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Anerobic Digestion of Blackwater and Kitchen Refuse

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Zusammenfassung

Anaerobbehandlung von Schwarzwasser und Bioabfällen

Ziel dieser Arbeit ist die Bewertung von Möglichkeiten und Grenzen der anaeroben Behandlung von Schwarzwasser (Toilettenabwasser) aus Vakuumtoiletten ohne und mit Küchenabfällen im Rahmen von neuen Sanitärkonzepten.

In einem volldurchmischten, mesophilen Laborreaktor wurde die Leistungsfähigkeit der Anaerobbehandlung von Schwarzwasser allein und gemeinsam mit Küchenabfällen ermittelt. Dazu wurden die Randbedingungen hydraulische Verweilzeit, Ammoniumkonzentration und Vorbehandlung variiert. Bei einer hydraulischen Verweilzeit von 20 Tagen im Reaktor zur Anaerobbehandlung von Schwarzwasser werden über 60 % des CSB zu Biogas umgesetzt, was 87 % des maximalen anaeroben Abbaugrades entspricht. Die einwohnerspezifische Methangasmenge aus Schwarzwasser ergibt sich zu 14 l/Person/Tag. Die Anaerobbehandlung von Schwarzwasser verläuft trotz hoher Ammoniumgehalte ungehemmt. Bei künstlicher Steigerung der Ammoniumkonzentration zeigt sich eine Hemmung durch freies Ammoniak, die bei 300 mg/L NH_3-N startet. Bei hydraulischer Auslegung des Reaktors ergibt sich ein erhebliches Potential an organischer Auslastung, was durch die Zugabe von Küchenabfällen genutzt wurde. Der Anaerobreaktor kann bei minimal 15 Tagen hydraulischer Verweilzeit und einer maximalen Fracht von 5 kg CSB/($m^3_{Reaktor}$·d) ohne Hemmung betrieben werden. Sinkt die Verweilzeit oder steigt die CSB Fracht, gibt es ein Stadium des gehemmten stabilen Zustands, bei dem der Reaktor bei niedrigerer Methanproduktion weiter betrieben werden kann, bevor der biologische Prozess zusammenbricht.

Die mesophile Anaerobbehandlung hat ein Hygienisierungspotential von 2 log-Reduktion für das Indikatorbakterium E.-coli. Weitere 2 log-Reduktion wurden durch eine anschließende 40tägige Lagerung des Reaktorablaufes bei Umgebungstemperatur erzielt. Pasteurisierung und Vorversäuerung wurden auf ihre keimreduzierende Wirkung ebenfalls untersucht, aber kein Behandlungsverfahren erreicht den international als hygienisch sicher eingestuften Wert von 1.000 KBE/g TR (WHO 2006, Anonymous 1993) im Reaktorablauf.

Das Anaerobic Digestion Model No. 1 (ADM1) der IWA wurde mit der Software Aquasim implementiert. Die erforderlichen kinetischen Daten basierend auf den ermittelten experimentellen Daten wurden so durch Anpassung ermittelt. Schlüsselparameter waren dabei die Desintegration- und Hydrolysekonstante k_{dis} und die Konstanten für Ammoniak und pH-Hemmung. Das erfolgreich kalibrierte Modell diente der Simulation von verschiedenen Grenzzuständen der Laboranlage sowie der Umsetzung auf eine großtechnische Anlage. Damit gibt es für Planer alternativer Sanitärkonzepte jetzt ein Werkzeug, mit dem Anaerobreaktoren effizient ausgelegt und die Leistungsfähigkeit im Voraus abgeschätzt werden kann.

Im Vergleich zum konventionellen schneidet das auf Anaerobtechnologie basierende vorgeschlagene Sanitärkonzept für Haushaltsabwasser bezüglich der Energiebilanz und der CO_2-Emissionen deutlich günstiger ab.

Schlagwörter: Schwarzwasser, Vakuumtoilette, Ammoniakhemmung, Anaerobbehandlung, Vergärung, Hygienisierung, Biogas, ADM1

Abstract

Anaerobic Digestion of Blackwater and Kitchen Refuse

The main objective of this thesis was to assess the anaerobic treatment of blackwater (toilet wastewater) from vacuum toilets without and with kitchen refuse and its potential for resources management sanitation concepts.

In a bench scale continuous stirred tank reactor (CSTR), the performance of mesophilic anaerobic digestion of blackwater without and with kitchen refuse was investigated at varying conditions for hydraulic retention time (HRT), ammonia concentration and pre-treatment. At HRT of 20 days, more than 60 % of the input COD was converted to biogas which represents 87 % of the maximum biodegradable COD. At these conditions, the biogas contained 75 % methane and the specific rate was 14 l CH_4/cap/d. The digestion runs uninhibited despite high ammonia concentration of 1,111 mg NH_4/l. When increasing ammonia artificially about two times, the inhibition by free ammonia starts at concentration of 300 mg NH_3/l. Designing of the CSTR based on hydraulic criteria leads to a high unused potential of organic load. When adding kitchen refuse, the reactor can be operated at 15 days HRT and a maximum load of 5 kg COD/($m^3_{reaktor}$·d) in an uninhibited state. It results to an increase of methane production of 10 times compared to blackwater digestion. If the HRT decreases or the COD load increase, the reactor runs at an inhibited steady state at lower methane production until the process breaks down.

The mesophilic anaerobic digestion process has a relevant hygienization performance of 2 log reduction for the pathogen indicator E.coli. Additionally 2 log reduction can be reached in case of post-storage of 40 days at ambient temperature. Pre-pasteurization and Pre-acidification were studied, but no treatment is able to meet safely the standard of 1.000 CFU/g TR set by WHO (2006) and US EPA (Anonymous 1993).

The Anaerobic Digestion Model No. 1 (ADM1) by IWA was implemented with the software Aquasim. The model was calibrated based on the experimental results. The key parameter were the disintegration and hydrolysis constant k_{dis} and the inhibition constants for free ammonia and pH. The calibrated model was applied for the simulation of process limits and of a technical scale reactor. This model can now be used as a tool to design and predict the performance of an anerobic CSTR for combined blackwater and kitchen refuse treatment.

The finally proposed sanitation concept for household wastewaters based on anaerobic technology has major advantages in terms of energy balance and CO_2 emissions compared to the conventional aerobic system.

Keywords: Blackwater, vacuum toilets ammonia inhibition, anaerobic digestion, hygienization, biogas, ADM1

TABLE OF CONTENTS

CHAPTER 1 INTRODUCTION 1

1.1 The rationale behind separate collection and anaerobic digestion of blackwater 2

1.2 Examples of anaerobic digestion of blackwater world wide 4
1.2.1 European case studies 4
1.2.2 Asian examples 6

1.3 Objectives and procedure of this thesis 7

CHAPTER 2 BASICS OF ANAEROBIC DIGESTION 9

2.1 Anaerobic digestion process 11
2.1.1 Impact of high ammonia concentrations 13
2.1.2 Hygienic aspects 16

2.2 Types of anaerobic reactors 19
2.2.1 Continuous stirred tank reactor (CSTR) 19
2.2.2 Upflow anaerobic sludge blanket reactor (UASB reactor) 20
2.2.3 Batch reactor 21

CHAPTER 3 EXPERIMENTAL WORK 22

3.1 Material and methods of the experimental work 22
3.1.1 Substrates 22
3.1.2 Experimental set-up 23
3.1.3 Chemical and biological analysis 26
3.1.4 Data processing and calculations 28

3.2 Determination of the characteristics and daily loads of blackwater from vacuum toilets and kitchen refuse 30
3.2.1 Blackwater 30
3.2.2 Kitchen refuse 33

3.3 Anaerobic biodegradability 35

3.4 Performance of blackwater digestion in a CSTR 37
3.4.1 COD mass balance 37
3.4.2 Removal of COD fractions, TOC and VS 38
3.4.3 Removal of total nitrogen and ammonium 39
3.4.4 Biogas production 39
3.4.5 Output quality 41

3.5 Impact of increasing ammonia concentration 43

3.6 Co-digestion with kitchen refuse and assessment of different HRT 45
3.6.1 COD mass balance 45
3.6.2 Removal of COD fractions, TOC and VS 46
3.6.3 Biogas production....48

3.7 Pre-treatment and post-storage....49
3.7.1 Microbiological results....49
3.7.2 Impact of pre-treatment on AD 55

3.8 Chapter conclusions 56

CHAPTER 4 MATHEMATICAL MODELING AND SIMULATION 58

4.1 The mathematical model....58
4.1.1 Implementation of biochemical processes....59
4.1.2 Implementation of physico-chemical processes....66
4.1.3 Implementation in the simulation software AQUASIM 67

4.2 Calibration and adaptation of the model BWADM to the bench scale results....71
4.2.1 Reference conditions 71
4.2.2 Increasing ammonia concentration....78
4.2.3 Co-digestion with kitchen refuse....82
4.2.4 Impact of pre-treatment on AD 85

4.3 Up-scaling of the model....86
4.3.1 Simulation of the scenario *Flintenbreite* 87
4.3.2 Varying HRT and COD load....88

4.4 Chapter conclusions 92

CHAPTER 5 PROPOSED ANAEROBIC DIGESTION SYSTEM FOR HOUSEHOLD WASTEWATER....94

5.1 Description of the proposed AD system in comparison to an aerobic system 94

5.2 Energy balance....97
5.2.1 System boundaries....97
5.2.2 Methodology....98
5.2.3 Results 101

5.3 CO_2 emissions....104

5.4 Chapter conclusions 105

CHAPTER 6 CONCLUDING SUMMARY AND OUTLOOK....107

CHAPTER 7 REFERENCES 111

LIST OF FIGURES

Figure 1: Scheme of the blackwater and greywater flows in resources management sanitation concepts ... 2
Figure 2: Scheme of the pilot project *Flintenbreite* in Lübeck ... 5
Figure 3: Scheme of the procedure in the thesis ... 8
Figure 4: General processes of AD, redrawn from Batstone et al. (2002a) ... 12
Figure 5: Scheme of the CSTR ... 25
Figure 6: Composition of the VFA in blackwater based on COD (3 samples) ... 31
Figure 7: First batch serie (B1), total COD, dissolved and particulate COD removal with raw high-strength and low-strength blackwater ... 36
Figure 8: Second batch serie (B2), total COD, dissolved and particulate COD removal with raw blackwater ... 36
Figure 9: COD mass balance based on accumulated COD loads for AD of blackwater at 20 days HRT ... 38
Figure 10: Specific biogas production rates after feeding with high-strength and low-strength blackwater ... 39
Figure 11: NH_4-N concentrations and pH in the reactors with addition of urea and ammonia salts ... 43
Figure 12: Methanisation (%), weekly values, in the CSTR with different NH_4-N concentrations and pH due to different feeding and the kinetic approach for non-competitive inhibition ... 44
Figure 13: COD mass balances based on accumulated loads for AD of BW+KR at 10, 15 and 20 days HRT, COD mass balance for kitchen refuse was calculated ... 46
Figure 14: VFA-COD concentration and pH in the reactor with BW+KR at different HRT ... 47
Figure 15: E.coli, total coliforms and enterococcus in input and output of blackwater AD at 20 days HRT (15 samples) ... 49
Figure 16: Removal of E.coli and total coliforms during post-storage reactor output ... 50
Figure 17: Removal of E.coli, total coliforms and enterococcus after pre-pasteurisation (15 samples) ... 51
Figure 18: VFA-COD concentration, pH and E.coli removal in BW, BW+KR and BW+synth. KR after pre-acidification at 20 and 37 °C ... 52
Figure 19: Comparison of three selected treatment cycles in terms of removal of E.coli ... 54
Figure 20: Differences in dissolved COD concentrations in raw and pre-treated mixture of blackwater and kitchen refuse ... 55
Figure 21: COD mass flow for a particulate composite as used for ADM1. Propionic acid (HPr), Butyric acid (HBu) and Valeric acid (HVa) are grouped in the figure for simplicity (Batstone et al. 2002b) ... 59
Figure 22: Different kinetics of biochemical reactions ... 62
Figure 23: Impact of pH enzymatic pH inhibition on AD processes ... 64
Figure 24: Impact of non-competitive inhibition by free ammonia ... 65
Figure 25: Scheme of a single-tank digester (Batstone et al 2002a) ... 68
Figure 26: Calibration results of blackwater AD at 20 days HRT ... 73
Figure 27: Calibration results of AD of blackwater plus urea at 20 days HRT ... 79

Figure 28: Calibration results of AD of blackwater plus ammonia salts at 20 d HRT 81
Figure 29: Calibration results of AD of BW+KR at 10 and 20 days HRT 83
Figure 30: Simulation results of AD of combined blackwater and kitchen refuse with different distribution of lipids/carbohydrates/proteins in the particulate COD..........................84
Figure 31: Simulation results of AD of raw and pre-treated mixture of blackwater und kitchen refuse 86
Figure 32: Simulation results of AD of blackwater plus kitchen refuse for the biogas plant *Flintenbreite* 87
Figure 33: Simulation of scenario A in *Flintenbreite* with varying HRT from 6 to 24 days........88
Figure 34: Simulation of scenario B in *Flintenbreite*, increasing COD load 89
Figure 35: Comparison of hydraulic and COD load of the simulated scenarios...........................90
Figure 36: AD process stability related to COD load and HRT based on simulation results 91
Figure 37: Scheme of the proposed AD system for household wastewater 95
Figure 38: Consumption of heat and electricity for variant Vstandard (toilet using 0.7 – 1.0 l/flush)..102
Figure 39: Consumption of heat and electricity for Vadvanced (toilet using 0.25 l/flush).........102
Figure 40: Energy balance of the different AD variants and the aerobic treatment....................103
Figure 41: CO_2 emissions of the different AD variants and the aerobic treatment.....................105

LIST OF TABLES

Table 1: Blackwater characteristics....3
Table 2: General benefits and drawbacks of anaerobic digestion (van Velsen 1981, Zeeman 1991, Elmitwalli 2000)....9
Table 3: Characteristics of substrates applied for anaerobic digestion 10
Table 4: Inhibition thresholds of free ammonia for mesophilic anaerobic digestion....15
Table 5: Relevant guidelines and standards for the use of digested blackwater in agriculture....17
Table 6: General benefits and drawbacks of continuous stirred tank reactors (CSTR) 20
Table 7: Overview of the experimental steps....22
Table 8: Characteristics of raw blackwater, concentrations ± standard deviation 30
Table 9: Blackwater loads in *Flintenbreite* compared to reference data....32
Table 10: Characteristics of kitchen refuse (2 samples) and the mixture of blackwater and kitchen refuse (25 samples), concentration ± standard deviation....34
Table 11: Loads of kitchen refuse and mixture of blackwater and kitchen refuse....34
Table 12: Results of the batch tests B1 and B2 with average values 37
Table 13: Removal of organic matter and total N based on accumulated loads for AD of raw blackwater at 20 days HRT 38
Table 14: Biogas composition of blackwater AD with standard deviation (15 samples) 40
Table 15: Methane production based on accumulated loads for AD of blackwater at 20 days HRT 41
Table 16: Effluent characteristics of blackwaterAD in terms of organic matter, micro- and macronutrients and heavy metals as pollutants 42
Table 17: Investigation phases of co-digestion with kitchen refuse at different HRT....45
Table 18: Removal of organic matter based on accumulated loads for AD of BW and BW+KR at different HRT 47
Table 19: Biogas composition of AD of BW+KR with standard deviation (11 samples) 48
Table 20: Methane production based on accumulated loads for AD of untreated blackwater and kitchen refuse at 20, 15 and 10 days HRT....48
Table 21: Kinetic coefficients for acetate uptake based on Haldane kinetics with un-ionised acetate as substrate (experiments under mesophilic conditions)....63
Table 22: Required input parameter for BWADM to determine reactor feeding 71
Table 23: COD input for blackwater into the BWADM 72
Table 24: Kinetic parameter for disintegration and hydrolysis....75
Table 25: Kinetic parameter for acidogenesis, acetogenesis and methanogenesis (changes to ADM1 are pointed out in light grey)....77
Table 26: Kinetic parameter for inhibition by pH, hydrogen and free ammonia (changes to ADM1 are pointed out in light grey)....78
Table 27: COD input for kitchen refuse into the BWADM 82
Table 28: Up scaling the frame conditions of the model....87
Table 29: Expected methane production in *Flintenbreite* 92
Table 30: Design values for the variants of the proposed anerobic concept....96

CHAPTER 1 INTRODUCTION

Water is essential for life and the presence of clean water is a core issue for public health and economic growth. Human faeces are however the main source of water pollution in terms of pathogenic pollution. The rapid population growth along with an increasing water pollution and scarcity calls for action on a global level. Since 1980, the United Nations have addressed the world wide water problem, with the first International Water Decade (1981-1990) and now the second Water Decade “Water for Life” (2005-2015). The targets to halve the proportion of people without access to clean water and adequate sanitation until 2015 (Millenium Development Goal 7) were set in 2000 and 2002 by the UN. To meet these targets many efforts are being taken, e.g. the World Bank finances water projects only combined with sanitation. However, in 2006 there were still 1.6 billion people without access to safe sanitation (WHO and Unicef 2006).

Sanitation has a strong link to agriculture, as the nutrients nitrogen and phosphorus contained in human excreta are needed as fertilizer by farmers. The prices for chemical fertilizer increase, e.g. in Germany the market price for nitrogen rose in the year 2007 by about 40 % compared to 2006 (Anonymous 2008). Phosphorus resources are getting depleted and more and more polluted by cadmium. Consequently, the demand for organic fertilizer is growing. Organic fertilizer has an additional advantage of improving humous soil fertility which is urgently needed as many soils world wide suffer from substantial soil degradation.

An alarming aspect world wide is the depletion of non-renewable energy sources and predominantly oil. The fast growing demand, the market tightness of oil and depletion of possible extraction fields lead to further increasing oil prices. Alternative energy carriers such as biomass become more and more important (Kaltschmitt et al. 2007). The danger of global warming is associated with the consumption of non-renewable energy sources and the production of CO_2 which is the most relevant greenhouse gas. The Kyoto target to reach a CO_2 reduction of 5 % and 8 % world wide and in the EU respectively, cannot be met before 2012 by continuing with the use of fossil energies. The European Commission announced even more ambitious plans for an EU energy policy that included a unilateral 20 % reduction in greenhouse gas emissions by 2020 (Barroso 2007). Therefore new concepts and technologies are required which need less energy or produce renewable energy like biogas.

In the field of water management, anaerobic treatment can play a role in overcoming the addressed problems. Anaerobic digestion is one of the oldest technologies applied for wastewater treatment. Its main advantages are the gain of biogas and the smaller sludge production compared to aerobic treatment. The fact that the nutrients nitrogen and phosphorus are not removed is an advantage as well if the effluent is applied in agriculture to replace fertilizer. Historically, anaerobic digestion has been regarded with scepticism. The main drawbacks were considered to be its process instability and difficulties to operate efficiently, the need for high-strength influent, the need for mesophilic temperature and a minimum hydraulic retention time (HRT) of 10 days. Based on the knowledge of the last 15 years, these perceptions are incorrect under most circumstances (Stuckey 1998). The anaerobic technology has a considerable potential to treat domestic wastewater to a sufficient standard to recycle. Ultimately, anaerobic digestion can considerably reduce the cost of wastewater treatment, and help decreasing the CO_2

emissions as it produces energy in terms of biogas. Biogas has the potential to be used directly for various energy purposes. After purification, biogas can even reach the quality standard of natural gas and thus replacing it.

In the field of domestic wastewater treatment, the development of anaerobic digestion has followed two major lines: First, high-rate systems like UASB (upflow anaerobic sludge blanket) reactor followed by post-treatment, e.g. ponds were proven to be efficient and sustainable predominantly in warm climates. Many wastewater treatment plants were equipped with this concept during the last years in Latin America. Secondly, the low-tech digesters in household and bigger size fed by human excreta, animal manure and organic waste have been success stories in countries like China, Nepal and Vietnam. They have been supported mostly in the framework of national biogas programs in order to overcome the lack of energy in rural areas.

1.1 The rationale behind separate collection and anaerobic digestion of blackwater

In recent years, a new approach to sanitation and wastewater management has been developed and implemented in many projects world wide, which is referred to as Resources Management Sanitation, Ecological Sanitation (EcoSan), or Decentralised Sanitation and Reuse (DESAR) (Otterpohl et al. 1997, Lens et al. 2001, Otterpohl and Oldenburg 2007).

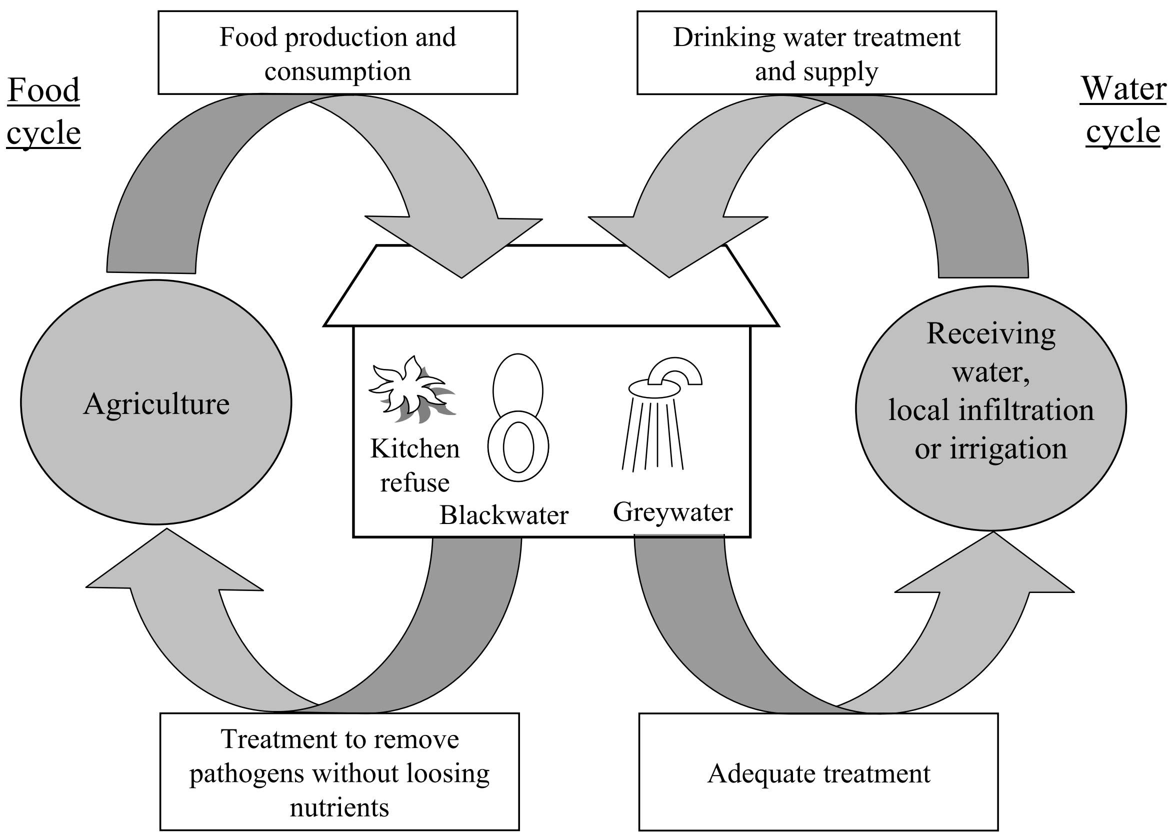

Figure 1: Scheme of the blackwater and greywater flows in resources management sanitation concepts

As an answer to the tremendous global sanitation problems as well as problems realized with centralized conventional wastewater treatment (Wilderer 2001), the new approach focuses on source separation of wastewater flows and organic waste on a household level, followed by an appropriate treatment of each stream in decentralized or semi-centralized systems and consequent reuse of water and nutrients (Figure 1).

Wastewater generated on household level consists of blackwater (BW) and greywater (GW).

Greywater (GW) is the wastewater generated in kitchen and bathroom. Greywater (comprising washing water from kitchen, shower, washing basins and laundry) is a low diluted wastewater stream big in quantity and can be treated separately in simple and low-cost treatment. Because of its characteristics, it belongs to the water cycle and is infiltrated locally, discharged to the receiving water or reused for irrigation (Figure 1).

Table 1: Blackwater characteristics

Parameter	Unit	BW from vacuum toilets, NL (Kujawa-Roeleveld et al. 2006)	Synthetic BW using primary sludge and toilet paper (Luostarinen 2005)	BW from vacuum toilets in Sneek, NL (Zeeman et al. 2007)	Synthetic BW using faeces, urine and water (Wolff 2000)
Total COD	mg/l	9,500 - 12,300	950	19,000	
Dissolved COD	mg/l	1,400 - 2,800	120	5,000	
VFA-COD	mg/l	500 - 1,900		1,300	
Particulate COD	mg/l	7,000 - 9,600	820	14,000	
TS	mg/l		670		10,370
VS	mg/l		490		7,570
TOC	mg/l				
NH_4-N	mg/l	600 – 1,000	4.5	1,400	692
Total N	mg/l		32		
Total P	mg/l	90 - 140	17	280	12
Ratio particulate COD to total COD	-	76 %	86 %	74 %	
COD/N/P	-	95/10/1	56/2/1	68/5/1	

Blackwater (BW) is wastewater coming uniquely from toilets and thus comprises urine, faeces, toilet paper and flushing water. Blackwater contains most of the nutrients, around half of the domestic COD load, the major part of the pathogens (Otterpohl 2002, Vinnerås et al. 2006). Only few references of blackwater analyses can be found which are summarized in Table 1. Because of its specific composition, blackwater requires separate collection, adequate treatment and final recycling of the nutrients in agriculture. Anaerobic digestion can play a key role as a known adequate treatment technology for concentrated wastewaters. Low flushing water consumption is helpful to achieve a low dilution of blackwater and an efficient process. That is why low-flush toilets or vacuum toilets are preferential for the collection of blackwater before anaerobic digestion. On the market, gravity toilets, so called pour-flush toilets, are available which need only 1 l per flush. The standard vacuum toilet requires 0.7 to 1.0 l/flush. Currently, there are advanced technologies under implementation in airplanes that need even less, around 0.25 l/flush. Additionally anaerobic digestion does not remove nutrients valuable as fertilizer and presents therefore an appropriate blackwater treatment for the food cycle (Figure 1).

In practice, there are several main drivers for anaerobic digestion of blackwater within resource management sanitation:

- Safe sanitation: The hazardous compounds in excreta, pathogens and medical residues, are not spread in the water cycle which is a severe danger to public health.
- Production of biogas for cooking, lighting and electricity: The produced biogas is a reliable renewable energy source.
- Water saving: The application of pour or low-flushing technology reduces the consumption of high-quality drinking water.
- Production of organic fertilizer for agriculture: Due to the remaining nutrients and organic matter, the digested blackwater can replace chemical fertilizer.

1.2 Examples of anaerobic digestion of blackwater world wide

Separate collection and digestion of blackwater has already been applied world wide in different contexts. In the following the development and the reasons behind in European and Asian countries are highlighted.

1.2.1 European case studies

In Europe, anaerobic digestion of blackwater has been applied within pilot projects to demonstrate its feasibility and its potential within resource management sanitation concepts.

Lübeck-Flintenbreite, Germany

In Northern Germany in Lübeck, the separation of domestic wastewater (blackwater, greywater, rainwater) at source was realized in a housing estate called *Flintenbreite* for about 400 inhabitants in the year 2000 (Figure 2) (Wendland and Oldenburg 2003).

For blackwater collection, a vacuum system was implemented to provide a low diluted blackwater flow. The blackwater is transported via vacuum toilets to a collecting tank in the basement of the central building by two vacuum pumps. The resulting production of blackwater is 5.0 l/cap/d. The low water amount for toilet flushing leads to a drinking water consumption of less than 80 l/cap/d in the housing estate (Oldenburg et al. 2008). Kitchen refuse is collected on household level in bins and is transported manually to the feeding unit in the central building. Other organic waste can be added, too.

Greywater is treated in vertical flow constructed wetlands and locally infiltrated into the soil as well as the rainwater.

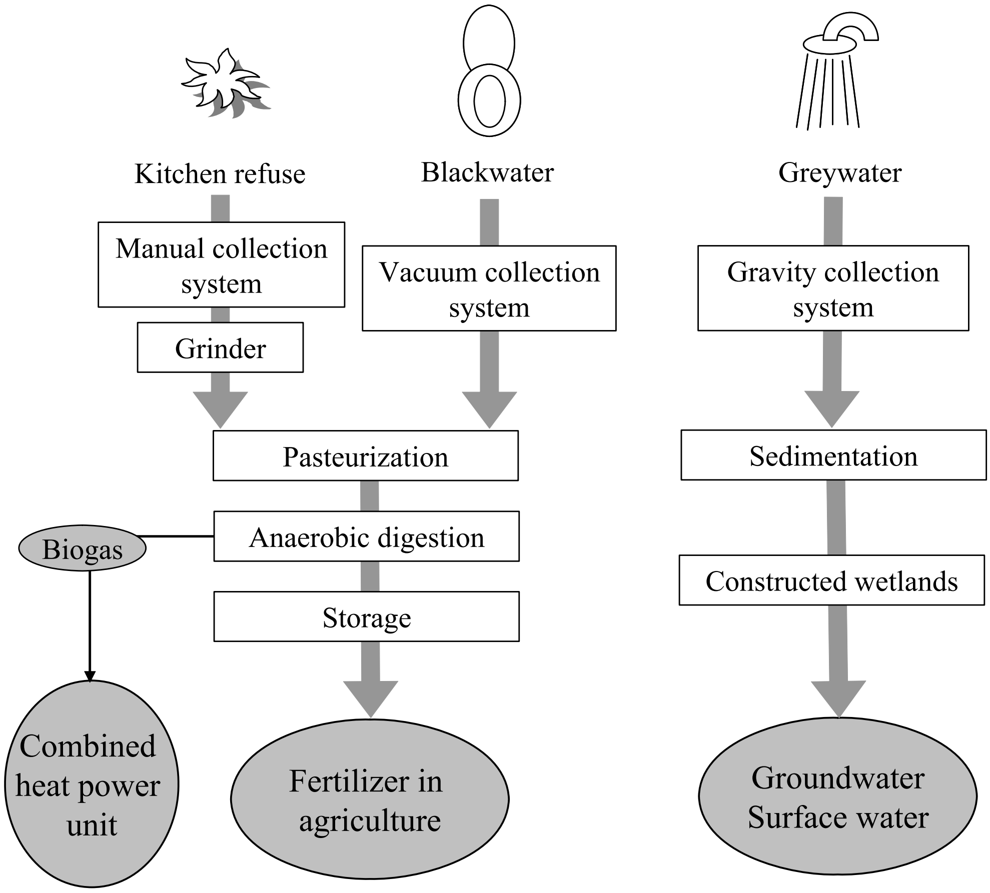

Figure 2: Scheme of the pilot project *Flintenbreite* in Lübeck

The treatment of kitchen refuse and blackwater comprises first the shredding of kitchen refuse in a grinder to maximum 2 mm. Then kitchen refuse and blackwater are mixed in a feeding tank, the mixture is pasteurized in a vessel before being fed into the anaerobic digester. The biogas is utilized in a combined power and heat unit (CHP). The digested effluent is stored in a tank. A farmer utilizes it as liquid fertilizer in agriculture. The anaerobic digestion system is now in a start-up phase.

Hamburg, Germany

A resource management sanitation concept in a settlement of 2,000 inhabitants in Hamburg is in the planning stage by the public water company Hamburg Water. Within the so called Hamburg Water Cycle, blackwater is supposed to be separated in a vacuum system and treated in a biogas digester together with other organic waste (Pauls 2007).

Sneek, The Netherlands

In Sneek, a city in the Northern part of the Netherlands, a similar blackwater system was realized in 2006, a housing estate of 32 houses is provided with a collection, transport and anaerobic treatment system for blackwater from vacuum toilets. The anaerobic technology there is different, the reactors are two UASB septic tanks of 6 m^3 each. The effluent of the UASB is subjected to a post treatment where residual COD is removed and NH_4 and phosphate are recovered (Zeeman et al. 2007).

The technical feasibility of UASB septic tank system for blackwater was demonstrated in laboratory scale before by Kujawa-Roeleveld et al. (2005). According to Zeeman et al. (2007b) an implementation of this size is not economical, but the technical feasibility of the concept including post-treatment shall be demonstrated here. Up-scaling of the system is planned for the coming years in Sneek.

1.2.2 Asian examples

On the Asian continent, the biogas technology has always been popular and has been applied for centuries. Where the frame conditions of tropical climate and small-scale agricultural activities meet a lack of energy, the low-tech biogas digesters are very successful. The biogas development in China and India will shortly be described, the situation also being similar in smaller countries like Nepal and Vietnam.

China

China is one of the leading countries in biogas technology in Asia. The main driver has been the energy production which led to the concentration of biogas plants in those areas where there are no small power stations, coalpits or possibilities for planting wood, like in the Sichuan Province (Marchaim 1992). In 1975, a big Chinese campaign calling “biogas for every household” led to huge number of construction of household biogas plants (1.6 million per year) but in 1980, 50 % of the plants were found to be defective or not in use (Marchaim 1992). Based on these experiences, the policy changed and focused on proper management (policy development, proper design, organization and financing of training, proper operation and maintenance). It was also learnt that the most important issue for the success of biogas technology for households is the immediate and obvious benefit for the people. The awareness rising about the benefit of proper effluent utilization in agriculture is therefore an important issue.

Today there are more than 5 million family sized plants of 6, 8 and 10 m^3 in operation in China. They are fed with animal waste (pig and cattle manure, and poultry), human excreta and organic waste such as kitchen residue. Constructed with bricks and cement, the household digesters are usually realized as fixed dome plants and operated as an accumulation digester (AC). It is generally underground and shaped as an egg; the headspace at the top is for gas collection.

In many Chinese regions, forest wood is increasingly used for cooking purposes causing environmental problems like deforestation and soil erosion. The restrictions by the government on tree felling and wood cutting to preserve the forest lead to an on-going interest in biogas (Knecht 2006). E.g. the project of 1,300 biogas plants in Shaanxi was awarded in 2006 with a sustainability award (Ashden 2006). In the framework of increasing energy demand in China, the Ministry of Agriculture is now addressing the importance of biogas again and wants to support the connection of households to biogas plants further. Additionally, there is also a growing interest in sustainable aspects such as water saving, so that sanitation projects started e.g. implementing vacuum toilets with blackwater digestion (Zhang 2008).

India

Similarly to China, national programs on biogas development were launched in the 1980s for mass diffusion of digesters in India. The Ministry of Energy supported the implementation of both family and community sized biogas digesters by developing guidelines and allocating budgets for training and subsidies. Great emphasis has been given on implementation, training and monitoring.

Small biogas digesters serving one or a small number of households have thus become more and more popular. Their main goal is to produce biogas and provide the family with energy mainly for cooking. Up to now, the program has supported to build 3.1 million plants in the country (Ndzana 2004). An increasing number of public toilets have been installed in India as well. Around 100 large scale plants are in operation treating blackwater from public pour-flush toilets in biogas plants.

1.3 Objectives and procedure of this thesis

There is considerable knowledge regarding anaerobic digestion of blackwater in low-tech digesters and there is some research going on in the field of characterizing urine and faeces. However, there is a lack of research about characteristics and specific loads of blackwater from vacuum toilets. The design and kinetic parameters of digestion of blackwater with kitchen refuse have not been studied yet.

The objectives of this thesis are

- to understand the process of anaerobic digestion of blackwater with and without kitchen refuse, the limiting steps and the inhibitory impact,
- to define the design criteria for anaerobic digestion of blackwater from vacuum toilets with kitchen refuse and
- to determine the potential of digesting blackwater with kitchen refuse to recover energy and nutrients within resource management sanitation concepts.

The thesis is structured as presented in Figure 3.

First, the anaerobic digestion process was studied based on the literature. The literature research focused on inhibition by high ammonia concentration. As hygienic aspects are of crucial importance for the reuse of digested blackwater, relevant references were explicitly studied (chapter 2).

The next step was the experimental and analytical work. The characteristics of blackwater from vacuum toilets and kitchen refuse were defined and assessed statistically (chapter 3). After determining the anaerobic biodegradability of blackwater, the performance of anaerobic digestion of blackwater with and without kitchen refuse was investigated in a bench scale plant. The study comprised the impact of varying hydraulic retention time (HRT) and ammonia concentrations. Different pre- and post-treatment step were additionally under investigation to assess their potential to reduce pathogens (chapter 3).

Based on the experimental and analytical results, the mathematical model ADM1 (Batstone et al. 2002a) was calibrated. The bench-scale experiments were dynamically simulated with the calibrated model (BWADM). The anaerobic processes in an up-scaled reactor were simulated with varying HRT and organic load in order to determine design criteria for a technical reactor (chapter 4).

Finally, the experimental and simulation results were put in an overall concept for anaerobic domestic wastewater treatment. In this proposed anaerobic system, blackwater from vacuum toilets, kitchen refuse and greywater are treated under anaerobic conditions, blackwater and kitchen refuse in a continuous stirred tank reactor and greywater in a high-rate reactor (UASB). The energy balance and CO_2 emissions of the proposed concept are calculated in comparison to a conventional aerobic treatment (chapter 5).

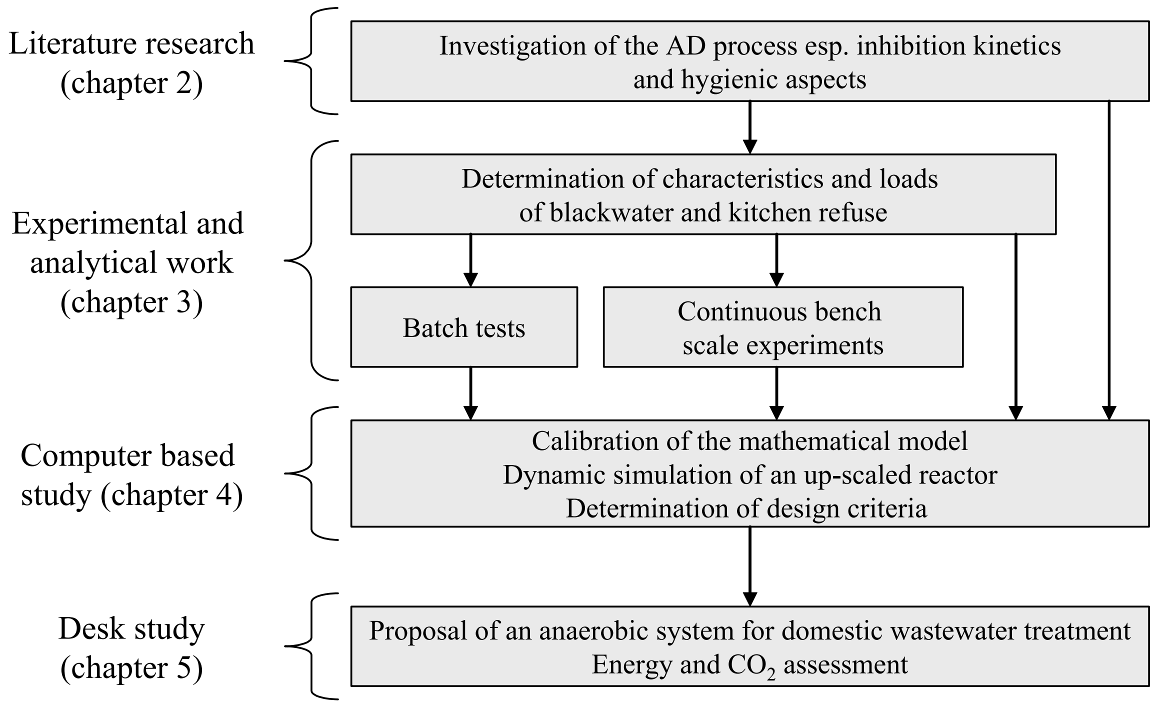

Figure 3: Scheme of the procedure in the thesis

CHAPTER 2 BASICS OF ANAEROBIC DIGESTION

Anaerobic digestion (AD) is the process of biological degradation of organic matter in the absence of oxygen under production of biogas which is a mixture of methane, carbon dioxide and traces of other gases.

