried out before and after the batch assays to determine the biodegradability and the quality of the WW. The different analytical methods carried out by the Wastewater Technology Department laboratory and their corresponding method numbers are listed in Table 2.

Gas analysis

Gas analysis (CH_4 and CO_2 content measurements) was done at least once a week or when the gas production was more than 50 mL, whichever came first. The biogas samples were collected from the headspace of the reactors prior to releasing the gas using 500 µL gas-tight syringes (Hamilton Gastight #1750, Bonaduz, Switzerland). For each reactor, three replicates of samples were taken. The biogas composition, methane and carbon dioxide content, were then analyzed using an AutoSystem GC gas chromatograph (GC, PerkinElmer, USA), and run using the software PerkinElmer TotalChrom Workstation Version 6.3.1.0504 at a run time of three minutes. The GC was equipped with a flame ionization detector and a capillary column (Agilent Technology, USA). Nitrogen was used as a carrier gas. The temperatures of injection inlet, oven, auxiliary and detector were 110 °C, 140 °C, 200 °C and 200 °C, respectively. The GC was calibrated using analytical grade gas composed of 40 percent CH_4 and 10 percent CO_2 (Linde, Germany) and a test gas was run prior to the first gas measurements to ensure that the calibration is still valid.

Table 2: Summary of analytical methods conducted for important WW quality parameters

Parameter analyzed	Method Number	BW	U	GTW	WW
Chemical Oxygen Demand, COD	DIN 38409–41	X	X		X
Total Solids, TS	DIN 38409–1	X	X	X	
Volatile Solids, VS	DIN EN 12879	X	X	X	
Suspended Solids, SS	DIN 38409–2–2	X	X		
Total Kjeldahl Nitrogen, TKN	DIN EN 25663	X	X		
Ammonium Nitrogen, NH_4-N	DIN 38406–5	X	X		X
Total Phosphorus, P_{Tot}	DIN 38414–12, DIN EN ISO 6878	X	X		
Phosphate Phosphorus, PO_4^{-3}-P	DIN EN ISO 6878	X	X		
Temperature	DIN 39404–4	X	X		
pH	DIN 38404–5	X	X		X

Results and Discussion

Anaerobic digestion of BW

Biogas production. The BW was anaerobically digested at four different ISRs. The cumulative biogas production curve is shown in Figure 2(a). The highest biogas production was achieved at A4 with 334 NmL and ISR of 52. At increasing substrate availability (decreasing ISR value), the biogas production was observed to be increasing.

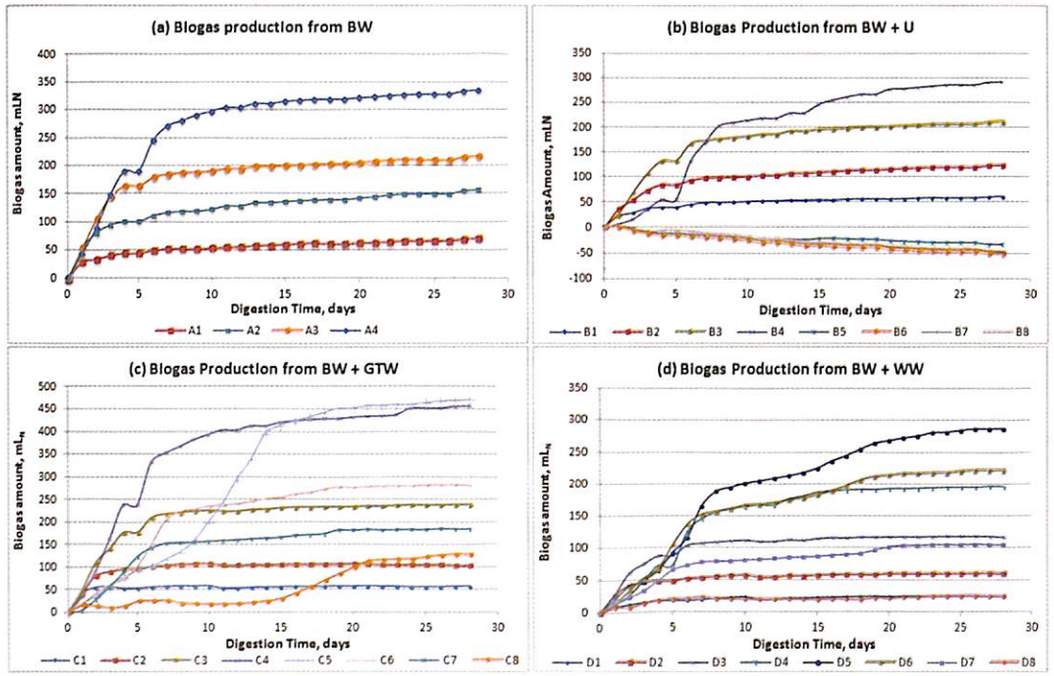

Figure 2: Biogas production from (a) BW, (b) BW+U, (c) BW+GTW, and (d) BW+WW, versus digestion time at various inoculum to substrate ratios (ISRs)

