ANALYSIS OF AN ANAEROBIC BAFFLED REACTOR TREATING COMPLEX PARTICULATE WASTEWATER IN AN ABR- MEMBRANE BIOREACTOR UNIT

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December 2010

DECLARATION

I**, J.K Bwapwa**, declare that

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As the candidate's supervisors, we have approved this thesis for submission

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December 2010

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ABSTRACT

Providing water and proper sanitation to poor communities by 2015 is one of the United Nations targets for this millennium. In South Africa many communities aspire to waterborne sanitation. However, there is a technology gap for decentralized and sustainable waterborne sanitation systems capable of treating domestic wastewater (Foxon et al., 2006). Although domestic wastewater is more commonly treated using aerobic processes, anaerobic processes may be more appropriate for decentralized applications since they do not require aeration. Research is currently being undertaken to understand the behavior of a combined ABR-MBR unit for treating domestic wastewater.

In this study, the anaerobic baffled reactor (ABR) was investigated by analyzing physico-chemical and biochemical data from experiments on a laboratory-scale ABR. This anaerobic reactor was treating complex particulate wastewater made up of sludge from the ventilated improved pit latrine toilets (known as VIP sludge). The main focus of this study was to establish the relationship between the increasing organic loading rates and the effluent characteristics (such as chemical oxygen demand: COD and extrapolymeric substances: EPS).

The present work was structured in two parts; in the first part the reactor was operated at constant hydraulic retention time (HRT) without controlling feed characteristics. In the second part, the ABR was operated with step increases in organic loading rates. It was logistically not possible to provide a feed of real domestic wastewater to the laboratory-scale equipment. Consequently, a pit latrine sludge diluted with tap water was used to feed the ABR. This feed was found to have different biodegradability characteristics compared to domestic wastewater. However, the results still give insight into the performance of the ABR and into the treatability of VIP sludge.

COD removal ranged from 52 to 80 % depending on the inlet COD. Some COD removal was due to solids retention in compartments, while it was estimated that only 28% of COD removal was due to biological degradation. Soluble extrapolymeric substances (proteins and carbohydrates) which are usually a by -product of anaerobic degradation were higher in the feed than in the effluent despite the increasing organic loading rates. However, more than 50 % of soluble extrapolymeric substances from the influent remained in the effluent and were found (in a parallel project) to influence membrane fouling in the membrane section of the experimental set-up (ABR-MBR unit).

Parameters such as pH, conductivity, alkalinity, total and volatile solids were also investigated in this study. The pH decreased slightly from the inlet to the outlet during all runs even though the loading rates were increased. Conductivity increased significantly from influent to effluent with the increasing organic loading rates. Large amounts of total solids were retained in the reactor during the treatment process. Low alkalinity production was recorded during the operation of the reactor. In most cases, the data recorded in this study showed a low biological activity taking place while the reactor was working at room temperatures.

Overall, up to 80% of removal efficiencies in terms of total COD and solids were recorded with increasing organic loading rates at constant hydraulic retention time. While these results do not allow the prediction of ABR-MBR performance during the treatment of real wastewater, it was concluded that:

- Most solids retention occurred in the feed tank.
- Most COD removal occurred as a result of solids retention and digestion.
- Loading characteristics did not strongly influence effluent EPS, pH or alkalinity, but did influence COD and conductivity.
- The relatively low biodegradability of the feedstock indicates that anaerobic digestion is not the most appropriate treatment for VIP sludge.

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LIST OF ABBREVIATIONS

ABR	Anaerobic baffled reactor
ASBR	Anaerobic sequencing batch reactor
BOD	Biological oxygen demand
BMP	Biochemical methane potential
BW	Blackwater
COD	Chemical oxygen demand
CSTR	Continuous stirred tank reactor
DEWATS	Decentralized water treatment systems
EGSB	Expanded granular sludge bed reactors
EPS	Extracellular polymeric substances
FTE	Feed tank effluent
HRT	Hydraulic retention time
OLR	Organic loading rate
SRT	Solid retention time
SMP	Soluble microbial products
TOC	Total organic content
TS	Total solids
TSS	Total suspended solids
UASB	Upflow anaerobic sludge blanket (reactor)
UD	Urine diversion (toilets)
VFA	Volatile fatty acids
VIP	Ventilated improved pit latrine toilets

VS	Volatile solids
VSS	Volatile suspended solids
WRC	Water research commission
WWTP	Wastewater treatment plant

CHAPTER 1: INTRODUCTION

One of the United Nations Millennium Development Goals (MDGs) relates to the provision of water and sanitation to previously unserved communities by 2015. South Africa like other countries in the world is committed to address this problem of water and sanitation to poor communities. However, this is hampered by a number of challenges, chief being the delivery of water and sanitation to areas located outside the sewage system. This includes the densely-populated areas that lack formal housing arrangements. Also, it is difficult to address all South African community needs through a centralized sewerage system approach. Hence, municipalities have adopted a decentralized approach to sanitation implementing on-site dry sanitation systems. These systems require no water and are less expensive and simple to implement than conventional sewage systems. However, there are many communities in need of waterborne sanitation. This situation is an indication that there is a technology gap for decentralized waterborne sanitation systems which can be sustainable for these areas, thereby prompting a study like the one reported in this thesis on the anaerobic baffled reactor treating blackwater made up of VIP sludge from low-income communities.

1.1 WATER AND SANITATION IN SOUTH AFRICA

- **Overview**

Water and sanitation are key issues when it comes to poverty eradication and economic development for any nation. This is because water and sanitation are very crucial factors in the process of social progress and economic development, as they deal with the wellbeing of the people. Hoffman et al., (2001) have reported that South Africa is among the driest countries in the world with 500 mm of rainfall per year. This represents about 42% less the world average of 860 mm of rainfall per year. As a semi-arid country, strong focus should be given to this field of water and sanitation to avoid challenges due to water scarcity and lack of basic sanitation in the near future.

- **Outcomes of water and sanitation program**

Overall, the program of delivering proper sanitation and water to every citizen has been very successful: 88% of households had access to potable water and 73% of households had access to basic sanitation in 2008 (WRC, 2009). In spite of these plausible statistics, more needs to be

done in this area if the MDGs are to be achieved by 2015 and poverty eradication is to become a reality. This is because the water and sanitation sector in South Africa is still facing delivery challenges despite considerable progress achieved since 1994. For instance, it has been estimated that approximately 16000 people die every year from diarrheal diseases directly linked to lack of clean water and proper sanitation (WRC, 2009). Furthermore, the effect of increasing urbanization in cities has become one of the major concerns for public health and has resulted in the development of informal settlements where sanitation and potable water facilities are very poor. The provision of sanitation in such areas (informal settlements) is a real challenge: conventional sanitation options such as pit and waterborne systems are not appropriate because of their costs, the space and connectivity issues. The eradication of these backlogs requires a concerted effort from all levels of government (local, provincial and national) as well as the private sector. This will speed up significantly the service delivery and meet the targets set by the government for the provision of water and sanitation.

1.2 CHALLENGES ASSOCIATED WITH THE DELIVERY OF WATER AND SANITATION IN INFORMAL SETTLEMENTS.

- **Informal settlements and water delivery**

The provision of sanitation in South Africa is mostly in the form of waterborne sewage connected to a centralized wastewater treatment or by on-site treatment system such as septic tanks, Ventilated Improved Pit latrine toilets (VIP toilets), Urine Diversion toilets (UD toilets), and rudimentary pits. Informal settlements around the world in general and in South Africa particularly, are well known to be densely populated. In these areas, the infrastructure for water and sanitation is either overloaded or cannot satisfy the needs of the population. Consequently, the implementation of waterborne sewage system is very complicated because the municipalities do not have control of housing arrangement and cannot stop the fast growing movement of properties in the informal settlements. Furthermore, the construction and maintenance costs of waterborne systems are expensive and they require huge amounts of water for flushing. A possible way to address sanitation needs in informal settlements is through the use of on-site sanitation systems such as chemical toilets or pit latrines. These systems have very low costs and are easier to build than waterborne sewage systems. However, on-site sanitation systems have shown low treatment efficiencies in many cases and they cannot be used on rocky ground where

groundwater level is high or in areas that are periodically flooded (Winblad and Simpson, 2004). In many instances the failure of these systems has led to groundwater pollution (Strenstrom, 1996). Consequently, an appropriate technology to address sanitation challenges in low- income communities was needed.

The anaerobic baffled reactor (ABR) was identified by the Pollution Research Group from the University of KwaZulu-Natal (School of Chemical Engineering) and a project was funded by the Water Research Commission and the eThekwini municipality to investigate on the ABR. The design advantages of the ABR in treating soluble industrial wastewater have been well documented (Polprasert et al., 1992; Barber and Stuckey, 1999; Bell, 2000). In addition, the system has the ability to reduce biomass washout and to separate the spatial arrangement of anaerobic microbial consortia, this confers greater protection from variations in parameters such as pH and temperature (Barber and Stuckey, 1999; Bell, 2000). The versatility and the ability of the ABR in removing organic material have been demonstrated in previous studies on various wastewater sources including domestic wastewater (Barber and Stuckey, 1999).

In 2005, the Pollution Research Group assessed the performance of a pilot ABR (treatment capacity of 3000 l) treating domestic wastewater. The aim of this project was to establish if the ABR was an appropriate sanitation technology for low- income communities. The outcomes of this study showed higher treatment rates (about 80% COD removal at 40-44h HRT), improved recovery times from shock loads and flexibility compared to septic tanks. Furthermore, it has been proposed that there is a potential to reuse the effluent generated by the ABR for horticulture as limited nutrient reduction occurs by anaerobic digestion (Foxon et al., 2005). However, the pathogen load was sufficiently high to make the effluent a potential hazard to public health and the environment. Consequently, membrane filtration combined with the ABR system was recommended as a potential post-treatment option for pathogen removal.