AD is one of the oldest processes applied for the treatment of domestic wastewater (McCarty 1985). The oldest and simplest method is the septic tank which combines settling with digestion. The first reported installation for the anaerobic treatment of domestic wastewater was developed by Louis Mouras in France about 1860 (Dunbar 1908). AD of domestic wastewater was studied by various researchers like Imhoff in 1905 which led to the development of several anaerobic treatment systems, the best known is the Imhoff tank (McCarty 1985). More than 12 million people in Germany were served by versions of the Imhoff tank until 1945. In the following decades, AD became less popular than aerobic treatment and it remained limited to small scale plants. AD was considered to be easy to build and maintain but the predominant drawback (Table 2) was the risk of odor nuisance from the reduction of sulphate to sulphide and the requirement of post-treatment to fulfill discharge standards (Imhoff and Imhoff 2006). With the development and large scale implementation of aerobic processes for wastewater treatment, AD was predominantly applied for sewage sludge treatment to stabilize the sludge.

Table 2: General benefits and drawbacks of anaerobic digestion (van Velsen 1981, Zeeman 1991, Elmitwalli 2000)

Benefits	**Drawbacks**
Efficient in the removal of organic material, especially at temperature > 20 °C	Low removal efficiency of particulate organic material at low temperature as the growth rate of methanogenic microorganisms is low
Biogas production which can be used for renewable energy production	Requirement for pre- or post-treatment to reach the effluent standards, depending on the requirements for reuse or discharge
Reduction of formation of malodorous compounds in case of sludge AD and manure AD	Risk of odor nuisance in case of sewage AD from the reduction of sulphate to sulphide
Lower sludge production	Long start-up period when inoculum sludge is not available
Low operation and maintenance costs, especially in tropical regions, as energy consumption is low	In cold regions, heating is necessary which increases construction and operation costs
Low construction cost possible in tropical regions	

However, in the past decades high-rate anaerobic systems have been developed for the treatment mainly of industrial wastewater containing high concentrations of dissolved organic matter. In these systems, anaerobic filters (AF) and the upflow anaerobic sludge blanket (UASB) reactor (Lettinga et al. 1980), the wastewater passes through the anaerobic biomass, where dissolved substrate is then digested and the particulate matter firstly is captured and then digested (Elmitwalli 2000). The main advantage of these high-rate systems is the high efficiency at high sludge retention time (SRT) but low hydraulic retention time (HRT) that brought AD of domestic sewage a milestone forward.

AD has been applied for decades in the field of agricultural residues like manure. Here, the interest in research and application of AD has followed the demand and prices of fossil fuels. After the Second World War, the digestion of agricultural residues was applied due to the energy shortage (Zeeman 1991). After the energy crisis in 1973, AD became again more popular. In the last years, AD of energy crops alone or jointly with manure is becoming more interesting. E.g. in Germany, there are currently 2,000 biogas plants running based on manure which were pushed due to governmental subsidies for biogas production.

AD of biodegradable waste like kitchen refuse (KR) and organic waste has become more interesting within the discussion about global warming, as the energy produced by AD can replace fossil energy sources. Important is the capture and use of the produced methane because the release of methane in to the atmosphere causes worse climatic effects than CO_2 as a green house gas.

Some dry systems for AD of organic waste have been developed in the 1990s (Ten Brummeler 1993). However, separately collected biodegradable wastes are mostly treated aerobically (composting) or are rarely co-digested in biogas plants.

Table 3: Characteristics of substrates applied for anaerobic digestion

Substrate	Total COD	TS	VS	Total N	VS/N	Total P	Reference
Unit	mg/l	mg/l	mg/l	mg/l		mg/l	
Untreated domestic sewage	800	1,230	950	70	14/1	12	Metcalf and Eddy 2003
Raw primary sludge		60,000	39,000	1,500	26/1	960	Metcalf and Eddy 2003
Swine manure		25,000 – 130,000	13,000 – 80,000	3,900 – 8,000	8/1		Eder and Schulz 2006
Cattle manure		70,000 – 170,000	33,000 – 110,000	3,300 – 9,900	11/1		Eder and Schulz 2006
Kitchen refuse		375,000		7,500 [a]		1,400	STOWA 2005

[a] here NH_4-N

Table 3 gives an overview of substrates that are treated by AD (without industrial substrates). Their characteristics have a wide range. For total solids (TS), the range is dependent on the treatment status, in case of pre-thickening the substrate, the value is much higher. But also the Volatile solids/nitrogen (VS/N) ratio varies in the wide range from 8/1 to 26/1, for swine manure and raw primary sludge, respectively.

2.1 Anaerobic digestion process

AD of complex substrates containing carbohydrates, proteins and lipids such as domestic wastewater is a multi step process of series and parallel reactions. Its complexity is presented in Figure 4. These biochemical and physicochemical processes are commonly classified into the following major four stages:

- Disintegration and hydrolysis: Complex organic matter is broken down within an extracellular process into organic material with smaller particles which can be further hydrolyzed (disintegration). Particulate organic matter is converted by extracellular enzymes to monomeric or dimeric components, such as amino acids, single sugars and long chain fatty acids (LCFA). Such compounds can pass through the cell membrane (hydrolysis).
- Acidogenesis: Hydrolysis products are fermented or anaerobically oxidized to volatile fatty acids (VFA), alcohol and ammonia.
- Acetogenesis: Alcohol and VFA are converted to acetic acid or hydrogen and carbon dioxide.
- Methanogenesis: Acetic acids, carbon dioxide and hydrogen are converted to methane and carbon dioxide.

The rate-limiting step is dependent upon the substrate characteristics. In the case of high particulate matter content such as organic waste, it is usually disintegration and hydrolysis (van Velsen 1981a, Zeeman 1991). Both steps can hardly be disaggregated in practice, so that they are often summarized under hydrolysis. For predominantly dissolved organic wastewater, the rate-limiting steps are the acetogenesis and the methanogenesis via acetate as these bacteria groups have the slowest growing rates (Gujer and Zehnder 1983).

Complex wastewaters contain carbohydrates, proteins and lipids which are degraded in anaerobic process steps with different metabolites (Figure 4). Kinetics and modelling of the AD process is further explained in detail in chapter 4.

For bacterial growth, other elements are essential as well such as sulphur and trace elements (Ni, Co, Mo, Fe, Se, Wo, Zn, Cu, Mn). In domestic wastewater, there is usually no lack of such substances as they originate from urine and faeces in sufficient concentrations.

Several environmental factors influence AD, primary factors are pH and temperature:

The pH in the digester is influenced by the composition of the substrate (especially carbonate, VFA and ammonia) and the operation conditions. For hydrolysis, Madigan et al. (1997) reported that the optimum pH is around 6.0. An optimum pH near neutrality (6.5 to 7.5) should be maintained for methanogenesis (Grady and Lim 1980, van Haandel and Lettinga 1994). Some authors found the optimal pH of acetoclastic bacteria between 6 and 7 (Mudrack and Kunst 1991). If the rate of methanogenesis becomes lower than acidogenesis, the pH might reach values below 6 due to VFA increase, which is fatal for the methanogenic bacteria (Koster 1989) as the toxic un-ionized molecules of VFA increase. Generally, the alkalinity of domestic wastewater is however high enough to provide a stable AD process.

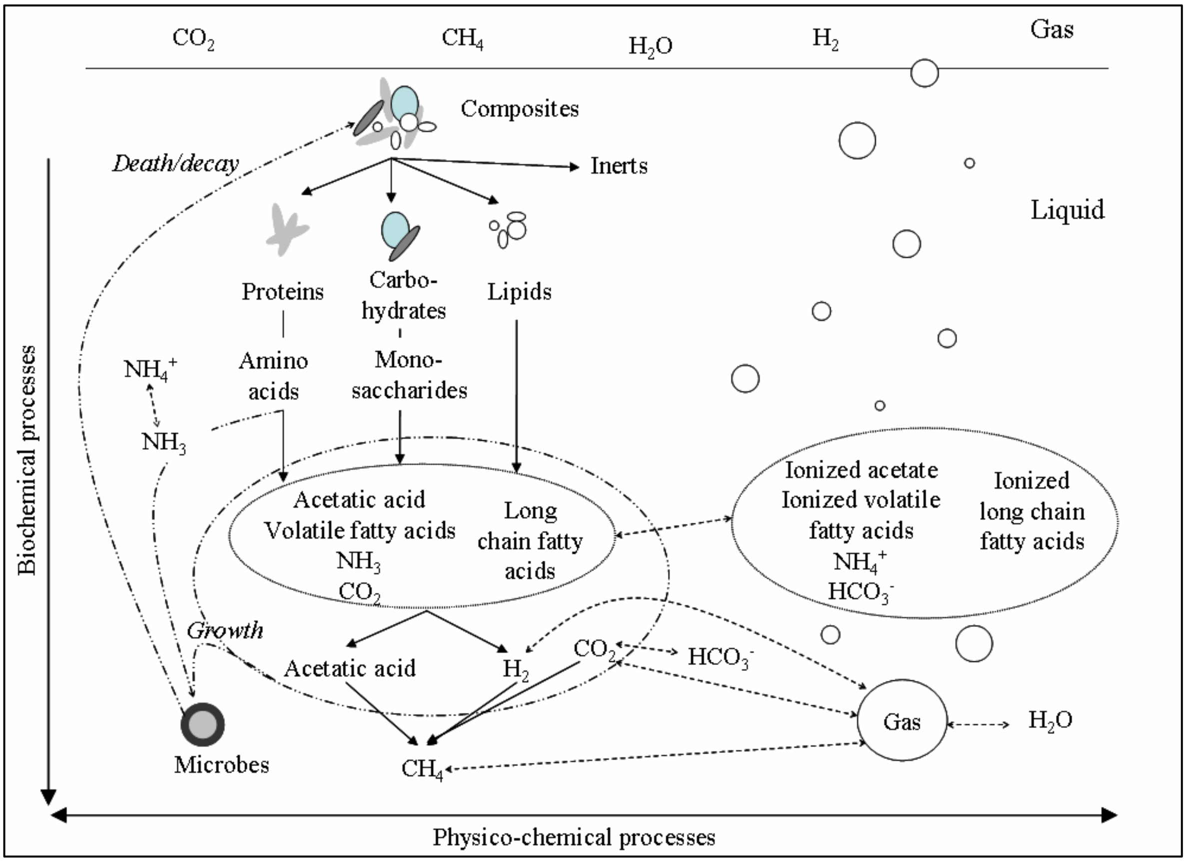

Figure 4: General processes of AD, redrawn from Batstone et al. (2002a)

Three ranges of temperature classify three types of AD: Psychrophilic (10 to 20 °C), mesophilic (20 to 40 °C) and thermophilic (50 to 60 °C) with each specific microflora. Most of AD systems are operated at mesophilic conditions because the relatively robust mesophilic bacteria and their large variety of species secure an easy and stable process. Psychrophilic operation has not been in focus due to the slower bacterial growth rate resulting in longer retention times and larger reactor volumes. However, low-temperature anaerobic wastewater treatment has gained renewed attention, especially in areas with low seasonal ambient temperatures (Elmitwalli et al. 2003, Luostarinen 2005). In the case of a fluctuating reactor temperature from 10 to 30 °C, e.g. due to seasonal temperature variation, real psychrophilic bacteria are hardly present but psychrotolerant bacteria. Psychrotolerant bacteria are able to grow slowly at 0 °C but have a temperature optimum also at mesophilic condition (Madigan et al. 1997). Thermophilic digestion can reach higher growth rates but is more sensitive to operational problems. The primary reason for its selection is the purpose of higher pathogen removal requirements.

2.1.1 Impact of high ammonia concentrations

Ammonia plays a key role in the performance and stability of anaerobic reactors operated on substrate with a high protein content. Ammonia has a positive impact on AD, as bacteria need nitrogen for their cell mass synthesis, they obtain it directly from NH_4-N.

In fresh blackwater, most nitrogen is present in the form of urea, a product of protein degradation. Urea ($CO(NH_2)_2$) is decomposed by bacteria via the following enzymatic catalyzed reaction (Fidaleo and Laveccio 2003):

$$CO(NH_2)_2 + 2\,H_2O \rightarrow 2\,NH_4^+ + CO_3^{2-} \qquad (1)$$

Ammonia and the buffer capacity

One of the most important parameters affecting reactor stability concerns the buffer capacity of the system. The bicarbonate-ammonia buffer is the primary parameter controlling the pH and process stability. The buffer capacity of a solution is determined from the concentrations of each buffer present, here ammonia and carbonate, their pK values and the pH of the solution. Due to the decomposition of urea, ammonia and carbonate are present in high concentrations in blackwater and therefore the system has a high buffer capacity. Consequently, a dramatic pH-fall below 6 as a critical value hardly occurs (van Velsen and Lettinga 1980). However, the formation of VFA (HAc) decreases the buffer capacity but the formation of NH_4^+ produced at the same time increases the bicarbonate concentration:

$$H\,Ac + NH_4\,HCO_3 \rightarrow NH_4\,Ac + H_2CO_3 \qquad (2)$$

Additional buffer capacity has sulphate contained in the substrate. As sulphate is of importance within AD, its behavior is shortly described here with the following equations:

$$SO_4^{2-} + 4\,H_2 \rightarrow H_2S + 2\,H_2O + 2\,OH^- \qquad (3)$$

$$SO_4^{2-} + CH_3\,COOH \rightarrow H_2S + 2\,HCO_3^- \qquad (4)$$

The sulphate reduction leads to a COD decrease and an increase of pH and buffer capacity. Partly the H_2S is leaving the system with the biogas flow.

If the buffer capacity is high, the AD process is very stable. If the pH increases due to high ammonia or sulphate concentrations, it subsequently inhibits the methanogenic bacteria and the declined bacterial activity leads to a VFA accumulation so that a new steady state process will be established. If the pH drop is too drastic, the AD process may irreversibly break down.

Inhibition by ammonia

High ammonia concentrations can cause inhibition of microbiological activity in the AD process. Ammonia is frequently considered as a cause for digester failure (El Mashad 2003). AD of swine manure as sole substrate was sometimes unsuccessful and the reason was assumed to be the high content of ammonia of more than 4,000 mg/l NH_4-N in this waste (van Velsen 1979). An ammonia concentration of 4,000 mg/l NH_4-N was shown to be inhibitory during digestion of cattle manure (Angelidaki and Ahring 1994). Ammonia concentrations exceeding 3,000 mg/l NH_4-N are toxic regardless of the pH according to van Velsen (1979) and Koster and Lettinga (1984). For unadapted methanogenic cultures, ammonia inhibition has been observed to start at concentration of 1,500 to 2,500 mg/l NH_4-N (van Velsen 1979b, Hansen et al. 1998). By adaptation of the biogas process to ammonium, tolerance to more than 4,000 mg/l NH_4-N has been demonstrated for swine as well as for cattle manure (Hashimoto 1986, Angelidaki et al. 1993). But still it is not clear, why AD of swine manure with similar NH_4-N concentrations is more sensitive to ammonia inhibition than AD of cattle manure (El-Mashad 2003).

Ammonia inhibits predominantly the methanogenesis (Koster and Lettinga 1984, Kroiss 1986, Angelidaki et al. 1993). Acetate utilizing methanogenic bacteria were found to be more sensitive to ammonia than hydrogen consuming ones (Poggi-Varaldo et al. 1997). The active component causing ammonia inhibition is considered to be the free ammonia concentration (Hansen et al. 1998). Two different mechanisms were attributed to ammonia inhibition: Firstly methane synthesizing enzymes are directly inhibited by free ammonia. Secondly the bacteria cell wall is far more permeable to undissociated molecules than to ions. In the cell, free ammonia is rapidly converted to NH_4^+ changing intracellular pH conditions (Kadam and Boone 1996).

The concentration of the ammonia ion (NH_4^+) and free ammonia (NH_3) are interrelated via the pH and temperature according to the following equation:

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \qquad (5)$$

The higher the pH and the temperature, the higher the free ammonia concentration is. If rising free ammonia concentration starts to inhibit the methanogenic bacteria, VFA concentration increases. This leads to a decrease of the pH and the free ammonia concentration again. In a certain range, the process stabilizes itself (Kroiss 1986).

Acetate utilizing bacteria adapted to ammonia were shown to develop with a free ammonia concentration of up to 700 mg/l NH_3-N (Angelidaki et al. 1993), while many lower free ammonia concentrations have been reported for initial inhibition of an unadapted process (Hansen et al 1998). The threshold value of free ammonia is in the range of 80 to 200 mg/l NH_3-N (Table 4).

Table 4: Inhibition thresholds of free ammonia for mesophilic anaerobic digestion

Substrate	Free ammonia concentration	pH	Reference
Unit	mg/l NH_3-N		
Potato juice	80 – 150	7.5	Koster and Lettinga 1984
Organic fraction of municipal solid waste	80 – 100		De Baere et al. 1984
Wastewater from seafood processing industry	200		Omil et al. 1995
Synthetic wastewater	200		Calli et al. 2005

Several authors found that methane fermentation of high ammonia containing wastewaters is more easily inhibited at thermophilic temperatures than at mesophilic temperatures (Hansen et al. 1998). This coincides with the fact that free ammonia concentration increases with increasing temperature. Furthermore, the biogas process becomes more sensitive towards ammonia when pH increases (Koster 1986) which again increases the concentration of free ammonia.

Hansen et al. (1998) suggested that the interaction between free ammonia, VFA and pH leads to an "inhibited steady state" which is a condition where the process is running stably but with a lower methane rate.

Thus, the ammonia concentration at mesophilic condition has two antagonistic effects, free ammonia causes an inhibition of the process but it also controls the pH and therefore inhibition by accumulation of VFA is avoided and the overall process improved (Vidal et al. 2000). Too high free ammonia concentrations inhibit the process but the extent of the inhibition depends additionally on other factors, such as the adaptation of the microflora and the characteristic of the feedstock.

Prevention of ammonia inhibition

Although the mechanisms of ammonia inhibition are not fully understood, key issues are the free ammonia concentration dependent on pH, and temperature and adaptation conditions. Many researches were dedicated to finding means to improve the performance of AD at high ammonia concentrations, some successful means most relevant for blackwater digestion are the following:

pH control and temperature adjustment: The concentration of free ammonia can be kept lower to prevent ammonia inhibition, if the operation is pH-controlled (Braun et al. 1981). Concerning temperature, mesophilic temperature is apparently more suitable to prevent ammonia inhibition than thermophilic.

Adaptation: Many references mention the importance of bacterial adaptation to ammonia concentrations but it is not clear whether the adaptation is the result of internal changes in the predominant species of methanogenic bacteria or of a shift in the methanogenic population (El-Mashad 2003). According to Koster (1986) the adaptation of the methanogenic population to high ammonia concentrations is not caused by growing of a new type of bacteria but by slow adaptation of the original population during the period of stagnation of the methane production which lasted in his experiments for around six months.

The adaptation period depends on the applied ammonia concentration and organic loading rate. The time required for complete acclimation increases with the ammonia concentrations. In un-adapted cultures, free ammonia levels of 150 mg/l NH_3-N cause growth inhibition but much higher concentrations can be tolerated by methanogenic bacteria which have undergone a period of gradual adaptation. Adequate adaptation may take two months or even longer (El-Mashad 2003). A linear correlation between the lag phase and the ammonia concentration was found by van Velsen (1981). At ammonia concentrations of 2,000 and 5,000 g/l NH_4-N, the lag phase was approximately 20 and 50 days, respectively.

Dilution of the feedstock: By diluting the feedstock, inhibition is prevented by decreasing ammonia concentrations. But this measure increases the required size of the digester and the energy needed to heat the wastewater to operational temperature. Diluting should be applied only in exceptional cases if e.g. the feedstock is too concentrated to be pumped.

Adjusting VS/N or COD/N ratio of the feedstock: More suitable to provide better operational conditions for AD is the addition of substrate that has high organic and low nitrogen content which produces better VS/N ratio for AD. Although the ammonia concentration does not decrease significantly, the process improves in terms of kinetics and becomes more stable.

2.1.2 Hygienic aspects

Anaerobic treatment reaches a relevant inactivation of pathogens, measured most often as microbiological indicators E.coli, total coliforms, enterococcus, salmonella, or helminth eggs.

In a UASB septic tank for blackwater treatment (HRT around 25 days) at 15 and 25 °C, E.coli were removed about 1 log and 3 log, respectively (STOWA 2005). The reduction of helminth eggs by blackwater AD is modest with on average 50 % inactivation or 0.5 log reduction of viable helminth eggs (Feachem et al. 1983, Gantzer et al. 2001).

Watanabe et al. (1997) analyzed mesophilic digested sludge of several wastewater treatment plants and found a decrease of around 2 log for total coliforms. Still, there were 10^3 CFU/g TS total coliforms in the digested sludge regardless of the HRT, 10^2 to 10^5 CFU/g TS enterococcus and 1.8 to 30 CFU/4 g TS salmonella. Horan et al. (2004) found a relationship of pathogen die-off and HRT in mesophilic digestion of sewage sludge. Additionally, the extent of die-off was a function of the number of pathogens in the feedstock. The log removal of pathogens increases when increasing either the HRT or the number of pathogens in the substrate.

Although mesophilic digestion has a potential to inactivate pathogens, its performance is often not sufficient to reach standards for reuse. The guidelines considered here are the guidelines of the World Health Organization (WHO), Vol. 2 and 4 (WHO 2006), US EPA guidelines for biosolids (Anonymous 1993) and the German ordinance on organic waste recycling (Anonymous 1998) because they cover or relate closest to digested blackwater. Whereas the WHO and the US EPA guidelines set standards as threshold concentrations or reduction levels which are summarized in Table 5, the German ordinance requires certain treatment such as pasteurization.

Table 5: Relevant guidelines and standards for the use of digested blackwater in agriculture

	Helminths viable eggs	**Bacteria**		
		faecal coliforms	**E.coli**	**Salmonella**
WHO Guidelines for the safe use of wastewater, excreta and greywater (WHO 2006)				
Volume 4: Excreta and greywater use in agriculture				
Use of treated faeces and feacal sludge in agriculture [b]	< 1 number/g TS		< 1,000 number/g TS [c]	
Volume 2: Wastewater use in agriculture				
Unrestricted irrigation [b]	< 1 number/l [a]	6 - 7 log pathogen reduction required		
Restricted irrigation [b]	< 1 number/l [a]	3 - 4 log pathogen reduction required		
Localized (drip) irrigation [b]	< 1 number/l [a]	2 - 4 log pathogen reduction required		
US EPA Standards for biosolids 40 CFR Part 503 (Anonymous 1993)				
Unrestricted use (Class A)	< 1 number/4 g TS	< 1,000 number/g TS		< 3 number/4 g TS
Restricted use (Class B)		$< 2 \cdot 10^6$ number/g TS		

[a] When children under 15 years are exposed, additional health protection measures should be used

[b] Recommendation for 10^{-6} DALY (Disability adjusted life year)

[c] for large systems

Pathways to reach a further inactivation of pathogens that are relevant for blackwater and kitchen refuse are the following:

Thermophilic digestion

For complete inactivation, AD at thermophilic condition has proven to be efficient for sewage sludge by many researchers. Watanabe et al. (1997) found on several wastewater treatment plants 1 CFU/g TS faecal coliforms and enterococcus and 1.8 CFU/4 g TS salmonella in the effluent of the thermophilic digester. Due to the high concentration of organics in the effluent, the bacteria are however easily reactive and re-grow again (Iranpour et al. 2005).

Pre-treatment

Pasteurization: A second option for inactivation of pathogens is a pre-treatment before AD. Pasteurization as thermal pre-treatment can be applied, either 24 hours at 55 °C or 1 hour at 70 °C is assumed to inactivate pathogens sufficiently and is required by the German ordinance on organic waste recycling (Anonymous 1998). The heating must be operated in a batch modus to guarantee the pasteurization effect.

Acidification: Some research was carried out to inactivate pathogens by acidogenic pre-digestion (acidification). VFA in high concentrations, especially the un-ionized molecules, are toxic to bacteria similarly to free ammonia. Kunte et al. (2004) showed in laboratory scale-experiments with pre-treatment of blackwater that complete inactivation of salmonella typhi, shigella dysenteriae and vibrio cholerae were achieved at levels above 15,000 mg/l VFA and pH around 6 (30 °C and 5 days HRT). They observed still a methane production on a low level in the acidogenic digester.

Puchajda and Oleszkiewicz (2004) analyzed faecal coliforms in the effluent of an acidogenic batch digester for sewage sludge at 21 and 37 °C. After 5 days, the sludge at 21 °C achieved a complete inactivation of faecal coliforms at pH of 5.4 and un-ionized VFA concentration of 700 mg/l while at 37 °C, 10^3 CFU/g TS faecal coliforms were analyzed in the effluent at pH of 6.4 and un-ionized VFA concentration less than 50 mg/l. Even by increasing the organic loading rate, the pH decreased further in the mesophilic experiments. This might be explained by the methanogenic bacteria that consume the VFA faster in mesophilic condition than at lower temperature. Thus, the key parameter is a pH less than 6 for inactivation of pathogens by un-ionized VFA. To achieve such a low pH in the pre-treatment step, the reactor temperature should be lower than mesophilic. This pre-treatment has not been applied in large scale.

Post-treatment

Pasteurization: Another option for pathogen inactivation is the post-treatment of the AD effluent. As well as for pre-treatment, pasteurization is also a suitable post-treatment to inactivate pathogens. Roediger et al. (1990) reports an important drawback of post-pasteurization. In contrast to the pre-pasteurization, the bacteria, also the anaerobic microorganisms, are killed and substrate is hydrolysed again. This can lead to a quick re-growth of pathogens which easily enter the substrate thereafter e.g. during storage. That is why post pasteurization plants are rarely operated in practice.

Liming is an old method to treat digested sewage sludge. Slaked lime ($Ca(OH)_2$) or quick lime (CaO) can be applied, both inactivate pathogens due to pH increase. In case of quick lime, additionally the temperature rises to between 55 and 70 °C due to the exothermic reaction which causes a second efficient effect of killing bacteria. For sewage sludge, German guidelines recommend to apply slaked lime to achieve pH of 12.5 in the sludge and to store at least three months for safe decontamination (ATV 1998). Addition of quick lime requires two hours at pH of 12.5 and 55 °C or more (Bischofsberger et al. 2005). Helminth eggs are most resistant to liming, Capizzi-Banas et al. (1994) determined the inactivation threshold for the kinetics where a negligible level of Ascaris eggs was reached. Depending on the experimental situation, the inactivation threshold period was found to fluctuate between 5 and 75 minutes at 55 °C and between 1 and 8 minutes at 60 °C.

Post-storage: For inactivation of pathogens, the storage of faecal sludge and faeces is recommended before application on the fields by Haible (1989) and WHO (2006). Haible (1989) studied the long-term open storage of sewage sludge at ambient temperature in Germany. She found no salmonella and no enterovirus after 5 and 12 months of storage, respectively. After 12 months, enterococcus were reduced to 10^5 CFU/g TS.

Composting is a simple technology to reduce pathogens due to the biologically produced heat but it can only be applied to dry material (TS > 30 %). For adequate composting, digested sludge or blackwater must be dewatered which requires a mechanical device such as centrifuge.

2.2 Types of anaerobic reactors

The general types of anaerobic reactors which are relevant in this thesis are shortly presented here.

2.2.1 Continuous stirred tank reactor (CSTR)

This reactor is characterized by continuous and constant rates of both feeding and discharging. The system has a complete mixing of substrate and bacteria. Both bacteria and wastewater have the same retention time, so hydraulic retention time (HRT) equals solid/sludge retention time (SRT). The term steady state can be applied to the CSTR under condition that the system is well adapted to the substrate and the loading is constant. Steady state conditions will ensure stable biogas production and output quality. This system was applied mostly for sewage sludge and manure digestion at mesophilic conditions. Benefits and drawbacks of CSTR are summarized in Table 6 (Fischer et al. 1986).

The CSTR system operates usually well at HRT above 15 or 20 days at mesophilic conditions. In general, it can be stated that mesophilic CSTR can be applied when the input is so concentrated as to at least provide enough biogas to produce the heating energy for the system (STOWA 2005). The higher the concentration of organic matter to be degraded, the more surplus energy is produced. Another important economic benefit is that the reactor volume becomes smaller assuming that the same HRT can be applied for a diluted and concentrated wastewater.

Low-tech systems operated at ambient temperature and fed discontinuously, e.g. daily, are also comprised under CSTR. They mostly have higher HRTs of up to 100 days.

Table 6: General benefits and drawbacks of continuous stirred tank reactors (CSTR)

Benefits of CSTR	**Drawbacks of CSTR**
High reliability	Big reactor volumes as HRT = SRT
Applicable for substrate with high suspended solid concentration	Required energy for mixing
Good contact between substrate and micro-organisms	Possibility of undigested sludge substrate leaving the reactor
Homogeneous temperature throughout the tank	
Prevention of scum layer formation	

The accumulation reactor (AC) is a variant of the CSTR, both systems being continuously fed. While the output from the CSTR is continuously removed, the AC reactor is always filled until the effluent is only removed once, at the end of the filling period. The CSTR has a constant digestion volume while that of the AC system is increasing with time. The AC reactor has been applied for manure digestion (Wellinger and Kaufmann 1982) and for mixtures of manure and household waste and wastewater. The main benefit of AC reactor is the combination of digestion and storage, as in agricultural application, a storage tank is usually required to overcome the periods where organic fertilizer cannot be applied on the fields (El-Mashad 2003). In the last years, the application of the UASB septic tank as a combination of high-rate and accumulation reactor was investigated as a promising alternative.

2.2.2 Upflow anaerobic sludge blanket reactor (UASB reactor)

Several high-rate anaerobic systems were developed during the past decades, like the anaerobic filters (AF) (Young & McCarthy 1969), the upflow anaerobic sludge blanket (UASB) reactor (Lettinga et al. 1980) and the baffled reactor (Bachmann et al. 1985). The common feature between these reactors is that they operate at long SRT and short HRT. Amongst these systems, the UASB reactor is the most widely applied. The success of the UASB reactor can be attributed to its capability to retain high concentration of active suspended biomass with simple and low cost means. The formation of granular sludge which has a high methanogenic activity and is better settlable than flocculant sludge improves the maximum loading rate of the UASB system especially for high-strength wastewater with a high dissolved COD fraction. UASB reactors are also applied for domestic wastewaters; although no formation of granular sludge takes place, they are efficient at ambient and mesophilic temperature (Elmitwalli 2000, Wendland et al. 2007). The generated sludge within the UASB reactor is stabilized simultaneously and is discharged from time to time, depending on the suspended solid fraction in the influent (Halalsheh 2002).

For onsite treatment of domestic wastewater, a variant of the UASB reactor, the so-called UASB-septic tank was first investigated by Bogte et al. (1993). The major difference in relation to the conventional UASB systems is that the UASB-septic tank is designed also to accumulate and stabilize the sludge. In relation to the conventional septic tank system, it differs by the up-flow mode implemented, achieving an improved suspended solid removal and better biological conversion (STOWA 2005). Bogte et al. (1993) reported a high removal efficiency at a temperature above 12 °C. Below 12 °C, the conversion of VFA to methane was too slow. In that case, Zeeman and Lettinga (1999) propose the pre-treatment in a UASB reactor to support hydrolysis.

2.2.3 Batch reactor

The batch reactor starts with a certain amount of inoculum and is filled with raw substrate. After the digestion, the reactor is emptied and refilled again. This reactor is very simple in design and in operation. Predominantly, batch reactors are applied to determine biodegradability at bench scale.

At technical scale batch reactors have been rarely applied e.g. for dry AD of solid wastes (Ten Brummeler 1993). Its operational drawbacks are firstly the accumulation of metabolites in the start-up phase and secondly great differences in the gas production rate over the digestion time. To produce a more continuous biogas production, Zeeman (1991) proposed operating several batch digesters consecutively.

CHAPTER 3 EXPERIMENTAL WORK

The focus of the experimental work is to find out the performance of AD in a CSTR with blackwater from vacuum toilets and together with kitchen refuse. The experimental steps are presented in Table 7. To obtain reference values for the further research, the maximum anaerobic biodegradability of blackwater and kitchen refuse is determined in batch experiments. The performance of AD of pure blackwater is first investigated at HRT of 20 days. Special attention is drawn to the role of ammonia because high concentrations due to a higher proportion of urine in the blackwater may affect the AD process significantly. As the HRT plays a key role for the investment costs of a CSTR, the limits of varying HRT were studied. Finally the pre-treatment step as required by German legislation is studied in terms of pathogen removal and consequences for the AD process.

Table 7: Overview of the experimental steps

Experimental step	Substrate	Reactor
1. Determination of the characteristics and loads	BW and KR	-
2. Anaerobic biodegradability and hydrolysis constant	BW and KR	Batch test (multiple flasks system)
3. Performance of BW AD	BW	Bench scale CSTR (10 l)
4. Impact of increasing ammonia concentration at stable and increasing pH	BW	Bench scale CSTR (10 l)
5. Co-digestion with KR and assessment of different HRT	BW and KR	Bench scale CSTR (10 l)
6. Impact of pre-treatment on microbiological parameter	BW	Bench scale CSTR (10 l)

3.1 Material and methods of the experimental work

3.1.1 Substrates

Blackwater

Blackwater (BW) was taken from the settlement *Flintenbreite* in Lübeck, Germany. The installed vacuum toilets use 0.7 to 1.0 l per flush and the blackwater was sucked with a pressure of 0.3 to 0.5 bar by the vacuum pumps into a vacuum tank. The blackwater was stored around two days in this tank before being pumped into a bigger storage tank outside. The blackwater samples were taken every week or every two weeks from this vacuum tank.

For the batch experiments, the blackwater was taken and used at once.

For the CSTR experiments, the blackwater was stored in the refridgerator at 7 °C until use. The storage time was not longer than five days which is acceptable as the retention time in the blackwater vacuum collection system is in the same range. One hour before feeding, it was taken out of fridge and heated up to 37 °C in the water tub.

Kitchen refuse

Kitchen refuse (KR) was collected from the student restaurant left over at TUHH which represents the waste from food preparation as well as the refuse from the dishes over one day. The students can choose between salad plus bread and five different dishes including different soups and desserts. It can be concluded that the mixture of one day-KR was containing raw and cooked food, bread, meat and therefore representative of kitchen refuse. This was additionally controlled when taking the samples.

For the batch experiments, two charges of kitchen refuse were taken and used at once.

For the CSTR experiments, two big charges of several kg are taken, prepared for the reactor feeding (by homogenization and shredding below 2 mm) and frozen in small portions in the deep freezer. Two hours before use, a portion was taken out and heated up to 37 °C in the water tub.

For single experiments investigating the acidification as pre-treatment, synthetic kitchen refuse was produced to guarantee a definite composition. Therefore, half stewed apples and half bread crumbs were mixed.

Inoculum sludge

Inoculum sludge was taken from a mesophilic sludge digester from a municipal wastewater treatment plant Seevetal, Landkreis Harburg. The digester is operated at HRT of more than 20 days.

3.1.2 Experimental set-up

Batch experiments

For the determination of anaerobic biodegradability, batch tests were carried out. Pre-tests with blackwater according to a German standard method (Anonymous 1985) resulted in unreliable and hardly reproducible data. Thus a multiple flasks system was selected where several flasks of polyethylene (0.5 ml) represent one batch “reactor” (Sanders 2001). At the beginning, all flasks had the same content. It is assumed that the anaerobic processes are similar in all flasks. All flasks were closed, the biogas escaped through an airtight tube which purges in a water tub but was not collected. The multiple flask system was stored in a room at 30 °C. After definite durations, the “reactor” was sampled, three flasks were emptied, the content was analyzed and the concentrations were averaged.

Two series of batch tests were carried out:

Batch test serie B1 was carried out with homogenized blackwater only. Each time the “reactor” was sampled (five times in defined intervals within 22 weeks) three flasks were emptied, the content was analyzed and the concentrations were averaged.

Batch test serie B2 was carried out with blackwater and inoculum sludge. Therefore a big quantity of blackwater and inoculum sludge from a mesophilic sewage sludge digester was mixed half/half and homogenised. Half of the flasks were filled with 400 ml of the mixture. Similarly, another half of the flasks were filled with 400 ml inoculum only. Each time the "reactor" was sampled (five times in defined intervals within 14 weeks) three flasks were emptied, the content was analyzed and the concentrations were averaged. Similarly, three flasks with inoculum sludge were emptied and analyzed. Due to the reference flasks with inoculum, the data for pure blackwater could be calculated.

CSTR experiments

At the Institute of Wastewater Management and Water Protection, the bench scale plant was installed (Figure 5). It consisted of three parallel continuous stirred tank reactors (CSTR). Made of PVC cylindrical material, each reactor has 10 l capacity corresponding to a 190 mm diameter and 355 mm height. The horseshoe mixer was actuated by an electric engine. The reactors were kept at mesophilic temperature (37 °C) in a tub filled with water. The water temperature in the tub was controlled by a thermostat. Water losses through evaporation were reduced by spherical plastic balls covering the water surface. The reactors were filled up to 8.5 l and 1.5 l was headspace.

All hose connections of the reactors were placed at the top. Firstly, the headspace of each reactor was connected to a gas counter via a flexible PVC hose (diameter 7.5 mm). In front of the gas counter, a gas mouse was placed enabling the sampling of biogas for composition analysis. Secondly, a flexible PVC hose (diameter 7.5 mm) was connected at the top of the reactor to a tube with a length of 120 mm that was inserted from the top vertically down inside the reactor. This hose was used for taking output samples from the reactor centre and feeding via an electrical peristaltic pump. Thirdly, each reactor was equipped with a flexible U-tube for pressure equilibration during feeding. Having an inside diameter of 38 mm the U-tube was partly filled with a sealing liquid of pH 2. This liquid (200 g sodium chloride and 5 g citric acid per 1 l deionized water) prevents the dissolution of CO_2 from the biogas. Methyl orange indicator was added to check the acidity. Fourthly, at the top of the reactor a hole for a pH-electrode was plunged into the reactor which measured the pH value online. Due to an airtight sluice, the pH electrode could be checked and cleaned regularly. Each hose was equipped with a lab glass valve for maintenance and easy disconnection of parts of the installation. Most important was the water and airtight installation of the whole bench scale plant which was controlled regularly with the empty plants by pressure tests.

The daily amount of produced biogas was continuously measured by gas counter connected to each reactor. The gas counter was constructed based on the development of the Milligascounter by Scherer (2002). It works by liquid displacement and allows the precise measurement of small and fluctuating gas flows.