Biodegradability. A summary of the final pH, volatile solids (VS) and chemical oxygen demand (COD) reductions, methanation and average methane content of the produced biogas from the AD of BW is presented in Table 3. The final neutral pH of the mixtures A1-A4 (7.16–7.29) indicate that there was no accumulation of acids or inhibition by ammonia. The COD and VS reduction for the BW assays is obviously influenced by the ISR, that is, higher removal occurred with the lower inoculum to substrate ratios. The CH_4 content of the biogas increased at increasing substrate amount until A3 after which it decreased a little.

TABLE 3: Final pH, % of methanation, % reduction of VS and COD and average methane content of the biogas produced from the different BW mixtures in the batch assays

	ISR[a]	pH	Methanation[b] (%)	VS Reduction (%)	COD Reduction (%)	Ave CH_4 content (%)
A1	707	7.29	45	7.83±0.50	19.79±1.06	31.26±11.14
A2	314	7.24	55	10.47±3.25	30.37±2.28	41.81±4.97
A3	118	7.16	58	13.75±1.34	35.75±2.04	51.88±2.46
A4	52	7.16	51	18.39±2.04	44.31±1.78	50.79±5.25
B1	725	7.31±0.01	36.03	7.13±1.94	14.41±1.06	32.82±9.51
B2	322	7.31±0.02	38.17	8.65±0.98	21.32±2.11	34.54±11.87
B3	121	7.31±0.02	44.09	9.57±0.58	32.76±2.34	46.71±7.04
B4	54	7.35±0.00	43.82	17.82±1.02	42.12±2.44	50.10±11.71
C1	8.2	7.48	29.74	86.08±0.41	-	31.80±7.20
C2	3.6	7.39	33.87	87.29±0.55	-	41.06±5.47
C3	1.4	7.40	42.69	89.54±0.68	-	44.71±7.99
C4	0.6	7.34	61.76	91.02±0.20	-	50.26±9.15
C5	0.58	7.38	68.26	13.34±0.85	-	58.81±25.65
C6	0.53	7.32	33.53	17.48±0.80	-	52.33±16.19
C7	0.48	7.18	14.67	21.16±1.32	-	38.29±13.72
C8	0.43	6.36	8.42	2.91±3.43	-	36.38±4.20
D1	1152	7.44±0.05	19.82	-	15.61±0.74	36.41±9.75
D2	512	7.35±0.02	26.41	-	19.09±3.01	31.58±11.94
D3	192	7.37±0.06	27.66	-	26.05±0.36	33.55±13.00
D4	85.3	7.34±0.02	36.33	-	34.70±0.64	38.53±12.46
D5	53.3	7.43±0.02	40.65	-	31.01±2.00	49.78±18.13
D6	66.1	7.39±0.01	43.60	-	25.53±0.84	51.75±16.11
D7	127.2	7.35±0.01	20.82	-	18.10±0.81	27.43±15.88
D8	413.3	7.33±0.02	11.34	-	7.12±0.87	20.56±8.69

ISR: Inoculum to substrate ratio on a g VS/g COD basis; [a] For C1-C8 ISR of on a gVS/gVS basis

[b] %Methanation for C1-C4 is based on a theoretical SMY of 4.41 mL/g VS, for C5-C8 is based on a theoretical SMY of 4.87 mL/g VS

Anaerobic co-digestion of BW with other substrates

Biogas production. In the anaerobic co-digestion (ACD) experiments with BW + U, two sets of experiments were conducted. The B1–B4 had different inoculum amounts with U added at 10 percent by volume of the substrate. The B5–B8 had the same inoculum amount but at different BW to U fractions.

The biogas production (NmL) of B1–B4 is shown in Figure 2(b). After twenty-eight days of digestion, the biogas produced was found out to be 206, 250, 306, and 356 NmL, respectively. The resulting cumulative curves indicated normal biogas production, characterized by a steep increase in the gas production in the first few days of the digestion. A lag of about five days at the beginning was observed for B4, most probably due to the high substrate availability but low number of microorganisms that will consume them.