1.3 BOUNDARY OF THE STUDY

This study is limited to the performance of the ABR section of the ABR-MBR unit. But, it forms part of the ABR- MBR unit bigger project carried out in a parallel study (Pillay et al., 2007) using microfiltration and ultrafiltration membranes as post-treatment options.

Both studies (ABR and ABR-MBR) are very important, they are investigating on the capacity of the ABR technology as one of the sanitation options for the low-income communities.

1.4 HYPOTHESES

ABR can be used for treating domestic wastewater with different organic loading rates. In most low-income communities, the sludge in the VIP toilets usually comprises of faecal material mixed with domestic wastewater. It is known that domestic wastewater is biodegradable.

Therefore, two hypotheses were suggested:

- The biodegradability of VIP sludge (sludge from the VIP toilets) obtained would be similar to that obtained if domestic wastewater only was treated.
- By increasing organic loading rates, effluent characteristics such as chemical oxygen demand (COD) and extrapolymeric substances (EPS) should be affected. Effluent COD is expected to increase for any increase in loading rates. EPS are expected to be produced from the feed to the effluent for any increase in loading rates. This is due to biological stress that occurs during the digestion process.

To test these hypotheses, the analysis of biochemical and physico-chemical data from the operation of a laboratory-scale ABR treating VIP sludge was undertaken in this study.

1.5 OBJECTIVES OF THE STUDY

The main objective chosen to investigate the reactor performance is:

- To understand how the organic loading rates affect the effluent characteristics: to characterize the type of effluent produced by the ABR with increasing loading rates for a reactor treating wastewater from VIP sludge.

The more specific objectives are described as follows:

- Performing a COD mass balance for the system.
- Identifying possible membrane foulants such as extrapolymeric substances (EPS).
- Obtaining operational data from different inlets and outlets for selected parameters.
- Evaluating the variation of some physico-chemical parameters in compartments.

CHAPTER 2: LITERATURE REVIEW

2.1 BLACK WATER CHARACTERISTICS: Overview

The literature on VIP sludge and blackwater characteristics is very limited, this constitutes a gap to be filled in the future. The present study is concerned with the anaerobic treatment of complex particulate wastewater comprised of VIP sludge, urine, domestic wastewater from domestic activities such as washing and many other materials thrown into the toilets located in peri-urban communities. Normally, typical blackwater should not consist of other material except faeces and urine, but due to poor habits and understanding within such communities, it has become common to find these other materials in the blackwater. There is a difference between blackwater from VIP sludge and blackwater from waterborne sewage: blackwater from waterborne sewage contains only fresh faeces while in blackwater from VIP sludge faeces are not fresh.

Blackwater in comparison to greywater contains most of the nutrients, around half of the domestic COD load, and a large portion of pathogens (Otterpohl, 2002; Vinnerås et al., 2006). Because of its specific composition, blackwater requires separate collection, adequate treatment and final recycling. The anaerobic digestion can play a key role as a known adequate treatment technology for concentrated wastewaters (Claudia, 2008). 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 (Claudia, 2008). 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 per flush. Practically, there are several main drivers for anaerobic digestion of blackwater within resource management sanitation:

- Safe sanitation: the hazardous compounds in excreta are not spread in the water cycle.
- 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 because of its high content in nutrients such as nitrogen and phosphorous.

Table 2.1 presents different types of blackwater (BW) including parameters such as total COD, VS, and TS.

Table 2.1: some indications on blackwater (BW) characteristics (Claudia, 2008)

Parameter	**Unit**	**BW from vacuum toilets**	**Synthetic BW using primary sludge and toilet paper**	**BW from vacuum toilets in Sneek**	**Synthetic BW using faeces, urine and water**
Total COD	mg/l	9500-12300	950	19000	
Dissolved COD	mg/l	1400-2800	120	5000	
VFA-COD	mg/l	500-1900		1300	
Particulate COD	mg/l	7000-9600	820	14000	
TS	mg/l		670		10370
VS	mg/l		490		7570
TOC	mg/l				
NH_4N	mg/l	600-1000	4.5	1400	692
Total N	mg/l		32		
Total P	mg/l	90-140	17	280	12
Part.COD/Total COD	-	76%	86%	74%	
COD/N/P	-	95/10/1	56/2/1	68/5/1	

BW: blackwater

2.2 ANAEROBIC DIGESTION

The anaerobic digestion is a microbial degradation of an organic compound in the absence of oxygen. There is a conversion of organic matter to CO_2 and CH_4 gases next to a sequence of biochemical reactions during an anaerobic process (Bailey and Ollis, 1986). As a result, a breakdown of organics is occurring during the digestion, this is made possible by anaerobic microorganisms. The anaerobic digestion of an organic matter follows stages which are organized by different categories of microorganisms. Most biodegradable organic matter is converted to gases while only a small amount (about 10%) is converted to new cell mass through microbial growth (Speece, 1996). Methane produced by anaerobic digestion can be used to run a treatment plant; this is an economic advantage of the anaerobic over the aerobic digestion. Tables 2.2 and 2.3 present the advantages and disadvantages of an anaerobic digestion in terms of costs, start up, sludge generation and buffering capacity.

Table 2.2: Merits of anaerobic digestion process (Lettinga et al., 1997; Lettinga, 1995; Seghezzo et al., 1998)

- **The operating costs** for an anaerobic treatment plant are relatively very low compared to an aerobic treatment plant.
- **Low energy consumption** and production of biogas for further applications such as the production of electricity; also the system does not require external energy for its operation.
- **The flexibility** of an anaerobic system allows the technology to be applied on either a small or a large scale.
- **Low sludge generation** compared to aerobic systems due to a lower yield co-efficient.
- The excess sludge is well **stabilized**.
- **Low nutrient** and **chemical requirement**: this is due to the small biomass production during the course of an anaerobic process; consequently, the nutrients requirement is proportionally less.

Table 2.3: Disadvantages of anaerobic digestion (Seghezzo et al., 1998)

- **Long start-up**: the start up period is longer than in aerobic systems because of the slow growth rate.
- **High buffer requirements** for the pH control: the required pH for anaerobic digestion should be included in the range of 6.5 to 8. Also, chemical addition, mostly in industrial wastewater, may be indispensable for the control of pH with inadequate buffering capacity.
- **High sensitivity of microorganisms**: methanogens are sensitive to pH and temperature, they are assumed to have less resistance toward toxic compounds.
- **Low pathogen and nutrients removal**: effluents from anaerobic digestion are characterized by low removal of pathogens and nutrients. A post-treatment process such as membrane filtration must be required to meet the discharge guidelines aiming to protect the environment.

2.2.1 Anaerobic microbiology

Reactions involved in an anaerobic degradation of complex organic waste can be categorized in four main steps described as follows: hydrolysis, acidogenesis, acetogenesis and methanogenesis (Seghezzo et al., 1998). The mechanism of degradation of organic compounds and different intermediate products obtained during an anaerobic process is shown in Figure 2.1.

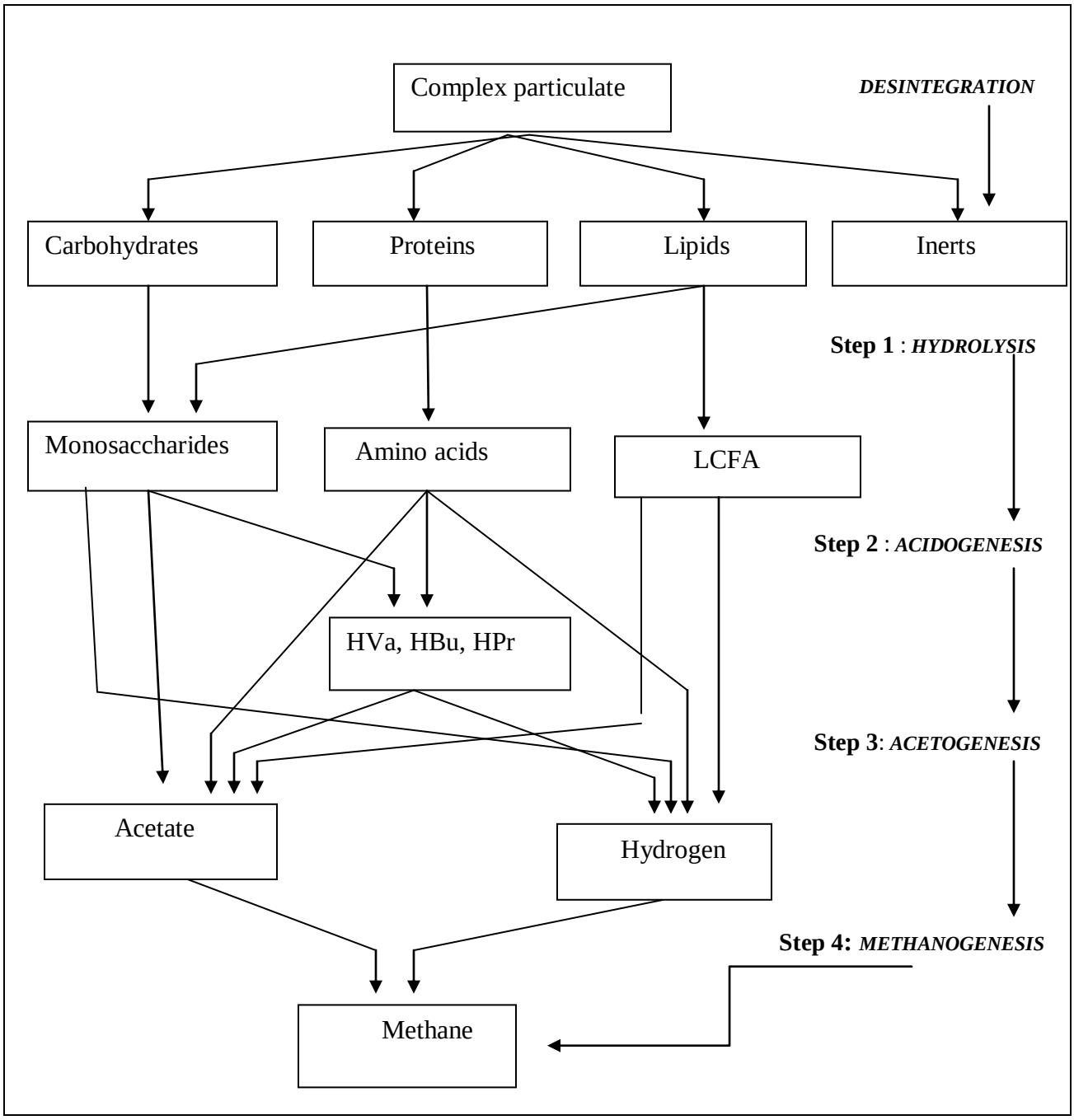


Figure 2.1: Schematic representation of an anaerobic process [(from Batstone et al., (2002)]