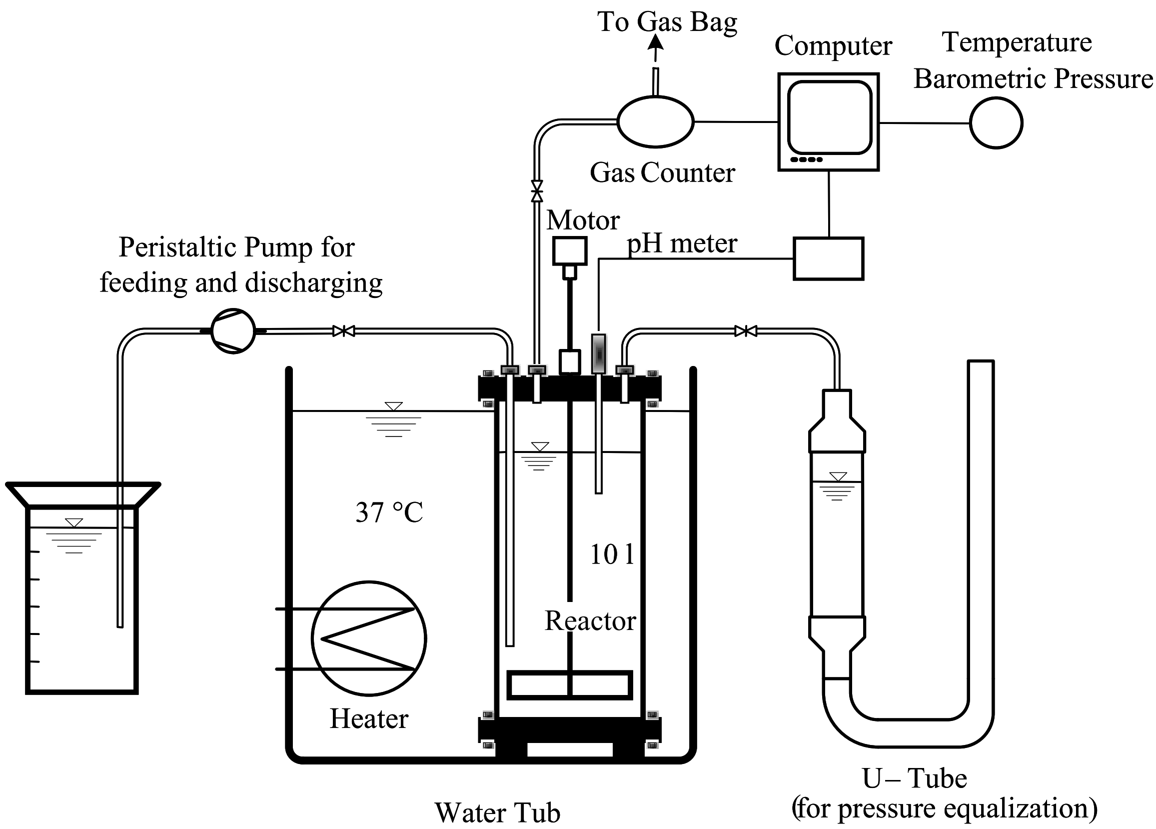

Figure 5: Scheme of the CSTR

The gas counter device consists of a cylindrical tube of PVC (diameter 135 mm and height 165 mm) and is filled with 1.5 l of the same sealing liquid as the U-tube. It is equipped with a chamber with two equal compartments made of acrylic glass which rotates via an axis when one compartment is filled with gas. The gas flow produces a flipping of the chamber which is detected via a magnetic signal. A sensor delivered the signal every minute to the data processing computer online. By means of online measurement of air temperature and barometric pressure, the produced biogas was recalculated continuously (every minute) for standard temperature and pressure (T = 0 °C, p = 1,013.25 mbar) according to Anonymous (1985).

Pre- and post-treatment

Pre-pasteurization of the feedstock was carried out according to the German organic waste ordinance (heating at 70 °C for one hour or 55 °C for 24 hours) (Anonymous 1998). Blackwater or mixture of blackwater and kitchen refuse was put in a pot on a hot plate, heated and mixed from time to time manually. After reaching 55 and 70 °C in the feedstock, the pot was put in a hot cabinet for one hour and 24 hours, respectively.

Pre-acidification: Additionally, investigations into acidification of the feedstock were carried out in terms of microbiological parameters. Therefore, the blackwater or the mixture of blackwater and kitchen refuse was divided in several polyethylene flasks. The flasks were closed and put at constant temperature (20 or 37 °C) in a cabinet for up to six days without mixing. Upon each analysis, one flask was utilized (multiple flasks system).

Post-storage: From the digested output, four samples at different times were taken. Several 250 ml polyethylene flasks were filled up to 200 ml with the output. The flasks were closed but still allowing a gas exchange and stored at ambient temperature inside. The temperature varied seasonally from 15 to 27 °C. The storage time was up to 90 days. The multiple flask system was applied again for sampling.

3.1.3 Chemical and biological analysis

For the batch experiments, the chemical analysis was carried out from the input and the output, only the biogas quantity was measured continuously and the biogas quality at once.

During the CSTR experiments, the chemical analysis was carried out every week in input and output, the biogas quantity and pH was measured continuously, the biogas composition around every two weeks according to the requirements.

Chemical oxygen demand (COD)

COD was determined photometrically after 120 min oxidation time at 148 °C with cuvette tests. The organic material was oxidised with $K_2Cr_2O_7$ and the quantity of $Cr_2O_7^{2-}$ spent was calculated as O_2.

For determination of total COD, the samples were homogenized with a rotor stator homogenizer. For analysis of kitchen refuse, the sample was 20 times diluted and homogenized. Dissolved COD concentrations were measured after membrane filtration at 0.45 µm.

Volatile fatty acids (VFA)

For determination of VFA, H_2PO_4 was added to the sample and the solution was distilled with water steam (Büchi 321, Switzerland). After 10 min boiling in the Büchi apparatus, the solution was cooled down, spiked with phenolphthalein and titrated with NaOH solution (0.1 mol/l) until the pink colour appeared. The concentration of organic acids was determined in terms of equivalents (mol VFA/l) and recalculated stoichiometrically in acetic acid equivalents and finally COD-equivalents.

Total organic carbon (TOC)

TOC was analyzed with the TOC/TN Analyzer multi N/C 3000 by Analytik Jena, Germany. After acidification of the sample with phosphorus acid, the generated CO_2 in the gas phase was determined as inorganic carbon. Then the whole sample was evaporated and the organic parts were oxidized with O_2 at max. 1,000 °C. The generated CO_2 was measured again by infrared absorption.

Total solids and volatile solids (TS and VS)

To determine TS, a small sample volume was stored in a glass envelope at 105 °C for at least 24 hours until the total moisture was evaporated. After cooling down, the sample was weighed again. The difference in weight between the envelope and the sample before and after evaporation represented the TS.

To determine the VS, the sample was further burnt in an oven at 550 °C for two hours. After cooling down, the sample was weighed again. The difference in weight between the envelope and the sample before and after burning represented the VS.

Total nitrogen (TN)

Total nitrogen was analyzed with the TOC/TN Analyzer multi N/C 3000 by Analytik Jena, Germany, as well as with cuvette tests. In the TOC/TN Analyzer, all nitrogen components were transferred to NO by thermo-catalysis according to German standard methods. The determination of NO was carried out by chemiluminescent detection.

The photometric determination was carried out with cuvette tests. A comparison of both analytic methods showed a very good correlation but the photometric test had a higher standard deviation. However, the analytic methods were both used adequately.

Ammonium (NH_4-N)

Ammonium was analyzed with cuvette tests. Salicylate and hypochlorite were added to produce monochloramine which was then measured photometrically.

Total phosphorus (TP)

Total phosphorus was analyzed with cuvette tests. The generated blue colour of phosphorus molybdenum was determined photometrically after adding molybdate, antimony and ascorbic acid.

E.-coli, total coliforms and enterococcus

The analysis was based on colony forming units (CFU) which developed over 24 hours for E.coli, total coliforms and 48 hours for enterococcus on a nutrient selective agar at 37 °C. Chromocult-Coliformen-Agar and Chromocult-Enterokokken-Agar was chosen as selective nutrient agar for determination on E.coli and total coliforms.

The determination of the microorganisms in the raw blackwater required a dilution series because of its high concentration. 100 µl of the homogenised diluted sample was put directly on the agar which resulted in a detection limit of 1,000 CFU/100 ml.

In the treated blackwater, 100 µl of the homogenised sample was put directly on the agar. If CFU were not countable, 1 ml of the sample was filtrated (0.45 µm) assuming that the bacteria were completely attached to the suspended solids. Then the filter was put on the agar which resulted in a detection limit of 100 CFU/100 ml.

All microbiological analyses were executed in duplicate and averaged. If required, the bacteria concentration was calculated from CFU/100 ml to the unit CFU/g TS by using the TS value.

3.1.4 Data processing and calculations

As blackwater and kitchen refuse are very heterogeneous substrates, the statistical calculations are playing an important role to assess the processed data and calculate with them in a proper way.

Statistical methods and error assessment

For determination of the characteristics of blackwater, it is assumed that the measured concentrations are following the normal distribution. It is further assumed that the amount of 95.5 % within this distribution is exact enough. Thus, all figures which were above or below the confidence interval (average ± 2 * standard deviation) were eliminated ({Bokranz, 1986 52 /id}).

To assess the overall error of sampling and analysis, the COD mass balance was calculated for all experiments. For the batch experiments, the COD mass balance was calculated based on the overall COD loads. For the continuous experiments, the COD mass balances were calculated based on accumulated COD loads at least over one HRT.

Calculations

For the CSTR experiments with no changing of frame conditions, the calculations were done based on total loads summed up over the whole duration. In case the frame conditions were changing like HRT or nitrogen concentration, all calculations were done based on weekly loads as the analysis was carried out weekly. The data were processed after attaining steady state conditions which was usually two HRTs after the modification.

Anaerobic biodegradability (AB) is usually determined in batch tests and represents the methanogenesis or methanisation after a defined period. After that period, it is assumed that the further produced methane is neglectable. If AB cannot be determined in a reproducible way, the total COD removal can be taken instead as anaerobic biodegradability.

$$AB = \frac{\text{accumulated } CH_4 \text{ - COD} \cdot 100}{\text{total COD input}} \quad (6)$$

Where AB = Anaerobic biodegradability (%)

CH_4 – COD = methane load in COD equivalents (350 l CH_4 is stochiometrically equivalent to 1 kg COD at STP (g)

total COD input = COD load fed to the reactor (g)

COD removal was calculated for total COD, diss COD and part COD. These processes should consistently be called COD conversion. However, as mostly used in scientific literature the equation is the following:

$$\text{COD removal} = \frac{(\text{COD input - COD output}) \cdot 100}{\text{COD input}} \quad (7)$$

Where: COD removal (%)

COD input = COD load fed to the reactor (g)

COD output = COD load discharged from the reactor (g)

Free ammonia concentration was calculated according to:

$$NH_3\text{ - N} = \frac{NH_4\text{ - N}}{1 + 10^{(\text{pKa-pH})}} \quad (8)$$

Where: NH_3-N = concentration of free ammonia (mg N/l)

NH_4-N = concentration of ammonia (mg N/l)

pK_a = dissociation constant for ammonia ion, dependent on temperature

3.2 Determination of the characteristics and daily loads of blackwater from vacuum toilets and kitchen refuse

The characteristics of the substrates were determined for organic matter, nutrients and microbiological indicators. Based on the measured concentrations and the specific flow in *Flintenbreite*, the daily loads were calculated.

3.2.1 Blackwater

Compared to typical raw domestic wastewater, blackwater from vacuum toilets is relatively homogeneous due to the pressure of 0.3 to 0.5 bar in the vacuum system.

The analyzed concentrations of the typical wastewater parameter (Table 8) are much higher than typical German wastewater due to the low water demand for flushing the vacuum toilets. The daily water consumption for the vacuum toilets was calculated to be 5.0 l/cap/d in *Flintenbreite*.

Table 8: Characteristics of raw blackwater, concentrations ± standard deviation

Parameter	Unit	No samples	Blackwater (BW)
Total COD	mg/l	119	8,060 ± 2,950
Dissolved COD	mg/l	22	2,440 ± 670
VFA-COD	mg/l	19	1,640 ± 470
Particulate COD	mg/l	20	6,010 ± 2,790
TS	mg/l	123	6,530 ± 2,110
VS	mg/l	119	4,090 ± 1,830
TOC	mg/l	121	2,410 ± 720
NH_4-N	mg/l	115	1,111 ± 137
Total N	mg/l	126	1,495 ± 244
Total P	mg/l	9	175
pH		357	7.7
Alkalinity	mmol/l	3	93
Ratio particulate COD to total COD [a]	-	20	69 %
COD/N/P	-		53/10/1
VS/N/P	-		24/10/1
E.coli	CFU / 100 ml	13	$9.1*10^7$
Total coli	CFU / 100 ml	13	$1.3*10^8$
Enterococcus	CFU / 100 ml	13	$3.1*10^7$

[a] calculated as average of the ratios of the correspondent values

The concentrations are significantly varying, especially for the organic parameter COD, VS and TOC. The high standard deviations result from the high fraction of solids and the sample taking.

The dissolved COD is represented by biodegradable COD (mainly VFA) and inert dissolved COD. The major part of the VFA is acetic acid with 63 % (see Figure 6), followed by propionic acid with 24 %. The particulate COD (> 0.45 µm) in blackwater represents 69 % of the total COD which fits in the typical range of domestic or municipal wastewater from 65 to 80 % as reported by Wang (1994) and Elmitwalli et al (2002).

The ratios COD/N/P of 53/10/1 and VS/N/P of 24/10/1 demonstrates that there is a high part of nitrogen and phosphorus in the blackwater. The nutrients are present in relatively high fractions compared to typical ratios of 100/10/1 for municipal wastewater which can be explained by the absence of greywater. The ratio is also lower compared to the other substrates used in AD (see Table 3).

As expected, the indicator microorganisms E.coli, total coliforms and enterococcus are highly concentrated in raw blackwater. They are ranging from 10^7 to 10^8 CFU/100 ml.

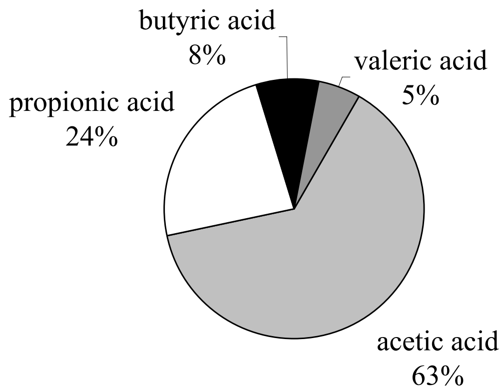

Figure 6: Composition of the VFA in blackwater based on COD (3 samples)

Table 9: Blackwater loads in *Flintenbreite* compared to reference data

Parameter	Unit	*Flintenbreite*		Vinnerås et al. 2006	Otterpohl 2002	Del Porto and Steinfeld 2000
		Own analyses	Assumed that 100 % BW is collected	Analyses in Swedish housing estates	Calculated from medical data	North-American and European studies
Quantity	l/cap/d	5.0	8.3			
total COD	kg/cap/year	14.7 [a]	24.5 [a] 17.9 [b]		17.7 [b]	18.3 [b]
Ratio *Flintenbreite* to reference	-				83 %	81 %
TS	kg/cap/year	11.9	19.9	32.0		
Ratio *Flintenbreite* to reference	-			37 %		
VS	kg/cap/year	7.5	12.5			
TOC	kg/cap/year	4.4	7.3			
TN	kg/cap/year	2.7	4.6	4.6	4.4	4.8
Ratio *Flintenbreite* to reference	-			60 %	63 %	58 %
TP	kg/cap/year	0.32	0.53	0.55	0.68	0.73
Ratio *Flintenbreite* to reference	-			67 %	55 %	51 %

[a] including toilet paper
[b] excluding toilet paper

Considering the daily loads of blackwater, the values are significantly lower than the loads in human excreta reported by other authors (Table 9). Many references present loads but they are varying highly and are often based on average estimated design values. Latest efforts were carried out by Vinnerås et al. (2006) who evaluated extensively the data of two Swedish housing estates with separating systems. It is apparent that for all parameters, the loads in *Flintenbreite* are less than in the reported articles.

For the parameter nitrogen, the ratio *Flintenbreite* to reference is between 58 and 63 %. Regarding the phosphorus load, the differences of 51 to 67 % are varying similarly to nitrogen. For the COD load, the ratio *Flintenbreite* to reference is with 81 and 83 % higher than for N and P. There is significant effect of the quantity of used toilet paper on the COD load. This is reflected in high differences of the TS load as well.

Based on these data, it is assumed that the nitrogen loads are most reliable and it can be concluded that 60 % of the totally produced nitrogen load in BW is collected. The missing part is caused mainly by two facts:

Firstly, the housing estate *Flintenbreite* is a living space, the employed persons are not present most of the day. Secondly, about 40 % of the inhabitants are children who produce less excreta and are half-time not at home either, but in kindergarten and at school. It can be further assumed that the distribution of urine and faeces in blackwater appears to occur consistently distributed throughout the day (Hellström and Kärrman 1996, Pharmacia 2000).

Assuming 100 % collection of blackwater, the resulting COD of 24.51 kg COD/cap/year is relatively high. Taking the toilet paper consumption into account, the average use of toilet paper is around 5.5 kg/cap/year which results in 6.6 kg COD/cap/year (Dockhorn 2007). The COD load of blackwater without toilet paper would be 17.9 kg COD/cap/year, which fits exactly to the reference values. This confirms the fact that 60 % of the total blackwater load is collected in *Flintenbreite*.

3.2.2 Kitchen refuse

Kitchen refuse is a part of the organic waste which is collected separately in many Western European cities and villages. The specific load of organic waste and kitchen refuse is of course varying even more than blackwater. The degree of collection is mainly depending on the urbanization rate as shown for the Netherlands by CBS (2003). Separate collection of kitchen refuse, small garden waste and bulky garden waste is more usual in villages than in towns. CBS (2003) ranked the urbanization rate from very low to very high. The higher the urbanization rate, the lower the volume of collected organic waste per capita. In most rural areas the volume of collected organic waste is 122 kg/cap/year as opposed to large cities with only 22 kg/cap/year. However, the big part of bulky garden waste collected in the villages is not appropriate for AD but much more suitable for composting on site.

The quantity highly depends on diet and region, but world wide data apart from Western Europe are rarely known as organic waste is not used to be collected separately throughout the world. Assuming that 30 % of the total solid waste load is organic waste, the figure ranges from 438 kg/cap/year in Singapore to 60 kg/cap/year in Vietnam but only a part of it might be feasible to collect separately.

In Germany, there are detailed data for separately collected organic waste but often not disaggregated by source. For this work, data for kitchen refuse in Germany from several authors (summarized by Wendler 2005) were averaged resulting in 73 kg/cap/year. The kitchen refuse used in this work was taken from the student restaurant at TUHH and represents a good mixture of kitchen refuse. For the representative mixture of blackwater and kitchen refuse, 73 kg/cap/year adequates a ratio 40 g kitchen refuse per l blackwater.

The characteristics in Table 10 are based on analyses of the used kitchen refuse after grinding and the mixture of blackwater and kitchen refuse that was fed to the reactors. The synthetic kitchen refuse was also analyzed and had the same characteristics in terms of organic matter, only nitrogen and phosphorus concentrations were much lower.

The COD is much higher in kitchen refuse than in blackwater due to higher TS values while the ratio particulate COD to total COD is with 73 % similar and only little higher than in blackwater. When adding kitchen refuse to the blackwater, the concentration of particulate COD decreases to

59 % although it should increase. This difference is assumed to be caused by the fact that there is an immediate start of hydrolysis after mixing of blackwater and kitchen refuse and there is a time difference up to 24 hours between mixing, sample-taking and analyzing. Regarding the ratio COD/N/P, the part of organic matter is much higher which leads to a better mixture of blackwater and kitchen refuse for anaerobic treatment.

Table 10: Characteristics of kitchen refuse (2 samples) and the mixture of blackwater and kitchen refuse (25 samples), concentration ± standard deviation

Parameter	Unit	KR	BW+KR
Total COD	mg/l	297,210	17,690 ± 4,530
Dissolved COD	mg/l	80,330	6,780 ± 1,070
Particulate COD	mg/l	216,880	10,260 ± 3,620
TS	mg/l	190,500	11,080 ± 3,040
VS	mg/l	172,370	7,920 ± 3,240
TOC	mg/l	80,690	5,420 ± 1,770
NH_4-N	mg/l	301	1,148 ± 111
Total N	mg/l	4,901	1,503 ± 155
Total P	mg/l	521	171
Ratio particulate COD to total COD [a]	-	73 %	59 %
COD/N/P		570/9/1	75/6/1
VS/N/P		330/9/1	46/6/1

[a] excluding toilet paper calculated as average of the ratios of the correspondent values

The COD load of kitchen refuse is in the same range as the COD load of blackwater (Table 11), similarly for the other organic parameter. In contrast, the main load of the nutrients N and P derives from blackwater, particularly from urine.

Table 11: Loads of kitchen refuse and mixture of blackwater and kitchen refuse

Parameter	Unit	KR	BW+KR
		Calculated based on statistic average quantity (73 kg/cap/year) and performed analyses of KR	Assumed that 100 % BW is collected
total COD	kg/cap/year	21.7	46.2
TS	kg/cap/year	13.9	33.8
VS	kg/cap/year	12.6	25.0
TOC	kg/cap/year	5.9	13.2
Total N	kg/cap/year	0.4	4.9
Total P	kg/cap/year	0.04	0.65

3.3 Anaerobic biodegradability

Batch tests were carried out to determine the maximum anaerobic biodegradability (AB) of the blackwater from vacuum toilets. Because of the high variations in the concentration of total COD in blackwater caused mainly by varying particulate COD, different tests were carried out for high-strength (with high particulate organic matter) and low-strength (with low particulate organic matter) blackwater to assess the impact on AB.

Therefore, the definition of high-strength blackwater of >10,000 mg/l total COD and low-strength blackwater < 7,000 mg/l total COD was chosen in this work.

By preliminary batch tests, it was found that the determination of the COD removal with the multiple flask system gave the most reliable results for AB of blackwater (see chapter 3.1.2). Two series of batch tests were carried out, one without inoculum sludge (B1) and one with inoculum sludge (B2).

The results of B1 (Figure 7) for high and low-strength blackwater show similar results for total COD removal of 72 and 75 %. The lag-phase of both blackwaters was about 10 days and they follow a similar curve for the total COD removal which seems to be independent of the blackwater strength.

The conversion of dissolved and particulate COD, however, looks different for both blackwaters. During the first 35 days, hydrolysis in the high-strength blackwater results in increasing dissolved COD concentrations whereas the particulate COD removal follows a similar curve as the total COD removal. Hydrolysis is not predominant in the low-strength blackwater. The particulate COD removal in the high-strength blackwater is significantly higher (70 %) than in the low-strength blackwater (51 %). There is no apparent reason why the particulate COD in the low-strength blackwater is apparently less biodegradable at these conditions. The results of B1 give a picture on the varying biodegradability of the particulate COD in blackwater. The dissolved COD removal is similarly high (78 and 83 %). For further assessment, the values for high and low-strength blackwater are averaged (Table 12).

Within the second batch experiment B2, blackwater was mixed with inoculum sludge to enhance the AD process. The anaerobic digestion process was much faster than in B1 (no lag-phase) (Figure 8). The maximum COD removal was achieved after 20 days while in B1 only after 85 days. The high removal of dissolved COD of 78 % already after 6 days underlines the result of B1 that the conversion of dissolved COD is faster than the conversion of particulate COD which indicates that hydrolysis is the slowest step in blackwater digestion. For this low-strength blackwater, the particulate COD removal is 66 % which lies in the range of the results of B1.

In Table 12, the results of both batch tests B1 and B2 are summarized.

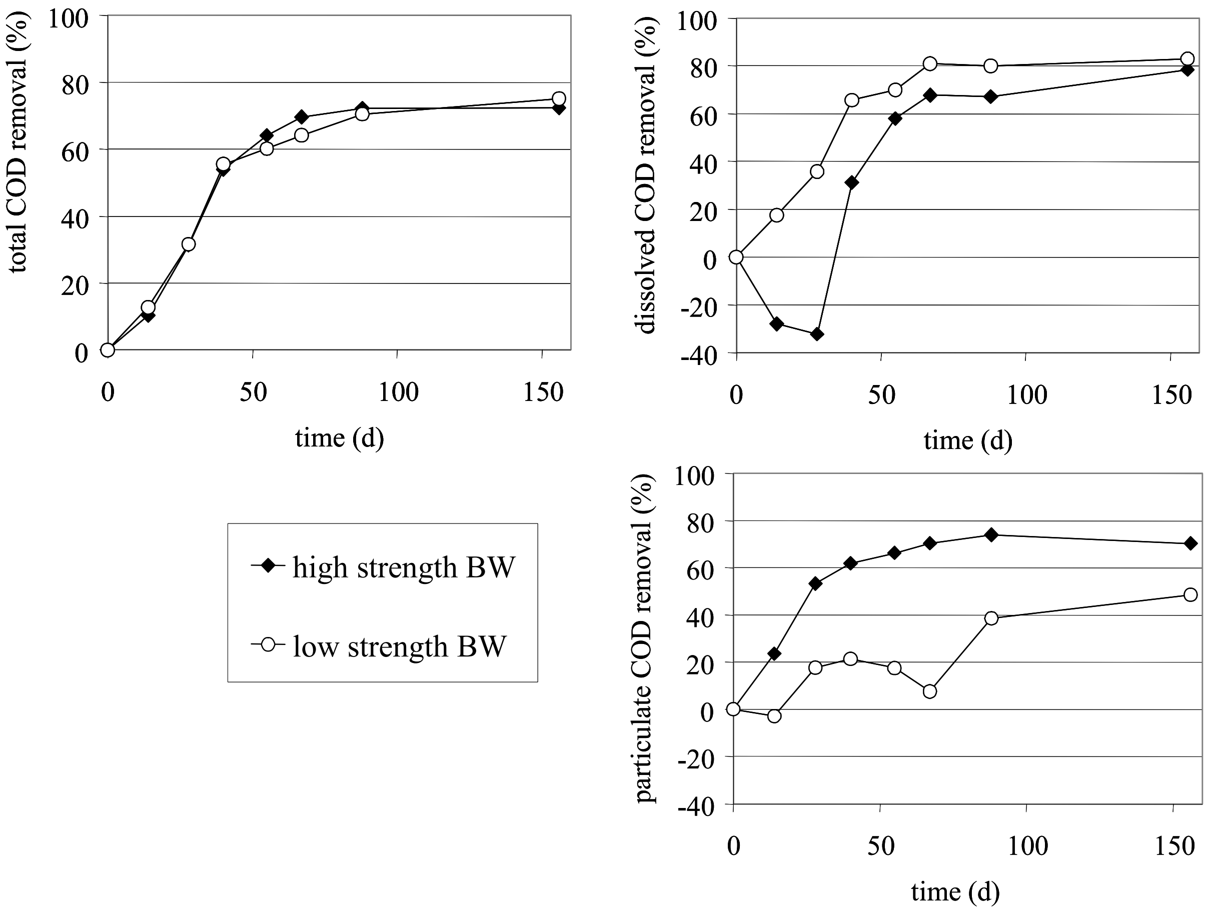

Figure 7: First batch serie (B1), total COD, dissolved and particulate COD removal with raw high-strength and low-strength blackwater

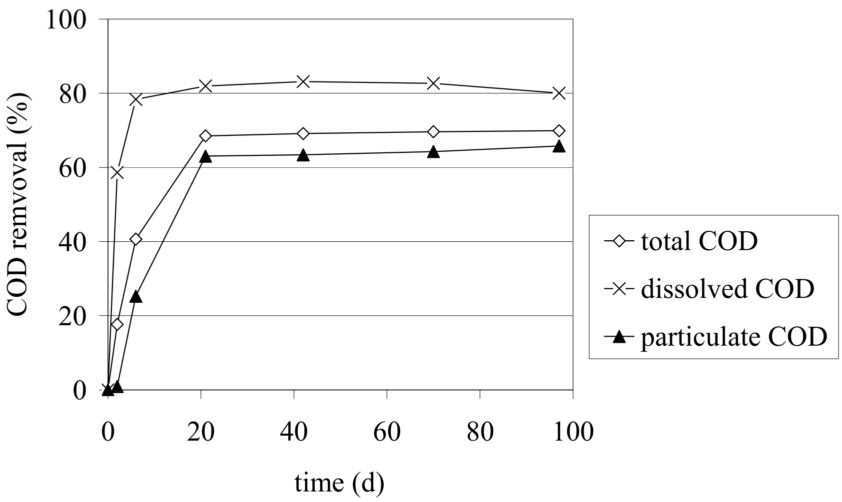

Figure 8: Second batch serie (B2), total COD, dissolved and particulate COD removal with raw blackwater

Table 12: Results of the batch tests B1 and B2 with average values

Performance	Unit	First batch test B1			Second batch test B2	Average
System		**Multiple flasks system without inoculum**			**Multiple flasks system with inoculum**	
BW characteristics		High-strength	Low-strength	Average	Low-strength	
Total COD	mg/l	13,560	4,520		5,220	
Particulate COD	mg/l	10,090	1,030		2,990	
Total COD removal	-	72 %	75 %	74 %	70 %	72 %
Dissolved COD removal	-	78 %	83 %	81 %	80 %	80 %
Particulate COD removal	-	70 %	51 %	60 %	66 %	63 %

3.4 Performance of blackwater digestion in a CSTR

These experiments were carried out with the bench scale CSTR to get basic reference data for further investigations, the frame conditions were as follows:

- Reactor temperature: 37 °C
- HRT: 20 days
- Average COD load: 0.45 kg COD/m^3/d

The reactor was started with inoculum sludge from a mesophilic sludge digester, and was fed three times per week with raw blackwater. The start-up phase took around three months. The reactor was operated at steady state for 420 days. The pH is in the reactor ranged between 7.5 and 7.7 and the VFA-COD concentration was always below 80 mg/l. These low VFA concentrations and the stable pH value prove that the digestion process is stable at HRT of 20 days. Not all parameters were analyzed during the total period. The following evaluation is based on a period of 140 days running at steady state where all parameters were analyzed and statistically assessed.

3.4.1 COD mass balance

The COD mass balance (Figure 9) shows the COD conversion during the anaerobic process. The calculated accumulated COD loads of input and output show a difference of 1.5 %. This might be caused due to errors in measurement or due to biomass accumulation in the CSTR e.g. biofilm on the stirrer.

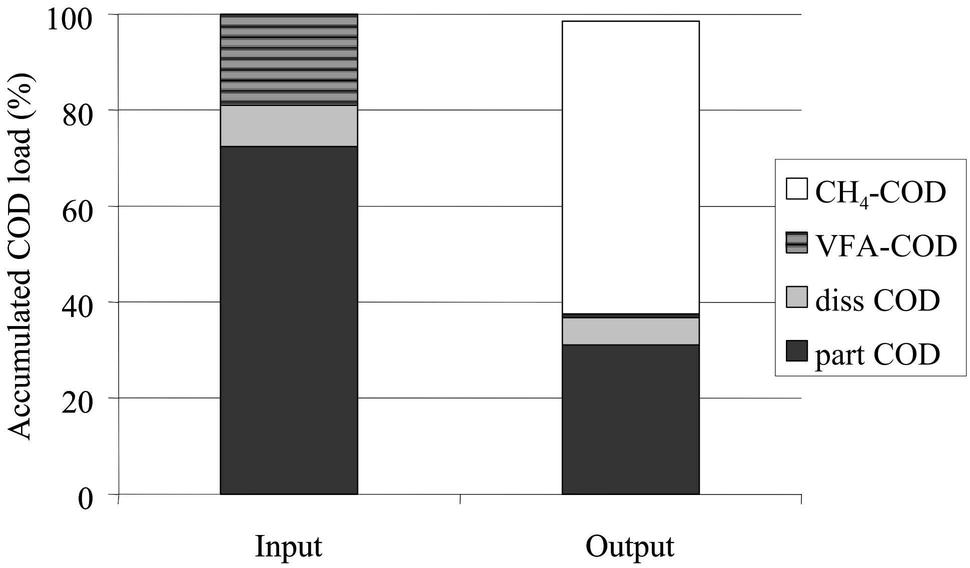

Figure 9: COD mass balance based on accumulated COD loads for AD of blackwater at 20 days HRT

3.4.2 Removal of COD fractions, TOC and VS

The removal of total and particulate COD is calculated based on the accumulated values during steady state conditions and is shown in Table 13. The removal of total, dissolved and particulate COD is 61 %, 66 % and 59 % respectively. The results show that 87 % of the anaerobic biodegradability of total COD is achieved at 20 days HRT. For the particulate COD, the efficiency is with 94 % even higher. Thus, an increasing HRT of above 20 days leads to a slightly higher overall efficiency but does not improve hydrolysis significantly.

Table 13: Removal of organic matter and total N based on accumulated loads for AD of raw blackwater at 20 days HRT

	total COD	**dissolved COD**	**particulate COD**	**VS**	**TOC**	**NH_4-N**	**total N**
Removal	62 %	66 %	59 %	56 %	57 %	-10 %	-1 %
Percentage of the anaerobic biodegradability	87 %	83 %	94 %				

The parameter VS and TOC represent the organic matter similar to COD. The VS is a value for organic solids like particulate COD and the TOC is similar to total COD. The results show that the VS removal correlated mainly to the particulate COD removal as well as the TOC removal to the total COD removal. These values are underlining the results discussed above.

3.4.3 Removal of total nitrogen and ammonium

The analysis of total nitrogen in input and output show that as expected there is no significant nitrogen removal. There is an increase in ammonium of 10 % in the output caused by the conversion of organic nitrogen to ammonium due to protein hydrolysis.

3.4.4 Biogas production

The biogas production was measured online and accumulated over time. The biogas production rates are highly varying. For comparative reasons, the specific biogas production rate related to total COD input feeding was calculated and put in one diagram with feeding time at zero (Figure 10). The data from typical low-strength BW (A) and high-strength blackwater (B) were chosen to illustrate the differences in the AD process.

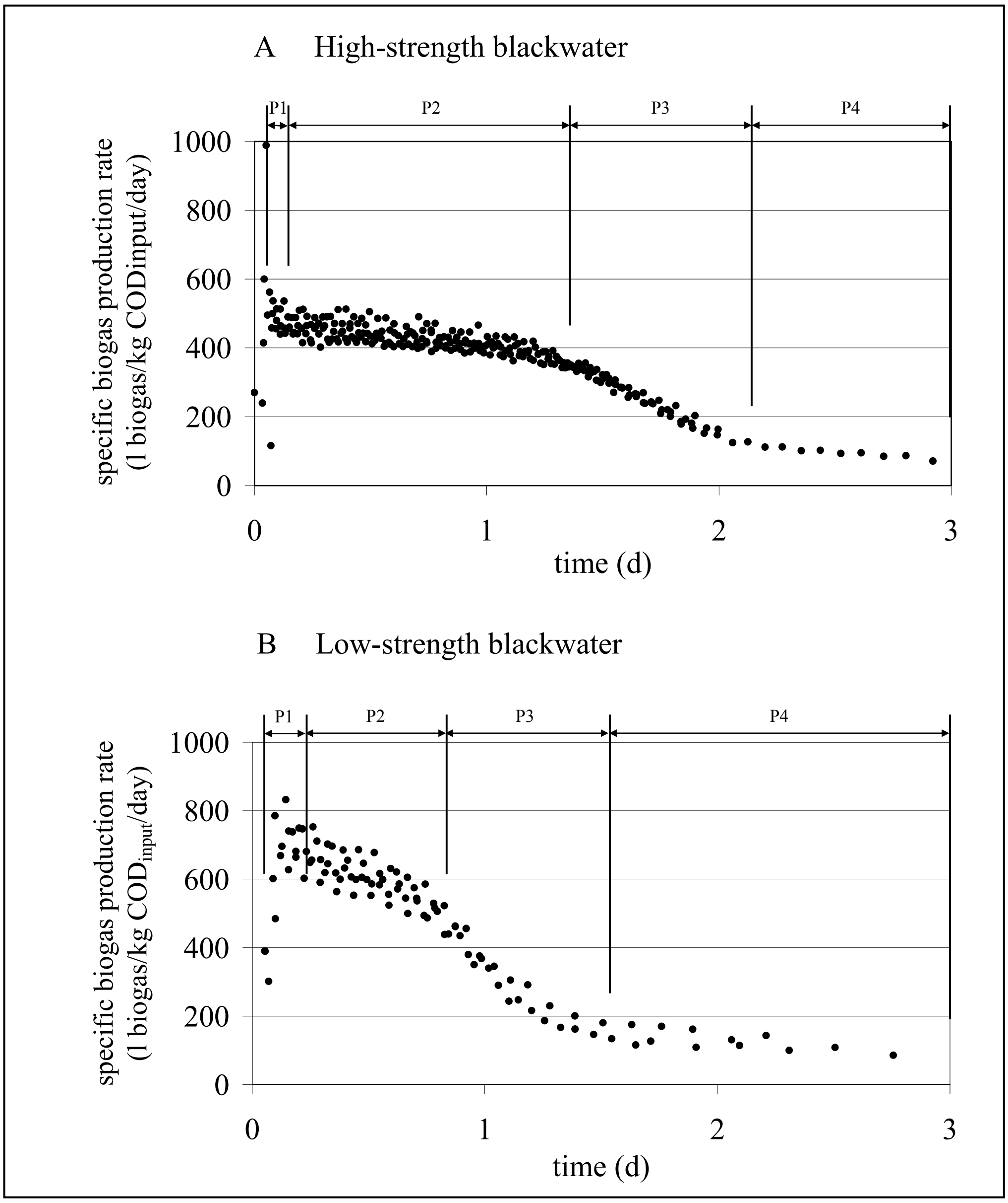

Figure 10: Specific biogas production rates after feeding with high-strength and low-strength blackwater

The biogas production rate forms a half bell shape curve which can be divided into different phases (Figure 10). Firstly, there is a very high peak where the acetic acid in the blackwater is taken up by the methanogenic bacteria present in the reactor. Therefore, it is important to know about the concentration of acetate in the input.

Then in the second phase, the production rate reaches highest values of about 700 l biogas/kg COD_{input}/day for low-strength blackwater which is around 50 % higher than the rate for high-strength blackwater. This phase for high-strength blackwater takes much longer and remains on a stable level of 450 l biogas/kg COD_{input}/day (Figure 10 A). It represents the first stage of disintegration and hydrolysis, thus is much shorter if the particulate COD concentration in the input is smaller. The biogas production rate remains on this level or decreases slowly.

Afterwards, the third phase occurs where the biogas production rate drops down quickly. Methanogenic bacteria start to use the acetate produced by the previous metabolic stages such as hydrolysis, acidogenesis and acetogenesis. By decreasing acetate, the biogas production is limited. This drop in the rate is faster for low-strength blackwater as for the previous steps because the metabolites can be taken up faster as well (Figure 10 B). The final phase is entered after 1.5 and two days for low-strength and high-strength blackwater, respectively. Here, the rate stays at a very low level of around 100 l biogas/kg COD_{input}/day. The feeding input is almost completely degraded and a new feeding could start.

The biogas composition is measured every second week after attaining steady state. The biogas sample was taken in changing intervals after feeding until the next feeding time, so that potential different biogas compositions were collected. However, the measurements of the biogas composition showed very stable values with low standard deviation (Table 14). Therefore, the biogas composition was thereafter controlled only from time to time.

Table 14: Biogas composition of blackwater AD with standard deviation (15 samples)

Parameter	**CH_4**	**CO_2**	**N_2**	**O_2**
% per weight	75 ± 2.5	22 ± 2.4	2.7 ± 2.2	0.6 ± 0.4

This composition is typical for substrate rich in protein like blackwater and fits well to literature values (ATV 2002).