The biogas production from B5-B8, where the inoculum amount remained constant but at increasing fractions of U of 30 percent, 50 percent, 70 percent and 100 percent, respectively, is also shown in Figure 2(b). It showed a negative net biogas production for B5-B8, indicating that there is a strong or complete inhibition (VDI 2006, 48). Strong inhibition by ammonia was already observed beginning at B5, with a total NH_4-N concentration of 708 mg/L, which is significantly lower than what has been reported in literature of 1,500 to 2,500 mg/L (Velsen 1979, 995-999; Hansen, Angelidaki and Ahring 1998, 5-12). This inhibition occurred in these experiments at an earlier stage due to uncontrolled pH, and a faster shift to pH values of 8.20–8.58 which resulted in the inhibition of the process.

Figure 2(c) illustrates the cumulative biogas production from the ACD of BW with 10 percent GTW at varying inoculum amounts. The biogas produced from C1, C2, C3 and C4 are found out to be 58, 102, 237 and 457 NmL. The C4 produced the highest biogas amount, showing a remarkable increase in biogas production vs. the amount of biogas produced by BW alone at 334.3 NmL. This increased the biogas production by about 37 percent.

Further ACD experiments of BW and with higher fractions of GTW showed that at constant inoculum amounts and higher GTW contents, the

biogas produced decreased (Figure 2(c)) from as high as 471 NmL for C5 to as low as 128 NmL for C8. Based on the figure, there was generally a long lag observed for C5 of about ten days compared to experiments with BW (A1-A4) where no lags were observed, indicating an accumulation of long-chain fatty acids (LCFAs) in the system [15] and suggesting retarded degradation [12]. Inhibition was highest for C8 containing 100 percent GTW in the first eighteen days, signifying a shock loading of LCFAs in the system (Hanaki, Matsuo and Nagase 1981, 1591-1610). A higher increased biogas production of about 41 percent was achieved when 30 percent by volume GTW was added to BW (C5). A decrease in biogas production was observed at higher GTW fractions.

The suitability of high strength domestic WW as a co-substrate for the AD of BW was also investigated. Biogas production curves for D1–D8, shown in Figure 2(d), demonstrate a normal biogas production. Comparing the amount of biogas produced from the AD of BW alone (B4 = 334.33 NmL) to that of D4-D8, which contained more or less the same amount of inoculum sludge (VS basis), it was observed that there was a lower production of biogas. One reason could be the dilution of the available organics as COD. The domestic WW used in the experiments, although classified as high-strength WW, had a COD content that is five times lower than that of the COD of BW, resulting in lower biogas production. The COD concentration of the substrates in D8 was only 594 mg/L, which is lower than the recommended COD value of >1,500 - 2,000 mg/L for the production of sufficient methane (Tschobanoglous, Burton and Stensel 2003).

Biodegradability. After twenty-eight days, the pH, VS and COD eliminations and percentage (%) of methanation of the eight different mixtures of BW and U, BW and GTW, and BW and WW were determined and the results are listed in Table 3. The final pH of the mixtures with 10 percent U (B1–B4) were slightly higher than with BW alone, between 7.31-7.35. The B5-B8, however, showed a final basic pH ranging from 8.20–8.58, indicating accumulation of ammonia in the system and confirming that, indeed, inhibition by ammonia occurred. The final pH appeared to decrease at increasing GTW fractions (C5-C8), with C8 having a final acidic pH

of 6.36, suggesting that there was an accumulation of acidic fermentation products, most probably LCFAs. The pH dependence of LCFA degradation is assumed to be similar to that of acetate conversion so that at pH = 6.3, 50 percent inhibition is reached (Siegrist, Vogt, Garcia-Hera and Gujer 2002, 1113-1123), which somehow confirms the observed inhibition. The final pH of the mixtures of BW+WW were observed to be in the neutral range, ranging from 7.33–7.44.

The highest VS reduction was achieved from the experiment with BW and GTW (C4) at 91.02 percent, while the highest COD reduction was achieved from the experiment with BW (B4) at 44 percent. A percentage methanation value of as high as 68 percent was achieved from the ACD of BW and GTW (C5).

Conclusion and Recommendations

The AD of BW in resource-oriented sanitation concepts has a potential for recovering energy. The anaerobic batch digestion tests were successfully applied as a simple, low cost laboratory scale treatability study for BW alone and BW with U, GTW and WW using digested sludge as the inoculum at different inoculum to substrate ratios. The anaerobic batch digestion tests were performed at a temperature of 35°C for twenty-eight days to achieve complete digestion.

Co-digestion of BW with U was possible only at 10 percent by volume addition (B1-B4), with the highest biogas production of 292 NmL at ISR = 54. At higher U additions, the digestion process seems to be completely inhibited. At 30 percent U addition or higher, where an ammonium concentration is added starting from 396 mg/L, methane-formation is completely inhibited by the formation of ammonia. Co-digestion of BW and U did not enhance the biogas production.