With LCFA: long chain fatty acids, HBu: Butyrate, HVa: Valerate, and HPr: propionate

2.2.1.1Hydrolysis

During this first step, complex macromolecules are converted into more simple compounds including organic monomers such as sugars, fatty acids and amino acids (Pohland, 1992; Van Haandel and Lettinga, 1994). This is a combination of extracellular, enzymatic biological and non-biological processes (Batstone et al., 2002). Enzymes facilitate the digestion process by converting complex polymers into soluble monomers. Consequently, substrates, which would otherwise be too large to pass through cell membranes, become available to other metabolic groups (Anderson et al., 2003; Bitton, 1994; Van Haandel and Lettinga, 1994). For this reason, this process is commonly referred to as solubilization or liquidification (Van Haandel and Lettinga, 1994).

2.2.1.2 Acidogenesis

During this second step, simple organic compounds and monomers are fermented and converted into volatile fatty acids, alcohols, ketones, acetate, lactic acid, butyric acid, succinic acid and inorganic compounds such as carbon dioxide, hydrogen, ammonia, and hydrogen sulphide (Van Haandel and Lettinga, 1994; Anderson, 2003). Organic compounds produced during this stage release hydrogen ions into the liquid phase resulting in a drop of pH (Anderson, 2003). Although obligate anaerobes are responsible for most acid fermentation, some organic matter is metabolized by facultative bacteria via an oxidative pathway (Van Haandel and Lettinga, 1994). Facultative bacteria play a significant role in anaerobic digestion by utilizing dissolved oxygen which would otherwise be toxic to obligate anaerobes (Van Haandel and Lettinga, 1994).

2. 2.1.3 Acetogenesis

Volatile fatty acids produced from the second step are converted into final products such as carbon dioxide, hydrogen and acetate for the production of methane (Van Haandel and Lettinga, 1994; McInerney, 1981). Van Haandel and Lettinga (1994) reported that the production of acetic acid during the acetogenic phase may be followed by carbon dioxide or hydrogen formation depending on the average oxidation state of the original organic matter. By-products generated during this step are the only substrates that are used by methanogens.

Two distinct metabolic groups of acetogens are described in anaerobic systems: obligate hydrogen-producing acetogens (OHPA) and homoacetogens (Anderson et al., 2003). To date, only a few OHPA species have been isolated and identified in a culture of microorganisms. The OHPA utilize the major fatty acid intermediates (propionic acid, butyric acid) produced by acidogenesis (Anderson et al., 2003; Bitton, 1994). They are also capable of metabolizing aromatic compounds as well as higher fatty acids (valeric acid, isovaleric acid, stearic acid, palmitic acid and myristic acid) produced in lipid hydrolysis via β-oxidation (Anderson et al., 2003). Under standard conditions (25° C, pH 7, 1 atmosphere of pressure, 1 M), fatty acids oxidation is energetically unfavorable due to a high free energy requirement ($\Delta G > 0$) (Lubberding, 1998).

The homoacetogens produce acetate from H_2 and CO_2, as a result, they maintain a low hydrogen partial pressure required by the OHPA. They are found in lower numbers than methanogens in anaerobic systems, and are thought to play a relatively minor role in the conversion process (Anderson et al., 2003).

2. 2.1.4 Methanogenesis

Methane is produced during the course of the fourth step from the reduction of CO_2 by H_2 using acetotrophic and hydrogenotrophic bacteria respectively. Also, it is produced from acetate (Van Haandel and Lettinga, 1994; Anderson, 2003). The acetotrophic methanogens produce up to 70% of methane by degrading acetic acid. The remaining 30% of methane is produced by the hydrogenotrophic methanogens by reducing carbon dioxide, formate and methanol. This is achieved by using the available hydrogen produced during the fermentative stage (Anderson, 2003). Although only a relatively small fraction of methane is produced via the hydrogenotrophic pathway, it is critical to the efficiency of the process, as it removes H_2 produced by hydrolytic and acidogenic bacteria (Gunnerson and Stuckey, 1986). Acetotrophic methanogenesis and hydrogenotrophic methanogenesis are described by the following reactions:

- **Acetotrophic methanogenesis**

$$CH_3COOH \rightarrow CO_2 + CH_4 \qquad (2\text{-}1)$$

- **Hydrogenotrophic methanogenesis**

$$4\ H_2 + CO_2 \rightarrow CH_4 + 2\ H_2O \qquad (2\text{-}2)$$

Methanogens are critical to the conversion process. In methanogenesis COD is finally converted to a form that leaves the liquid phase (CH_4 and CO_2) (Anderson et al., 2003). Methanogenesis is often recognized as the rate-limiting step due to slow growth of methanogens in anaerobic digestion. Furthermore, methanogens have a strictly defined pH range in which they can operate (6.5 to 8). Methanogenic bacteria are distinct from true bacteria (*Eubacteria*) and therefore, they belong to a different domain called *Archaebacteria* (also called *Archaea*) (Lubberding, 1998). Methanogens are able to utilize only a restricted number of substrates with acetate, CO_2 and H_2 being the most important (Anderson et al., 2003).

2.2.2 Parameters affecting the rate of an anaerobic digestion

There are various parameters that can influence the rate of an anaerobic digestion; these include pH, temperature, essential trace nutrients, toxic compounds, retention time and mixing. These parameters are key players on the stability of an anaerobic system and they can affect its performance.

2.2.2.1 Temperature

It has been widely documented that anaerobic digestion like any other biological process depends strongly on temperature (Van Haandel and Lettinga, 1994; Speece, 1996). Within an anaerobic process, mesophillic microbes exhibit maximum growth rates between 35°C and 40°C. Therefore, the optimum conversion rate of an anaerobic digestion takes place between 30°C and 40°C (Henze and Harremoes, 1997). Temperature between 40 and 50°C may lead to the failure of the entire process. For temperature above 50°C, thermophillic microbes can operate normally. There is little or no digestion taking place at temperatures above 70°C (Batstone et al., 2002; Henze et al., 1997). The effect of temperature on the anaerobic treatment can be physical, chemical as well as biological (Lettinga et al., 2001). For example, solubility of gases increases as temperature decreases below 20 ^{o}C leading to higher concentrations of gases such as methane, hydrogen and hydrogen sulphide into the effluent at low temperatures. Higher viscosity requires

more energy for mixing and the sludge bed may not be sufficiently mixed when there is low biogas production. The biological activity also decreases leading to poorer hydrolysis of solids and biogas conversion (Lettinga et al., 2001).

2.2.2.2 pH

A pH range between 6.5 and 8 is appropriate for methanogenic microbial activity (Batstone et al., 2002); below or above these values, methanogenic microbial activity decreases. If methanogenic microbes are affected by other factors such as temperature, their ability to convert hydrogen and acetic acid to methane will be reduced. Consequently, a subsequent drastic reduction of pH value and accumulation of volatile fatty acids will occur.

2.2.2.3 Nutrients

According to Henze et al., (1997), nitrogen, phosphorus and sulphur are the essential nutrients for an anaerobic process (as with any other biological processes). The need for nutrients is relatively small for anaerobic digestion because the process is characterized by low growth yields. Also, nutrient supplementation is not necessary in domestic wastewater because most nutrients are already present in this type of wastewater (Rittman and McCarthy, 2001). This applies also to industrial wastewater from food processing which can have an adequate supply of nutrients (Rittman and McCarthy, 2001; Anderson et al., 2003).

2.2.2.4 Toxicants or inhibitory compounds

- **Oxygen**

Oxygen is toxic to most anaerobic microorganisms. Its presence in an anaerobic reactor will result in a significant decrease in digestion rate. However, it is possible that facultative anaerobes metabolize the dissolved oxygen before toxic effects are noticeable (Zinder, 1994).

- **Volatile fatty acids (VFA)**

High concentrations of VFA are generally observed during the start-up or when there is organic overloading of the digester and they are usually associated with toxicity and inhibitory effects. Although it is generally understood that VFA inhibition is due to their accumulation and

subsequent pH reduction, some VFA are themselves toxic to anaerobic microbes (Anderson et al., 2003).

- **Free ammonia**

Free ammonia concentrations above 100 mg/l can cause inhibition, although the ionic form NH^+_4 will only cause inhibition at much higher concentrations (above 3000 mg/l) (Rittmann and McCarthy, 2001).

2.2.2.5 Retention time

A longer retention time will provide a greater degree of sludge stabilization and allows intimate contact between the biomass and the liquid flow during the treatment process (Keay, 1981).

2.2.2.6 Mixing

In a conventional anaerobic digester, mixing has been found to increase CH_4 yields and to increase digester stability (Forday and Greenfield, 1983). The effect of mixing is to bring a homogeneous environment and effective use of the entire digester volume. This is achieved by minimizing hydraulic dead zones and preventing build up of large pockets of unfavorable environmental conditions (low pH and high VFA). Consequently, the concentration of toxic agents throughout the reactor is diluted. Mixing promotes also the removal of excess CO_2 which is inhibitory at partial pressures greater than 0.2 atmosphere (Pulles et al., 2001).