The methane production is calculated to be 209 l/kg COD_{input} for blackwater AD at 20 days HRT (Table 15) which fits well to literature values for uninhibited AD. The value of 342 l/kg $COD_{removed}$ is close to the theoretical value of 350 l/kg $COD_{removed}$, the difference of 8 l/kg $COD_{removed}$ correlates to the missing part in the COD mass balance and is explained thusly.

The methane production amounts to 8.4 l CH_4/cap/day based on the COD load found out in *Flintenbreite* (see Table 15). If it is assumed that 60 % of the total blackwater is collected in the housing estate, the expected methane production for 100 % blackwater collection and treatment can be calculated accordingly to 14.0 l CH_4/cap/day. These figures are slightly different to those reported by Wendland et al. (2007) made with the same experimental set-up and substrate. The experimental duration there was much shorter there and the data had not been statistically assessed.

Table 15: Methane production based on accumulated loads for AD of blackwater at 20 days HRT

Methane production	**Unit**	**Raw BW**
based on COD	l CH_4/kg COD_{input} l CH_4/kg $COD_{removed}$	209 342
based on COD and related to capita and day	l CH_4/cap/day	8.4
Expected based on 100 % BW collection and treatment	l CH_4/cap/day	14.0

3.4.5 Output quality

For reuse purpose, the quality of the output is analyzed in terms of organic matter, macro and micro nutrients and heavy metals (Table 16). Macro nutrients are nutrients which are not available in sufficient concentrations in the soil and must be added for plant growth. Micronutrients are required for plant growth in relatively low concentrations (trace elements).

Nitrogen is not removed during AD and is thus in the same range as in raw blackwater. For phosphorus it is the same, here only the values for PO_4-P are given as it acts as fertilizer. Potassium as additional macronutrient was found in quite high concentrations. The concentration of SO_4-S decreased during AD as it is reduced to H_2S as seen in equations 3 and 4. Fertilizing experiments with the output of the CSTR showed very good results as quick acting nitrogen fertilizer in field and greenhouse tests (Simons and Clemens 2004) although the salt concentration is relatively high (565 mg/l Cl^-).

The very low concentrations of heavy metals are even below advanced standards for high quality water reuse as given e.g. in Asano and Levine (1998) and underline the excellent potential for use in agriculture and replacing chemical fertilizer. Only the copper concentration in the digested output is higher than the standard of 0.2 mg/l. Copper is however a trace element for growth but is in higher concentrations toxic. The concentration found here coincides with increasing copper concentration in many water bodies and sewage sludge in Germany.

The residual organic matter in the output has an additional potential to improve soil characteristics in terms of humidification.

Table 16: Effluent characteristics of blackwater AD in terms of organic matter, micro- and macronutrients and heavy metals as pollutants

Parameter	Unit	No samples	Effluent
Organic related compounds			
Total COD	mg/l	51	2,780 ± 420
Dissolved COD	mg/l	19	790 ± 160
Particulate COD	mg/l	19	1,990 ± 1,020
TS	mg/l	50	3,510 ± 770
VS	mg/l	50	1,620 ± 350
TOC	mg/l	53	990 ± 190
Macro nutrients			
NH_4-N	mg/l	54	1,246 ± 94
Total N	mg/l	54	1,531 ± 232
PO_4-P	mg/l	3	98
K	mg/l	3	140
SO_4-S	mg/l	3	19
Ca	mg/l	3	107
Mg	mg/l	3	38
Micro nutrients			
B	mg/l	2	0.52
Cu	mg/l	2	0.9
Fe	mg/l	2	1.38
Cl^-	mg/l	2	565
Mn	mg/l	2	0.3
Mo	mg/l	2	14.5
Zn	mg/l	2	1.46
Pollutants			
Pb	µg/l	2	47
Cd	µg/l	2	1.5
Hg	µg/l	2	0.94

3.5 Impact of increasing ammonia concentration

To increase the NH_4-concentrations in the reactors, three experimental series with CSTR over 170 days were carried out. The first run was with addition of urea and the second and third runs with addition of ammonium salts (NH_4Cl and NH_4HCO_3) to the feedstock. While the addition of urea leads to an increase of pH, the addition of ammonium salts can be done at stable pH around 7.5. The second and third runs were carried out with two different increase-ratios. Parallel to these three series, a reference reactor was operated only with blackwater. All were ran at 20 days HRT and at mesophilic condition.

Figure 11 shows the development of NH_4-N concentration in the reactors, reactor A with urea addition, B and C with ammonium salts addition but in different increase-ratios. In reactor A, the NH_4-N concentration rose in slow steps and doubled after 100 days operation. In reactor C, the same with ammonia salt addition but after 100 days the run stopped due to operational problems. In reactor B, the NH_4-N concentration rose in faster steps up to 3,500 mg/l after 150 days.

The pH in reactor A increased due to urea degradation up to 8.7 after 112 days. Reactor A was then pH-controlled by adding HCl, so that the pH fell again to 8.0. In reactors B and C the pH was kept stable between 7.4 and 7.7 due to the buffer capacity of the ammonium salts.

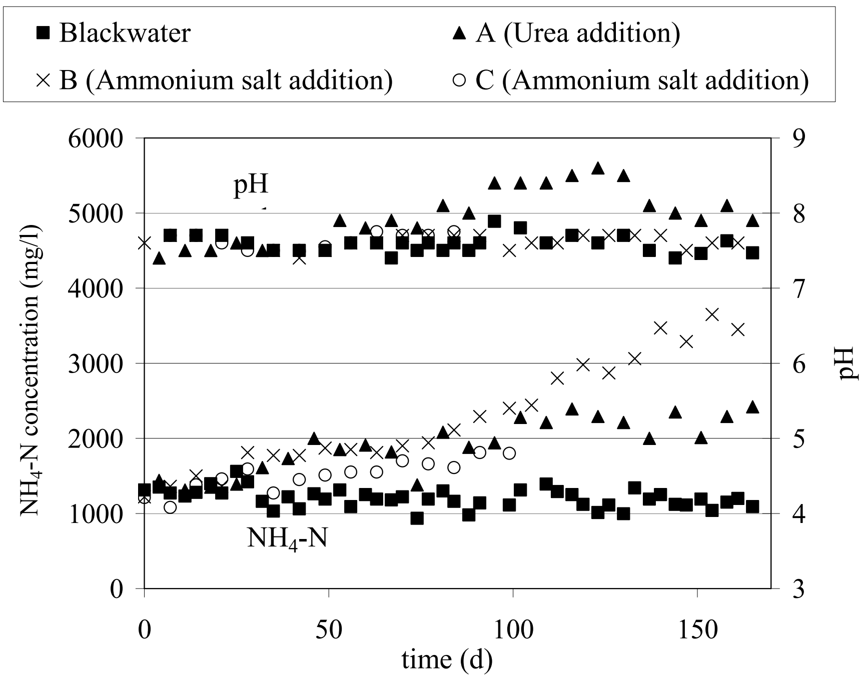

Figure 11: NH_4-N concentrations and pH in the reactors with addition of urea and ammonia salts

In the reference reactor as well as in reactors B and C with ammonia salts, the free ammonia concentration stayed below 150 mg/l NH_3-N and the weekly values for methanisation was varying around 55 to 65 % which indicates an uninhibited process. Even in reactor C where the NH_4-N concentrations are around 3,500 mg/l, there was apparently no inhibition due to a stable pH and a low free ammonia concentration. This can be explained by an adaptation of the process to ammonia, as the reactor content at the beginning of the experimental phases is already adapted to blackwater.

In reactor A however, the pH rose up to 8.6 and consequently the calculated free ammonia concentrations rose up to 780 mg/l NH_3-N which resulted in a significant decrease of methane production as seen in Figure 12. At calculated free ammonia concentrations around 300 mg/l NH_3-N, the methanisation decreases for most cases to below 20 and 35 %, reflecting this inhibition effect. The results are not as distinct as it is expected by the non-competitive inhibition kinetics but they fit to the range of kinetics approach (see Figure 24). The differences of the weekly methanisation values to the ideal curve are due to the varying blackwater characteristics and the AD processes exceeding one week.

This free ammonia threshold is higher than reported in the references (80 to 200 mg/l NH_3-N, see chapter 2.1.1) which is caused by a good adaptation of the process.

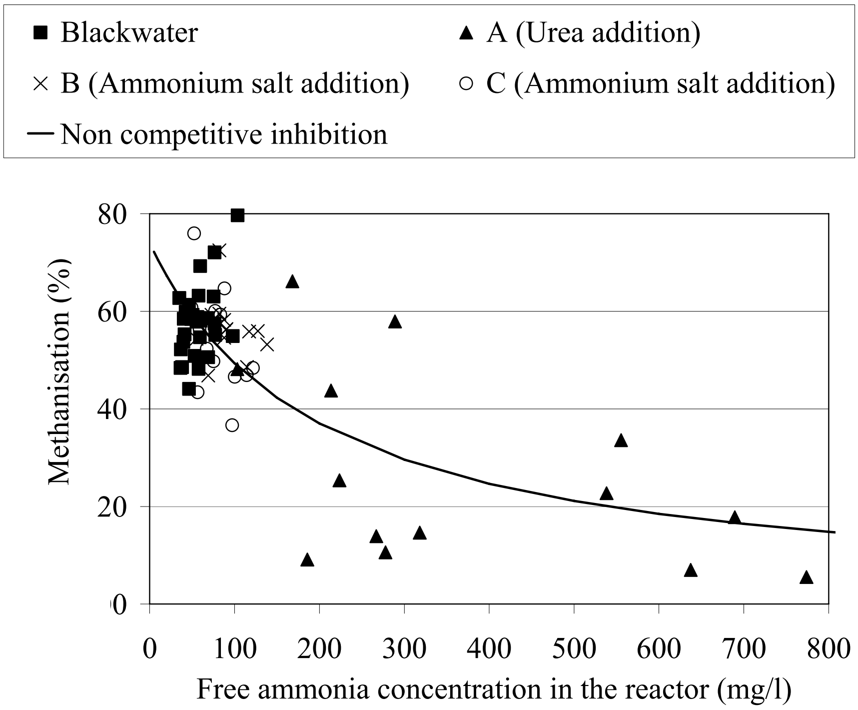

Figure 12: Methanisation (%), weekly values, in the CSTR with different NH_4-N concentrations and pH due to different feeding and the kinetics approach for non-competitive inhibition

3.6 Co-digestion with kitchen refuse and assessment of different HRT

The feeding mixture of blackwater and kitchen refuse was prepared weekly and subsequently fed to the reactors or stored at 6 °C until feeding. The reactor was fed discontinuously three times a week in case of HRT of 20 or 15 days and six times a week in case of 10 days HRT.

The addition of kitchen refuse to the blackwater was done according to statistical German values of 0.2 kg/cap/day (see chapter 3.2.2). This amounts to 40 g kitchen refuse per l blackwater. The CSTR started with adapted sludge (during previous experiments) and the feeding as shown in Table 17.

Table 17: Investigation phases of co-digestion with kitchen refuse at different HRT

Feeding	HRT	Duration
Raw BW + KR	20 days	70 days
Raw BW + KR	15 days	36 days
Raw BW + KR	10 days	54 days

3.6.1 COD mass balance

Because of the varying concentrations of organic matter in blackwater and kitchen refuse, it is important to calculate mass balances to find out the level of experimental error. The calculated COD mass balances for the different conditions are given in Figure 13. They are calculated based on the COD loads in each phase after at least one HRT. Only the mass balance for kitchen refuse was calculated based on the differences in loads for blackwater + kitchen refuse and blackwater at 20 days HRT. The total COD masses of input and output show relatively small differences with less than 3 %, which represents very small sampling or analytical errors.
The calculated COD mass balance for kitchen refuse shows that, as expected, the kitchen refuse has a higher biodegradability than blackwater: The total COD, particulate COD and dissolved COD removal of kitchen refuse is calculated at 81 %, 77 % and 92 %, respectively. Compared to particulate COD removal of blackwater, particulate kitchen refuse is also better hydrolysable and biodegradable under anaerobic conditions. These values are not representative for but give just a spot view on kitchen refuse, as only two samples were taken from the student restaurant.
In case of 15 days HRT, the particulate COD of the input was relatively low which is caused by fluctuations in the input material blackwater and kitchen refuse.

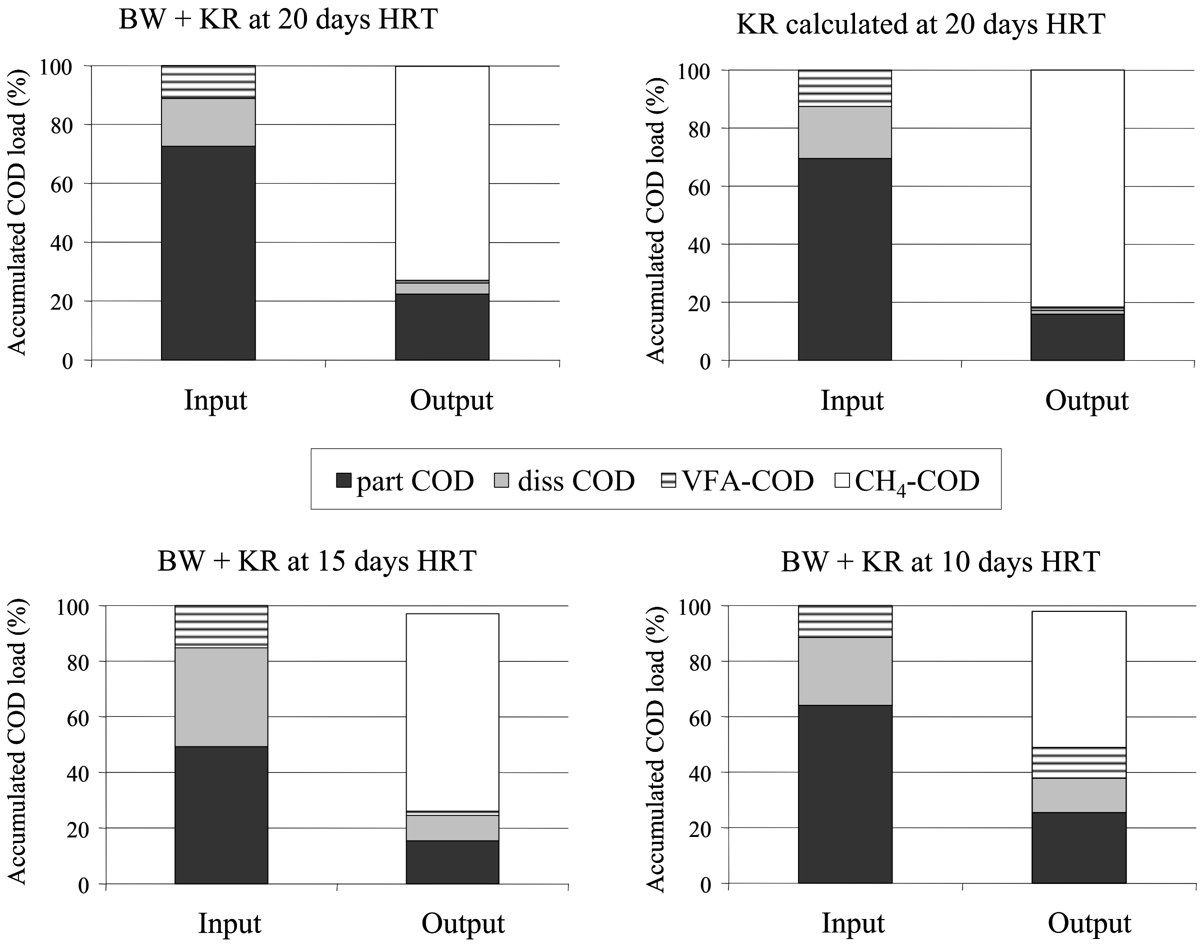

Figure 13: COD mass balances based on accumulated loads for AD of BW+KR at 10, 15 and 20 days HRT, COD mass balance for kitchen refuse was calculated

3.6.2 Removal of COD fractions, TOC and VS

The removal of organic matter in terms of COD, TOC and VS is summarized in Table 18. For blackwater digestion at 20 days HRT, the removal of total and particulate COD is 61 % and 53 % respectively. Low VFA concentrations in the output indicate that the process was stable and uninhibited. When adding kitchen refuse, the organic loading rate increased from 0.5 to 1.0 kg $COD/m^3_{reactor}$/day at 20 days HRT. The removal ratio was elevated to 71 % and 67 % for total and particulate COD because of the higher anaerobic biodegradability of kitchen refuse. At 15 days HRT, the total COD removal efficiency was even higher (75 %) whereas the removal of particulate COD was at the same level (Table 18). The increase of total COD removal performance with decreased HRT can be explained by varying particulate COD loads in both phases. The good and stable performance of the CSTR at 15 and 20 days HRT was further corroborated by the low concentrations of VFA. The VFA-COD concentrations (as criteria for the stability of the anaerobic process) were varying, but typically below 150 mg/l.

In Figure 14, the development of VFA-COD concentration and pH in the reactor are shown when decreasing the HRT from 20 to 10 days. At an HRT of 20 days, pH was stable around 7.5, which is lower than with blackwater AD. At HRT of 15 days, the pH slightly decreased to 7.3

but rose to 7.5 after a few days. When the HRT further dropped to 10 days and the organic loading rate increased to 2.0 kg COD/$m^3_{reactor}$/day, there was a rise of VFA-COD concentration up to 2,600 mg/l. During 20 days (2 HRTs), VFA accumulated in the CSTR and then remained constantly at that high level for more than 30 days (3 HRTs). Due to good buffering capacity of blackwater, the pH did not drop below 7.0 but a steady state process was achieved. The methanogenic activity and thus the COD removal were limited during the experimental phase at 10 days HRT. The COD removal was stable at 50 % which is 30 to 33 % below the removal at uninhibited conditions. Particulate COD removal was about 53 %, which is 20 % lower than the removal at 15 and 20 days HRT. Thus, also the hydrolysis is partly inhibited at 10 days HRT. However, these results show that a stable digestion with blackwater and kitchen refuse can be realized with major COD removal even at HRT of 10 days.

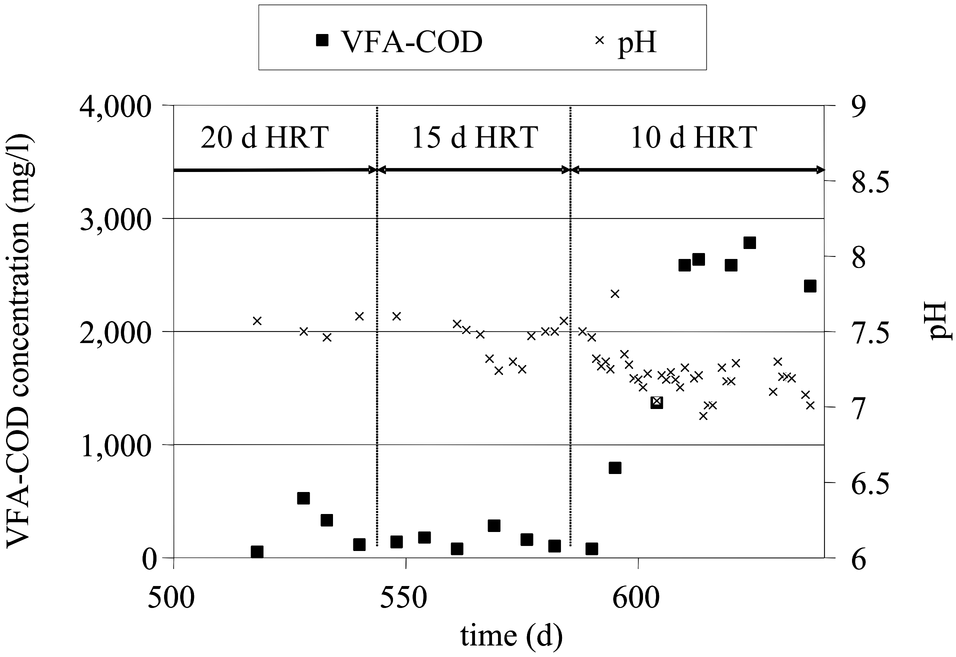

Figure 14: VFA-COD concentration and pH in the reactor with BW+KR at different HRT

Table 18: Removal of organic matter based on accumulated loads for AD of BW and BW+KR at different HRT

Parameter	**BW (20 days HRT)**	**BW+KR (20 days HRT)**	**BW+KR (15 days HRT)**	**BW+KR (10 days HRT)**
Total COD removal	62 %	71 %	75 %	50 %
Particulate COD removal	59 %	67 %	67 %	53 %
VS removal	56 %	65 %	69 %	51 %
TOC removal	57 %	72 %	71 %	52 %

The parameter VS and TOC represent the organic matter similar to COD. The VS is a value for organic SS like particulate COD and the TOC is similar to total COD. The removal efficiencies (Table 18) reflect these parallels.

3.6.3 Biogas production

The biogas produced by co-digestion with kitchen refuse had a different composition than blackwater AD. The methane concentration is lower (see Table 19) mainly because of the lower pH in the reactor. The lower the pH in the reactor, the more the gas-liquid-equilibrium of CO_2 leads to higher CO_2 concentrations in the gas phase.

Table 19: Biogas composition of AD of BW+KR with standard deviation (11 samples)

Parameter	**CH_4**	**CO_2**	**N_2**	**O_2**
% per weight	65 ± 2.0	34 ± 2.1	1.2 ± 1.2	0.4 ± 0.4

Methane production related to total input COD as well as related to capita and day for the different experimental phases were calculated (Table 20). If blackwater is treated together with kitchen refuse, the methane production is 270/280/205 l CH_4/kg COD_{input} and 27/28/21 l CH_4/cap/day at 20/15/10 days HRT respectively. If it is assumed that 60 % of the produced blackwater is collected in the housing estate as discussed before, the expected methane production would be 32 to 33 l CH_4/cap/day in case of combined blackwater and kitchen refuse digestion at 15 to 20 days HRT. These values correspond to 35.6 l CH_4/cap/day reported by Kujawa-Roeleveld et al. (2003) for an accumulation digester at longer HRT for a similar BW+KR load. The 10 % higher methane production might be due to the longer HRT in the AC.

Table 20: Methane production based on accumulated loads for AD of untreated blackwater and kitchen refuse at 20, 15 and 10 days HRT

Methane production	**Unit**	**BW (20 d HRT)**	**BW+KR (20 d HRT)**	**BW+KR (15 d HRT)**	**BW+KR (10 d HRT)**
based on COD	l CH_4/kg COD_{input}	209	255	248	172
	l CH_4/kg $COD_{removed}$	342	349	336	336
based on COD and related to capita and day	l CH_4/cap/day	8.4	25	25	17
Expected based on 100% BW collection and treatment	l CH_4/cap/day	14	32	31	22

3.7 Pre-treatment and post-storage

Pre-treatment and post-storage are treatment steps before and after digestion, which shall improve the hygienic quality of the output in order to allow its safe utilization in agriculture. Pasteurization for pre-treatment and acidification were investigated as well as post-storage in terms of microbiological indicators. Additionally, the pre-treatment was evaluated to see if it enhances the AD performance of blackwater and kitchen refuse.

3.7.1 Microbiological results

Without pre-treatment

The concentrations of pathogens in the blackwater and the mixture of blackwater and kitchen refuse are very high compared to sewage or sewage sludge because of the low dilution of the faeces. E.coli concentration is around one to two log higher than in sewage sludge.

E.coli, total coliforms and enterococci were significantly reduced by AD as the analyses after 125 days show (Figure 15). E.coli and total coliforms were similarly reduced by about 2.5 log (99.6 % reduction). Enterococci were reduced by about less than 1 log (96.9 % reduction). The lower reduction for enterococci is due to the round bacteria form which makes it more difficult to attack them in general.

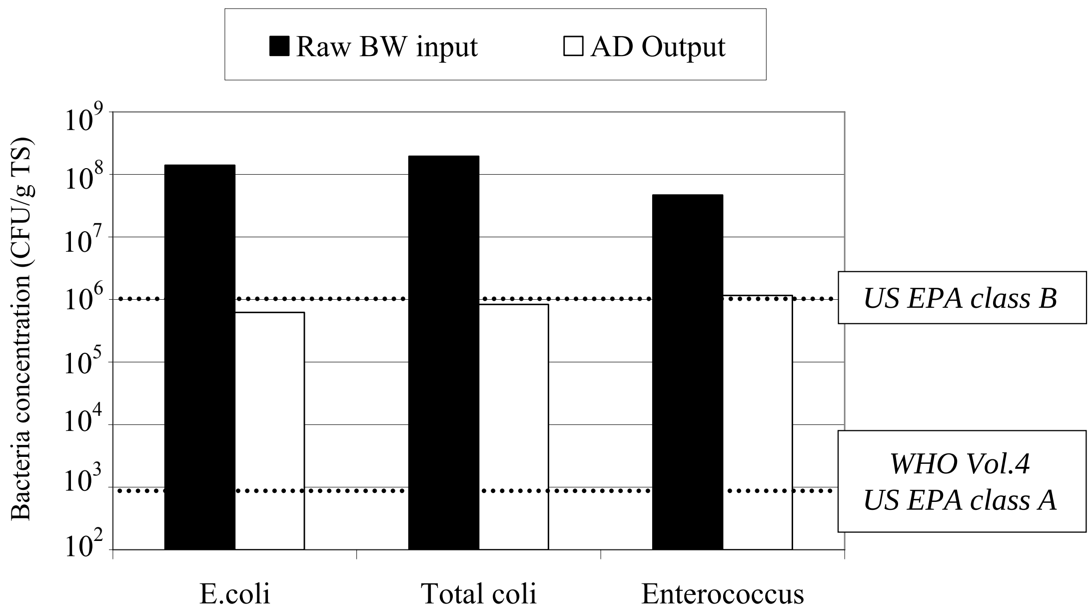

Figure 15: E.coli, total coliforms and enterococcus in input and output of blackwater AD at 20 days HRT (15 samples)

These results fit to literature values for AD of sewage sludge (Watanabe et al. 1997) and blackwater (STOWA 2005). 2.5 log removal for E.coli is higher than 1.66 as found by Horan et al. (2004) and it underlines his statement that with higher pathogen concentration in the feed the log-removal increases. However, the WHO guidelines Vol. 4, (WHO 2006) and the US EPA standards (Anonymous 1993) for use of biosolids cannot be met. Only the US EPA class B standard for restricted use in agriculture can be met on average, but there are some samples exceeding the standard of 10^6 CFU/g TS total coliforms.

The results for blackwater and the mixture of blackwater and kitchen refuse are very similar. In the data evaluation there are no significant differences between the two feeds to be found. Kitchen refuse increases the organic and the TS load but not the load of the pathogen indicators chosen here.

Post-storage of blackwater output

After blackwater AD, the output was stored up to 90 days at ambient temperature to check further log removal performance. The results in Figure 16 show that E.coli and total coliforms are further reduced during storage and keep stable after 40 days around 10^4 CFU/g TS.

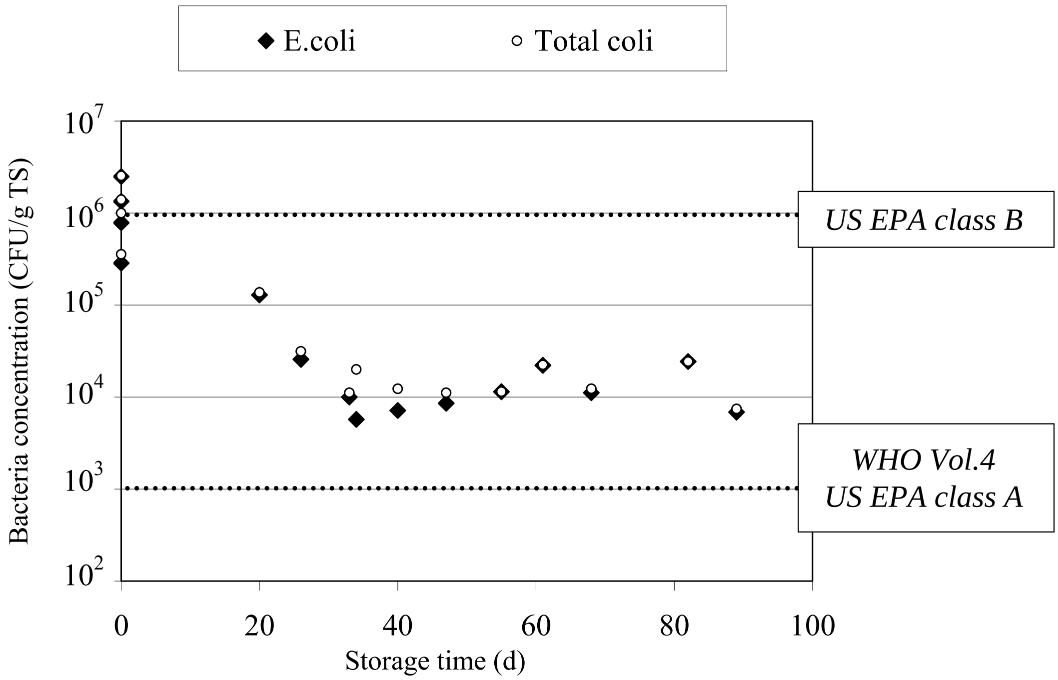

Figure 16: Removal of E.coli and total coliforms during post-storage reactor output

Thus the storage of more than 40 days provides a further reduction of around 2 log for the microbiological indicators. After the storage time of 40 days, the US EPA class B standards for restricted application on the fields can be safely met. This coincides with the results of Haible (1989) for digested sewage sludge who found the same range of reduction of pathogen indicators at ambient temperature.

Pre-pasteurization

Pasteurization was applied according to the German regulations (Anonymous 2003) at 55 °C for 24 hours and 75 °C for 1 hour.

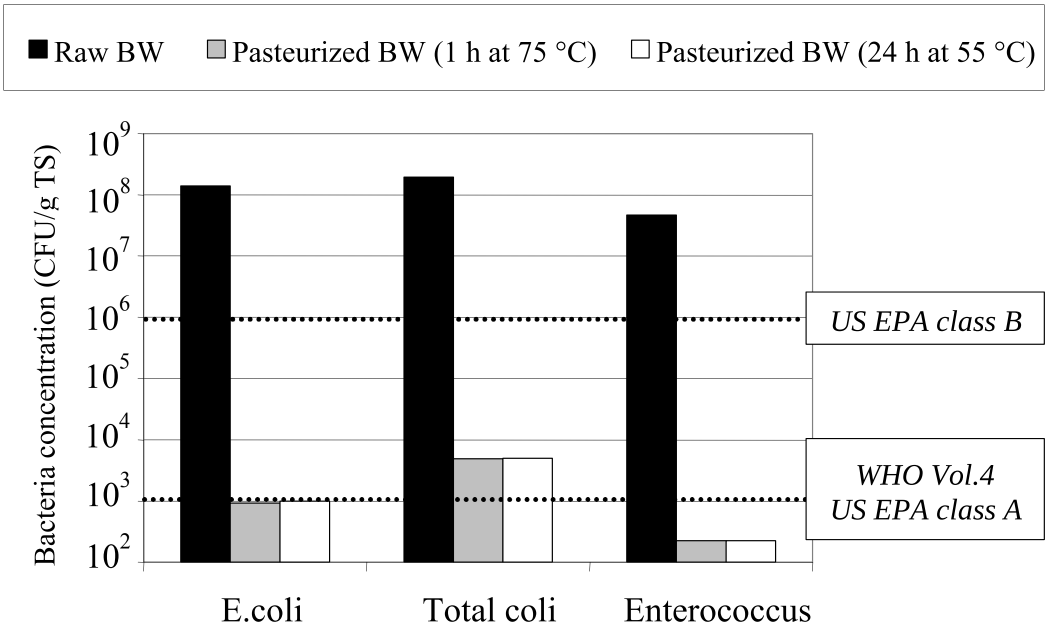

Figure 17: Removal of E.coli, total coliforms and enterococcus after pre-pasteurization (15 samples)

The results show high log removals of 5.1, 4.5 and 5.3 for E.coli, total coliforms and enterococcus, respectively (Figure 17). Blackwater cannot be disinfected totally as re-growth of bacteria starts immediately due to the high concentration of easily biodegradable matter in blackwater.

As the pathogenic indicators are very similar, the following discussion focuses mainly on E.coli as there are most reference data available.

Pre-acidification

The acidogenic digestion was investigated over a period of 6 days at 20 and 37 °C. The following substrates were taken: blackwater, blackwater and kitchen refuse and blackwater and synthetic kitchen refuse. As synthetic kitchen refuse, the definite mixture of half stewed apples and half bread crumbs provides a feed predominantly high in carbohydrates without lipids. For kitchen refuse, two loads were examined, first the expected load based on 100 % collection and second on 200 % collection.

The graphs of Figure 18 show the results for VFA-COD, pH and E.coli during 6 days of acidification. The VFA-COD concentrations quickly increase within the first day and then further to 2,400 mg/l VFA-COD for blackwater and 7,900 mg/l VFA-COD for BW+synthetic KR at 37 °C after 6 days. The other feeds are within these minimum and maximum curves. BW+synth. KR, BW+2·synth. KR and BW+2·KR at 37 °C produce the the highest VFA-COD concentrations after 6 days.

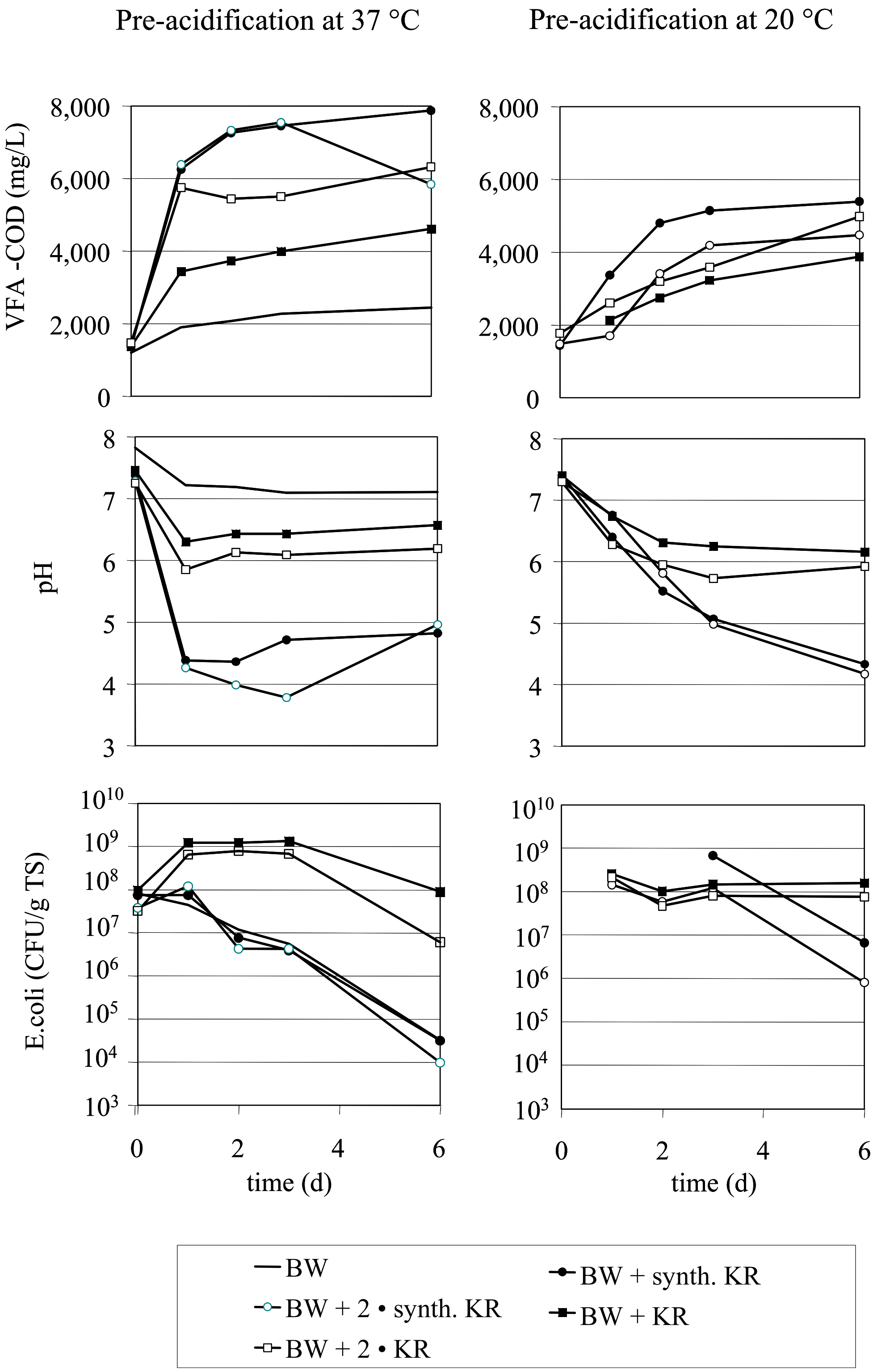

Figure 18: VFA-COD concentration, pH and E.coli removal in BW, BW+KR and BW+synth. KR after pre-acidification at 20 and 37 °C

The pH decreases the fastest during the first day for all probes. At 37 °C, the samples already reach their minimum after one day. At 20 °C, the pH continues to decrease slightly to a final value during the 6 days. For blackwater, the pH drops from 7.8 to 7.2 within the first day and then slightly to 7.1 at the end. This relatively small drop in pH corresponds to the low concentrations of VFA and is caused by the high buffer capacity of blackwater as well as the low organic load.

The pH curve has the lowest values between 4 and 5 for samples with synthetic KR. At 37 °C, the pH is the smallest, 4.4 for BW+synth. KR after 2 days and 3.8 for BW+2·synth. KR after 3 days. Then the pH rises again to values of 4.8 and 4.9, which corresponds to the decrease of VFA at that period. At 37 °C, acetoclastic bacteria might develop at an inhibited level. The mixtures with KR reached pH between 5.9 and 6.6 corresponding to lower VFA concentrations than those with synthetic KR. This can be explained by the higher content of lipids in KR. The degradation of lipids skips partly the acidogenesis, and the LCFA consuming bacteria produce acetate but have the slowest uptake rate of all.

The results for E.coli removal are reported in the last graph of Figure 18. For total coliforms, the results were similar and thus not shown here. Due to high VFA concentrations and low pH, acidification of BW+synth. KR achieved the highest log removal of E.coli. At 37 °C, the removal is 4 and 3.5 log, while at 20 °C 2.1 and 1.2 log. The inactivation started at a low rate and reaches these values only after 6 days although the pH and the VFA concentration were already achieved after 2 or 3 days. Here, the higher the organic load, the higher the log removal. These results indicate that six days at low pH and high VFA concentrations are required for an efficient pathogen die-off. The die-off might continue further when keeping the conditions for more than six days.

The acidification of BW+KR shows even an increase in E.coli numbers of 1 log and then drops on the initial figure or 1.2 log below. This insufficient removal corresponds to the graphs above with relatively high pH and low VFA concentrations for BW+KR. However, the acidification of blackwater reduces E.coli by about 3.5 log as well.

The results do not give clear functions between pH and VFA and removal of E.coli. However, there are some clear trends:

Firstly, at 37 °C the acidification is higher than at 20 °C. This does not fit to Puchajda and Oleszkiewicz (2004) who had better results at 21 °C than at 37 °C for sewage sludge. This difference might be caused due to the higher organic load in sewage sludge.

Secondly, the higher the organic load in terms of carbohydrates and proteins, the higher the log removal.