The co-digestion of BW and GTW showed enhanced biogas production, with the highest increase in production of 57 percent as compared to when digesting with BW alone. The highest methanation achieved was 68 percent at an ISR of 0.58.

Co-digestion experiments of BW and high-strength domestic WW indicated negative synergisms due to the low biogas production as compared to when digesting BW alone. The highest biogas production was 287 NmL at ISR=53.3. At increasing domestic WW fractions, signs of inhibitions were observed, as indicated by low amounts of biogas production and poor biogas quality. A fraction of 10-30 percent WW by volume can be the optimum, which can produce biogas with a methane content of 46-52 percent.

Inhibitions were observed especially on the co-digestion experiments of BW with U and with GTW, which showed that there were also antagonistic interactions that influenced the methane production. Further studies should be carried out to investigate the effect of these intermediates on biogas yield and the digestion performance.

Pre-treatment methods, which can potentially increase biogas production, could be tested for the BW and its co-substrates.

References

Deublein, Diether and Steinhauser, Angelika. 2011. *Biogas from waste and renewable resources, 2nd ed.* Weinheim, Germany: Wiley-VCH Verlag GmbH & Co. KGaA.

Gallagher, Neil T. 2010. *An advanced decentralized wastewater management planning study and demonstration project for the CSU Foothills Campus (master's thesis).* Colorado State University: Fort Collins, Colorado. Unpublished.

Graaff, Marthe Sophie de. 2010. *Resource recovery from black water (PhD dissertation).* Wageningen University: Wageningen, Netherlands.

Hanaki, Keisuke, Matsuo, Tomonori and Nagase, Michihiko. 1981. Mechanism of inhibition caused by long-chain fatty acids in anaerobic digestion process. *Biotechnology Bioengineering,* 23, no. 7: 1591-1610.

Hansen, Kaare Hvid, Angelidaki, Irini and Ahring, Birgitte Klaer. 1998. Anaerobic digestion of swine manure: Inhibition by ammonia. *Water Research* 32: 5-12.

Kabouris, John C. et al. 2009a. Mesophilic and thermophilic anaerobic digestion of municipal sludge and fat, oil, and grease. *Water Environment Research* 81, no. 5: 476–485.

Kabouris, John C. et al. 2009b. Methane recovery from the anaerobic codigestion of municipal sludge and FOG. *Bioresource Technology* 100, no. 15: 3701–3705.

Kujawa-Roeleveld, Katarzyna and Zeeman, Grietje. 2006. Anaerobic treatment in decentralised and source-separation-based sanitation concepts. *Reviews in Environmental Science and Bio/Technology* 5: 115-139.

Lim, Jun Wei. 2011. Anaerobic co-digestion of brown water and food waste for energy recovery. In *World wide workshop for young environmental scientists (WWWYES-2011)-Urban Waters: Resource or risks.* Arcueil: France.

Luostarinen, Sari, Luste, Sami and Sillanpää, Mika. 2009. Increased biogas production at wastewater treatment plants through co-digestion of sewage sludge with grease trap sludge from a meat processing plant. *Bioresource Technology* 100: 79-85.

Otterpohl, Ralf. 2002. Options for alternative types of sewerage and treatment systems directed to improvement of the overall performance. *Water Science Technology* 45, no. 3: 149-158.

Siegrist, Hansruedi et al. 2002. Mathematical model for meso- and thermophilic anaerobic sewage sludge digestion. *Environmental Science and Technology* 36:1113-1123.

Tschobanoglous, George, Burton, Franklin L. and Stensel, David H. 2003. *Wastewater engineering: Treatment and reuse, Metcalf & Eddy, Inc., 4th edn. Revised.* New York, USA: The McGraw-Hill Companies, Inc.

Velsen, A. F. M. van. 1979. Adaption of methanogenic sludge to high ammonia-nitrogen concentrations. *Water Research* 13: 995-999.

VDI. 2006. Vergärung organischer Stoffe. In *VDI-Richtlinie 4630, Handbuch.* Düsseldorf, Verein deutscher Ingenieure (Hrsg.).

Wendland, Claudia. 2008. *Anerobic digestion of blackwater and kitchen refuse (PhD dissertation).* Technische Universität Hamburg-Harburg: Hamburg, Germany. Unpublished.

Wilderer, Peter. 2001. Decentralized versus centralized wastewater management. In *Decentralised sanitation and reuse: Concepts, systems and implementation,* eds. P. Lens, G. Zeeman, G. Lettinga. London, UK: IWA Publishing, pp. 39-54.