2.2.2.7 Particle deposition

Wastewaters often have considerable amounts of colloidal and particulate matter in addition to soluble substances. Generally, physical properties of sludge aggregates such as size, density, porosity, as well as settling velocity, have significant influence on the efficacy of solid/liquid separation. Raskin et., al (1994) used column experiments with raw sewage under anaerobic conditions in an upflow anaerobic filter. From this study, it was concluded that the settling of solids in the reactor produced methane gas and acceptable effluent total BOD. Also, 68% COD removal and 63% COD stabilization were achieved at 23 $^{\circ}$C. As soon as sedimentation was prevented, COD removal and stabilization were only 45 and 55 % respectively. The difference in removals was attributable mainly to suspended particles. Solids retention time of particulate

organic matter can be significantly increased over the hydraulic retention time (HRT) if the sedimentation or filtration retains the particles. When particulate organic matter is retained, its concentration builds up and the removal kinetics expressed per unit of reactor volume, increase as well (Raskin et., al 1994).

2.2.3 Alkalinity in anaerobic digestion

Alkalinity represents the ability for a digester to neutralize volatile fatty acids formed during digestion (Ross et al., 1992). This parameter is useful for monitoring the reactor stability. Alkalinity is expressed as an amount of $CaCO_3$ per unit volume. The amount of buffer produced should be enough to balance the acids produced during digestion and maintain the pH between 6.5 and 8 for a proper methanogenic activity, i.e. the biological conversion of acids to methane (Ross et al., 1992).

2.2.4 Assessing and monitoring the performance of anaerobic processes

Anaerobic systems require a daily monitoring to prevent failure and to evaluate the efficiency of the process. The biodegradability and toxicity of a particular waste stream must be assessed before loading to an anaerobic system. Physical and chemical analyses are used to monitor the performance of an anaerobic system. According to Ross et al., (1992), the monitoring of the reactor pH, alkalinity and volatile fatty acids concentrations is very important. This allows predicting and preventing failure of the reactor due to the build–up of volatile fatty acids in the system. The COD is also used to assess the performance of an anaerobic system; a COD removal between 50 and 70% is expected in a properly functioning anaerobic system (Ross et al., 1992). The following section outlines some important parameters mostly used for the assessment of anaerobic reactors:

2.2.4.1 Physico-chemical parameters

Physico-chemical parameters have been traditionally used to monitor the performance of various wastewater treatment systems. Methods used to evaluate these parameters can allow fast and cheap quantification of process stability and performance.

- **Organic Content**

Domestic wastewater is primarily composed of organic compounds, such as carbohydrates, amino acids, peptides, proteins, fatty acids and their esters (Bitton, 1994). As it is not easy to measure all these compounds individually, three major tests are used to determine organic matter in wastewater: chemical oxygen demand (COD), biochemical oxygen demand (BOD), and total organic carbon (TOC). The first two (COD and BOD) are based on the oxidation of organic matter and the third (TOC) is based on the determination of the organic carbon concentration (Van Haandel and Lettinga, 1994). COD is often chosen over the other two tests because it is more accurate and can be carried out in a relatively short time (Van Haandel and Lettinga, 1994). Also, COD is a conserved species and it is possible to complete balances on COD for an anaerobic reactor. In this study, COD is used to determine the organic content of the complex particulate wastewater. The COD removal efficiency is considered as one of the key indicators of the reactor performance.

- **pH, Alkalinity, and Volatile Fatty Acids (VFA**)

The values of pH, bicarbonate alkalinity (acid neutralizing capacity) and VFA concentrations have considerable influence on the stability of an anaerobic system. These parameters serve as warning signals of digester failure, as changes in these parameters occur faster than a decrease in gas production (Malina, 1992). The methods used to determine these parameters are relatively simple and cheap.

- **Solids**

The anaerobic degradation pathway, especially hydrolysis, can become rate-limited in reactors treating wastewaters with high a concentration of solids, especially if solids are poorly degradable (Lettinga and Hulshoff, 1992). Also, high concentration of solids in wastewater can lead to the formation of a scum layer (floating layer consisting of suspended fats and lipids) in anaerobic reactors. These fats and lipids accumulate in the reactor and produce excess sludge. They slow down or inhibit the formation of flocculent or granular sludge by attachment, and contribute to ‘washout’ (Lettinga and Hulshoff, 1992). Therefore, the determination of solids concentration is very important. Normally the following tests are completed: total suspended solids (TSS), total dissolved solids (TDS), total solids (TS) (which is TDS + TSS), volatile

suspended solids (VSS) and volatile solids (VS). Solids removal efficiency is also known as one of the key performance indicators for an anaerobic reactor.

- **Gas production**

The relative proportion of gases produced during anaerobic digestion provides useful insight into the efficiency of the degradation process. Methane and CO_2 are produced in the largest quantities and are therefore the easiest to determine. Decrease of methane production or sudden increase of CO_2 can be a sign of process instability (Malina, 1992).

- **Nutrients**

Although anaerobic digestion often does not have any significant effects on the final concentration of nutrients, their measurement is nevertheless necessary for two reasons: firstly, it allows nutrient-deficiency or nutrient overload conditions to be predicted or established. Secondly, it helps to determine the requirement of a post treatment option (Van Haandel and Lettinga, 1994). Concentrations of nitrogen, phosphorus and to a lesser extent potassium are routinely measured. There are numerous methods used for the determination of nitrogen in the form of mineral ion (NH_4^+) or oxidized nitrogenous compounds (NO_2^- and NO_3^-), including spectrophotometric and titration methods, and in some cases, electrode methods (APHA, 1998).

- **Extrapolymeric substances (EPS)**

EPS are complex polymers made up essentially of proteins, carbohydrates (polysaccharides), and nuclei acids. They are excreted by bacteria and their role is to maintain a linkage between cells. In general, EPS are divided in two categories: bound EPS and soluble EPS or soluble microbial products (SMP) (Ye et al., 2005). Bound EPS are usually derived from polymers in the form of capsule, sheaths, and loosely bound polymers joined to organic material. SMP are composed of soluble, biodegradable macromolecules and they are a product of bound EPS dissolution (Nielsen et al., 1997). Operating conditions defined by parameters such as organic loading rates (OLR) and solids retention time (SRT) have an impact on EPS (or SMP) production (Barker et al., 1999). EPS (or SMP) production increases as the feed COD concentration increases (Barker et al., 1999), this means that by increasing OLR, the concentration of EPS (or SMP) in the effluent will be higher than the one in the feed.

This is explained by the fact that at high loading rates the digester is overloaded and it is not able to remove all organic substances from the liquid flow. However, at low loading rates the digester is underfed; the sludge decomposes and releases organic matter into the liquid flow (Baskir and Hansford, 1980; Pribyl et al., 1997). This is validated by Baker et al., (1999) who reported that with OLR in the range of 0.3 to 1.2 g COD/ gMLSS.d, there is a possibility to obtain minimum values of EPS. Therefore, a substantial increase or decrease, above or below this optimum range will result in EPS production because of biological stress taking place in the reactor.

2.2.4.2 Pathogen indicator parameters

Pathogens are not regularly measured in anaerobic reactors, except from those that treat various sludges to be re-used in land application. Indicator organisms such as coliforms are used to detect pathogens in wastewater.

2.3 THE ANAEROBIC BAFFLED REACTOR (ABR)

There are several types of anaerobic digesters such as the continuous stirred tank reactor (CSTR), the anaerobic contact process digester, the conventional mixed anaerobic digester, the anaerobic filters (AF), the upflow anaerobic sludge bed (UASB), the expanded granular sludge bed reactor (EGSB) and the anaerobic sequencing batch reactor (ASBR). The choice of the reactor depends on the type of wastewater to be treated. In this study, the focus is on the anaerobic baffled reactor treating blackwater from VIP sludge. Figure 2.2 is a schematic representation of the ABR with its hanging and standing baffles, the sample and gas ports, the inlet which is the feeding point and the outlet representing the effluent collection point.

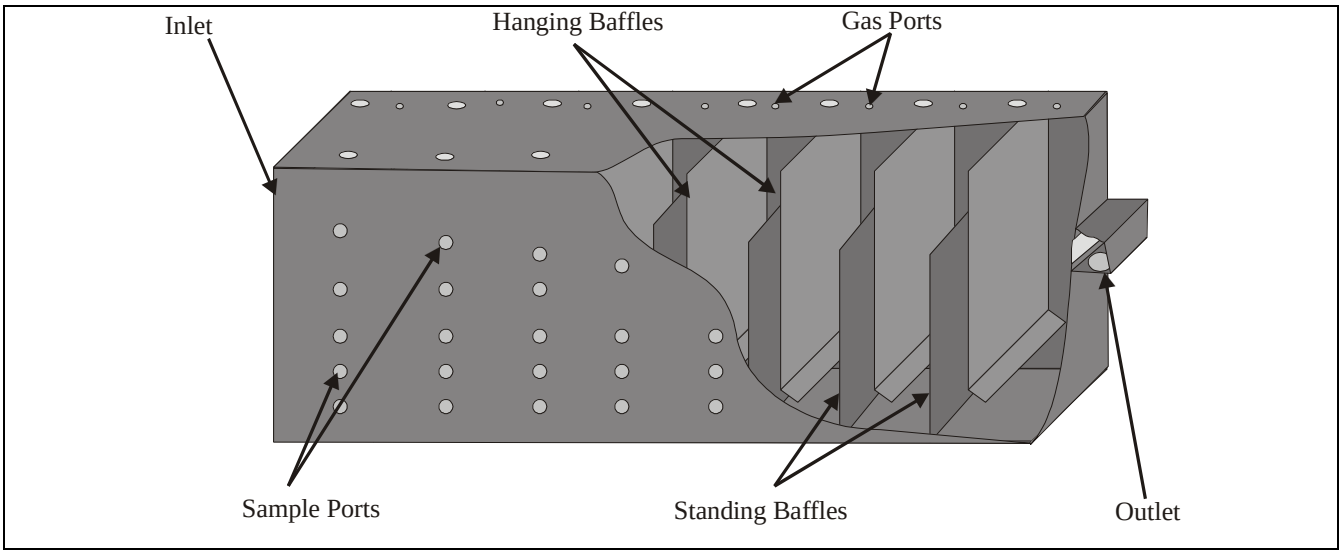


Figure 2.2: Diagram of an 8-compartment ABR (Dama et al., 2001)

2.3.1 Description of the Anaerobic Baffled Reactor (ABR)

The ABR is a reactor made up of a succession of baffles forcing raw wastewater to flow under and over (or through) vertical baffles as it passes from the inlet to the outlet (McCarthy and Bachmann, 1992). There is a gentle rise and settling of bacteria in the reactor due to the characteristics of flow and production of gas. However, the movement of bacteria within the reactor is low (Boopathy and Sievers, 1991). It allows the wastewater to be in contact with a high quantity of active biomass as it flows through the reactor (Grobicki and Stuckey, 1991).