Thirdly, acidification is more efficient with addition of synthetic kitchen refuse containing predominantly carbohydrates and proteins. As the difference between the two sorts of KR is the lack of lipids in the synthetic kitchen refuse, it is assumed that the lipids contained in the "original" kitchen refuse inhibit the acidification and thus the removal of pathogens.

Based on this discussion, the recommended treatment cycles for efficient pathogen removal are the following (Figure 19):

- AD and Post-storage for 40 days at ambient temperature achieve log removal of 4.1 for E.coli. As there are no big variations in the results, this treatment is considered as very reliable for blackwater and kitchen refuse.
- Pasteurization at 24 h at 55 °C or 1h at 75 °C followed by AD reach the highest log removal of 4.8 for E.coli. After pre-treatment, the log removal is 5.1 and thus even higher but it stays in the same range during AD. This pre-treatment is relatively independent of the typically changing characteristics of blackwater and kitchen refuse.
- Acidification for 6 days at 37 °C achieves 4.5 and 3.6 log removal for BW+synth. KR and BW, respectively. The subsequent AD is not investigated here but it can be assumed that the value stays stable or increases slightly (based on the results of pasteurization and Kunte et al. (2004)). This pre-treatment underlies high variations of log removal of E.coli when the feed changes in terms of organic load and composition of carbohydrates, proteins and lipids. As it is very difficult in practice to monitor the composition of blackwater and kitchen refuse continuously, this pre-treatment must be considered as insufficiently reliable. If it was possible to add continuously a co-substrate containing carbohydrates and proteins similarly to synthetic kitchen refuse, it would be possible to adjust the frame conditions for a high inactivation of pathogens as found in these experiments. But this pre-treatment requires higher effort in operation controlling.

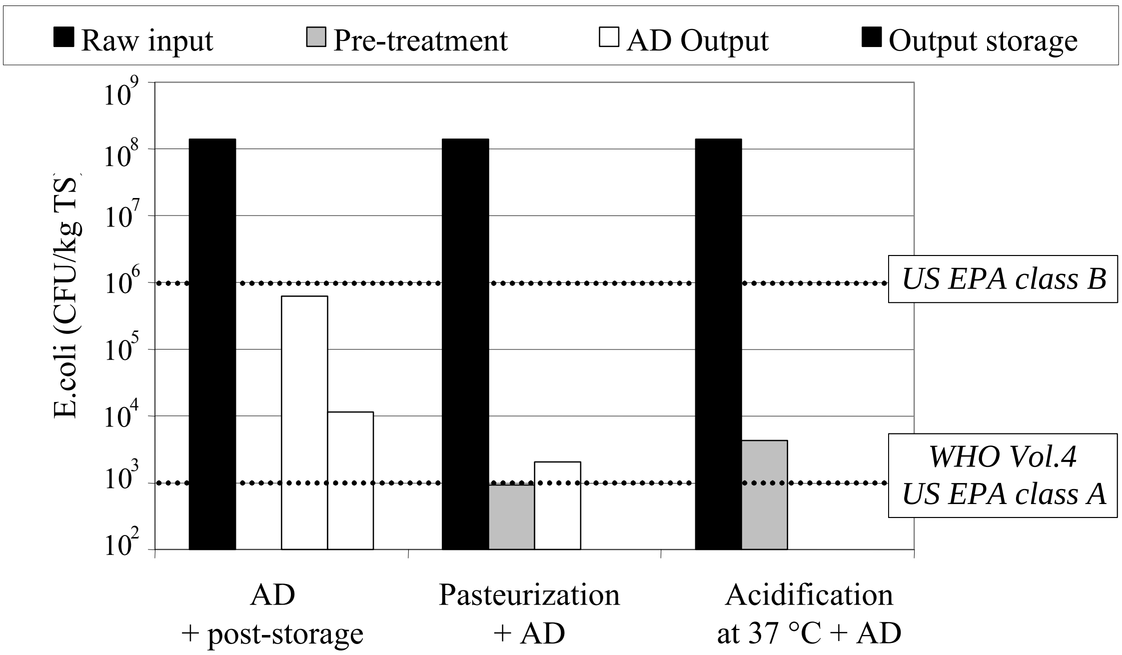

Figure 19: Comparison of three selected treatment cycles in terms of removal of E.coli

The comparison in Figure 19 shows that all treatment cycles reach log removals for E.coli from 4.1 to 4.8. Neither pasteurization followed by AD nor other studied treatment can safely meet the standards by WHO Vol. 4 (use of excreta and greywater in agriculture) and US EPA for the use of sewage sludge, class A. But all achieve the requirement of US EPA class B for restricted application on farm land. The value is for faecal coliforms but these results show high similarity between E.coli and faecal coliforms.

If, on the other hand, the WHO Vol. 2 (2006) for wastewater use in agriculture was considered, the treatment options are sufficient to meet restricted irrigation requirements. By complementing with other health measures, the required health based target of 6-7 log for unrestricted irrigation can even be met (Table 4). These measures can be e.g. localized (drip) irrigation on high (4 log reduction) or low (2 log reduction) growing crops and washing the field products (1 log reduction) with water (Mara and Kramer 2008). In summation, the treatment and the additional protection measures can reach 6-7 log removal which is typically required as health based target

Of particular interest is also the need to remove helminth eggs required by WHO Vol. 2 which is not investigated in this study.

3.7.2 Impact of pre-treatment on AD

When implementing a pre-treatment in order to enhance the removal of pathogens, it is important to know which impact this step exerts on AD. Therefore, the differences in terms of total and dissolved COD, NH_4-N and total N are evaluated.

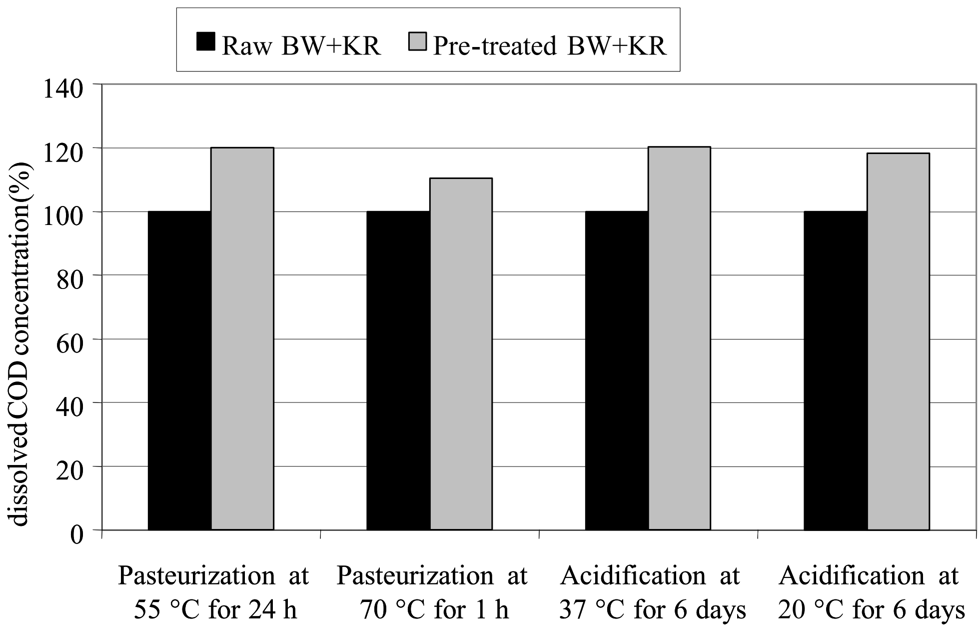

Figure 20: Differences in dissolved COD concentrations in raw and pre-treated mixture of blackwater and kitchen refuse

For total COD, NH_4-N and total N, the differences between raw and pre-treated material are negligible. But for dissolved COD there are significant differences (Figure 20). There is a trend of higher dissolved COD concentrations in pre-treated mixture of blackwater and kitchen refuse, 10 % and 20 % by pasteurization at 37 °C for 1 h and at 55 °C for 24 h, respectively, and around 20 % in case of acidification at 20 and 37 °C.

The comparison between AD of raw and pasteurized mixture of blackwater and kitchen refuse running over three months shows the same COD removal and the biogas production curve is similar as well.

3.8 Chapter conclusions

- Blackwater from vacuum toilets is concentrated wastewater with high concentrations of particulate COD, the nutrients N and P and pathogens. There are high standard deviations for COD especially for particulate COD.
- In *Flintenbreite*, only 60 % of the total blackwater load is collected. In the case of 100 % blackwater collection, the load is 24.5 and 17.9 kg/cap/year, with and without toilet paper, respectively. The addition of separately collected kitchen refuse leads to doubling of the organic load and improves the COD/N/P ratio for AD.
- The determination of COD removal with the multiple flask system is a reliable method to determine anaerobic biodegradability of blackwater. The batch test without inoculum sludge took more than 4 times longer (85 days) to achieve the maximum biodegradability than the batch test with inoculum (20 days). Both are suitable to determine the anaerobic biodegradability.
- Based on two batch test series with and without inoculum, the anaerobic biodegradability (AB) was determined as total COD removal of 72 %. Removal of particulate COD is on average lower but underlies a high variety (51 to 70 %) independent of the blackwater concentration.
- Mesophilic AD in CSTR is an appropriate technique to treat BW. At HRT of 20 days, more than 60 % of the input COD was converted to biogas which represents 87 % of the maximum biodegradable COD. At these conditions, the produced biogas contained 75 % methane and the biogas production rate was 8 and 14 l CH_4/cap/d for 60 and 100 % blackwater collection. The process at 20 days HRT was stable and uninhibited. For low-strength blackwater, the biogas production rate is higher than for high-strength blackwater, which indicates disintegration and hydrolysis as being the limiting step.
- At HRT of 15 and 20 days, a stable and uninhibited process was achieved when blackwater and kitchen refuse mixture was fed. Even at HRT of 10 days, a steady state process with a decreased COD removal of 30 to 33 % and decreased methane production by 19 to 21 % could be achieved, however with increased VFA concentrations in the output.
- The NH_4-N concentration in blackwater is relatively high, in average 1,111 mg/l, due to the degradation of urea from urine. The content of ammonia can be much higher, firstly if faeces and urine are not consistently composed. This could be the case in places with only part-time

use frequencies of the toilets like in public buildings. Secondly, if an advanced vacuum toilet needed only 0.25 l/flush, the NH_4-N concentration would be on average 2,920 mg/l.

- The results of the experiments with stepwise ammonia addition clearly show that AD of blackwater with NH_4-N concentration up to 3,500 mg/l can be operated uninhibited, if the pH is kept stable around 7.5. If the pH increases, the rising concentration of free ammonia starting from 300 mg/l NH_3-N inhibits the process. In that case, the digester must be pH-controlled by adding acid again or ammonia concentration of the feedstock must be reduced so that the bacteria can recover. The inhibition kinetics are further investigated in chapter 4.
- The reduction of microbiological indicators is significant during AD (2.4 log reduction for E.coli). Another 2 log reduction is achieved by post-storage at ambient temperature for more than 40 days of the output. However, the standard of 1,000 CFU/g TS E.coli (faecal coliform), could be safely met neither by post-storage nor pasteurization nor pre-acidification.
- Pre-acidification for 6 days at 37 °C can be an alternative pre-treatment step to pasteurization for pathogen removal. In the case that co-substrate containing carbohydrates and proteins are available, the pre-acidification is an efficient option if the operation is well controlled in terms of pH and VFA.
- The digested output can be used in agriculture as an organic liquid fertilizer in terms of macro- and micronutrients (especially nitrogen). Pollutants such as heavy metals are present in very low concentrations. Because of remaining pathogens and relatively high salt concentrations, restricted application is required.

CHAPTER 4 MATHEMATICAL MODELING AND SIMULATION

Mathematical modeling and simulation serve to analyze processes in a complex system such as AD and to simulate specific operational situations. Over the last three decades, many different anaerobic models have been developed, however their use was often limited due to their specific nature (Gavala et al. 2003). Based on the wide variety of anaerobic models available, the International Water Association (IWA) Task Group for Mathematical Modeling developed one model, namely the Anaerobic Digestion Model No 1 (ADM1) (Batstone et al. 2002a). Trying to serve as a generic platform, ADM involves a total of 19 biochemical processes with seven species utilizing eight intermediates, as well as three sorts of physiochemical processes. As benefits, the Task Group expects the following (Batstone et al. 2002b):

- Increased model application for full-scale plant design, operation and optimization
- Further development work on process optimization and control, aimed at direct implementation in full-scale plants
- Common basis for further model development and validation studies to make outcomes more comparable and compatible
- Assisting technology transfer from research to industry

Since then, the model has been extensively used and analyzed in academic and practical applications (Batstone et al. 2006). Critical analysis of the model has mainly focused on model structure reduction, hydrogen inhibition functions, and the default parameter set recommended by the Task Group. This default parameter set has largely been verified as a reasonable compromise, especially for wastewater sludge digestion (Batstone et al. 2006).

As one of the most sophisticated and most widely applied model, ADM1 is chosen to be the fundamental of this developed model called BWADM (Blackwater Anaerobic Digestion Model). Feng (2004) firstly applied ADM1 for blackwater digestion. His model is further developed in this work which is explained in the following chapters.

4.1 The mathematical model

The conversion processes in AD can be divided into biochemical and physico-chemical processes. The biochemical processes are at the core of the AD model. To describe the effect of the physico-chemical state such as pH on the biochemical processes, the physico-chemical conversions must be included in the model as well.

Figure 21 shows the COD mass flow through the biochemical processes for a composite particulate material as implemented in ADM1. It becomes clear that it is of most importance for the accuracy of the model to well define the input characteristics in terms of COD. Defining the biodegradable input COD is necessary, as a considerable fraction of the input COD may be anaerobically not degradable (Gosset and Belser 1982). Secondly, the determination of dissolved and particulate COD is recommended as it has a significant impact on the COD mass flow. It is thirdly recommended to determine the composition of carbohydrates, proteins and lipids in the composite particulate material.

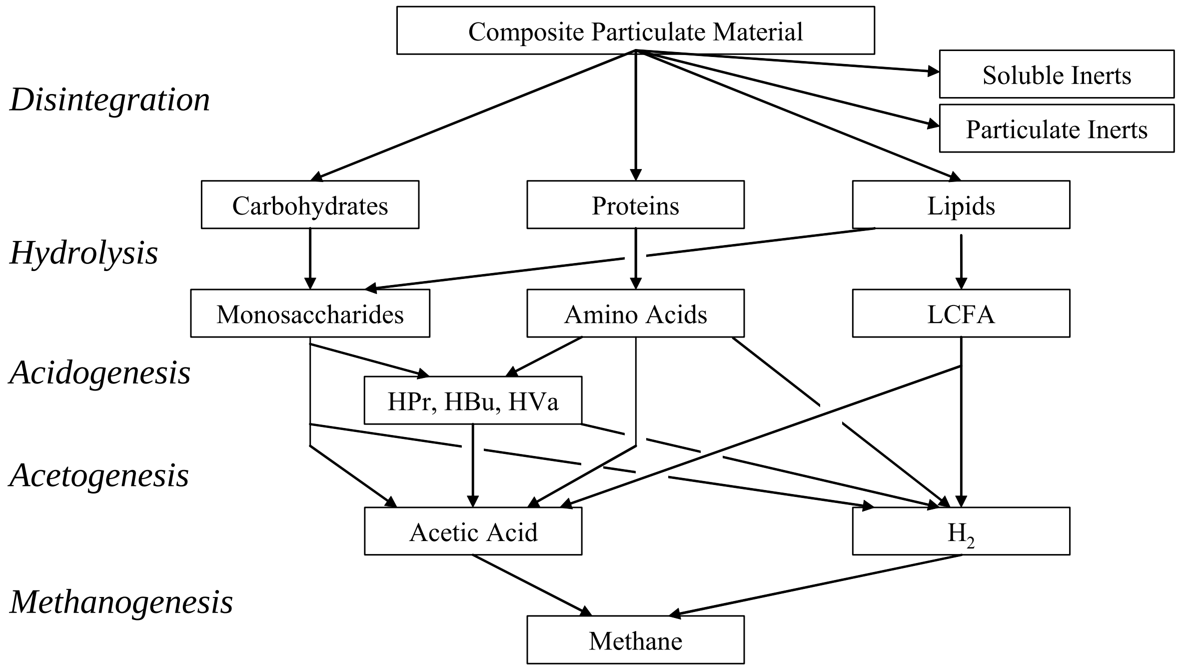

Figure 21: COD mass flow for a particulate composite as used for ADM1. Propionic acid (HPr), Butyric acid (HBu) and Valeric acid (HVa) are grouped in the figure for simplicity (Batstone et al. 2002b)

The structure of ADM1 is formulated by a Peterson Matrix. This matrix was developed by Peterson (1965) to construct chemical and biological models in a flexible way. In the matrix, each row represents one process and each column represents one component (see Batstone et al. 2002a).

4.1.1 Implementation of biochemical processes

The biochemical processes are implemented based on ADM1 and the modified model by Feng (2004). Modifications to those models are explained in the following parts.

Disintegration and hydrolysis

As shown in Figure 21, the first two steps of AD are disintegration and hydrolysis. These processes occur jointly together with the support of extra cellular-enzyme outside organisms in the liquid phase and are summarized in a single step in the literature. From the mathematical point of view, the processes with two steps make the model easier to be adjusted in order to be seasoned with different cases. Consequently, the composition of carbohydrates, proteins, and lipids must be determined by analysis to describe the first two steps accurately. In practice, this analysis is not common for wastewater, sludge and solid waste. In most references, these two steps are therefore not divided, and instead the processes are together investigated in terms of hydrolysis.

Disintegration and hydrolysis are very important for a good model as they are often the rate limiting step over the whole AD process if the input has a high particulate content (Eastman and Ferguson 1981). To implement disintegration, all literature models use first order kinetics Batstone et al. (2002b).

To implement hydrolysis, several mathematical models can be used. Vavilin et al. (1996) compared the four types of hydrolysis kinetics, i.e. the first-order, two-phase model (Vavilin et al. 1996), Monod type (Hobson 1983) and Contois model (Chen and Hashimoto 1980) with four different composites (swine waste, sewage sludge, cattle manure, cellulose), and the four kinetics gave similar simulation results. Sanders et al. (2000) reported on surface related growth of biomass and thus the importance of surface based kinetics.

As however the ADM1 task group recommends applying the first-order kinetics by default, which is most widely applied, for disintegration and hydrolysis the first-order kinetics are implemented and adopted in BWADM.

The common expression of first-order kinetics is shown as follows:

$$\rho_i = k_i X_i \qquad (9)$$

Where: ρ_i = uptake rate of substrate i (g COD/m^3/d)

k_i = parameter of first order kinetics of particulate component i (1/d)

X_i = particulate component I (g COD/m^3)

The parameter k_{dis} is used for disintegration, where the parameters k_{hyd_ch}, k_{hyd_pr} and k_{hyd_li} are used for the hydrolysis of carbohydrates (ch), proteins (pr) and lipids (li), respectively.

Substrate uptake

Following hydrolysis, the three steps of acidogenesis, acetogenesis and methanogenesis are orderly at its heel. They are used to describe the utilisation of substrates by microorganisms. Seven species are involved in three steps, namely sugar degraders, amino acids degraders, LCFA degraders, valerate and butyrate degraders, propionate degraders, acetate degraders and hydrogen degraders. Only valerate and butyrate are utilised by the same degraders in ADM1.

ADM1 uses Michaelis-Menten Kinetics for acidogenesis, acetogenesis, and methanogenesis to describe the uptake rate of substrate (Michaelis and Menten 1913) as Michaelis-Menten approach is based on enzyme related processes which is most realistic. Also the flexibility to include easily the different kinetics forms (e.g. inhibition) is one of the reasons that ADM1 uses Michaelis-Menten kinetics (substrate uptake rate) instead of Monod function (biomass growth rate) like in the aerobic model ASM (Batstone et al. 2002a).

Another approach is the Haldane kinetic (Haldane 1930), which considers that too high a concentration of substrate will inhibit the uptake of the substrate itself. Equations 10 and 11 give the formulas of Michaelis-Menten kinetics and Haldane kinetics, respectively.

$$\text{Michaelis-Menten: } \rho_j = k_{m,j} \cdot \frac{S_i}{K_{S,j} + S_i} \cdot X_i \cdot I_{i\_j} \qquad (10)$$

$$\text{Haldane: } \rho_j = k_{m,j} \cdot \frac{S_i}{K_{S,j} + S_i + \frac{S_i^{\,2}}{K_{H,j}}} \cdot X_i \cdot I_{i\_j} \qquad (11)$$

Where: ρ_j = reaction rate of process j (g COD/m^3/d)

$k_{m,j}$ = maximum specific uptake rate of process j (1/d)

$K_{S,j}$ = half saturation concentration of process j (g COD/m^3)

$K_{H,j}$ = Haldane saturation concentration of process j (g COD/m^3)

S_i = utilized dissolved component (i.e. substrate) i (g COD/m^3)

X_i = particulate component (i.e. biomass) i (g COD/m^3)

I_{i_j} = inhibition function from inhibitor i to process j (g COD/m^3)

In order to compare different kinds of kinetics easily, the typical curves of the zero order reaction, the first order reaction, the Michaelis-Menten kinetics and Haldane kinetics are shown in Figure 22.

For acidogenesis and acetogenesis, Michaelis Menten kinetics were applied successfully on blackwater AD by Feng et al. (2006a) with the default values of ADM1. For acetate, butyrate and propionate uptake, he made a parameter estimation of the kinetic coefficients by batch tests and could find similar k_m and K_s values. Feng et al. (2006) reported that the model is not sensitive to valerate and butyrate variations in case of blackwater AD so that the uptake of valerate and butyrate are optional in the model.

In BWADM, all substrate uptake steps are implemented as Michaelis-Menten kinetics.

The hydrogen-utilizing methanogenesis is the fastest step within the AD process and follows the Michaelis-Menten kinetics (Kus 1993). However, this step is of most importance as the thermodynamics of sytrophic acetogenesis and hydrogen-utilizing methanogenesis reactions are only possible in a narrow range of hydrogen or formate concentrations. This is important for modeling because the thermodynamic limitations largely determine the parameter for hydrogen inhibition, as well as half saturation coefficients and yields (Batstone et al. 2002a). The BWADM follows here the recommendation by ADM1.

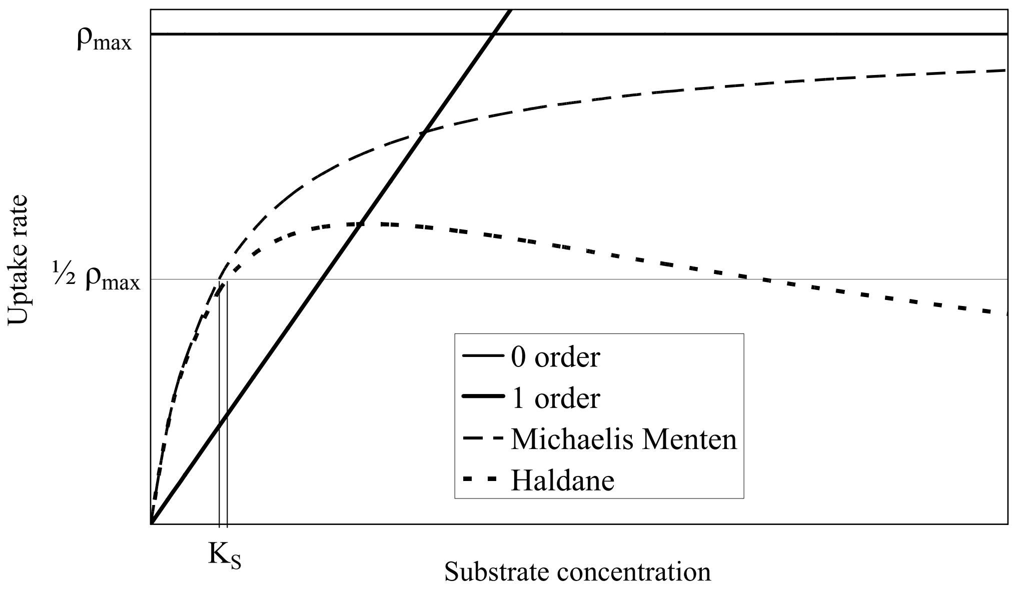

Figure 22: Different kinetics of biochemical reactions

As major methanogenic step, the aceticlastic methanogenesis is often the slowest step in AD of predominantly dissolved substrates (Wiesmann 1988). If the acetate concentration increases due to slow methanogenic activity, the pH decreases which might be a cause of failure of AD reactors. During the last two decades, Haldane kinetics were successfully applied on this step (Kus 1993) that requires particular attention in the model.

Some authors argue that the substrate for the aceticlastic methanogenesis is the un-inionized acetatic acid which must be considered in the Haldane kinetics (Andrews 1969, Duarte and Anderson 1982, Moletta et al. 1986, Bolle et al. 1986, Spieß 1991, Kus 1993) found that the uptake to acetatic acid as well as propionic acid can be accurately described by Haldane kinetics when taking the un-ionized part as substrate. These results also best reflect the microbiological reality as the un-ionized acid can pass through the cell membrane much easier. The un-ionized part of the acetatic acid can be calculated based on pH and pK_a values.

The resulting kinetic coefficients for Haldane kinetics found in the literature (Table 21) are varying between $4.67 \cdot 10^{-4}$ and 16 mg/l COD for K_S and 0.537 and 169 for K_H which makes it difficult to select the default value for the model.

In the BWADM, the uptake of acetatic acid was implemented as Haldane kinetic with un-ionized acetate as substrate.

The related yield coefficients Y_i for all species are taken from ADM1.

The decay of biomass is the indispensable step of all biochemical processes which is described as the first-order reaction. The recommended decay rates by ADM1 were chosen.

Table 21: Kinetic coefficients for acetate uptake based on Haldane kinetics with un-ionized acetate as substrate (experiments under mesophilic conditions)

Reference	**Reactor**	**pH**	**μ_{max} (1/d)**	**K_S (mg/l COD)**	**K_H (mg/l COD)**
Graef and Andrews 1973	Chemostat	6.95	0.4	2.13	42.7
Carr and O´Donnell 1977	Chemostat	-	0.11	1.15	35.2
Bolle et al. 1986	Batch reactor	7.0	0.038	2.1	169
Moletta et al. 1986	Batch reactor	-	0.144	16	46.7
Attal 1988	Batch reactor	varying	-	-	16
Fukuzaki et al. 1990	Batch reactor	varying	-	0.012	9.7
Fukuzaki et al. 1990	Batch reactor	varying	-	$4.67 \cdot 10^{-4}$	0.537

Inhibition

Inhibition is a very important part in AD processes. In spite of its complexity, inhibition is implemented in ADM1 in an easy manner. I are the inhibition functions multiplied with the uninhibited uptake kinetic (Batstone et al. 2002a). This is not always possible, because the inhibition function is integral in the uptake equation.

In ADM1, three different kinds are considered:

- pH inhibition,
- non-competitive inhibition from free ammonia and hydrogen and
- competitive inhibition between valerate and butyrate.

pH inhibition is implemented in ADM1 as an empirical term assuming that until specifically high and low pH values, no or very little inhibition occurs (Angelidaki et al. 1993, Ramsay 1997).

As extreme pH values inhibit the enzymatic activity, it affects the organisms in all steps of the AD process. An enzyme based approach for pH inhibition called the “bell-shaped” curve (Fersht 1999) could better reflect reality, although this approach assumes that only one charged form of the enzyme is active and the enzyme is a single ionized species, whereas it could contain a mixture of different ionized groups. However, the “bell-shaped” relation is proposed in this work and applied in the BWADM. The pH inhibition with an optimum pH of 7.0 is applied on all intracellular processes. This relatively small narrow range curve (Figure 23) considers the acetoclastic methanogenic bacteria as they are more sensitive than the other groups (Mudrack and Kunst 1991).

The extracellular processes disintegration and hydrolysis as well as acidogenesis and acetogenesis have their pH optima around 6.0 (Madigan et al. 1997) but have a greater tolerance towards pH variations (Figure 23).

$$\text{pH-inhibition: } I_{pH\_j} = \frac{1}{1 + 10^{pK_{I1} - pH} + 10^{pH - pK_{I2}}} \qquad (12)$$

Where: I_{i_j} = inhibition from inhibitor i to process j

pK_I = 50 % inhibitory value for pH

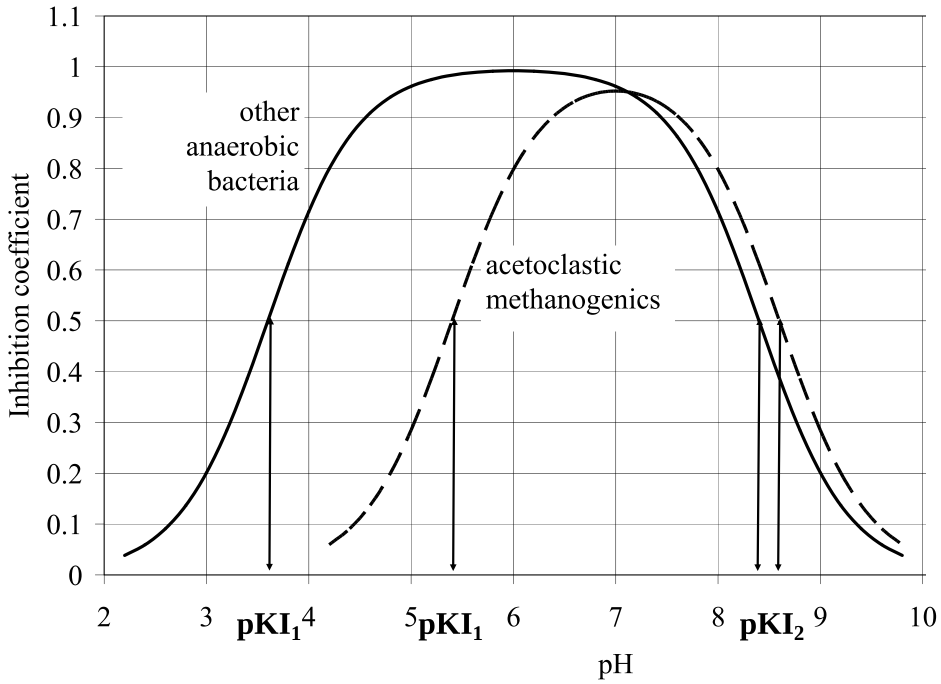

Figure 23: Impact of pH enzymatic pH inhibition on AD processes

Equation 13 corresponds to non-competitive inhibition (Pavlostathis and Giraldo-Gomez 1991. It is used to implement the inhibition of acetogenesis and methanogenesis by hydrogen and free ammonia, respectively.

The free ammonia is taken in to account, as it is accepted that the free ammonia in the liquid phase causes the inhibition. As example, Figure 24 shows the non-competitive inhibition by free ammonia.

$$\text{non-competitive inhibition: } I_{i\_j} = \frac{K_{I,i\_j}}{K_{I,i\_j} + S_{I,i}} \qquad (13)$$

Where: I_{i_j} = inhibition from inhibitor i to process j

$K_{I.i\_j}$ = 50 % inhibitory concentration of inhibitor i to process j, (g N/m^3 for ammonia; g COD/m^3 for hydrogen)

$S_{I,i}$ = concentration of inhibitor I, (g N/m^3 for ammonia; g COD/m^3 for hydrogen)

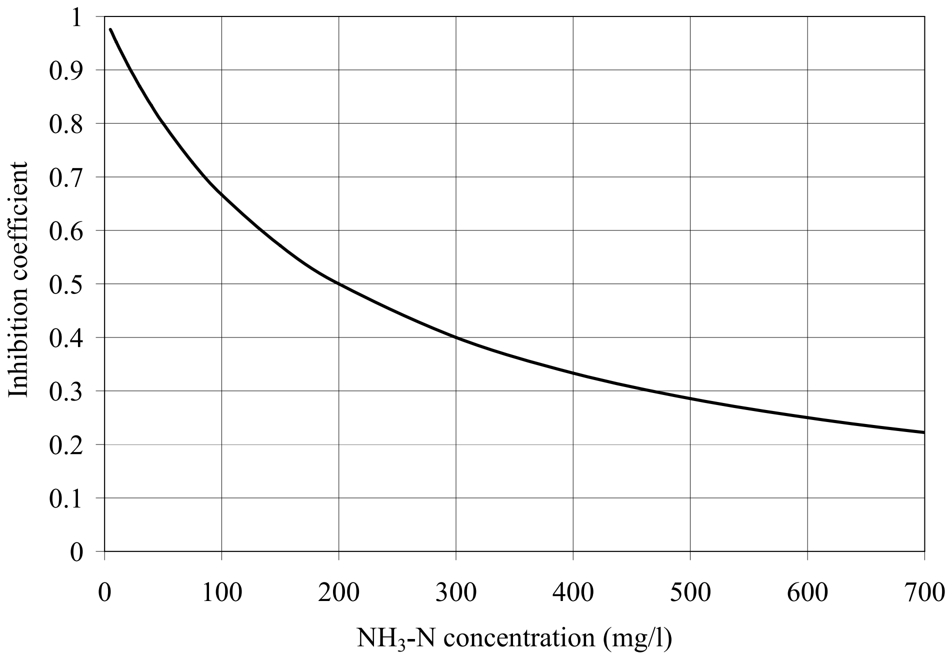

Figure 24: Impact of non-competitive inhibition by free ammonia

For hydrogen the inhibition approach is similar. The liquid phase hydrogen concentration is used for hydrogen inhibition.

As valerate and butyrate are metabolized by the same kind of microorganisms, the competition inhibition is introduced by ADM1:

$$\text{competitive inhibition: } I_{i\_j} = \frac{S_j}{S_j + S_i} \tag{14}$$

Where: $I_{i,j}$ = competitive inhibition from substrate i to substrate j

S_i = concentration of substrate i (as inhibitor) (g COD/m^3)

S_j = concentration of substrate j (which is utilised) (g COD/m^3)

As another important limiting factor, the C:N ratio is controlled also as a inhibition factor, i.e. when S_{IN} is too low in the reactor, AD will be inhibited. This inhibition (or, say, limitation) exerts on uptake processes of all substrates.

$$\text{Low IN limitation: } I_{IN\_j} = \frac{S_{IN}}{K_{I,IN\_j} + S_{IN}} \tag{15}$$

Where: I_{IN_j} = inorganic nitrogen (IN) as a inhibition function when IN → 0

K_{I,IN_j} = 50 % inhibitory concentration of IN to process j (g N/m^3)

S_{IN} = concentration of IN (g N/m^3)

In summary, pH inhibition affects all processes. IN limitation affects the uptake processes of all substrates. The inhibition of hydrogen affect on the uptake of LCFA, valerate, butyrate and propionate, and the free ammonia only exerts an influence on the uptake of acetate.

4.1.2 Implementation of physico-chemical processes

AD is sensitive to physical conditions, therefore physico-chemical processes are integrated into the model. In ADM1, three physico-chemical processes are considered, i.e. liquid-liquid processes, liquid-gas processes, and liquid-solid processes.

Liquid-liquid processes are characterized by ion association and dissociation with hydrogen and hydroxide ions. These are called the acid-base-reactions. ADM1 proposes two different possibilities to integrate the acid-base-reactions into the model: As they are so rapid, they can firstly be referred to as equilibrium process. Secondly, they can be described as dynamic process with high kinetic coefficients. Feng et al. (2006) studied both approaches for the relevant processes which are NH_4^+/NH_3, CO_2/HCO_3 and HCO_3/CO_3^{2-}. He found that the differences between the approaches are so small that they can be ignored. Thus, the ADM1 recommendation to use the equilibrium approach is followed in this work.

Liquid-gas processes are most important in the model as the production of biogas is one of the benefits of AD. The biogas contains CH_4, CO_2, and water vapour, as well as some other trace gases like N_2 and H_2S. Because of their strong impact on biological processed or outputs, CH_4, CO_2 and H_2 are considered in ADM1 (Batstone et al. 2002b). N_2 is not included as its solubility is so high that the mass flow to gas is negligible compared to that in the output. H_2S is not included because sulphate reduction is not included either in the model.

The liquid-gas processes for these three components are implemented by Henry's law.

The liquid-solid processes mainly include precipitation, sediment, and adsorption. Due to their complexity, ADM1 escapes these processes. The absence of these processes has no significant impacts on the simulation results under the common conditions. However, if these processes are decisive or required to be performed under the particular conditions, they can be implemented by the similar methods as dynamic liquid-liquid processes (Musvoto et al. 2000b, Musvoto et al. 2000a). In BWADM, the precipitation is not included, as it is not critical for the reactors. Nevertheless, the precipitation such as $CaCO_3$, $Mg(OH)_2$, etc. could be integrated into the model according to Musvoto et al. (2000a, 2000b).

The physico-chemical processes are implemented according to ADM1 and the modified model by Feng (2004).

Temperature is one of the key issues for all reactions. The standard temperature in this model is set to 37 °C like in the experiments. ADM1 proposes the implementation of the Arrhenius equation for disintegration and hydrolysis according to Sanders (2001) and the Van´t Hoff equation for biochemical and physico-chemical reactions (Batstone et al. 2002a). However, temperature variation is not further investigated in this study.

4.1.3 Implementation in the simulation software AQUASIM

Software to solve differential equation matrices is required for simulation. For this work, the program AQUASIM 2.1d was chosen, which was developed by EAWAG (Swiss Federal Institute for Environmental Science and Technology) for simulating aquatic systems (Reichert 1994).

The software AQUASIM offers the following options:

- Implementation of mathematical models
- Choice between different types of reactors such as CSTR and plug flow
- Input of the stochiometric and kinetic parameter according to the Peterson matrix
- Introduction of data sets from external files
- Output of results in terms of diagrams or file list

The structure of AQUASIM is divided in four parts: Variables, processes, compartments and links. In the part “variables”, all variables need to be defined such as program variables, kinetic parameter, other constants, external input figures). In the “processes”, all the processes with stochiometric and kinetic parameters are implemented according to the Peterson matrix of ADM1. The reactors are specified in “compartments” where processes are activated and input variables are given. The “links” define the links between the reactors. A detailed description can be found in Reichert (1994) and in the AQUASIM manual and tutorial (Reichert 1998b, Reichert 1998a).

The reactor implemented in the software is a single-stage CSTR. The model is based on mass balances as visualized in Figure 25. In AQUASIM, besides all necessary parameters and variables, as well as processes (both biochemical and physicochemical processes), the model further contains two compartments, i.e. the reactor and the headspace, which represent the liquid phase and headspace, separately. One diffusive link is established between the reactor and the headspace to transfer the biogas.

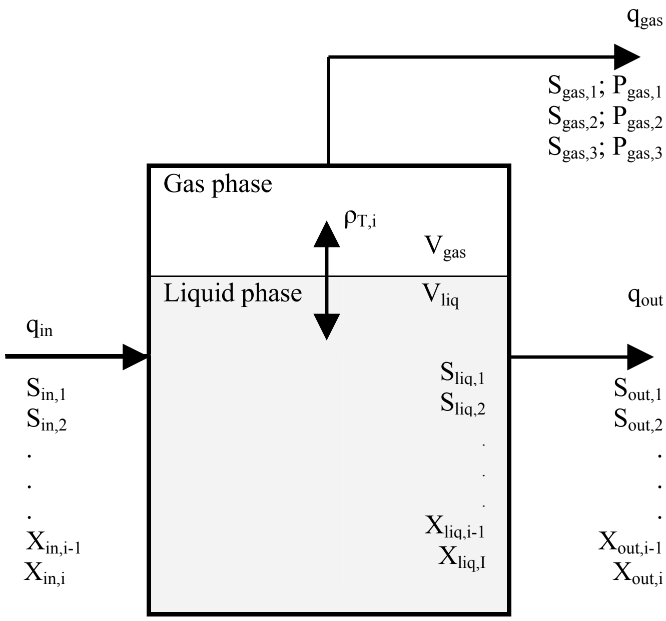

Figure 25: Scheme of a single-tank digester (Batstone et al 2002a)

Equations in the liquid phase

According as the mass balance, the state of each component in liquid phase can be expressed as follows:

$$\frac{dS_{liq,i}}{dt} = \frac{q_{in}S_{in,i}}{V_{liq}} - \frac{q_{out}S_{liq,i}}{V_{liq}} + \sum_{j=1-19}\rho_j \nu_{i,j} \qquad (16)$$

ρ_j is the reaction rate of process j. Here, the reactor is fed discontinuously and the liquid phase volume is constant, so during the non-feeding period, the equation 16 can be simplified as:

$$\frac{dS_{liq,i}}{dt} = \sum_{j=1-19}\rho_j \nu_{i,j} \qquad (17)$$

Equations the in gas phase

In the gas phase, the three main gases CH_4, H_2 and CO_2 present in biogas are dealt with. Likewise, the following equation can be obtained based on the mass balance:

$$\frac{dS_{gas,i}}{dt} = -\frac{q_{gas}S_{gas,i}}{V_{gas}} + \rho_{T,i}\frac{V_{liq}}{V_{gas}} \qquad (18)$$

As the liquid gas transfer is of importance in this model, the equations for implementation are presented as follows.

Henry's law can be applied when the liquid phase is dilute. Based on these, the gas transfer rates can be presented in the form of equation 19.

$$\rho_{T,i} = k_L a \cdot \left(S_{liq,i} - K_{H,i} P_{gas,i}\right) \qquad (19)$$

Where $\rho_{T,i}$ = specific mass transfer rate of gas i,
(g COD/(m^3·d) for CH_4 and H_2; mole C/(m^3·d) for CO_2)

$K_L a$ = overall mass transfer coefficient K_L times the specific transfer area a (1/d)

$S_{liq.i}$ = concentration of gas i in liquid phase, (
g COD/m^3 for CH_4 and H_2; mole C/m^3 for CO_2)

$P_{gas,i}$ = partial pressure of gas i in gas phase (bar)

$K_{H,i}$ = Henry's law coefficient of gas i (mole/m^3/bar)

For equation 19, the partial pressure of each gas is necessary and can be calculated by ideal gas law:

$$P_{gas,i} = S_{gas,i} \cdot R \cdot T \qquad (20)$$

Where $S_{gas.i}$ = concentration of gas i in gas phase,
(g COD/m^3 for CH_4 and H_2; mole C/m^3 for CO_2)

R = gas law constant ($8.314 \cdot 10^{-5}$ bar·m^3/mole/K)

T = absolute temperature (K)

The software AQUASIM provides the diffusion link between compartments, which follows the Henry's law with the transformation of the formula (Reichert 1998b). So equation 19 can be achieved in the model, but the coefficients and the transfer sequence of compartments need to be chosen carefully in AQUASIM. The three main gases CH_4, CO_2, and H_2 are transferred between two phases with the rates from equation 19.

The partial pressure of water vapour in gas phase is also needed in order to calculate the total gas pressure, and it is given by the empirical equation:

$$P_{gas,H_2O} = 0.0313 \cdot \exp\left(5290 \cdot \left(\frac{1}{298} - \frac{1}{T}\right)\right) \qquad (21)$$

The total gas pressure $P_{gas,total}$ is as follows:

$$P_{gas,total} = P_{gas,CH_4} + P_{gas,H_2} + P_{gas,CO_2} + P_{gas,H_2O} \qquad (22)$$

Introducing the pipe resistance coefficient k_p (m^3/bar/d), hereby the gas flow rate q_{gas} (m^3/d) can be obtained as equation 23:

$$q_{gas} = k_p \left(P_{gas,total} - P_{atm}\right) \qquad (23)$$

k_p is the pipe resistance coefficient in m^3/d/bar for calculating the gas flow according to Batstone et al. (2002b). The gas flow is not only depending on the difference in pressure (ΔP) but might be restricted by an orifice. k_p must therefore be chosen according to the outlet of the biogas and the reactor volume.

Siegrist et al. 2002 names k_p the proportional control constant and suggests $k_p > 100\ q_{gas}$ in order to keep the offset $\Delta P < 0.01$ bar. This is the case where the biogas is regularly withdrawn and ΔP is kept small. If k_p is smaller than 100 q_{gas}, the biogas flow rate will be consequently reduced by the model.

Afterwards the biogas products are dried and transformed to standard temperature and pressure conditions by equation 24 in order to make them comparable.

$$q_{gas,STP} = q_{gas} \cdot \frac{\left(p_{gas,air} - p_{gas,H_2O}\right)}{p_{gas,STP}} \cdot \frac{273.15}{T} \qquad (24)$$

Where: $q_{gas,STP}$ = dried biogas volume at standard temperature and pressure (m^3_{STP}/d)

q_{gas} = measured biogas volume from the gas counter (m^3/d)

$P_{gas,air}$ = air pressure (bar)

$P_{gas.H2O}$ = water vapour pressure at temperature T (bar)

$P_{gas,STP}$ = standard atmosphere pressure (1,013.25 mbar)

T = temperature (K)

For more detailed information on the implementation of the model, it is referred to Batstone et al. (2002a) and Feng (2004).

4.2 Calibration and adaptation of the model BWADM to the bench scale results

Based on the experimental data from the bench scale CSTR, the following scenarios are studied reflecting the investigation program see chapter 3:

- Reference conditions with blackwater at 20 days HRT
- Increasing ammonia concentration
- Co-digestion with kitchen refuse

4.2.1 Reference conditions

In the first scenario the reactor run as described in chapter 3 (fed discontinuously three times a week which resulted in a HRT of 20 days). The default parameters and coefficients for the model were taken from Feng (2004) and calibrated with the input data and results of different experimental weeks. The weeks have all the same frame conditions but the blackwater characteristics are different. As reported in chapter 3, the blackwater characteristics are highly fluctuating especially in terms of particulate COD which leads to different curves of the biogas production rate in AD. That is why it is particularly important to specify the blackwater input as most precise as possible in order to calibrate the model reasonably.

For all scenarios, the blackwater of each feeding was characterized by the following parameters which must be introduced separately. Total and dissolved COD, NH_4-N, TIC were analyzed for each feeding, whereas VFA-COD was not analyzed regularly. Anions and cations were not analyzed.

Table 22: Required input parameter for BWADM to determine reactor feeding

Input Parameter	Unit	Name in BWADM	Determination
Total COD	g/m^3	_in_rawBW	Analysis each feeding
Dissolved COD	g/m^3	_in_rawS	Analysis each feeding
VFA-COD	g/m^3	_in_rawVFA	Random analysis
Total N (here NH_4-N including NH_3-N)		_inS_IN	Analysis each feeding
TIC	mM/m^3	_inS_IC	Analysis each feeding
Cations	mM/m^3	_inS_an	-
Anions	mM/m^3	_inS_an	-
Feeding time	day	_inQ_dyn	Each feeding
Feeding volume	m^3	_inQ_vol	Each feeding

Important for the COD mass flow is the composition of the COD. The particulate COD is composed of a degradable and inert part which are analyzed by determination of the anaerobic biodegradability (see chapter 3) and which is 63 and 37 % for degradable and inert in part COD of blackwater, respectively. The division of the degradable part into carbohydrates, proteins and lipids are not analyzed but assumed to be equally distributed (Table 23), a little bit higher for lipids as also reported for blackwater by Kujawa-Roeleveld et al. (2003). The inert part of particulate COD is composed of particulate and dissolved COD which is analyzed to be 82 and 18 %, respectively.

The dissolved COD is assumed to be totally degradable. It is composed of VFA and non-VFA, the composition of VFA was analyzed (Figure 6) whereas the non-VFA is assumed to distribute equally on the products sugar (monosaccharides), amino acids and LCFA.

After this input determination, the stochiometric parameter for the COD mass flow through the AD process are taken from the ADM1.

Table 23: COD input for blackwater into the BWADM

<table>
<tr><th colspan="5">Particulate COD</th><th colspan="7">Dissolved COD</th></tr>
<tr><th colspan="3">Degradable (63% of part COD) [1]</th><th colspan="2">Inert (37% of part COD) [1]</th><th colspan="4">VFA</th><th colspan="3">Dissolved non-VFA</th></tr>
<tr><td>Carbo-hydrates (32% of degradable)[3]</td><td>Proteins (32% of degradable)[3]</td><td>Lipids (36% of degradable)[3]</td><td>Diss inert (18% of inert)[1]</td><td>Part inert (82% of inert)[1]</td><td>Acetic acid (63% of VFA)[2]</td><td>Propio-nic acid (24% of VFA)[2]</td><td>Butyrate acid (8% of VFA)[2]</td><td>Valerate acid (5% of VFA)[2]</td><td>Sugar (33% of diss non-VFA)[3]</td><td>Amino acid (33% of diss non-VFA)[3]</td><td>LCFA (33% of diss non-VFA)[3]</td></tr>
</table>

[1] Percentage based on anaerobic biodegradabiltiy see chapter 3.3
[2] Percentages based on analysis see chapter 3.2
[3] Assumption

To achieve steady state conditions, the simulation started 60 days before the scenario, whereas the last three weeks (one HRT) are already implemented with the real experimental data. It is thus guaranteed that the pre-history of the AD process is considered in the simulation.

The three weeks which are chosen as reference weeks for blackwater AD simulation shall reflect low as well as high-strength blackwater AD with different pre-histories. They are also well checked for their consistency in the COD mass balance.

Figure 26 shows the calibration results in a summarized way. In the graph, the specific biogas production rate related to input COD load is given. With the arrows on the top, the feeding times are marked as well as their total COD concentration. Additionally in the text on the right, the variables pH and NH_4-N are given. As the two variables are stable in steady state, there is no need to show the graph.

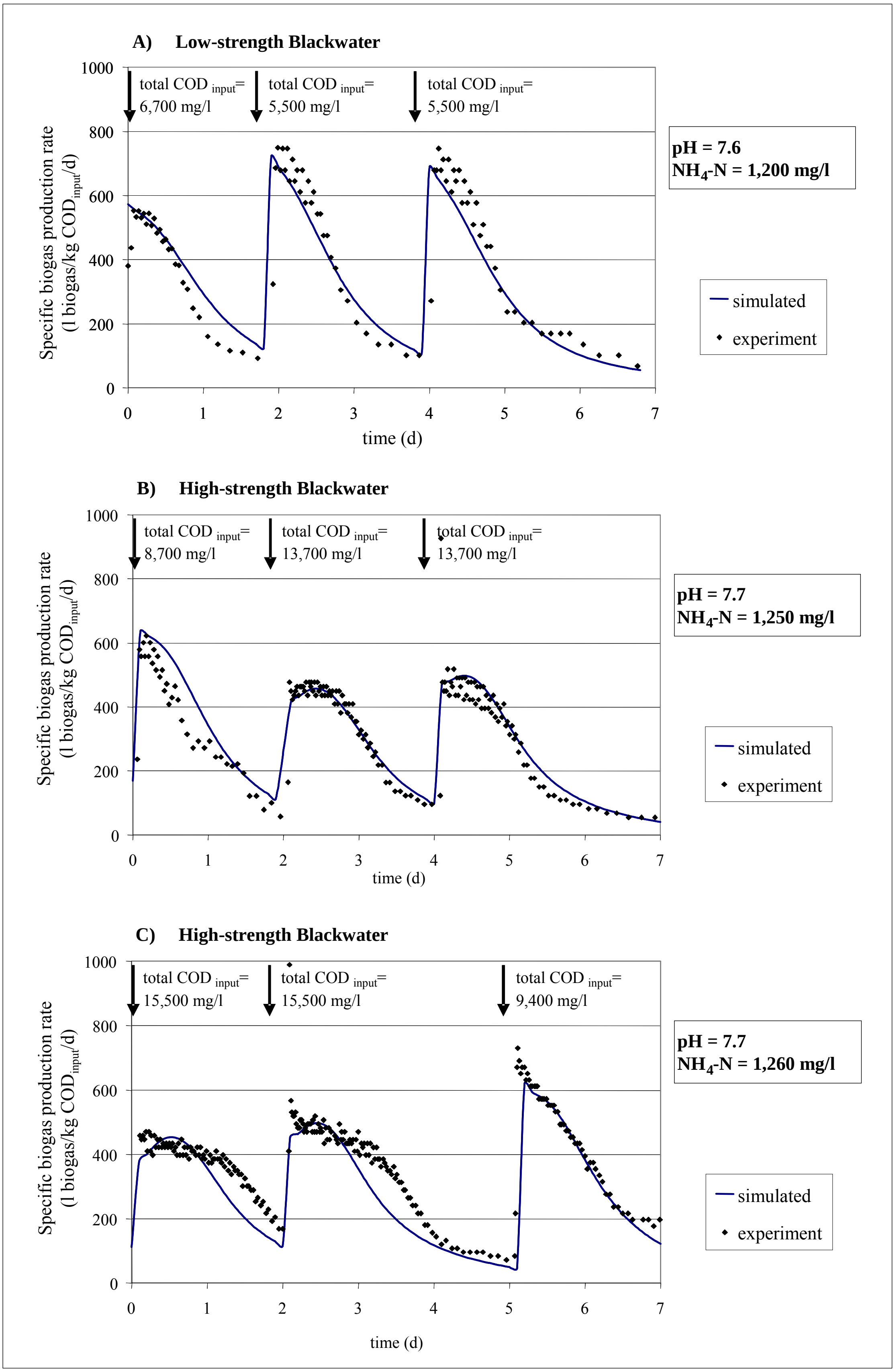

Figure 26: Calibration results of blackwater AD at 20 days HRT

For calibration after giving the input data, first the pH, second the output COD and the NH_4-N concentration are controlled. If they are not consistent with the experimental data, the input variable cations and anions are changed accordingly. The output COD and the biogas composition are also checked but not given explicitly here. If the results of experiments and simulation are met in all these parameters, finally the gas flow is studied. In the biogas production rate curve, the AD process is very well reflected and can be best fine tuned.

Figure 26 shows the typical half bell shape curves. As discussed in chapter 3, the biogas production rate shows different curve characteristics for high and low-strength blackwater. For the high-strength blackwater, the hydrolysis is rate limiting, which is characterized by a plateau during phase 2 where the rate stays constant until it drops down. The low-strength blackwater digestion is not limited by hydrolysis as the particulate COD concentration is much smaller. That is why the rate shows almost no plateau but a single peak.

The model shows good compliance with low-strength blackwater as seen in Figure 26 A, the high experimental peak is not exactly reflected for the second and third feeding whereas the last phase not in the first and second feeding. A possibility to better adapt the curve could be to change the input COD in terms of VFA/dissolved/particulate COD ratio slightly, which could lead to an exact adaptation with the curve. However, this would mean to doubt the analysis. This slight error could as well result from analytical or experimental variations such as the blackwater for feeding not being homogeneously divided into three feeding portions. The differences in figure A are slight and can be accepted. Figure 26 B shows an excellent correspondence between the experimental and simulated data for the high-strength blackwater, which demonstrates the correct selection of k_{dis}. However, in case of the blackwater with even higher COD concentration in the feeding (see Figure 26 C), the simulation cannot exactly reflect the long lasting plateau, where the disintegration and hydrolysis are predominant. However, the third feeding can be reflected perfectly again.

This calibration is discussed in detail in the following part.

Characteristics of blackwater is crucial for the model as predicted before. Most important is the detailed information about the composition of dissolved and particulate COD and the content of VFA in the input. VFA in the input means that the first three steps of AD are skipped and VFA is utilised directly by the acetogenic and acetoclastic bacteria. The distribution of VFA into acetic, propionic, butyrate and valerate acid is not sensitive. This result was reported for butyrate and valerate by Feng (2004) when he skipped both. However, also for a change in concentrations of acetic and propionic acid of up to 20 %, no significant change for blackwater AD is observed. That is due to the high kinetic values for uptake of propionic acid.

Disintegration and hydrolysis: These fist two steps are reported to be often the rate-limiting steps in AD. Disintegration is often the crucial step in case of activated sludge digesters (Shang et al. 2004). Secondary sewage sludge contains many microorganisms and their cell membranes need more time to disintegrate than e.g. primary sewage sludge. Hydrolysis is often the rate-limiting step in AD of complex substrate (Sanders 2001).

In practice, these two steps cannot be separated easily and thus, in most references, both processes are investigated together. Based on 21 reviewed references, ADM1 suggests the kinetic parameters listed in Batstone et al. (2002b). They recommend for solids digesters fed with a relatively homogeneous substrate to carefully apply k_{dis} as subsequent hydrolysis is commonly much faster. That is why they recommend for k_{hyd} = 10 1/d.

This approach is successfully applied in BWADM and k_{dis} is found to be 2.5 1/d after extensive testing with the reference reactor. This value is five times higher than the recommended value by ADM1 and the reference value cited by Batstone et al. (2002a). The reason might be that the blackwater comes from vacuum toilets. In the vacuum system, the pressure of 0.8 bar exerts a lot of energy which leads not only to a well homogenized substrate, but also enhances disintegration. This is however in contrast to the results by Elmitwalli et al. (2006) who found very low values for k_{hyd} of 0.1 and 0.14 1/d for combined blackwater and kitchen refuse (Table 24).

Feng et al. (2006) determined a k_{dis} value of 4.5 1/d in his model., This value is probably high because he calibrated the model with a low-strength blackwater where disintegration is not very sensitive. In this case, BWADM is tested with nine feedings with different characteristics which leads to a more reliable calibration.

When testing BWADM, it is found that disintegration or hydrolysis is the rate-limiting step in the case of high-strength blackwater (Figure 26). When varying k_{dis}, there is a big impact on the course of the biogas production rate. This is not the case for low-strength blackwater, there is only a slight impact. Feng (2004) underlines this fact as for his relatively low-strength BW, hydrolysis is not found to be rate-limiting. The composition of high-strength BW is closer to complex substrates that are often proven to show hydrolysis as the rate-limiting step.

As reported by Batstone et al. (2002b) and Feng et al. (2006), the model is not sensitive to the distribution ratio of carbohydrates, lipids and proteins. In this model tests, the variation of ± 20 % in the distribution ratio causes only slightly different biogas production rates.

Table 24: Kinetic parameter for disintegration and hydrolysis

Parameter	Unit	Description	ADM1 Batstone et al. 2002b	Elmitwalli et al. 2006b	Feng 2004	BWADM
			Mesophilic solids	BW+KR at 30 °C	BW at 37 °C	BW at 37 °C
k_{dis}	1/d	Disintegration rate of composites	0.5	0.1 – 0.14	4.5	2.5
k_{hyd_CH}	1/d	Hydrolysis rate of carbohydrates	10		10	10
k_{hyd_PR}	1/d	Hydrolysis rate of proteins	10		10	10
k_{hyd_LI}	1/d	Hydrolysis rate of lipids	10		10	10

Acidogenesis and acetogenesis: Both processes are successfully implemented in BWADM with Michaelis Menten kinetics as it proven by Feng (2004). The parameters for this model are mainly taken from ADM1 or slightly modified. The acidogenic and acetogenic bacteria have relatively high uptake rates and are therefore not crucial for the process. This is proven by model tests that some changes in the kinetic parameters have no significant effect on the biogas production. In addition, Feng (2004) reported that valerate and butyrate have very little impact and can be skipped. For acetogenesis, it is accepted that the bacteria need the un-ionized form of the VFA which could be adequately implemented into the model. However, this step is assumed to not be crucial so that the suggested parameters by ADM1 are taken into BWADM. The extensive tests of the BWADM at 20 days HRT showed that these steps between hydrolysis and methanogenesis are not critical under these frame conditions.

Methanogenesis: This last step in AD is known to be often the rate-limiting step in AD and as well the most sensitive one comprising the two. It contains the acetoclastic methanogenesis which is investigated further in this study. The suggested Michaelis Menten kinetics with total acetate as substrate as suggested by ADM1 is applied firstly and successfully in this model.

However, as discussed before, it is accepted today that only the un-ionized form of acetate is substrate that can be taken up by the bacteria. Also it is known that the acetoclastic bacteria are inhibited by the substrate itself (substrate inhibition) which can be reflected by Haldane kinetics (Haldane 1930). The Haldane kinetics with un-ionized acetate was successfully applied by different authors (see table 5). Secondly, when the Haldane kinetics are applied within the model, the results are similar to the first approach. The maximum uptake rate is the same for both. The half saturation constant is much lower as it is based on un-ionized acetate concentration (0.15 % of the constant for total acetate). This fits to the calculated concentration of un-ionized acetate at pH 7.5. The Haldane inhibition constant is chosen to be 10 mg/l COD but alterations to this constant show only slight impact on the biogas production under these reference conditions.

By extensive testing of the model with different feedings, it becomes apparent that the determination of the input is crucial in terms of content of VFA and acetate. The higher the ratio of dissolved COD and particularly of VFA, the more dominant position the acetoclastic methanogenesis has. In the case of low-strength blackwater, the content of particulate COD is low and then the acetoclastic methanogenesis becomes the rate-limiting step.

The inhibition of free ammonia on the acetoclastic methanogenensis is investigated in detail in this model. The 50 % inhibitory concentration is determined to be 200 mg/l NH_3-N, which is eight times higher than the value proposed by ADM1. The reason for this high value is the long adaptation process in the bench scale plant. It was reported by many authors (see chapter 2) that adaptation is a crucial step for substrate with high ammonia concentrations. The inhibition threshold is depending on the adaptation conditions of the anaerobic microorganisms.

Table 25: Kinetic parameter for acidogenesis, acetogenesis and methanogenesis (changes to ADM1 are pointed out in light grey)

Parameter	Description	ADM1	Feng (2004)	BWADM
		Mesophilic solids	Mesophilic BW	Mesophilic BW
k_{m_su} (1/d)	Max uptake rate of sugar degraders	30	30	30
k_{m_aa} (1/d)	Max uptake rate of amino acid degraders	50	50	50
k_{m_fa} (1/d)	Max uptake rate of LCFA degraders	6	6	6
k_{m_c4} (1/d)	Max uptake rate of valerate and butyrate degraders	20	18	18
k_{m_pro} (1/d)	Max uptake rate of propionate degraders	13	14	14
k_{m_ac} (1/d)	Max uptake rate of acetate degraders	8	13	13
$k_{m\_ac\_haldane}$ (1/d)	Max uptake rate of acetate degraders (Haldane kinetic)			13
k_{m_H2} (1/d)	Max uptake rate of hydrogen degraders	35	35	35
K_{S_su} (mg/l COD)	Half saturation constant of sugar degraders	500	500	500
K_{S_aa} (mg/l COD)	Half saturation constant of amino acid degraders	300	300	300
K_{S_fa} (mg/l COD)	Half saturation constant of LCFA degraders	400	400	400
K_{S_c4} (mg/l COD)	Half saturation constant of valerate and butyrate degraders	300	110	110
K_{S_pro} (mg/l COD)	Half saturation constant of propionate degraders	300	120	120
K_{S_ac} (mg/l COD)	Half saturation constant of acetate degraders	150	160	160
$K_{S\_ac\_haldane}$ (mg/l COD)	Half saturation constant of acetate degraders (Haldane kinetic)			0.25
$KI_{AC\_ac\ haldane}$ (mg/l COD)	Haldane inhibition constant of acetate degraders (Haldane kinetic)			10
K_{S_H2} (mg/l COD)	Half saturation constant of hydrogen degraders	0.007	0.007	0.007

Table 26: Kinetic parameter for inhibition by pH, hydrogen and free ammonia (changes to ADM1 are pointed out in light grey)

Parameter	Description	ADM1	Feng (2004)	BWADM
		Mesophilic solids	Mesophilic BW	Mesophilic BW
KI_{H2_fa} (mg/l COD)	50 % inhibitory concentration for LCFA uptake by hydrogen	0.005	0.005	0.005
KI_{H2_c4} (mg/l COD)	50 % inhibitory concentration for butyrate and valerate uptake by hydrogen	0.01	0.01	0.01
KI_{H2_pro} (mg/l COD)	50 % inhibitory concentration for propionate uptake by hydrogen	0.0035	0.0035	0.0035
KI_{NH3_ac} (mg/l N)	50 % inhibitory concentration for acetate uptake by free ammonia	25.2	200/50	200
pKI_{1_ac} (-)/ pKI_{2_ac} (-)	50 % inhibitory lower/upper value for acetate uptake by pH			5.4 / 8.6
pKI_{1_other} (-)/ pKI_{2_other} (-)	50 % inhibitory lower/upper value for all other processes by pH			3.6 / 8.4

The simulating curve of biogas production rate complies well with the experimental data, although there are some slight differences for some cases. It can be summarized for these nine selected feedings that BWADM covers well the different blackwater characteristics. Considering the uncertainties in terms of experiments and analysis, the BWADM is a sufficient tool to simulate blackwater AD. The adapted kinetic parameters can be found in Table 25 and Table 26.

4.2.2 Increasing ammonia concentration

Based on the successful calibration of the reference conditions, the model is tested for the scenarios with increasing ammonia concentrations by urea and ammonia salts. Therefore, three weeks are selected to represent each scenario at different NH_4-N concentrations and different pH in the reactor.

Addition of urea

When adding urea to blackwater, the pH increases due to the conversion from organic nitrogen to ammonia (equation 5). In the reactor, the NH_4-N concentration rises up to 2,100 mg/l and the pH to 8.4. Figure 27 shows the development of the specific biogas production rate at increasing NH_4-N concentrations in the reactor.

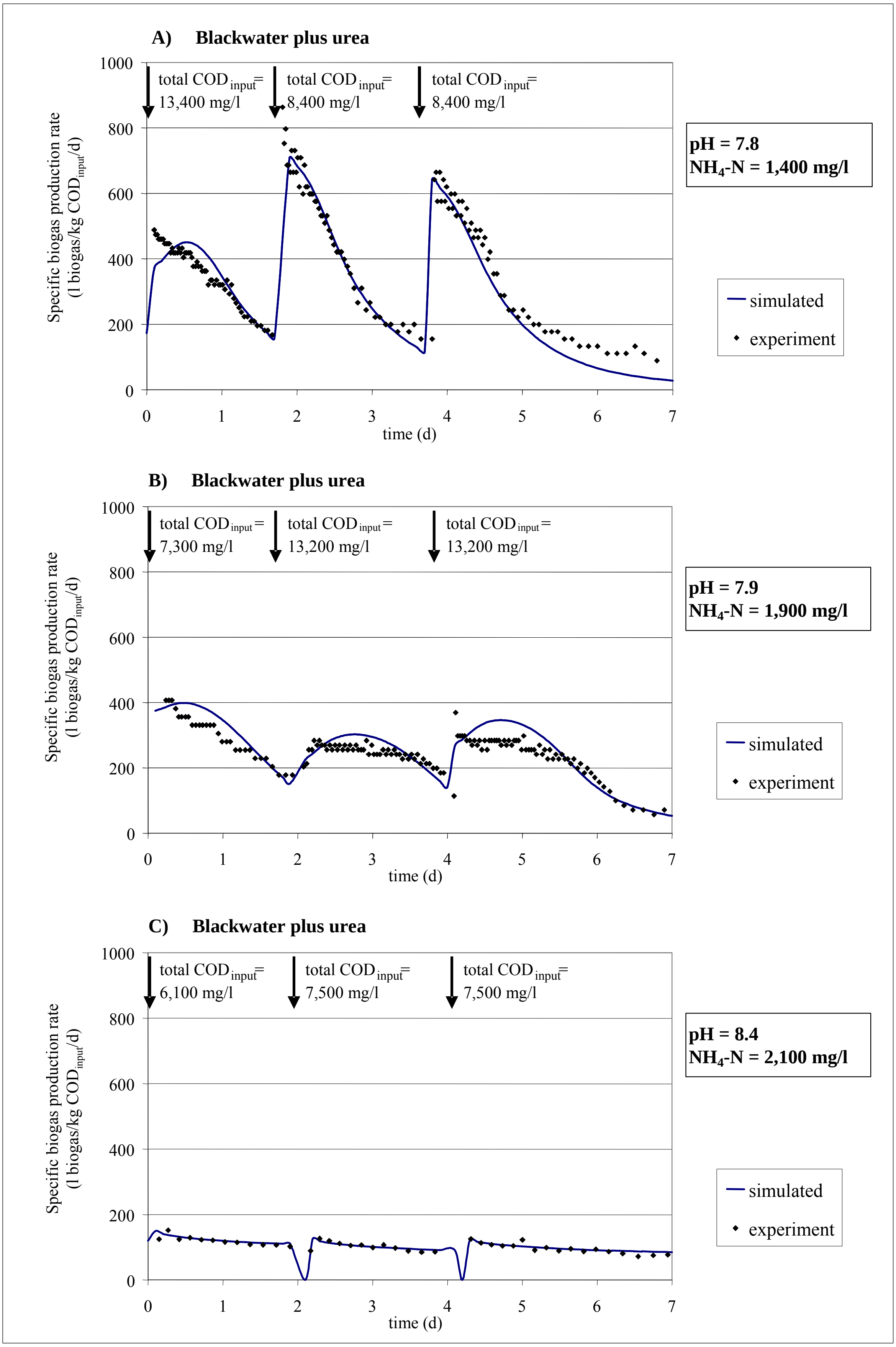

Figure 27: Calibration results of AD of blackwater plus urea at 20 days HRT

When increasing pH, inhibition occurs in two ways. Firstly, the pH inhibition is applied on all processes with different coefficients for acetoclastic methanogenesis and the other processes (see table 5). Secondly, the pH rise leads to an increasing free ammonia concentration which inhibits the acetoclastic methanogenic bacteria.

In Figure 27 A, the NH_4-N concentrations are around 10 % higher and the pH is 0.1 higher than with pure blackwater. Although the free ammonia concentration is around 100 mg/l, the biogas production rate is uninhibited like in Figure 26 which is well reflected by the model results.

When the NH_4-N concentrations increase further to 1,900 mg/l, the pH only slightly increases to 7.9 because of the high buffer capacity with blackwater and urea. Mainly the acetoclastic methanogens are inhibited by free ammonia concentration of around 200 mg/l. The inhibition coefficient $I\_NH_3\_ac$ is 0.55 in this scenario. The scenario is with high-strength blackwater and again the rate develops a long lasting plateau, but on a lower level than pure high-strength blackwater. These data can only be simulated adequately by applying an inhibition factor on the disintegration (and hydrolysis together). By testing the model extensively, a good correspondence can be achieved by applying the same pH inhibition like on the other processes apart from acetoclastic methanogenesis. The pH inhibition of hydrolytic bacteria is reasonable as it was discussed in chapter 3 that the hydrolytic bacteria have their optimum at around 6 similar to the acetogenic bacteria. The simulation can well reflect the inhibited biogas production rate but the long lasting plateau is not perfectly simulated, as it is the case of Figure 27 C.

The increase of NH_4-N concentration up to 2,100 mg/l leads to a pH of 8.4. All AD processes are inhibited with both I_pH_other and I_pH_ac = 0.55. The acetoclastic methanogens are additionally inhibited by free ammonia concentration of 580 mg/l which leads to $I\_NH_3\_ac$ of 0.27. Under these inhibitory conditions, the AD process continues on a very low level with a biogas production rate of 130 l biogas/kg COD_{input}/day. The experimental data can be very well reflected by BWADM.

Addition of ammonia salts

The addition of ammonia salts (NH_4Cl and NH_4HCO_3) is done in order to stabilize the pH. In the reactor, the NH_4-N concentration rises up to 2,100 mg/l as for urea addition but the pH keeps stable at 7.5. Figure 28 presents the development in the reactor.

The specific biogas production rate at ammonia concentration of 1,600 mg/l in the reactor represents the typical bell shape but the peak of the curve is around 25 % lower than comparable for a similar feeding with low-strength blackwater (see Figure 26 A). These lower rates result from an inhibition by free ammonia on the acetoclastic methanogens ($I\_NH_3\_ac$ = 0.71). The fact that the inhibition coefficient correlates nearly totally to the biogas production rate, underlines again that the methanogenesis is the rate limiting process for low-strength blackwater.

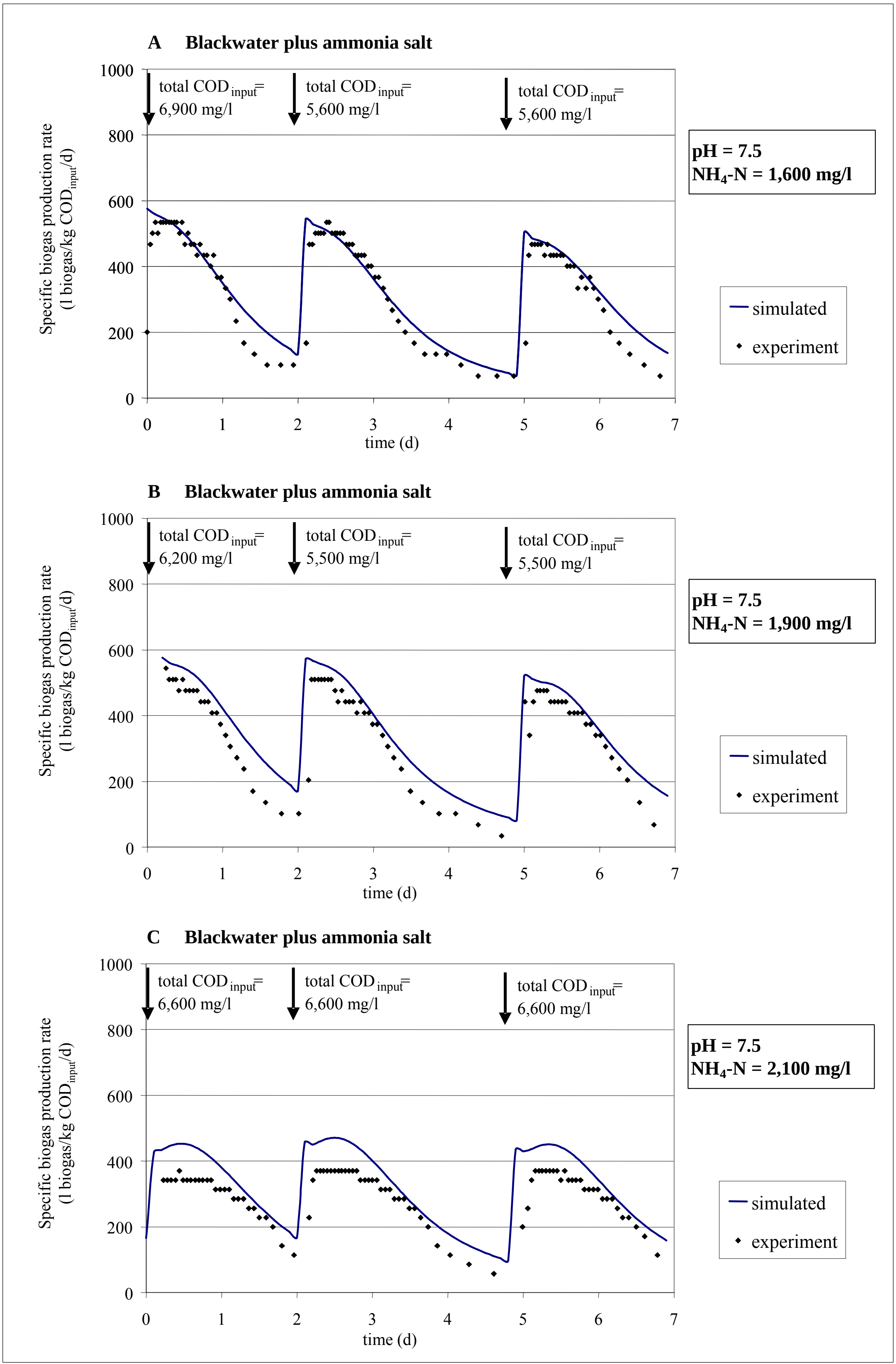

Figure 28: Calibration results of AD of blackwater plus ammonia salts at 20 d HRT

While increasing the ammonia concentration to 1,900 mg/l NH_4-N (Figure 28 B) at the same pH in the reactor, the simulated curve shows similar rates than for 1,600 mg/l NH_4-N (Figure 28 A). The inhibition by acetoclastic methanogens is slightly higher but the experimental results show an even higher inhibition than expected by free ammonia. The simulation rate is higher also for 2,100 mg/l NH_4-N (Figure 28 C). After checking all possible parameters, it is most probable that the high salt content has an inhibitory effect itself. At an ammonia concentration of 1,900 mg/l NH_4-N, the addition of ammonia salts is 5 g/l. Some authors reported on inhibiting effects on all microorganisms by increasing osmotic pressure (Kargi and Dincer 1998, Kempf and Bremer 1998, Vyrides and Stuckey 2007). The higher the salt content is, the higher the inhibitory effect on the biogas production rate. The salt inhibition is however not reflected in ADM1.

The good simulation results for pH from 7.5 to 8.4 at different ammonia concentrations prove the correct selection of the kinetic coefficients (particularly for the inhibition) to be worthy. BWADM can be applied accurately even if the urine content in blackwater is higher than consistently distributed.

4.2.3 Co-digestion with kitchen refuse

Co-digestion is implemented in BWADM by a separate set of input parameter including separate disintegration coefficient for kitchen refuse. Following disintegration and hydrolysis, the same coefficients are applied as for AD of blackwater.

As for BW AD the detailed composition of the COD composition of kitchen refuse is essential for the COD mass flow (Table 27). Certainly, the composition of kitchen refuse is varying much more than blackwater and thus the biodegradability can highly fluctuate. To calibrate the model, the data are based on the experimental framework where only two charges of kitchen refuse are taken for the experimental run.

Table 27: COD input for kitchen refuse into the BWADM

Particulate COD (76 %) [3]					Dissolved COD (24 %) [3]						
Degradable (77% of part COD)[1]			Inert (23 % of part COD)[1]		VFA				Dissolved non-VFA		
Carbo-hydrates (32% of degradable)[2]	Proteins (32% of degradable)[2]	Lipids (36% of degradable)[2]	Diss inert (13% of inert)[1]	Part inert (87% of inert)[1]	Acetic acid (63% of VFA)[2]	Propio-nic acid (24% of VFA)[2]	Butyrate acid (8% of VFA)[2]	Valerate acid (5% of VFA)[2]	Sugar (33% of diss non-VFA)[2]	Amino acid (33% of diss non-VFA)[2]	LCFA (33% of diss non-VFA)[2]

[1] Percentages based on calculation see chapter 3.2 and 3.6
[2] Assumption
[3] Percentages based on analysis see chapter 3.2

The anaerobic biodegradability is determined based on the calculation presented in chapter 3.6 which is 77 and 23 % for degradable and inert in particulate COD, respectively. The division of the degradable part into carbohydrates, proteins and lipids is not analyzed but assumed to be equally distributed based on the visual impression. The inert part of particulate COD is composed of particulate and dissolved COD which is analyzed to be 87 and 13 %, respectively. For the dissolved COD and the VFA-COD, the same distribution as in blackwater is assumed.