2.3.2 Significant advantages of the ABR

Jianlong et al., (2004) reported that the most important advantage of this reactor is its ability to separate acidogenesis and methanogenesis phases longitudinally down the reactor. This is explained by the fact that different conditions develop at different points during digestion relating to pH, temperature and substrate concentration. Different zones result in the development of different microbial populations that are adapted to the prevailing conditions, specifically, acidogenesis in front and methanogenesis at the end. Therefore, bacteria grow under most favorable conditions defined by the pH and the temperature. Furthermore, the ABR can be cost-effective at large-scale operation (Orozco, 1997). The reactor can be operated without electricity as wastewater could be channeled to the reactor by gravity (Foxon et al., 2004).

2.3.3 Effect of phase separation in ABR

The separation of acidogenesis and methanogenesis phases is very important for the operation of the ABR as mentioned in section 2.3.2. However, the hydrolysis of particulate organics to soluble substrates is generally a rate-limiting step during the degradation process. Therefore, the rate of degradation for particles in the reactor is generally slower compared to soluble organics (Eastman and Ferguson, 1981). The effect of phase separation allows the reactor to behave as a two-phase system without control problems and high costs usually associated with two-phase systems (Weiland and Rozzi, 1991). Phase separation in an ABR is thought to encourage the hydrolysis of particulate matter at a low pH without affecting the methanogenesis phase (Langenhoff et al., 2000).

2.3.4 Design of an ABR

The ABR can be compared to a modified septic tank divided in compartments by vertical hanging and standing baffles. The design with baffles presents an advantage by limiting biomass washout as solids cannot bypass from the first to last compartment (Polprasert et al., 1992; Barber and Stuckey, 1999). Also, it has the potential to allow high treatment rates compared to the traditional septic tank at similar hydraulic loadings (Foxon et al., 2006). In this reactor, the interactive association of microorganisms confers great protection against toxic substances (Barber and Stuckey, 1999). Furthermore, it may improve the hydrolysis of particulate organics in the front of the reactor due to a low pH. In addition, previous studies have indicated that the baffled design of an ABR results in a residence time distribution that can be approximated by a number N of completely mixed tanks in series, where N is the number of real compartments of the ABR (Foxon, 2009). The design objective is to maximize the contact between the biomass and the wastewater made up of dissolved and suspended substances. This is achieved both by maximizing the hydraulic retention time (which is the treatment time) and solids retention within the constraints of space and capital cost (Foxon et al., 2006). Foxon et al., (2006) have identified the following key parameters in the design of an efficient ABR:

- *Mean hydraulic retention time*: it affects the contact time for the treatment of wastewater.
- *Number of compartments*: it affects the internal velocity of the liquid within the reactor, therefore, the solids retention capacity of each compartment can be affected if the number of compartments is high. Also, it affects the capital cost of the reactor.
- *Design upflow velocity*: it affects the sludge retention characteristics such as settling velocity.
- *Upflow-to-downflow area ratio*: it affects the fluid dynamics in the sludge bed.
- *Compartment length-to-width ratio*: the length –to- width ratio between 1:3 and 1:4 can be used depending on the space available at the installation site.
- *Hanging baffle clearance*: this must be adequately large to prevent the occurrence of blockages by the sludge bed.
- *Reserve Capacity*: The total volume of the reactor should be double the working volume for a 36 h retention time design.

2.3.5 Effect of baffles in the ABR

Wanasen (2003) undertook a comparison study between a conventional septic tank and modified septic tanks with 1 and 2 internal baffles to make a 2 and 3-compartment ABR. These reactors were fed with a mix of university wastewater and matured septic tank sludge. It was recorded that at hydraulic retention time of 48 h, the conventional septic tank had approximately the same removal efficiencies in terms of COD, BOD, TS and TSS as the baffled septic tanks. However, at hydraulic retention time of 24 h, the removal efficiency was reduced by up to two- fold compared to the baffled reactors. The removal efficiencies for the three-baffled septic tank were 10 to 15% higher than observed in the conventional septic tank. The outcomes of this study showed that stabilization of biodegradable material was achieved at hydraulic retention time of 48 h. However, the extent of stabilization decreased more rapidly in the conventional septic tank with reduced hydraulic retention time than in the baffled systems (Wanasen, 2003). Boopathy (1998) treated swine manure in four laboratory-scale ABRs which respectively had two, three, four and five compartments. At OLR between 6 and 12 kg COD/ m^3.d, it was recorded that COD removal, solids removal and methane production rates all increased with an increasing number of

compartments. Therefore, it can be deduced that the number of baffles can influence the removal efficiencies of an ABR.

2.3.6 Application of anaerobic digestion theory to an ABR

The ABR is a high rate digester used to treat municipal sludge and high industrial wastewater under anaerobic conditions. All four steps shown in Figure 2.1 are involved in the treatment of wastewater within the reactor. There is a complex anaerobic microbial conversion of organic substrate to methane during the treatment process within the reactor. The treatment process is achieved by means of seeded and active micoorganisms through phase separation as mentionned in section 2.3.2. These retained microorganisms are operating anaerobically at the bottom of each compartment and they are constantly in contact with the liquid flow. Substrates are hydrolyzed to simple organics which are fermented to volatile fatty acids by the acidogens. Volatile fatty acids are converted to acetate and H_2 gas by acetogens. Finally, these intermediates (acetate and hydrogen) are converted to methane by methanogens (see Figure 2.1). Methane is collected from each compartment, channeled by a piping system, stored and used for energy production. An effluent with reduced COD and solid matter can be collected at the end of the compartment train.

2.3.7 ABR Start-up

The start-up of an anaerobic reactor can be successfully achieved by developing a suitable microbial culture (the biomass) for the waste stream (Stuckey and Barber, 1998). Once the biomass is established, either as flocs or granular particles, the reactor operation will be fairly stable. Several factors are important in the start-up of high rate reactors, they include wastewater composition, biomass activity, growth rates, saturation constants, yield, adaptation, ability to excrete polysaccharides, size and properties of granules, reactor configuration, geometry, size and ability to immobilize biomass, loading rate, HRT, mixing characteristics, pH, temperature, and the availability of nutrients and trace elements. During start-up, fluctuations in parameters such as pH, temperature, HRT, and recycle ratio are avoided and efforts are made to maintain consistent organic loading rates (Barber and Stuckey, 1998). Also, Henze and Harremoes (1993) have suggested that the start-up should be operated with low loading rates to prevent overloading of slow growing microorganisms.

2.3.8 Granulation in ABR

Granulation is the immobilization of active biomass into discrete macroscopic aggregates. These aggregates display better settling characteristics and are less prone to be subjected to a washout during the operation of the reactor (Uyanik et al., 2002b). Foxon et al., (2005) reported that granulation also depends on the relative availability of substrate, constrained by concentrations and diffusion rates.

According to Wirtz and Dague (1996), granulation can:

- allow concentrations of high biomass in continuous reactors
- lead to physico-chemical gradients inside of aggregates
- lead to heterogeneous structured populations of syntrophic microorganisms
- allow continuous operation for reactors beyond normal washout flow rates
- allow biomass to be manipulated as a single phase
- affect overall stoichiometry, metabolism and rates of growth
- allow the manipulation of growth rate independently of the dilution rate
- allow the generation of an effluent with low suspended solids

Granulation is not essential to the ABR operation but an advantage because it is one of the factors affecting the settling properties in the ABR (Stuckey, 1998).

CHAPTER 3: EQUIPMENT DESCRIPTION AND OPERATION

The experimental study was undertaken at the Biochemical Engineering Laboratory (School of Chemical Engineering, University of KwaZulu-Natal) on a laboratory-scale ABR. The reactor was already constructed, installed, and was in working order since 2007. No modification on the design was undertaken during the course of this study.

This chapter is presented in three sections: the first section includes the equipment description and the auxiliary equipment. Materials and methods constitute the second section while the third section describes the reactor operating conditions.

3.1 EQUIPMENT

3.1.1 The laboratory-scale ABR

The laboratory-scale ABR was designed according to the guidelines developed by Foxon et al., (2006). Few modifications were made to the original design of the ABR. The modified reactor had wider and fewer compartments (only four) than the reactor used for the pilot study (used in 2005). Also, the reactor had a large feed tank which is a component of the ABR system included in the treatment train. In the compartments, hanging and standing baffles are represented respectively by downflow and upflow pipes. In terms of treatment capacity, the reactor can treat an average of 100 l of wastewater per day. Table 3.1 shows the base operating conditions for which the laboratory-scale ABR was designed.