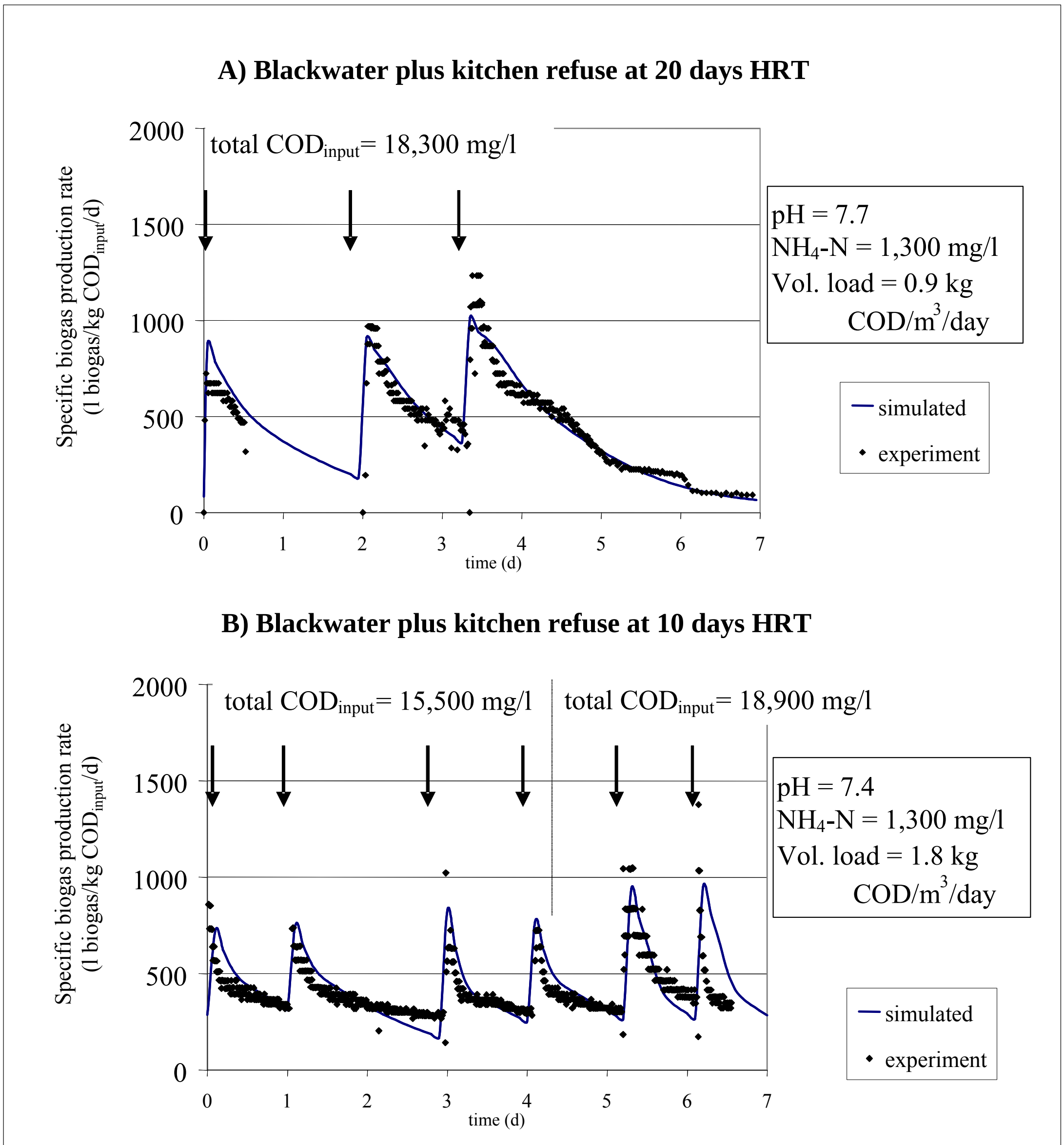

Figure 29: Calibration results of AD of BW+KR at 10 and 20 days HRT

The calibration results for AD of blackwater and kitchen refuse at HRT of 20 and 10 days in Figure 29 are generated in the same manner than previously. The graph at 20 days HRT is similar to reference conditions but with a volumetric load of 0.9 kg COD/m^3/day, which is twice higher than without kitchen refuse.

The specific biogas production rate follows the bell shape curve as for low-strength blackwater (Figure 26 A) but it reaches higher peaks up to 1,000 l biogas/kg COD$_{input}$/day. This is explained by the higher anaerobic biodegradability of kitchen refuse compared to blackwater. Shortly, the peaks are sometimes even much higher up to 2,500 l biogas/kg COD$_{input}$/day. Due to the higher concentrations of dissolved COD or VFA, the first two or three steps are skipped and the methanogens metabolize the substrate immediately to methane. After this high peak, the biogas production rate drops very fast down to a level below 200 l biogas/kg COD_{input}/day. The curve can be well simulated by the model. The pH can also be well reflected as well as the methane concentration.

At 10 days HRT, the biogas curve is similar but the feeding frequency is daily so that the curve does not reach the final phase until the next feeding time (Figure 29 B). This status is the so-called ‘inhibited steady state’. This means that the process is stable but suffers from constant inhibition. The peaks are at the same level as for 20 days HRT as it is well simulated in the model.

Characteristics of blackwater and kitchen refuse are crucial for the model especially because kitchen refuse composition can fluctuate highly. As proven with blackwater AD, the distribution of dissolved and particulate COD and the anaerobic biodegradability have a major impact, which is the same for blackwater and kitchen refuse. As the distribution of particulate COD on carbohydrates, lipids and proteins varies more than in blackwater, their impact is investigated here for HRT of 20 and 10 days. The distribution of the reference blackwater plus kitchen refuse is assumed to be 0.3/0.25/0.25 for lipids/carbohydrates/proteins. Each portion is then increased by about 100 % and simulated in BWADM. Figure 30 shows the simulation results at 20 days HRT and the results for 10 days are similar in respect to this issue. They show slight changes in the biogas production curves, which are of minor importance compared to the range of the experimental data.

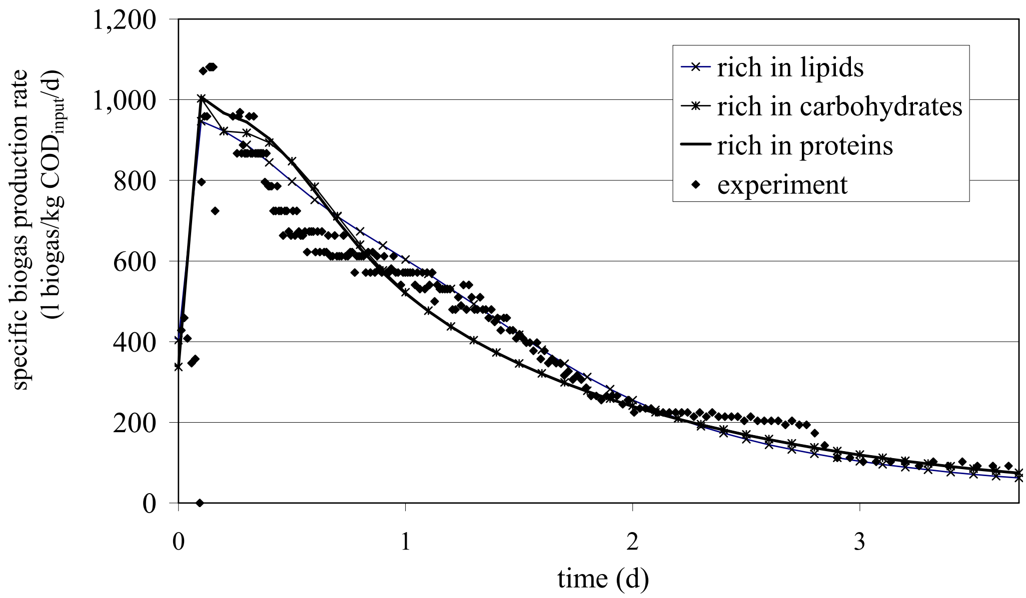

Figure 30: Simulation results of AD of combined blackwater and kitchen refuse with different distribution of lipids/carbohydrates/proteins in the particulate COD

Disintegration and hydrolysis: The characteristic of kitchen refuse is of special importance for well defining the kinetics for disintegration and hydrolysis. The disintegration rate is dependent on the pre-treatment of kitchen refuse. The kitchen refuse here is shredded to a maximal diameter of 2mm, which enhances these first processes. In the model, k_{hyd} is kept at 10 1/d for carbohydrates, lipids and proteins, similarly to blackwater. k_{dis} for kitchen refuse is found to be 0.3 1/d. It is significantly lower than for blackwater.

Veeken and Hamelers (1990) report k_{hyd} values of 0.1 to 0.35 1/d for six different wastes (wholewheat bread, leaves, bark, straw, orange peelings, grass and filter paper). Their statement that smaller values are for the outdoor or garden waste and the higher values for indoor waste such kitchen refuse fits well to the chosen value. Feng et al. (2006) also applied successfully k_{hyd} = 0.3 1/d for kitchen refuse. However, Rusdi et al. (2005) determined far lower values for $k_{hyd\_ch}/k_{hyd\_pr}/k_{hyd\_li}$ of 0.01-0,09/0.01/0.01 1/d (when keeping k_{dis} at 0.5 1/d) for paprika, tomato leftovers and mayonnaise as typical kitchen refuse. Explanations for these varying values are firstly the different frame conditions (pre-treatment and experimental set up) and secondly the impact of the inoculum or other substrates added like blackwater in this case. It can be assumed that blackwater has an enhancing impact on the process.

Acidogenesis, acetogenesis and methanogenesis: The model is applied for blackwater and kitchen refuse with the same kinetics as for blackwater and reflects well the biogas production rate. However, the model shows shortcomings in simulating the process at 10 days HRT. The inhibited steady state process is very complex, the acetoclastic methanogenesis is working well at VFA-COD concentrations of 2,500 mg/l (see Figure 29). The model reflects slightly lower (about 15 %) VFA-COD concentrations. The COD and VFA concentrations determined in the experiments cannot be perfectly simulated in BWADM. Although the biogas production rate is adequately simulated the COD removal (62 %) is about 10% higher than the experimental (50 %).Also, selecting the $KI_{AC\_ac\_haldane}$ well cannot solve the problem. Under these conditions there is no significant difference between Michaelis-Menten and Haldane kinetics and the uptake of un-ionized acetate.

4.2.4 Impact of pre-treatment on AD

As the characteristic of the input is crucial for the AD process, the impact of pre-treatment is investigated by simulation. As shown in chapter 3.1.8, the pasteurization as well as pre-acidification cause an increase of dissolved and VFA COD up to 20 %. This was introduced into BWADM in comparison to typical values for blackwater and kitchen refuse (Figure 31). As expected based on the discussion above, the higher the dissolved COD concentration, the higher the first peak where the acetoclastic methanogens are rapidly consuming acetate. An increase of dissolved COD concentration of 10 % and the VFA concentration accordingly produces a higher peak of 12 % in the biogas production. Also the hydrolysis might be slightly enhanced by pre-treatment.

Subsequently, the total process is faster and the HRT can be decreased when pre-treatment is applied. In practice however, this effect gets lost in the high standard deviation of the blackwater organic matter.

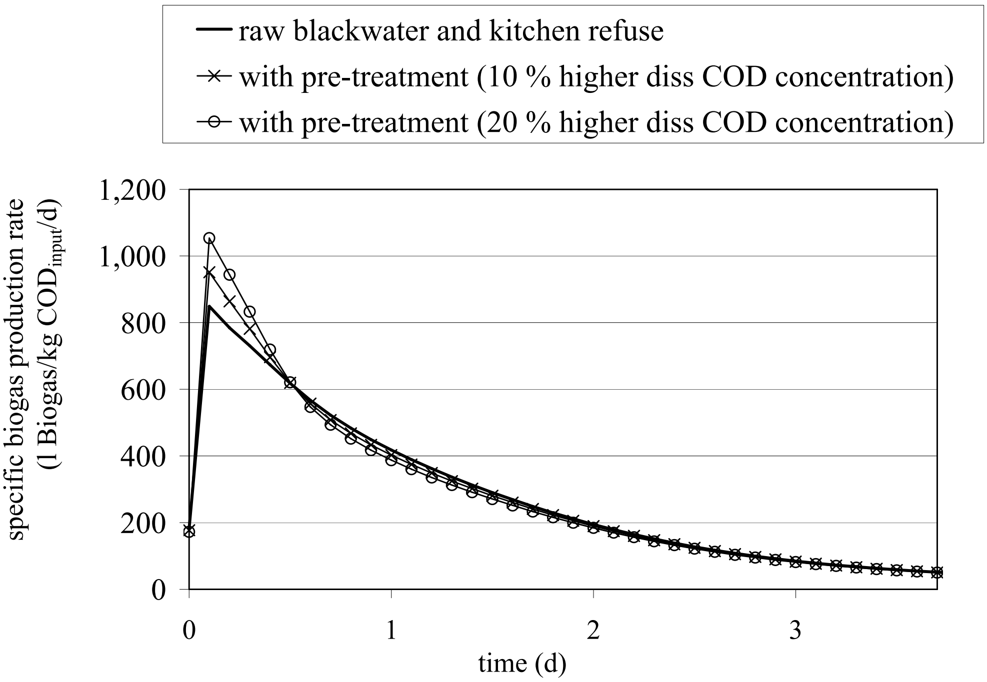

Figure 31: Simulation results of AD of raw and pre-treated mixture of blackwater und kitchen refuse

4.3 Up-scaling of the model

After calibrating successfully on blackwater and kitchen refuse, the BWADM can be applied and used for simulation purposes. Based on the results, the performance of a large scale biogas plant, e.g. *Flintenbreite*, is simulated in virtual scenarios. The main objective is to evaluate the limits of the biogas plant in terms of hydraulic and organic load.

The frame conditions are therefore scaled up, the differences between the lab-scale reactor and an up-scaled biogas plant in *Flintenbreite* are summarized in the following table.

The plant in *Flintenbreite* has been modeled here as a CSTR. Although it is a horizontal flown reactor, its design is more close to a CSTR than to a plug flow, especially at relatively high HRT. The headspace volume related to the total volume is slightly higher than in the bench scale reactor. As k_La is dependent on the surface area between liquid and gas, it is increased by about 50 times. k_p is proportional to the total gas flow and is increased by about 100 times (Table 28).

For simulation, all other coefficients are taken from the calibration of the bench scale plant. The average blackwater and kitchen refuse characteristics are selected as feed. For all scenarios, the simulation is carried out until steady state conditions, the following results are taken at steady state.

It is assumed that the feeding frequency is daily as the pre-pasteurization does not allow a continuous feeding to guarantee safe hygienization.

Table 28: Up scaling the frame conditions of the model

	Bench scale CSTR	Up-scaled biogas plant
Total reactor volume (m^3)	0.01	73
Headspace (m^3)	0.002	23
k_p (m^3/bar/d)	100	10,000
k_La (1/d)	20	1,000
Feeding frequency	3 times per week	daily

4.3.1 Simulation of the scenario *Flintenbreite*

With BWADM, the conditions in *Flintenbreite* are first simulated, 400 inhabitants with 5 l/cap/d blackwater and 0.2 kg/cap/d kitchen refuse. The results are shown in Figure 32.

The specific biogas production rate is similar than for the bench scale plant. The curve is however smoother which is caused by up-scaling in general and the bigger headspace related to the total volume. All other parameters like pH and biogas composition are similar to the bench scale results.

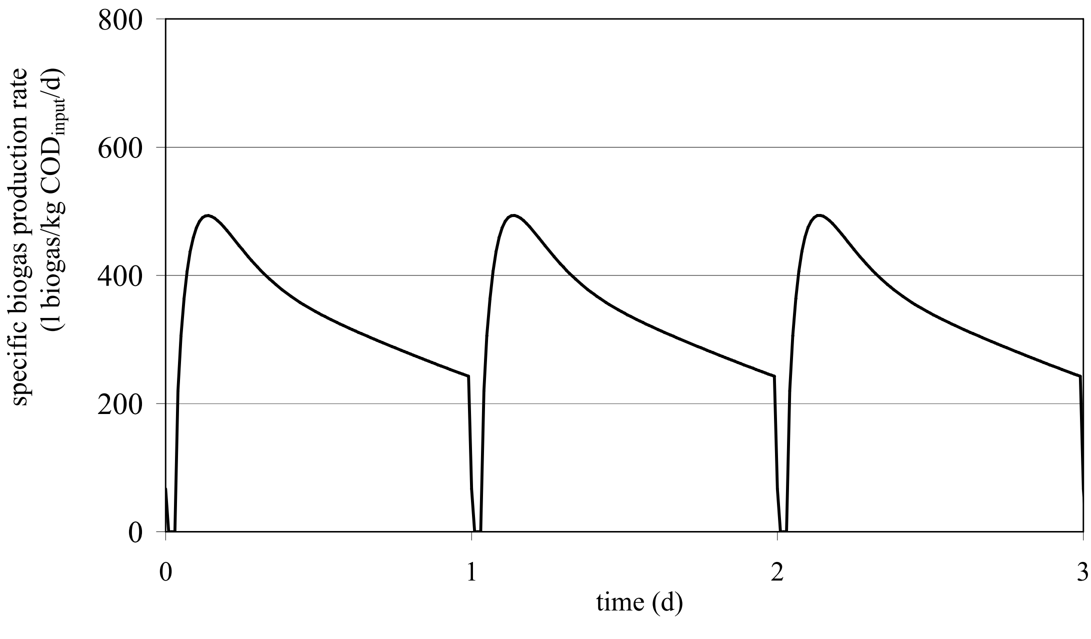

Figure 32: Simulation results of AD of blackwater plus kitchen refuse for the biogas plant *Flintenbreite*

The simulation analysis show that the curve is varying with k_p and k_La. The lower both values are, the smoother the biogas production curve is. k_p and k_La chosen here cannot be proven in practice yet as the biogas plant in *Flintenbreite* is not operating. The chosen values and their results are however reasonable due to the correspondence with the lab-scale results.

4.3.2 Varying HRT and COD load

With BWADM, two scenarios are studied further:

Scenario A: Varying the HRT from 24 to 6 days. Assuming that the number of inhabitants (PE) increases and the specific blackwater plus kitchen refuse production stay the same, the hydraulic load rises with the same organic concentrations and the HRT decreases.

Scenario B: Varying the COD load from 0.8 to 10 kg/m^3/d. Assuming that the number of inhabitants (PE) stays 400 and there is more kitchen refuse or other organic substrate available to be treated in the plant, then the COD load predominantly rises.

The results are presented in terms of COD removal.

Scenario A

The *Flintenbreite* conditions are 400 PE resulting in 24 days HRT. When increasing PE and thus decreasing HRT, the COD removal slightly drops from 74 % to 66 % at 24 and 10 days HRT, respectively. At 6 days HRT, the process is disturbed and the COD removal is 15 % only. Compared to the bench scale results, the situation at HRT > 13 days is very well reflected, there being a slight inhibition impact. The process around 10 days HRT is characterized by an inhibited steady state.

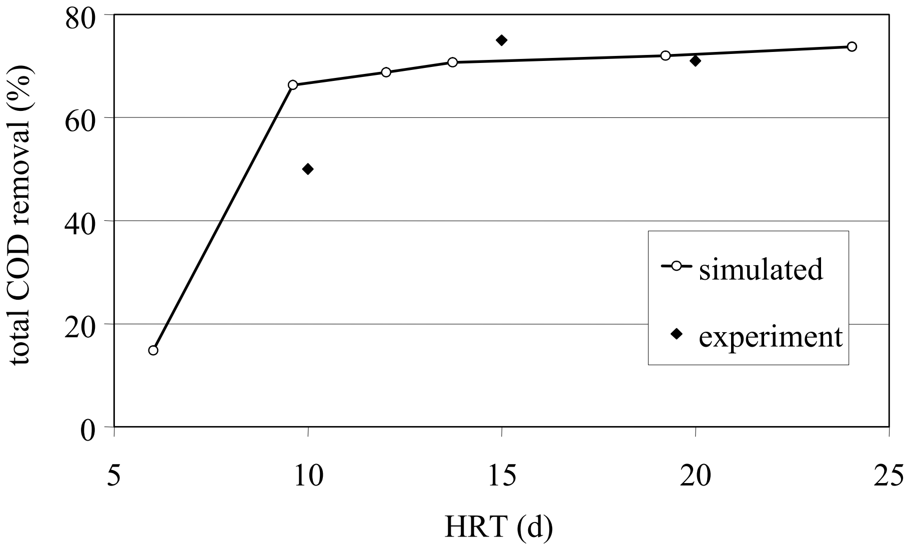

Figure 33: Simulation of scenario A in *Flintenbreite* with varying HRT from 6 to 24 days

The COD removal achieved by simulation is 15 % higher than the experimental data at HRT of 10 days. In the technical plant, the feeding is simulated more homogeneously with average concentrations which might result in a better performance and lower inhibition by short feeding peaks as simulated for the bench scale plant. This fact could partly explain the differences between experiment and simulation. However, the typical inhibited steady state apparently cannot be reflected by the BWADM perfectly, which was already observed before.

Scenario B

The *Flintenbreite* COD load at 400 PE is 0.8 kg/m^3/d. When keeping PE constant and increasing the kitchen refuse load, the COD load rises and subsequently the hydraulic load slightly as well. In five steps, kitchen refuse load is rose times 3, 5, 10, 15 and 20 with COD loads of 1.8, 2.7, 5.1, 7.5 and 9.9 kg/m^3/d, respectively. HRT drops from 24 to 22, 21, 18, 16 and 14 days, respectively.

The results show that the COD removal decreases continuously from 74 to 66 % at 7.5 kg/m^3/d (16 days HRT). When further increasing the COD load, the process breaks down to 16 % at 9.9 kg/m^3/d.

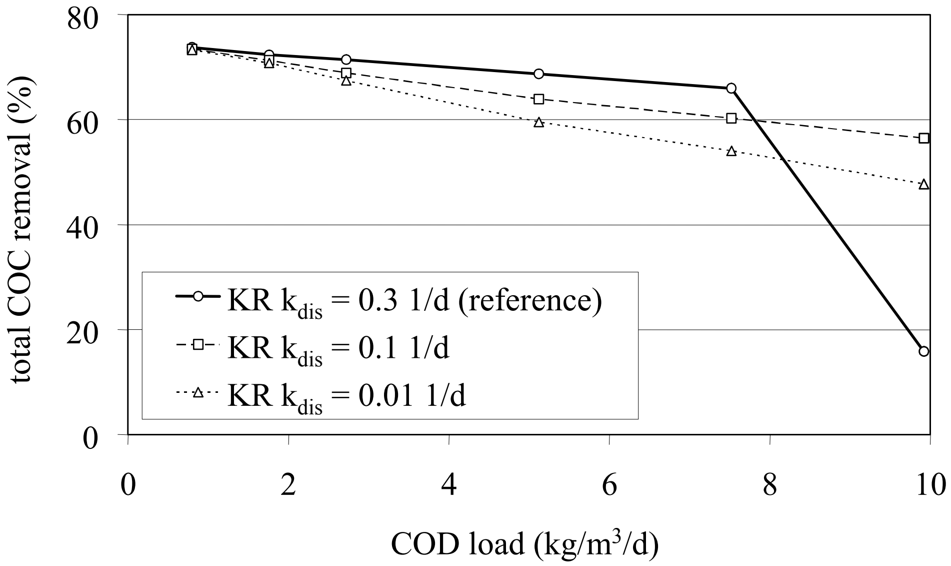

Figure 34: Simulation of scenario B in *Flintenbreite*, increasing COD load

These simulation results are not verifiable by experiments here but they correspond to common design criteria for maximal COD load in AD (Bischofsberger et al. 2005). The results show that there is a large potential for additional feeding of kitchen refuse or other organic solid wastes.

The composition of kitchen refuse or other organic waste might vary, thus a sensitivity analysis is carried out. The limiting parameter is k_{dis} in this model and k_{dis} is reported to be even smaller in many references. k_{dis} is therefore investigated in the range from 0.3 1/d (reference in this model) and 0.01 1/d in this sensitivity analysis. At reference conditions, the COD removal is the same for the three cases (Figure 34) because HRT is high and COD load is low. While increasing the COD load, the COD removal decreases to 60 and 54 % at 7.5 kg/m^3/d for k_{dis} of 0.1 and 0.01 1/d, respectively. When increasing the COD load further, the process does not break down as for k_{dis} of 0.3 1/d, but the COD removal decreases constantly to 56 % and 47 % at k_{dis} of 0.1 and 0.01 1/d, respectively. These results clearly show that disintegration and hydrolysis are the bottle neck steps which control the velocity of the total process. To sum up, at 7.5 kg/m^3/d

COD load the decrease of k_{dis} results in a decrease in COD removal of 7 % and 13 % at k_{dis} of 0.1 and 0.01 1/d, respectively.

The results show clear limits in terms of hydraulic and organic load in the AD process. For the composition of blackwater from vacuum toilets plus kitchen refuse, the process is more limited by hydraulic than by organic load. There is a potential up to 15 times addition of kitchen refuse or other organic waste.

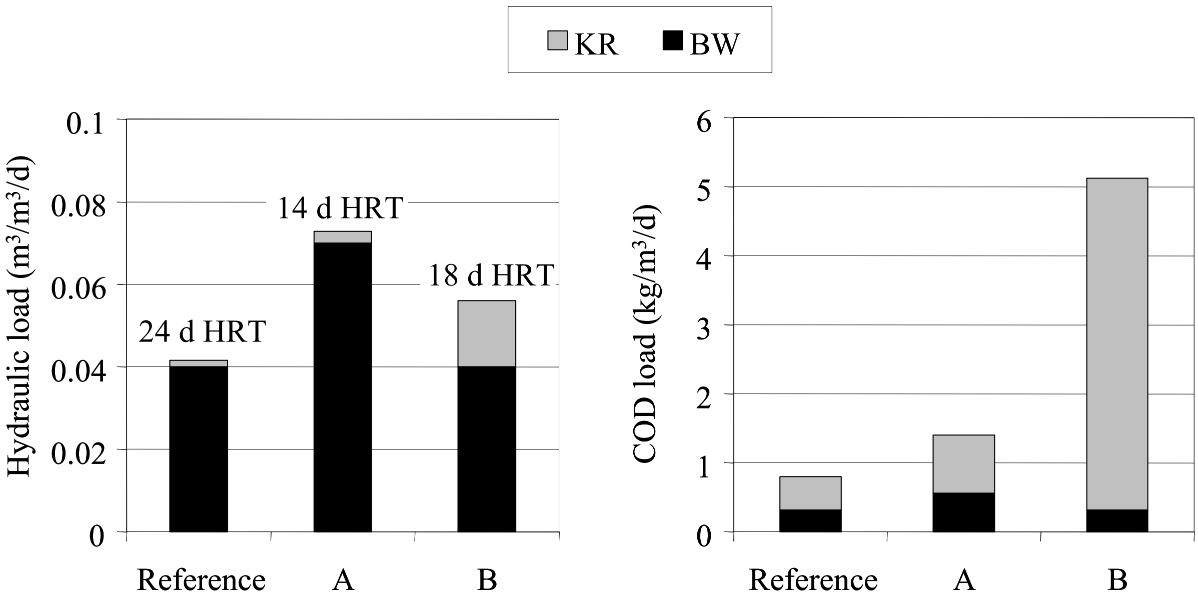

Figure 35: Comparison of hydraulic and COD load of the simulated scenarios

In practice, it is reasonable to consider a security factor because both blackwater and kitchen refuse are highly fluctuating in terms of COD characteristics. The practical limits in terms of hydraulic and organic load are presented in Figure 35 and 36. Based on the experimental and simulation results, the HRT can decrease to minimum 14 days and the COD load increase to maximum 5 kg/m^3/d in order to achieve COD removal > 70 %. If disintegration and hydrolysis are slower (k_{dis} = 0.1, 0.01 1/d), the COD removal is 64, 60 %, respectively.

The limits of AD in terms of hydraulic and organic load based on the simulation results of reference scenario, scenario A and B are clearly summarized in Figure 36. Therefore the results are clustered into 3 different status:

(1) Uninhibited steady state process with COD removal of 66 to maximal 75 % and

(2) inhibited steady state process with COD removal of 50 to 65 % and

(3) inhibited process characterized by COD removal < 50 %.

In general, the higher the COD load, the higher the HRT is required to achieve the same COD removal performance. If the COD load is below 2 kg/m^3/d, the HRT can drop to 12 d to still have an uninhibited process. If the COD load increases to 5 kg/m^3/d, the HRT must be higher than 15 d to achieve an uninhibited digestion. The border between inhibited steady state and inhibited process is determined averagely and must be understood within a range because the simulation does not perfectly reflect the status of inhibited steady state. The space at the top right of Figure 36 (long HRT at high COD load) cannot be reached by the substrate characteristics blackwater and kitchen refuse.

The reference conditions at *Flintenbreite* are 24 d HRT at 0.8 kg/m^3/d which indicate again the high potential in terms of hydraulic and COD load.

If the reactor volume could be newly designed and the amount of additional co-substrate can be selected then the limit is given by the solid concentration of the input mixture which should be not higher than 60 g/l to ensure a proper operation (Leschber and Loll 1996).

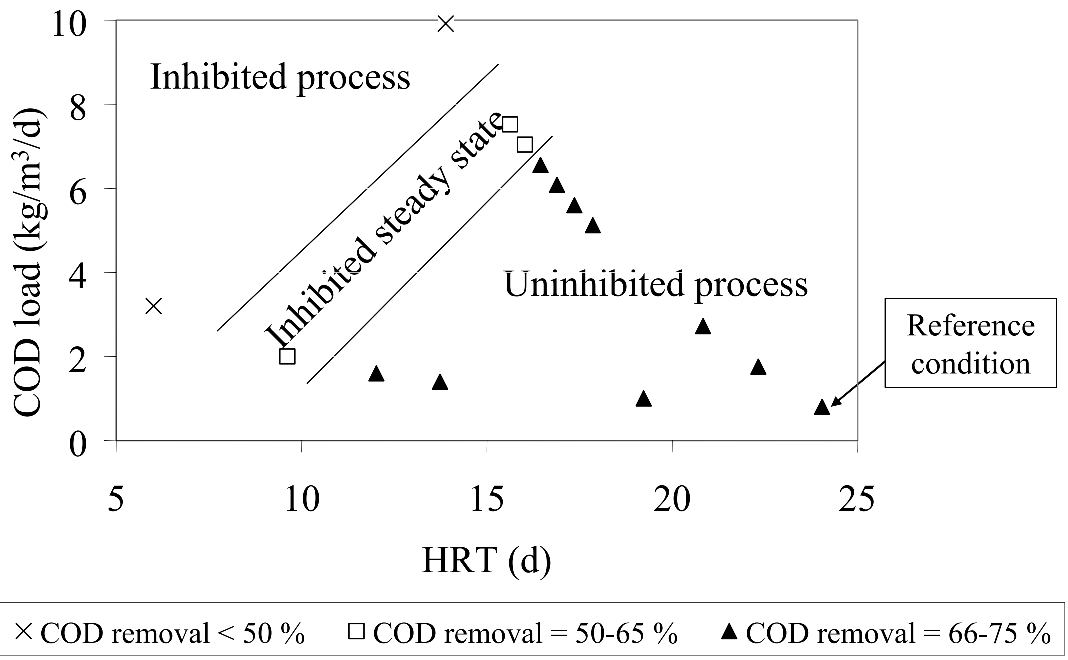

Figure 36: AD process stability related to COD load and HRT based on simulation results

Methane production in Flintenbreite

Based on the results above, the expected methane production is 3,768 m^3/year for the reference conditions. If 100 % of the blackwater was collected in *Flintenbreite*, the methane production would rise by about 24 % (Table 29). If it was possible to add a similar co-substrate as kitchen refuse, the methane production could be 6 times higher than for reference conditions. As blackwater from vacuum toilets and collected kitchen refuse in consistent composition can reach the limit in terms of hydraulic load by appropriate reactor design but not in terms of organic load, it is recommended to add additional solid organic waste.

Table 29: Expected methane production in *Flintenbreite*

Parameter	**Unit**	***Flintenbreite***	**Assumed that 100 % BW is collected**	**Addition of co-substrate** [a]
Inhabitants	PE	400	400	400
BW	l/cap/year	1,825	3,042	1,825
KR	kg/cap/year	73	73	73
Additional co-substrate [a]	m^3 COD/year			78.8
Total COD load	$kg/m^3/d$	0.8	1.0	5.1
HRT	d	24	15	18
CH_4-production	m^3/year	3,768	4,660	22,467

[a] similar to kitchen refuse

4.4 Chapter conclusions

- The input characteristics are crucial in terms of distribution of dissolved/particulate COD and degradable/inert COD. Variations in the ratios for carbohydrates, proteins and lipids do not show a significant impact on the biogas production rate. These ratios can be assumed, whereas the first should be determined carefully by analysis.
- As pre-treatment by pasteurization or acidification cause a significant increase in dissolved COD and VFA concentrations, its impact must be considered within the model.
- The applied k_{dis} (including disintegration and hydrolysis) for blackwater and kitchen refuse are 2.5 and 0.3 1/d, respectively. They are higher than the values found in the literature. The hydrolytic bacteria are inhibited at high pH which is implemented by an enzyme based approach in the model with the same function as for the other processes apart from acetate uptake.
- The 50 % inhibitory constant for free ammonia inhibition (200 mg/l) is higher than suggested by ADM1 and other authors which might be due to long adaptation time of more than six months.
- The enzyme based approach for pH inhibition with two different functions for the acetoclastic bacteria (optimal pH at 7) and the other bacteria grouped together (wider range and optimal pH at 6), respectively, is successfully implemented in the range of pH from 7.3 to 8.4 which is a typical range of blackwater digestion.
- Haldane kinetics for acetate uptake is successfully applied under these conditions. For all other biochemical processes Michaelis-Menten kinetics as enzyme based approach work well.
- Based on the *Flintenbreite* conditions of 400 inhabitants, the daily blackwater load of 5 l/cap/d and kitchen refuse load of 0.2 kg/cap/d, the simulation of the biogas plant

Flintenbreite shows results far under the reactor´s limits in terms of hydraulic and organic load.

- It is an assumption how much organic waste the inhabitants collect separately. Based on the reference data, the organic load can be increased by up to 10 times (5 kg COD/m^3/d) without operational problems. This potential can be exploited by adding co-substrate. The methane production increases by about 6 times if the additional co-substrate has similar characteristics than kitchen refuse.
- The simulation results show that the variation of disintegration and hydrolysis rates from 0.01 to 0.3 1/d has no significant impact on the performance. Even by increasing the COD load, there is still a stable process at inhibited steady state conditions taking place. This situation is characterized by higher VFA concentrations, lower pH in the reactor and lower CH_4-content in the biogas.
- The impact of shock loads is not analyzed within this study as the conditions in *Flintenbreite* provide the possibility of equalising the flows in the collection, mixing and pasteurization tank in front of the biogas plant. Only for additional co-substrate is there a lack of storage on site. However, the long HRT > 15 d leads to a process stabilization against shock loads itself.
- The calibrated and tested model BWADM is able to serve as a tool for planner to design a CSTR for anaerobic digestion of blackwater and kitchen refuse and predict its operational performance.

CHAPTER 5 PROPOSED ANAEROBIC DIGESTION SYSTEM FOR HOUSEHOLD WASTEWATER

A full assessment of a treatment concept contains an economical and ecological analysis. A lot of research has been carried out during the last years dealing with these aspects of resource management sanitation projects which are shortly mentioned.

As a case study, a defined part of the city Berlin with around 5,000 inhabitants was studied in terms of costs and ecological footprint for different wastewater systems. Peter-Fröhlich et al. (2005) found that a system based on vacuum technology and blackwater AD has higher investment and lower operation costs, and the overall costs are equivalent to an aerobic system. The ecological performance and sustainability of different systems by using a life cycle assessment were carried out by Remy and Jekel (2007) for the same case study. They reported significant advantages of the system based on vacuum technology and blackwater AD, especially concerning the replacement of chemical fertilizer and less eutrophication of the receiving waters.

Meinzinger et al. (2007) presented preliminary results of a material flow analysis of different sanitation systems for the city Hamburg: A generally higher nutrient recovery in terms of total nitrogen, phosphorus, potassium and sulphur could be achieved in de-centralized systems based on AD as compared to the more centralized and conventional systems.

Several authors calculated energy data for anaerobic treatment concepts based on AD with resulting energy production of e.g. 200 MJ/cap/year (Otterpohl et al. 1997, Zeeman et al. 2007a).

The references are hardly comparable but two aspects can be found in common:

- Decentralized sanitation concepts based on AD are favorable due to ecological aspects such as nutrient recovery.
- A positive energy balance is assumed but there is mostly a lack of detailed energy calculations and CO_2 emissions.

5.1 Description of the proposed AD system in comparison to an aerobic system

The energy balance and CO_2 emissions are calculated in terms of specific per capita figures, although their values are dependent on the scale. For settlements with less than 1,000 inhabitants the concept with vacuum technology can be assumed to be too cost intensive in terms of investment. The concept is especially appropriate for application on a semi-centralized scale. If applied in big cities, the concept is supposed to be realized meters. Of course the energy balance and CO_2 emissions are not only dependent on settlement size but also on its infrastructure such as types of houses and sizes of properties.

The proposed anaerobic digestion system for full treatment of household wastewater consists of a CSTR for treatment of blackwater, kitchen refuse and other organic waste and a UASB reactor followed by constructed wetland for greywater treatment. Blackwater and kitchen refuse is collected and treated similarly as investigated in this work.

For greywater treatment, the common pre-treatment by settling is replaced by an anaerobic high-rate step which was proven to be more efficient in terms of COD removal (Elmitwalli et al.

2007a). The greywater has a relatively higher temperature (18 to 38 °C) than domestic wastewater as greywater is generated from hot sources such as shower, kitchen and laundry. Therefore, high-rate anaerobic system could run efficiently as on site treatment e.g. in the basement, even in low-temperature regions. In order to exploit all biogas potential in the household wastewater, the proposed concept includes the anaerobic treatment of greywater.

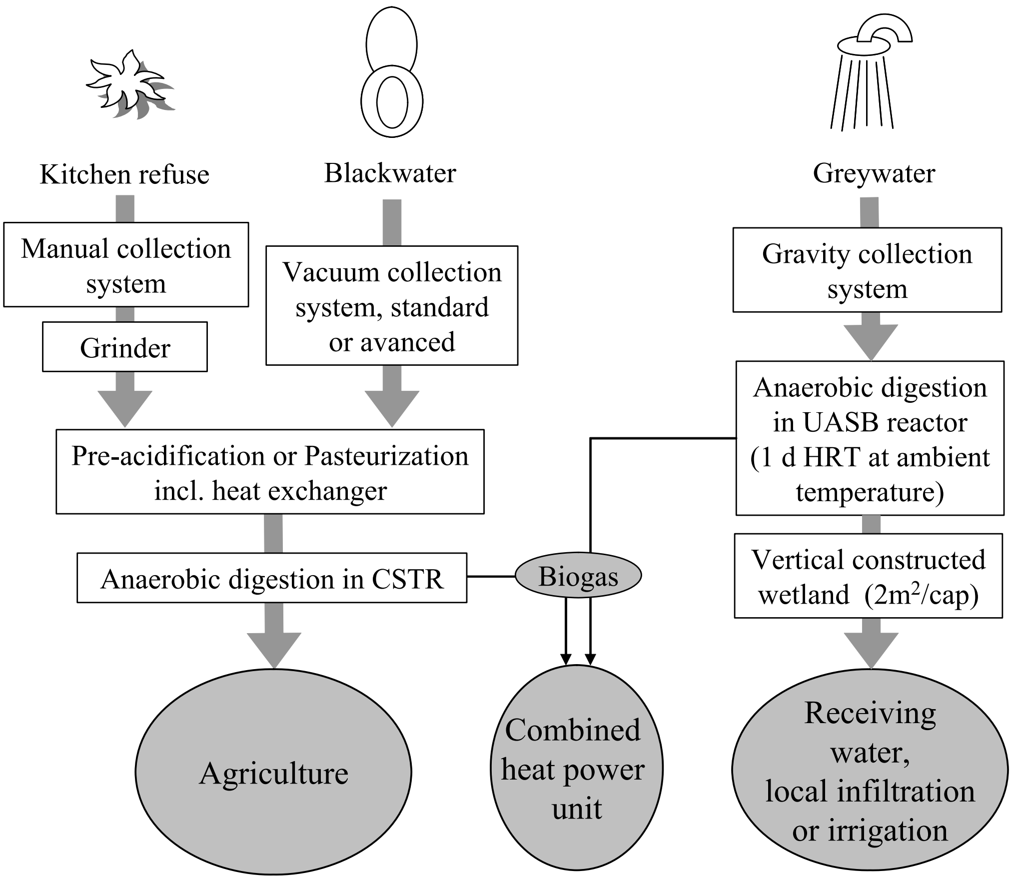

Figure 37: Scheme of the proposed AD system for household wastewater

In current research, greywater was successfully treated anaerobically. Elmitwalli et al. (2007) reported an anaerobic biodegradability of 79 % and a COD removal of 41 % at 24 °C for greywater in a UASB reactor, which represents a significantly higher COD removal than settling in a septic tank (14 %). The recommended HRT is 12 hours in the UASB reactor which results in relatively small treatment devices. Although the technology has not been applied on a large scale yet, it is expected that the up-scaling does not pose major problems as UASB is a well developed technology. One drawback of greywater AD is the relatively high amount of dissolved methane that escapes in the effluent. If dilution is high and/or temperature low, the part of dissolved methane rises to 21 % for greywater at 15 °C. This fact causes an undesirable impact on global warming and should be prevented. As diffusion is high, methane can be stripped easily.

The anaerobic sludge produced in the UASB reactor is stabilized due to long SRT and has to be discharged from time to time, e.g. twice a year. The sludge can be added to the blackwater digestion in the CSTR or directly to the effluent and recycled in agriculture. The effluent of the

UASB is treated in vertical flow wetlands for final treatment before discharge to a receiving water, infiltrated to the soil or applied in irrigation.

Based on the proposed anaerobic concept, two variants with different blackwater collection are investigated:

The first one called “V standard” is combined with the standard vacuum toilets using 0.7 to 1 l/flush. It is based on 100 % blackwater collection including toilet paper use, the data found in *Flintenbreite* and the average amount of organic waste collection. For greywater, the data of *Flintenbreite* greywater were taken which sums up to 18.3 kg COD/cap/year. The specific COD load fits well to literature values of greywater of 19 kg/cap/year by Vinnerås et al. (2006) for Sweden and 18 kg/cap/year by Dockhorn (2007) for Germany.

The second variant called “V advanced” considers vacuum toilets, here called advanced, which use only 0.25 l/flush in average. These toilets were already successfully applied in airplanes. The lower water consumption result in a decrease of specific water consumption of 61 %, see Table 30. A significantly lower dilution of blackwater would lead to lower reactor volume and less volume to be heated. A crucial issue would probably be a higher cleaning requirement of the vacuum piping system due to higher solid content.

Table 30: Design values for the variants of the proposed anaerobic concept

	V standard (using 0.7-1.0 l/flush)		V advanced (using 0.25 l/flush)	
	Input volume	Input COD	Input volume	Input COD
	l/cap/year	mg/l	l/cap/year	mg/l
Blackwater	3,042	8,000	1,172	20,767
Kitchen refuse	73	300,000	73	300,000
Input CSTR	**3,115**	**14,844**	**1,245**	**37,142**
Greywater	28,105	650	28,105	650

Efficiency would be increased by a variant that exceeds the maximum organic load by adding additional co-substrate. As shown in chapter 4, the co-substrate addition could highly increase the methane production and thus improve the energy balance. But compared to the conventional system, the co-substrate could be digested and produce biogas as well so that there is no net energy production.

For the selected variants, the following anaerobic conditions for blackwater and kitchen refuse are studied assuming that the anaerobic process is the same for all three temperatures and the anaerobic biodegradability is similar, although the microbiological growth rate at 30 °C is higher than at 37 °C resulting in a bigger reactor size:

(1) AD of blackwater and kitchen refuse at 30 °C

(2) Pre-acidification and AD of blackwater and kitchen refuse at 37 °C

(3) Pasteurizationof AD at 55 °C of blackwater and kitchen refuse followed by AD at 37 °C

(4) Pasteurization of AD at 75 °C of blackwater and kitchen refuse followed by AD at 37 °C

A conventional system based on activated sludge technology and composting of kitchen refuse is chosen for comparison purposes and is hitherto called the aerobic system. There is a wide range of options with different aerobic conditions to be selected but the focus here is on the reuse of water and nutrients on a semi-centralized level, so that an aerobic system targeting COD removal without nitrogen removal and sludge digestion is chosen for comparison. The effluent containing main parts of nitrogen in the form of ammonia and nitrate is supposed to be used for irrigation. It is therefore required that the effluent is disinfected, therefore UV radiation is selected here. The activated sludge is simultaneously stabilized, contains main part of phosphorus and should be applied on agricultural fields. Kitchen refuse is composted and reused in agriculture as well.

Due to this selection, the anaerobic and the aerobic systems are able to be assessed in the same framework.

5.2 Energy balance

The proposed anaerobic concept is going to be compared to a conventional aerobic system for domestic wastewater treatment. Calculations for energy and CO_2 are based on specific per capita figures for operation of the system. Energy consumption that is needed for the production and construction of the system is not taken into account.

5.2.1 System boundaries

Proposed anaerobic digestion system

The energy production and consumption of the collection and digestion of blackwater, kitchen refuse and greywater are calculated. The defined boundaries for the energy balance consider the following aspects:

- Energy consumption (electricity) of the vacuum technology
- Energy consumption (electricity and heat) of the biogas plant
- Energy production in terms of biogas from blackwater and kitchen refuse
- Energy saving due to reduced drinking water consumption by vacuum technology

Aerobic system

The aerobic treatment is based on activated sludge technology of blackwater and greywater and aerobic treatment of kitchen refuse. The defined boundaries for the energy balance consider the following aspects:

- Energy consumption (electricity) of the collection system by gravity
- Energy consumption (electricity) of the activated sludge treatment
- Energy consumption for composting of kitchen refuse

The following aspects are <u>not</u> taken into account:

Replacement of fertilizer: There is a significant energy credit by replacing fertilizer as the nutrients remain in the effluent during anaerobic treatment. But in case of aerobic treatment there is also the potential for reusing nitrogen if denitrification is prevented. For phosphorus, it is mainly contained in the sewage sludge. In the case of effluent, sludge and compost from kitchen refuse are applied in agriculture, and the nutrients are recycled. Thus, differences in the fertilizing efficiency between both systems seem not to be relevant.

Transportation: Transportation of the effluent is of course an important issue for energy balance and CO_2 emissions. Its impact is varying proportionally to the distance and can have a major impact in the case of high distances. In the proposed anaerobic system, the effluent is designed to be applied in agriculture as well as the effluent of the compared aerobic system. The effluent of both systems can be pumped or carried by trucks.

For the aerobic system, the sludge and the produced organic compost must be transported as well, but this is not within the scope of this study.

5.2.2 Methodology

For the energy balance, electricity and heat are separately calculated and balanced as they present different energy qualities.

Vacuum system

The electricity consumption for the standard vacuum system is calculated based on data by Oldenburg (2007) of 33 kWh_{el}/cap/year.

For the advanced vacuum toilet system, the electricity consumption is not known. Due to a higher requirement of air pressure, it is therefore assumed that the electricity consumption is 50 % higher related to the liquid flow. As the per capita flow is lower for the advanced vacuum system (Table 30), the electricity consumption for the advanced vacuum system is lower, calculated to be 19 kWh_{el}/cap/year.

Biogas plant (CSTR)

The heat requirement for pasteurization and mesophilic digestion are calculated according to the following equations.

$$P_H = c \cdot \Delta T \cdot \rho \cdot Q \qquad (25)$$

Where: P_H = required power for heating up from T_1 to reactor temperature T_R (kWh/d)

$c = 1.16$ kWh/m^3/K specific heat capacity of water, here assumed to be similar to water with ρ=1,000 kg/m^3

$\Delta T = T_R - T_1$, difference in temperature (K)

T_1 = Temperature of the input, here 15° C

Q = volume flow to be heated (m^3/d)

$$P_{HL} = k \cdot \Delta T \cdot A \qquad (26)$$

Where: P_{HL} = heat losses through the surface (kWh/d)

k = specific heat conductivity variable, dependent on the type and thickness of surface insulation (kW/m^2/K)

$\Delta T = T_R - T_2$, difference in temperature between reactor and outside (K)

A = surface (m^2)

In case of pre-pasteurization at 55 °C, the heat of the input to the CSTR covers the total heat demand of the mesophilic reactor. In case of pre-pasteurization at 75 °C, there is a surplus of heat in the input to the CSTR which can be recovered by heat exchange.

For pumps and mixing, the following figures are taken:

Vacuum pumps: 4.5 kWh$_{el}$/(m^3/h) for Vstandard calculated from Schneidmadl (1999)

2.0 kWh$_{el}$/(m^3/h) for Vadvanced assumed based on data in Table 30

Mixing: 4 W$_{el}$/m$^3_{reactor}$ (Leschber and Loll 1996)

Anaerobic digestion

The biogas produced by AD of blackwater and kitchen refuse is based on the results of chapter 3. It is assumed that blackwater and kitchen refuse are 100 % collected. AD takes place in a CSTR at mesophilic condition and 20 days HRT. In the case of 30 °C, the HRT is supposed to be accordingly higher to achieve the same anaerobic biodegradability.

Combined heat and power unit (CHP)

The CHP is a technical power unit equipped with a gas engine for gaining electricity and heat. The efficiency for gaining electricity is 30 % and heat 60 % (MURL 1999) which sums up to a total energy efficiency of 90 %. This high efficiency is the main advantage of the CHP and can only be entirely consumed if the heat is needed on site.

Drinking water consumption

Drinking water supply for Germany causes an electrical energy demand of 25 kWh/cap/year on average (Leist 2002).

Greywater treatment

For greywater treatment, pumping costs to the UASB and to the constructed wetlands are calculated. Operation of both units does not require additional energy as they run at ambient temperature.

The biogas produced by greywater digestion was calculated based on anaerobic biodegradability reported by Elmitwalli et al. (2007).

Aerobic treatment system

The energy consumption of wastewater treatment based on activated sludge technology with technical aeration depends largely on its design and purpose. Without tertiary treatment, the energy demand ranges from 20 to 54 kWh_{el}/cap/year (MURL 1999), depending mainly on the HRT in the aerated zone and the sludge treatment. Both systems are assumed to provide nutrients to agriculture. An aerobic system targeting COD removal without nitrogen removal and sludge digestion was selected with an energy consumption of 30 kWh_{el}/cap/year (MURL 1999) for comparison. Disinfection of the effluent with the purpose of reuse is done by UV radiation which needs 0.1 kWh_{el}/m^3 (TECE 2008), resulting in 4.7 kWh/cap/year.

For aerobic treatment of kitchen refuse, the commonly applied composting technology is selected. The electrical consumption for pre-treatment and composting is 49 kWh/Mg_{input} (Schmelz 2000) in Germany which amounts to 4 kWh_{el}/cap/year. Transportation is neglected here.

5.2.3 Results

The distribution of the energy carrier heat and electricity are calculated for the described variants "V standard" and "V advanced". For each variant the heat requirement differs according to the anaerobic conditions selected for blackwater treatment, see Figure 38 and Figure 39. For greywater treatment, the results show that it can be operated in a self-sufficient way at ambient temperature with a small surplus of heat of 15 kWh_{therm}/cap/year which can be used for the treatment of blackwater and kitchen refuse. The overall energy balance in terms of heat and electricity are presented in Figure 40.

V standard

The major electricity consumer is the vacuum system. The other consumers which are kitchen refuse grinder, CSTR and greywater pumps are of minor importance compared to the vacuum system. On the benefit side, the reduced drinking water brings a relatively small benefit in terms of electricity, the major part derives from the electricity production of biogas in the CHP unit. The total is 43 kWh_{el}/cap/year, from which 18 % is produced by greywater. V standard has a slightly negative electricity balance (-5 kWh_{el}/cap/year).

Concerning the heat balance, the biogas is the only heat producer via the CHP unit. The heat is needed for pasteurization and AD of blackwater and kitchen refuse. The different temperature levels required lead to huge differences in the heat demand. The heat demand is 60, 88, 145 and 163 kWh_{therm}/cap/year for AD at 30 °C, AD at 37 °C, pasteurization at 55 °C+AD at 37 °C and pasteurization at 75 °C+AD at 37 °C, respectively. These differences in heat requirement result in a range of overall balances. For the case of AD at 30 °C, the heat balance is positive: 25 kWh_{therm}/cap/year. For pasteurization at 55 °C + mesophilic AD and pasteurization at 75 °C + mesophilic AD, the balance is -59 and -77 kWh_{therm}/cap/year, respectively. The difference is not very high as in the case of 75 °C pasteurization, a heat exchanger was calculated.

The key issue is anyway the required temperature especially if the quantity to be heated is high. It would be a good option to use external surplus heat for mesophilic AD and pasteurization.

V advanced

The use of advanced vacuum toilets leads to a lower drinking water consumption and to a lower blackwater production that must be treated. The electricity benefit by reduced drinking water consumption is 14 % higher than Vstandard. The electrical consumption by the vacuum system is reduced by about 42 % compared to Vstandard. The other consumers are of minor importance compared to the vacuum system. In summation, there is a consequent positive electricity balance (15 kWh_{el}/cap/year).

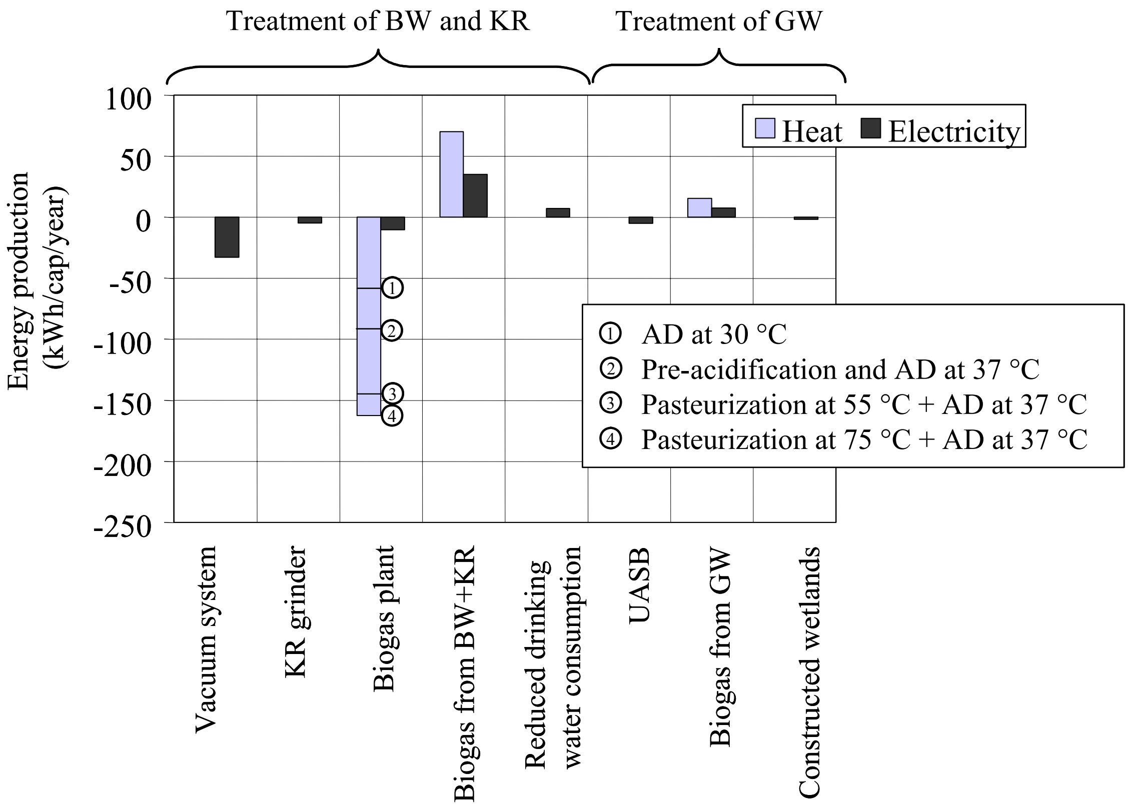

Figure 38: Consumption of heat and electricity for variant Vstandard (toilet using 0.7 – 1.0 l/flush)

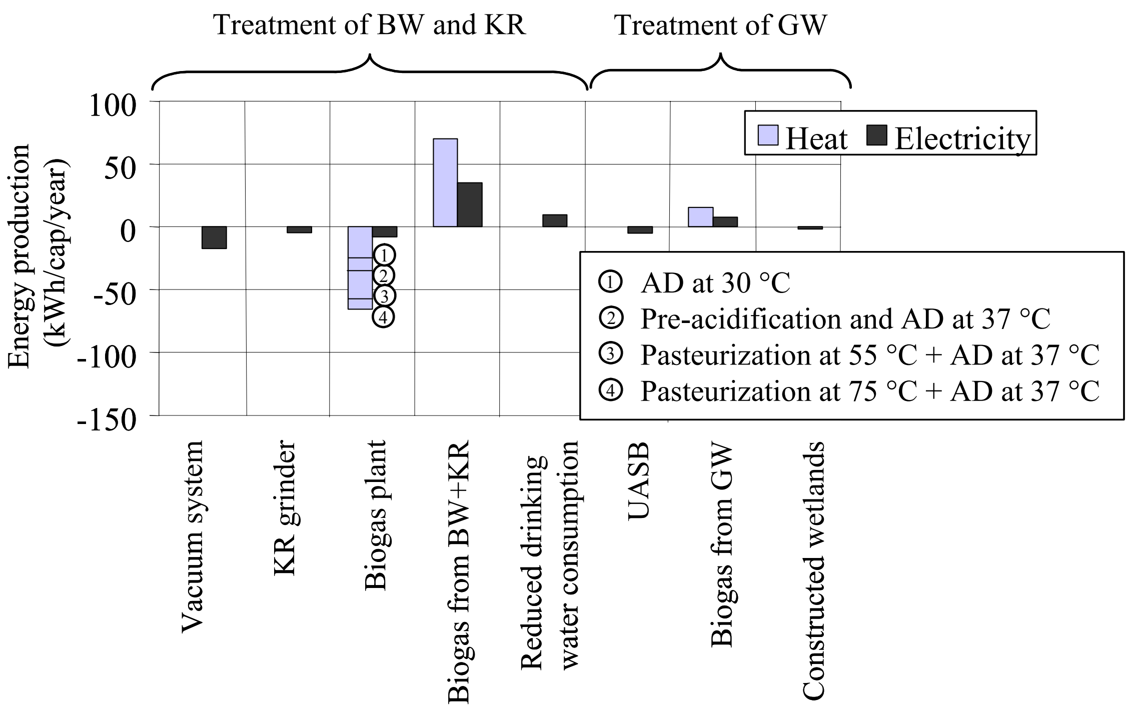

Figure 39: Consumption of heat and electricity for Vadvanced (toilet using 0.25 l/flush)

The use of advanced vacuum toilets has a positive impact on the heat balance. Due to the significant lower volume, the heat balance is positive for all variants. In contrary to electricity, a heat credit is only useful if the heat is required by an external consumer. In the cold season, indoor heating can be supplied with heat, in summer the heat might be released into the atmosphere without usage.

An option could be to use the additional heat either within a heat exchanger to increase the input temperature as for Vstandard proposed. Another option could be the use for volume reduction of the digested output e.g. by evaporation.

Comparing the variant based on the standard vacuum system to the aerobic system, the anaerobic system needs more energy especially in terms of heat. The aerobic system does not need any heat but electricity. If pasteurization is required, the heat demand for the anaerobic system is predominantly high. In terms of electricity it is almost self-sufficient.

Applying the advanced vacuum toilet, the proposed anaerobic system has a much better energy balance than the system based on the standard vacuum toilet and the aerobic system.

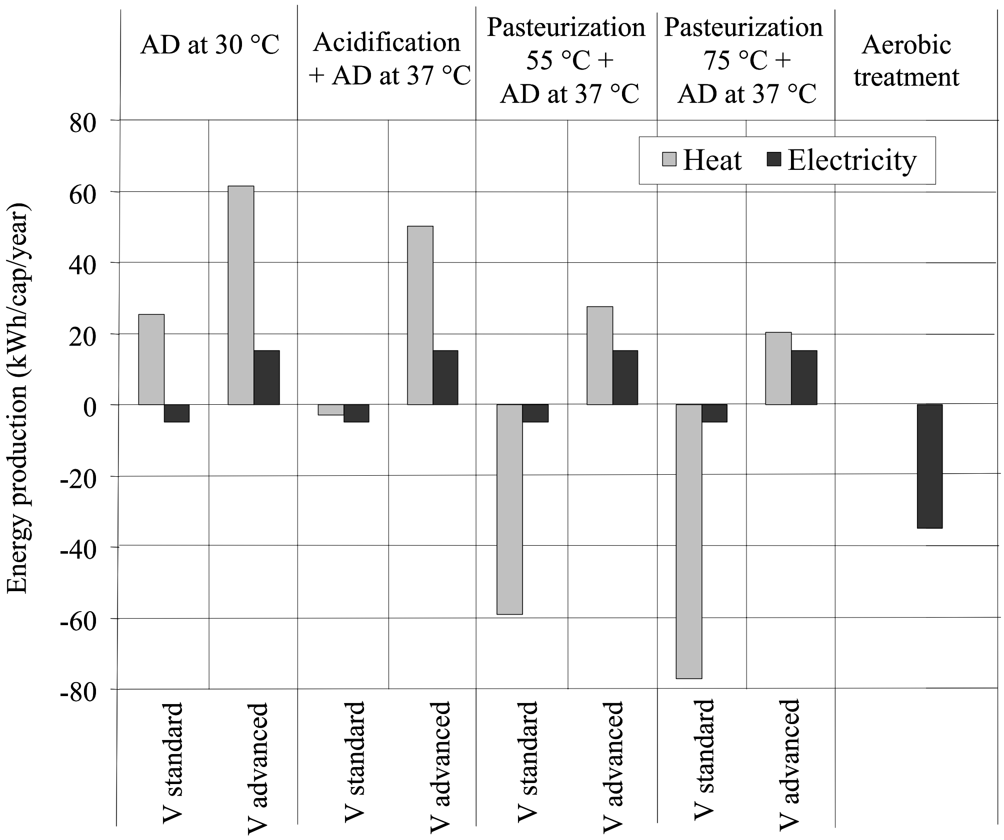

Figure 40: Energy balance of the different AD variants and the aerobic treatment

5.3 CO_2 emissions

CO_2 is the major greenhouse gas emitted by human activities. Methane and nitrous oxide (N_2O) are also important greenhouse gases with effective contribution to global warming for the next 100 years of 21 and 310 times, respectively. The release of methane is a potential drawback of AD, e.g. in dissolved form via the effluent. From the activated sludge of the aerobic system there might be an escape of methane as well, e.g. during storage. In any case, there are practical measures to be taken to avoid any unintended methane release. Nitrous oxide is a potential drawback for aerobic treatment but has not been quantified in relevant concentrations on large scale plants (Zeng et al. 2003). As these gases are hardly quantifiable they are neglected here.

The boundary conditions are analogous to the energy balance.

The bacterial activity also causes different CO_2 emissions for aerobic and anaerobic treatment; it is however not relevant in usual greenhouse gas calculations. Emissions of CO_2 generated from biomass sources should not be counted due to the assumption that over time re-growth of biomass equals consumption (Greenfield and Batstone 2005). Emissions of process generated CO_2 from human wastewater and waste are therefore not treated as net emissions. The CO_2 emissions are calculated based on the consumption of fossil energy sources.

To calculate the CO_2 emissions from heat and electricity the assumption based on the German energy average production was taken (Loga et al. 2001):

- 0.232 kg CO_2/kWh_{therm} for the supply of heat
- 0.689 kg CO_2/kWh_{el} for the supply of electricity

The results are shown in Figure 41. Positive numbers quantify an emission and negative numbers a capture of CO_2.

As for the energy balance, the heating has the major impact on the emissions. In the case of AD at 30 °C, CO_2 is captured 2 and 25 kg CO_2/cap/year for Vstandard and Vadvanced, respectively. For the other variants of Vstandard, the emissions are to 4, 17 and 21 kg CO_2/cap/year for AD at 37 °C, pasteurization at 55 °C and at 75 °C, respectively. Vadvanced does in all cases store significant amounts of CO_2. In comparison, the aerobic concept releases 24 kg CO_2/cap/year which is always worse than all anaerobic variants.

Thus the results are similar to the energy balance. The supply of heat does not produce as many emissions as electricity; that is why the relatively high heat demand of AD systems has not such a negative impact on the greenhouse gas emission as in the energy balance. The advanced vacuum system is highly favorable in terms of CO_2 emissions, but even the standard vacuum system even with pasteurization causes less CO_2 emissions than the conventional aerobic system.

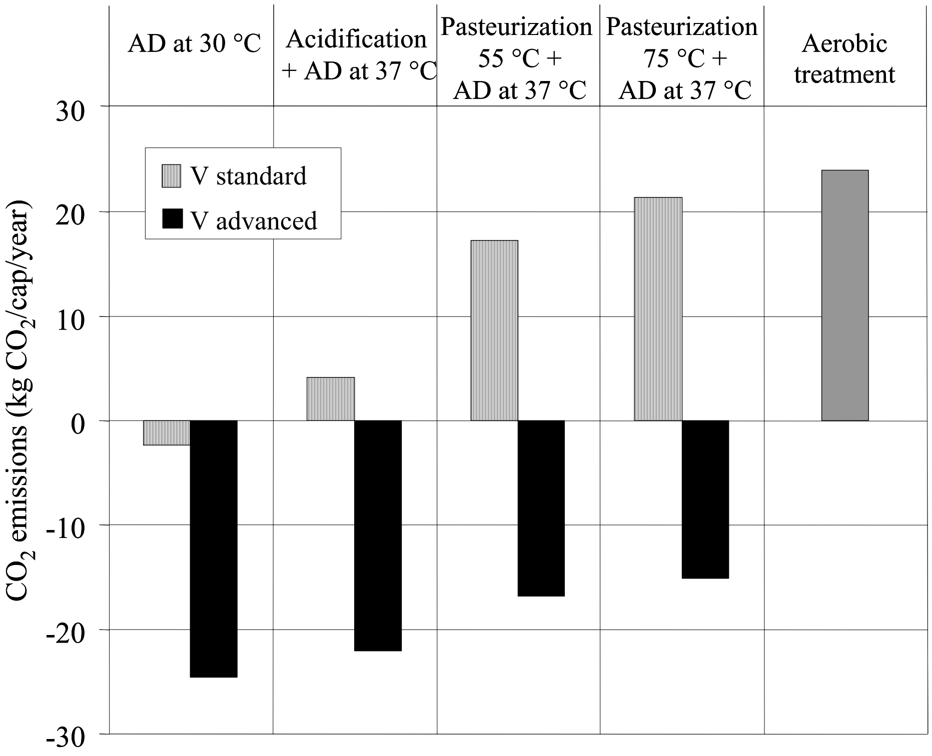

Figure 41: CO_2 emissions of the different AD variants and the aerobic treatment

5.4 Chapter conclusions

- For the proposed anaerobic system, the vacuum system is the major energy consumer in terms of electricity, the heating for pasteurization followed by AD in terms of heat demand.
- Although the electricity demand of the vacuum system is relatively high, the standard vacuum system with toilets using 0.7 to 1.0 l/flush has an almost self-sufficient system for electricity of total -5 kWh_{el}/cap/year due to the CHP unit. The advanced vacuum system using 0.25 l/flush has a better energy balance which is proven by an electricity credit of 15 kWh_{el}/cap/year.
- Concerning the heat balance, the advanced vacuum toilets show significant advantages to the standard vacuum system. Vstandard needs external heat for all treatment variants apart from AD than 30 °C.
- Compared to the aerobic system, the proposed anaerobic system has always a significantly better energy balance in terms of electricity. The crucial issue is the heat demand. Two aspects are relevant: (1) If pasteurization is required, 55 °C for 24 hours is recommended. More reasonable would be to pasteurize only the kitchen refuse which would highly decrease the volume to be heated. (2) Another measure to decrease the heat demand is to increase the

temperature of the influent e.g. by means of high-quality insulation of the pipes and by heat exchanger.

- The greywater treatment can be operated in a self-sufficient way at ambient temperature. The additional heat produced by the CHP unit can be used for combined blackwater and kitchen refuse treatment.
- In terms of CO_2 emissions, the pasteurization step in Vstandard causes a worse effect but all anaerobic variants are better than the aerobic system. Vadvanced has always a positive impact in terms of CO_2 emissions.

CHAPTER 6 CONCLUDING SUMMARY AND OUTLOOK

Anaerobic digestion (AD) was investigated as an option for treatment of blackwater and kitchen refuse within sustainable sanitation concepts. Because of the high dilution by flush water and high ammonia concentrations, AD of blackwater has been considered as inefficient. As part of resources management sanitation, blackwater collected in a vacuum system has gained attention. Based on the experimental and simulation results, it could be proven that AD of combined blackwater and kitchen refuse has major advantages in terms of energy and water efficiency compared to conventional aerobic treatment. The digested output is rich in nutrients and can be applied as organic fertilizer in agriculture.

Experimental work

Blackwater from vacuum toilets is concentrated wastewater (average COD = 8,060 mg/l) with high concentrations of particulate COD (average part COD = 6,010 mg/l) and macro- and micronutrients. There are high standard deviations for COD especially for particulate COD. The addition of separately collected kitchen refuse leads to doubling of the organic load and improves to COD/N/P ratio for AD. In the pilot project *Flintenbreite*, it was found that 60 % of the total blackwater is collected in the housing estate. Based on the data of 100 inhabitants, the blackwater loads were calculated to be 8.3 l/cap/d, 24.5 kg COD/cap/year (including toilet paper), 4.6 kg N/cap/year total nitrogen and 0.53 kg P/cap/year total phosphorus, in the case of 100 % blackwater collection. The anaerobic biodegradability was determined as total COD removal of 72 %. Although the solid concentration in blackwater highly fluctuates, the batch tests led to similar results for high-strength (> 10,000 mg/l COD) and low-strength (< 7,000 mg/l COD) blackwater.

The bench scale experiments with raw blackwater in a CSTR were carried out at HRT of 20 days and 37 °C. More than 60 % of the input COD was converted to biogas which represents 87 % of the maximum biodegradable COD. At these conditions, the produced biogas contained 75 % methane and the biogas production rate was 14 l CH_4/cap/d (at STP) in the case of 100 % blackwater collection. The process at 20 days HRT was stable and uninhibited. With low-strength blackwater, the biogas production rate was higher than with high-strength blackwater, so that the HRT could be decreased at the same efficiency.

When combined blackwater and kitchen refuse was fed to the reactor, a stable and uninhibited process was achieved at HRT of 20 and 15 days. Between 71 and 75 % of the input COD was converted to biogas which represents 90 % of the maximum biodegradable COD. At these conditions, the produced biogas contained 65 % methane and the biogas production rate was 32 l CH_4/cap/d (at STP) in the case of average kitchen refuse and 100 % blackwater collection. Even at HRT of 10 days, a steady state process with a decreased COD removal by 30 to 33 % and decreased methane production by 19 to 21 % was achieved, with increased stable VFA concentrations in the output.

The ammonia concentrations in blackwater are relatively high (NH_4-N = 1,111 mg/l) and can be even higher if the composition is not consistently distributed between faeces and urine or if the blackwater is more concentrated by vacuum toilets that require less flush water. In the case where the toilet needs 0.25 l/flush, the ammonia concentration increases to 2,920 mg/l NH_4-N. Therefore, bench scale experiments with increasing ammonia concentrations by adding stepwise either urea or ammonia salts were carried out to study the limits in terms of ammonia inhibition. The results clearly showed that AD of blackwater is uninhibited at NH_4-N concentration up to 3,500 mg/l, if the pH is kept stable around 7.5. If the pH rises, the increasing free ammonia starting from concentration of 300 mg/l NH_3-N inhibits the process. In that case, the digester must be pH-controlled by adding acid again or the ammonia concentration of the feedstock must be reduced so that the bacteria can recover.

Blackwater contains high concentration of pathogens ($9.1*10^7$ CFU/100 ml E.coli). The reduction of microbiological indicators is required for the agricultural use of the digested output. The reduction of E.coli during mesophilic AD at 20 days HRT is 2.4 log which is similar to activated sludge treatment. Another 2 log reduction is achieved by post-storage at ambient temperature for more than 40 days of the digested output. As pre-treatment steps, pasteurization and acidification were studied. Pasteurization followed by AD reaches a log removal of 4.8 for E.coli. Pre-acidification for 6 days at 37 °C can be an adequate pre-treatment step to pasteurization, in the case where a defined co-substrate rich in carbohydrates is available and the process is well controlled in terms of pH and VFA. Still, the process of acidification and the frame conditions for efficient hygienization needs further research.

The standard of US EPA, class B, (Anonymous 1993) for unrestricted use of sewage sludge in agriculture of 10^6 CFU/g TS faecal coliforms were safely met by the three chosen treatment systems (AD and post-storage, pasteurization and AD and pre-acidification and AD). But the standard of 1,000 CFU/g TS E.coli/faecal coliforms, set by WHO (2006) for the use of excreta in large systems (Vol. 4) and US EPA, class A, (Anonymous 1993) for unrestricted use could not be safely met by none of the systems. If, in contrast, the WHO guidelines (2006) for restricted use of wastewater in agriculture (Vol. 2) are taken into consideration, no threshold value must be met (see Table 5). Instead, the achieved log removal can be combined with additional health measures and the digested output may be applied in agriculture.

Concerning its composition, the digested output can be used in agriculture as an organic liquid fertilizer in terms of macro- and micronutrients (predominantly rich in ammonia). The heavy metal concentrations are very low. Restricted application is required because of relatively high salt concentration and remaining pathogens.

Mathematical Modeling and Simulation

The ADM1 model by IWA was chosen to model and simulate the AD process of blackwater and kitchen refuse. With the results of the bench scale experiments, the model, so called BWADM, could be successfully calibrated. The input characteristics are crucial in terms of distribution of dissolved/particulate COD and degradable/inert COD. Variations in the ratios for carbohydrates, proteins and lipids, in contrast, do not show a significant impact on the biogas production rate. These ratios can be assumed whereas the first should be determined carefully by analysis.

The applied k_{dis} (including disintegration and hydrolysis) for blackwater and kitchen refuse are 2.5 and 0.3 1/d, respectively. They are higher than the values found in the literature. The hydrolytic bacteria are inhibited at high pH which is implemented by an enzyme based approach in the model with the same function as for the other processes apart from acetate uptake. The results clearly show that hydrolysis is the limiting process step for high-strength blackwater and blackwater mixed with kitchen refuse. Low-strength blackwater is limited by acetoclastic methanogenesis.

The experimental results of increasing ammonia could be well reflected by the calibrated BWADM. The 50 % inhibitory constant for free ammonia inhibition (200 mg/l) is higher than suggested by ADM1 and other authors which might be caused by a long adaptation time of more than six months. The enzyme based approach for pH inhibition with two different functions, first one for the acetoclastic bacteria (optimal pH at 7) and the second one for the other bacteria grouped together (wider range and optimal pH at 6), respectively, was successfully adapted in the range of pH from 7.3 to 8.4 which is the typical range of blackwater AD. Haldane kinetics for acetate uptake fit well under these conditions. For all other biochemical processes, Michaelis-Menten kinetics as enzyme based approach was successfully applied.

The calibrated model BWADM was up-scaled and its limits in terms of hydraulic and organic loads were simulated. Based on the *Flintenbreite* conditions of 400 inhabitants, the daily blackwater load of 5 l/cap/d (60 % blackwater collection) and kitchen refuse load of 0.2 kg/cap/d, the simulation of the biogas plant *Flintenbreite* shows results far under the reactors' limits in terms of hydraulic and organic load.

Based on the reference data for kitchen refuse collection, the organic load by kitchen refuse can be increased by up to 10 times (5 kg COD/m^3/d) at HRT > 15 days without operational problems. This potential can be exploited by adding co-substrate. The methane production increases by about 6 times, if the additional co-substrate has a similar characteristic as kitchen refuse.

Energy Balance and CO_2 Emissions of the Proposed AD System

A system for AD in a CSTR for blackwater and kitchen refuse and AD of greywater in a UASB was proposed and compared to conventional treatment based on activated sludge and composting of kitchen refuse in terms of energy balance and CO_2 emissions. In contrast to the current vacuum toilet called Vstandard, a variant with advanced vacuum toilets leading to a more concentrated blackwater flow (using only 0.25 l/flush), called Vadvanced, was additionally studied.

For the proposed anaerobic system, the vacuum system is the major energy consumer in terms of electricity, the heating for pasteurization/AD in terms of heat demand. Although the electricity demand is high, the standard vacuum system with toilets using 0.7–1.0 l/flush has an almost self-sufficient system for electricity of total -5 kWh_{el}/cap/year due to the CHP unit. The advanced vacuum system using 0.25 l/flush has a better energy balance which is proven by an electricity credit of 15 kWh_{el}/cap/year. Concerning the heat balance, the advanced vacuum toilets show significant advantages to the standard vacuum system. Vstandard needs external heat for all variants higher than 30 °C.

Compared to the aerobic system, the proposed anaerobic system has always a better energy balance in terms of electricity, the crucial issue is the heat demand. If pasteurization is a requirement, 55 °C for 24 hours is recommended. If pasteurization was required only for treatment of kitchen refuse, it would be energy efficient to pasteurize only this small amount separately. In case of pasteurization at 75 °C, a heat exchanger is proposed to minimize the negative impact of energy consumption and CO_2 emissions.

The greywater treatment can be operated in a self-sufficient way at ambient temperature. The additional heat produced by the CHP unit can be used for the treatment of blackwater and kitchen refuse.

For the CO_2 emissions, the pasteurization step in Vstandard causes a worse effect but all anaerobic variants are better than the aerobic system. Vadvanced has always a significant positive impact in terms of CO_2 emissions.

Outlook

In order to further increase the sustainability of blackwater AD, the following research themes are proposed:

- The vacuum toilet should be improved in terms of flush water volume. There is a need of advanced vacuum toilets using less flush water which are applicable on household level with the same comfort as standard vacuum toilets. The implementation of a dual flush system with small quantity for urine and bigger quantity for faeces flushing is an essential asset.
- With the mathematical model BWADM, a tool for simulation of mesophilic AD of blackwater and kitchen refuse is now available. To further improve the energy efficiency of the anaerobic treatment, there is a need to investigate AD of blackwater and kitchen refuse at ambient temperature between 10 and 30 °C. The model should be calibrated on ambient temperature and able to simulate temperature fluctuations at various seasons.
- The transportation of the digested effluent to agriculture is a crucial aspect for sustainability. In addition to the flush water volume reduction mentioned above, it is recommended to investigate technical options of decreasing the volume of the digested effluent e.g. by evaporation.
- As hydrogen becomes more and more important as energy carrier, blackwater and kitchen refuse needs to be investigated as potential substrate for hydrogen production. Experiments with a hydrogenogenic reactor followed by a methanogenic reactor have already been successfully carried out with energy crops.

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