Table 3.1 Operating conditions for the laboratory-scale ABR (Foxon et al., 2006)

Parameter	Unit	Value
Residence time	h	40
Feed volume per day	l/d	100

3.1.1.1 Construction of the ABR **(**adapted from Pillay et al., 2006)

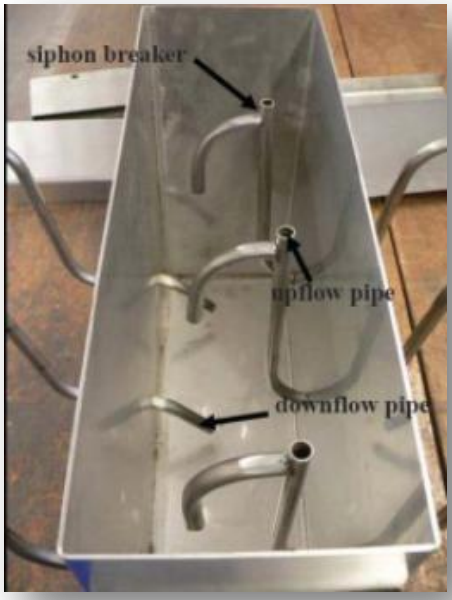
The laboratory-scale ABR consisted of five parts: a feed tank and four anaerobic compartment boxes. The feed tank was made from a sheet of stainless steel (3mm) which was laser cut, rolled and welded together to have an outer diameter of 940 mm (Photograph 3.1). The feed tank has the capacity to hold 220 l of wastewater. The feed tank lid was also made from stainless steel (3mm), laser cut, rolled and welded by Laser CNC. The reactor is fed through a sampling port (210 mm x 210mm) located on the top of the feed tank lid (Photograph 3.2).

Photograph 3.1: the feed tank **Photograph 3.2: feed tank lid**

Wastewater is pumped from the feed tank into a stainless steel splitter box (with the following specifications: length: 446 mm, breadth: 20 mm and height: 120 mm). It is thereafter channeled into four identical stainless steel anaerobic boxes connected in series. These boxes were laser cut to the following specifications: length: 445 mm, breadth: 150mm, and height: 350mm. They represent different compartments of the ABR and the design allowed for compartments to be added or removed from the laboratory plant (Photograph 3.3). Water-seal lids keep each compartment (or box) under anaerobic conditions.

Photograph 3.3: The stainless boxes representing ABR compartments

Inside the anaerobic boxes (compartments) there are three identical stainless tubes (outer diameter: 12mm) that enter from the outside to the inner bottom-half of each compartment (the location of biomass). These tubes (downflow pipes) replaced the hanging baffles**.** Standing baffles were replaced by three identical stainless tubes (outer diameter: 12 mm) that represent the upflow region of the box. Siphon breakers were placed between each box to prevent the clogging of the tubes (Photograph 3.4).



Direction of the flow

Photograph 3.4: The inside of an anaerobic compartment box with hanging and standing baffles represented by series of identical tubes

Figure 3.1 and Photograph 3.5 represent the complete experimental set up of the ABR from the feed to the effluent.

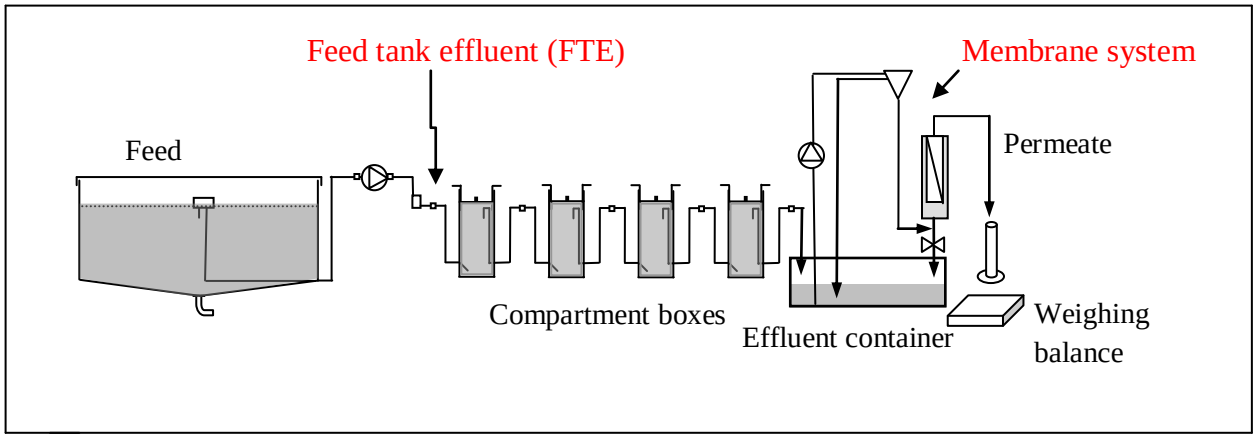


Figure 3.1 Schematic diagram of the laboratory ABR-MBR

Figure 3.1 includes the membrane system for the effluent treatment that is not part of this study.

Photograph 3.5: The laboratory-scale ABR with the feed tank, the four compartments and the membrane section at biochemical engineering laboratory, School of Chemical Engineering, University of KwaZulu-Natal

3.1.1.2 Auxiliary equipment

A peristaltic electrical pump (Watson-Marlow 323 DU), calibrated at 19 rotations per minutes was used to channel the wastewater from the feed tank to the last compartment. Tedlar bags were used as gas collectors via valves connected on top of each compartment lid. The effluent produced by the reactor was collected in a stainless steel container capable of holding 200 l, which is approximately "two days" effluent load.

3.1.1.3 Operation procedure of the laboratory-scale ABR

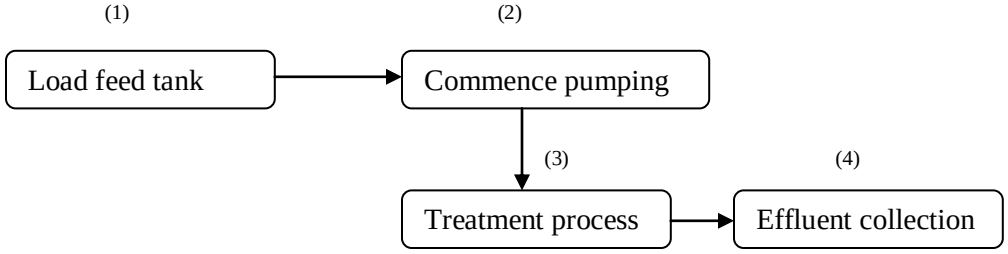


Figure 3.2: laboratory-scale ABR operation procedure

The operation of the laboratory-scale ABR presented in Figure 3.2 was semi-continuous; the feed was loaded into the feed tank batch-wise everyday. However, the flow between the feed tank and the compartment train was continuous. During the feeding time (step 1), the peristaltic pump was switched off. Once the prepared feed was loaded, the peristaltic pump was restarted (step 2), the raw wastewater was flowing within the reactor and the treatment process was taking place (step 3) since there was contact between the biomass and the liquid flow. The effluent was collected continuously (step 4) except during step 1 when the pump was switched off. The effluent tank was emptied before feeding activities.

3.2 MATERIALS AND METHODS

3.2.1 Choice of VIP sludge as a feed for the ABR.

Due to time limitations, in June 2007 the project team took the decision to change the focus of the study from the treatment of domestic wastewater to complex particulate wastewater made up of VIP sludge. One reason for this change was the challenge of getting daily domestic wastewater to run the reactor. An additional motivation for this shift was the necessity of desludging and disposal of VIP contents. Faecal material accumulates in the pits to a level where desludging becomes essential. This situation presents several problems to the municipalities. Firstly, the removal of pit contents can be difficult, especially in areas which are inaccessible to desludging equipment. Secondly, the disposal of pit contents has become an embarrassment to several municipalities, as many wastewater treatment plants (WWTP) are not able to reach their discharge limits due to the increase in the organic load. Anaerobic pre-treatment in an ABR was seen as a possible option for the treatment of VIP contents, as the pit contents are concentrated due to unavailability of water.

3.2.2 VIP collection and handling

The wastewater used to feed the reactor is made up of sludge from the ventilated improved pit latrines toilets (VIP toilets). The sludge was collected from various low-income-household communities in the eThekwini region that are served by VIP sanitation (non-flushing toilets). This required the endorsement and consent of the municipality and local community leaders to allow researchers to be in contact with the community for sampling.

Several households were visited in the area close to Newlands-KwaMashu where pits were opened and emptied. Earlier on, sludge was collected from Marianhill and Tongaat (Photograph 3.6). VIP toilets containing waste with a “solid” appearance were sampled as they were easy to empty with a spade; waste samples were stored in containers (Photograph 3.7). Undesirable objects, such as plastic bags, newspapers, hair, bricks and metals, were found dispersed in the waste (from the pits). They were all removed to fit the reactor requirements for the feeding operation (Photograph 3.8). Most of the larger and heavier objects were removed at the sampling point, and the sludge placed in plastic containers lined with plastic refuse bags. Once full, the containers were sealed with an airtight lid and disinfectant spray was used to sterilize the outside of the containers and placed in the transport vehicle. The areas surrounding the pits were also rinsed with soapy water and sprayed with a disinfectant as a precautionary measure.

Photograph 3.6: a view of the housing settlements where VIP waste is collected. Mariannhill (left), a township in Tongaat (right). Both areas are serviced with VIP toilets.

Photograph 3.7: Sampling of a VIP pit. Left: Plastic buckets (100 l) with sealable lids are used to collect VIP waste, Right: The pit is sealed once sampling is complete by placing the concrete slab over the pit.

Photograph 3.8: Sludge from VIP toilets that had been mixed with a shovel (large bucket). In the smaller bucket, undesirable material such as glass and plastic have been removed from the waste and placed in an autoclave bag for sterilization.

3.2.3 Experimental program

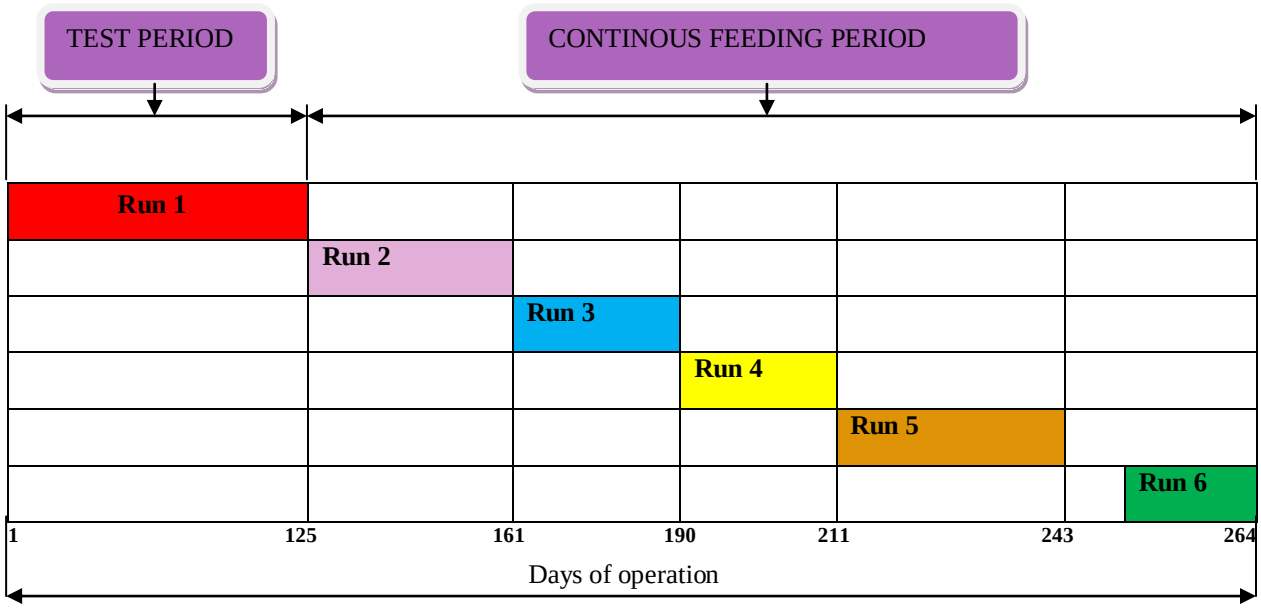


Figure 3.3: organization of the reactor operation

Figure 3.3 shows the experimental program of the laboratory-scale ABR during this study. The reactor operated for 264 days at two different periods (test period and continuous feeding period). These periods were divided in six runs. These runs operated at different inlet CODs (or organic loading rates) as shown in Tables 3.2 and 3.6.

3.2.4 Preparation of VIP slurry and wastewater supplied to the reactor.

During run 1 (test period) the inlet COD was measured per gram of VIP sludge. For run 2 to run 6 (continuous feeding period) the feed preparation procedure was changed. The feed was prepared by mixing 1.250 kg of VIP sludge into tap water to make 5 l of VIP slurry. Mixing was completed at high speed using a blender. Once the VIP slurry was homogenized, its total COD was determined by the open reflux method. Thereafter, it was added to the feed tank. The total COD of VIP slurry was very high (more than 30000 mg COD/l in most cases). Tap water was therefore added to the VIP slurry in the feed tank in order to achieve the correct inlet COD by dilution.

- **Amount of tap water added to the VIP slurry**

Table 3.1 shows the amount of water added to the VIP slurry to adjust wastewater to specific inlet COD (1000, 1500, 2000, 3000 mg COD/l) for run 2 to run 6.

Table 3.2: Amounts of tap water added to the reactor

inlet COD [mg COD/l]		amount of added water to the reactor [l]
Run 2	1000	From 86 to 190
Run 3	1500	From 83 to 165
Run 4	2000	From 64 to 99
Run 5	3000	From 35 to 45
Run 6	2000	From 77 to 80

The relevance of these dilutions in the context of full-scale operation was based on the previous studies completed on low, medium, and high strength wastewater (Barber and Stuckey, 1998). These dilutions allowed the investigation of the performance of the ABR treating wastewater from VIP sludge at low and medium strength applications (between 1000 and 3000 mg COD/l) [Ilda and Nuran, 2004].

3.2.5 Sampling

Samples were withdrawn daily from the inlet (middle of the feed tank), the outlet (effluent collection tank) of the reactor and from the splitter box (Feed tank effluent: FTE, see Figure 3.1). Feed samples were withdrawn after feeding the reactor before starting the peristaltic pump. Effluent samples were withdrawn at any particular time related to the FTE sample from 20 hours previously and the feed characteristics of the previous batch. The reactor was stopped before samples were withdrawn from each compartment. Supernatant (from the top of each compartment) and sludge samples (from the bottom of each compartment) were collected from each compartment periodically. Supernatant samples were clear liquid with floating solids and sludge samples were very dark and consisted essentially of solids. Polyethylene bottles (l l bottles) were used for sample collection. Samples were kept in the cold room at $4^{o}C$ with the remaining effluent or wastewater.

3.2.6 Desludging and sludge levels in compartments

Because of a daily loading operation, the feed tank was filled with settling solids from wastewater. This caused some disturbances such as clogging of upflow and downflow pipes. Consequently, the operation was sometimes disturbed. Desludging was undertaken at times to remove large amount of solids from the feed tank. The sludge was removed from the feed tank through a valve situated at the bottom of the feed tank. The volume of sludge was measured by beakers used for collection. Sludge levels were recorded in the compartments with a transparent cylindrical tube indicating the level of sludge. Results on sludge heights (or levels) in the compartments can be found in Appendix 4.

3.2.7 Analytical methods

All analyses were conducted according to standard methods (APHA, 1998). Details of analytical procedures can be found in Appendix 1.

3.2.7. 1 COD

COD is the amount of chemically oxidizable organic and inorganic material in the waste. Its determination provides an indication of the concentration of organic material since the organic oxidizable material in wastewater sludge is much greater than the inorganic oxidizable material. It represents the potentially biodegradable material in the raw wastewater. Feed and effluent total COD were measured by the open reflux method after digesting a sample for 2 hours in strongly acidic potassium dichromate solution. Silver sulphate was used as a catalyst and mercuric sulphate as masking agent to prevent chloride interference. Potassium dichromate is partially reduced by the oxidizable material present in the sample. The excess potassium dichromate is titrated with ammonium iron (II) sulphate, and the total COD is calculated from the amount of titrated potassium dichromate. Some samples were placed in a centrifuge (HERMLE model Z323) for 15 minutes at 15000 rotations per minute. The open reflux method (APHA, 1998) was used to determine COD concentration in the supernatant only. This measurement was taken to represent the soluble COD fraction.

3.2.7.2 Total and volatile solids

Solids concentration such as total solids (TS) and volatile solids (VS) were determined according to standard methods (APHA, 1998). A known volume of well mixed sample is weighed and evaporated to dryness in a porcelain crucible in hot air oven at 150 ^{o}C. Thereafter, the dried solids are cooled and weighed. This residual material in the crucible represents the total solids. Their determination provides an indication of the dry mass for the feed and the effluent samples. The residue from the previous method (total solids) is ignited to constant weight at 550°C. The remaining solids represent the fixed total, dissolved or suspended solids while the weight lost on ignition represents the volatile solids.

3.2.7.3 Temperature

The temperature was recorded with a temperature probe on samples withdrawn from the inlet and outlet of the reactor.

3.2.7.4 pH and Conductivity

These two measurements were recorded respectively with a pH meter and a conductivity meter. The pH was an indicator of the process stability while the conductivity was an indicator of production of total dissolved solids.

3.2.7.5 Alkalinity

This parameter was determined by potentiometric titration using 0.02N H_2SO_4 to an end-point pH value of 4.5. The aim of this measurement was to evaluate the buffering capacity of the laboratory-scale ABR treating VIP sludge.

3.2.8 Equipment, instruments and frequency of analyses

Tables 3.3 and 3.4 summarize the details about equipment and instruments as well as details concerning the frequency of analyses completed during this study.

Table 3.3: Instruments and equipment used for the measurement of various parameters

Parameters	Instrument/Equipment	Model/Manufacturer/ Specification
COD	Quickfit distillation apparatus	FMH INSTRUMENTS / LASEC
pH	pH meter	H193400 MICROPROCESSOR/HANNA INSTRUMENTS
Conductivity	Conductivity meter	EC 215
Temperature	Temperature probe	H193400 MICROPROCESSOR/HANNA INSTRUMENTS
Total solids (TS)	Oven Analytical balance	GALLENKAMP/ 166268
Volatile Solid(VS)	Muffle furnace Analytical fine balance	E160A-J4 METTLER AE 160
Alkalinity	Automatic Burette	BRAND/238638

Table 3.4 Frequencies of analyses

Parameters	Frequency of analyses	Sampling points
pH	Daily	Inlet , compartments, outlet, FTE
Temperatures	Daily	Inlet , compartments, outlet
Conductivity	Daily	Inlet , compartments, outlet, FTE
COD (total)	Daily	Inlet, compartments, outlet, FTE
COD (soluble)	Once a week	Outlet
Total and volatile solids	Twice a week	Inlet, compartments, outlet
Alkalinity	Daily (run 6 only)	Inlet and outlet
EPS	Twice a week	Inlet and outlet

FTE: feed tank effluent EPS : extrapolymeric substances

3.2.9 Statistical analyses

All statistical analyses were performed using Microsoft Excel. In all tables, data are represented as mean ± standard deviation except for pH and conductivity measurements where median values are reported. Confidence intervals at 95% are calculated for COD and conductivity. Minimum and maximum values are reported with N representing the number of samples analyzed for each parameter. The uncertainties are represented on all bar charts by error bars with standard error indicating the total range.

The student's T-test with unequal variances was used for parameters such as alkalinity and conductivity, to determine whether the changes from the inlet to outlet relating to these parameters were significant or not. The correlation between the inlet and outlet is also determined by the coefficient of correlation (for alkalinity and conductivity).

3.3 REACTOR OPERATING PARAMETERS

The work is presented for steady state operation because the feed flow was constant. However, it is only correct for the compartment train. The feed tank was batch fed as mentioned before. Consequently, the concentrations in the feed tank and the FTE were constantly changing. The effluent reached some kind of pseudo-steady state which was possibly affected by the oscillating feed conditions. These oscillating feed conditions were due to changes of organic loading rates (OLR).

3.3.1 Hydraulic retention time (HRT) and organic loading rates (OLR)

The purpose of changing OLR at constant HRT was to find out how the increasing organic loading rates impact on the effluent characteristics produced by the ABR. The total working volume of the reactor was 300 l, this includes four compartments of 20 l each and a 220 l feed tank.

The HRT was determined by calculation using the following equation:

$$\text{HRT} = \frac{WORKING\ VOLUME\ OF\ THE\ REACTOR}{PROCESS\ FLOW\ RATE} \qquad (3\text{-}1)$$

With HRT in hours, the working volume of the reactor in m^3 and the process flow rate in m^3/h.

From equation 3-1, the theoretical HRT calculation for each compartment was equal to:

$$\frac{20\ l}{4l/h} = 5 \text{ hours and HRT in the feed tank varies between 0 to 3 days.}$$

It was recorded that the HRT for the compartment train was very smaller compared to the overall HRT presented in Table 3.5 which includes both HRTs in compartments and feed tank. The possible effect is that most COD removal and digestion could occur in the feed tank than in the compartment train because of high HRT recorded in the feed tank.

The OLR was calculated from the following equation:

$$OLR = \frac{\text{FEED COD X REACTOR VOLUME}}{\text{HRT}} \quad (3\text{-}2)$$

The ratio between reactor volume and HRT is equal to the flow rate, therefore the equation 3-2 becomes:

$$OLR = \text{FEED COD x FLOW RATE} \quad (3\text{-}3)$$

With OLR in kg COD/ d and the feed COD in kg COD/m^3

Table 3.5: Operating parameters from run 1 to run 6

Run	Days	HRT(day) average	OLR (kg COD/d) min	average	max	Studies carried out
Run 1	1 to 124	3.4	0.075		0.398	Trial period
Run 2	125 to 160	3		0.096		Performance at constant OLR and HRT
Run 3	161 to 189	3		0.144		Same as in run 2
Run 4	190 to 210	3		0.192		Same as in run3
Run 5	211 to 243	3		0.288		Same as in run 4
Run 6	21	3		0.192		Same as in run 5

Table 3.5 is a summary of the operating parameters (OLR and HRT) recorded during 264 days of operation from run 1 to run 6. Run 1 aimed at identifying conditions and operating procedures that need to be controlled. From run 2 to run 6 the organic loading rates were increased from one run to another under a constant HRT. The HRT is not accurately known because the precise volume of the reactor taken up by solids and the residence time are not precisely known. For that reason, the HRT should be understood to be empty reactor volume / applied feed flow rate.

3.3.2 Reactor flow rate

Table 3.6 shows the hydraulic data recorded for the entire period of operation. Maintenance issues and electrical faults contributed to reactor downtimes as indicated in this table. Downtimes recorded describe only days when there were maintenance issues and electrical problems or public holidays during the week.

Table 3.6 Summary of hydraulic regimes used during the study

Run	Volume of treated wastewater[l]	Days of operation	Average flow rate [l/h]	Downtimes [days]
Run 1	4824	124	4.4	37
Run 2	1761	36	4	7
Run 3	1727	29	4.3	4
Run 4	961	21	4	6
Run 5	644	33	3.8	4
Run 6	960	21	4	0

CHAPTER 4: RESULTS AND DISCUSSION

Table 4.1 summarizes the COD data for the inlet and the outlet flow for run 1 to run 5. The COD data is discussed in section 4.1.1.

4.1 TOTAL COD

Table 4.1 COD data for run 1 to run 5

		Mean	Confidence interval [95%]	Minimum	Maximum	N
Test Period (Run 1) mg COD/l	**In**	1561±424	[1462,1686]	922	2691	55
	out	303±97	[279,321]	150	615	55
1000 mg COD/l (Run 2)	**In**	1000*	n.d	1000	1000	15
	out	309±75	[279,321]	113	409	15
1500 mg COD/l (Run3)	**In**	1500*	n.d	1500	1500	15
	Out	334±43	[309,353]	240	396	15
2000 mg COD/l (Run 4)	**In**	2000*	n.d	2000	2000	13
	Out	457±69	[419,495]	315	575	13
3000 mg COD/l (Run 5)	**In**	3000*	n.d	3000	3000	18
	Out	1439±153	[1367,1507]	1016	1643	18

n.d: not determined * unknown variance N: number of samples

The analysis of Table 4.1 shows that for run 2 to run 5 the effluent COD was increasing as the influent COD increased from 1000 to 3000 mg COD/l.

Table 4.2: COD data for run 6

This table presents the COD data for run 6.

		Mean	Confidence interval (95%)	Minimum	Maximum	N
COD[mg COD/l]	**In**	2000*	n.d	2000	2000	21
	Out	526±44	[507, 547]	473	592	21

n.d: not determined *variance unknown N: number of samples

Table 4.3: COD removal for different runs

RUN	Mean COD removal [confidence interval at 95%] %
Run 1	80 [72,88]
Run 2	69 [61,77]
Run 3	78 [70,86]
Run 4	77 [69,85]
Run 5	52 [44,60]
Run 6	74 [66,82]

The COD removal was calculated using the following expression:

$$\% \text{ COD} = \frac{Inlet\ COD - Outlet\ COD}{Inlet\ COD} \times 100 \quad (4\text{-}1)$$

Figure 4.1 represents the COD data recorded for run 1 to run 5 for the inlet (feed), outlet (effluent) and feed tank effluent (FTE). Feed tank effluent (FTE) designates the effluents from the feed tank being channeled into compartments (see Figure 3.1).

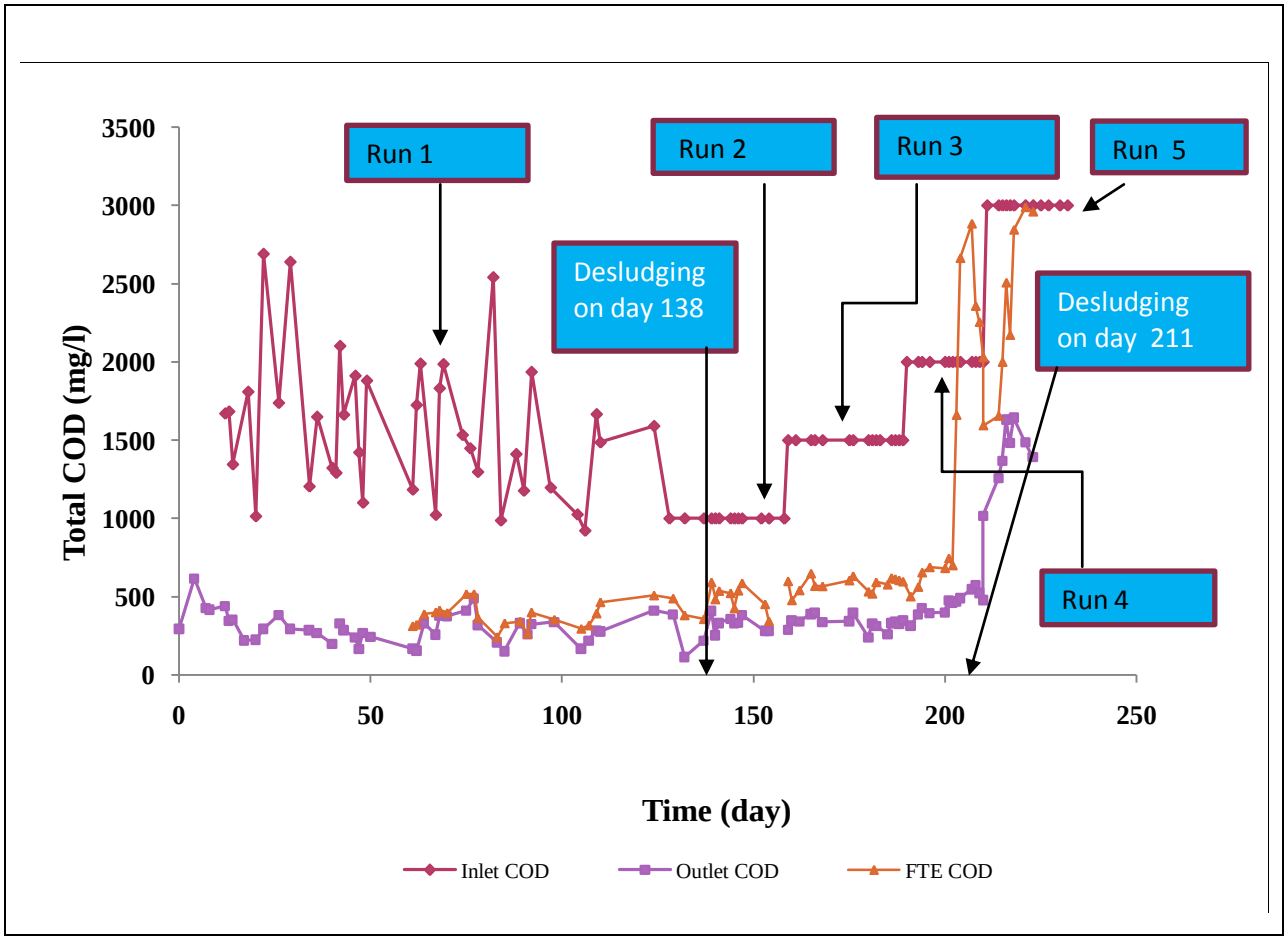


Figure 4.1: COD profiles for the inlet (feed), the feed tank and the outlet (effluent) flow (excluding run 6)

4.1.1 Performance analysis and relationship between the feed (inlet) and the effluent (outlet) total COD

- ***Performance of the reactor during run 1***

Run 1 lasted 124 days and recorded an average COD removal of 80% and an average OLR of 0.287 kg COD/d (see Table 3.5). The standard deviation on the average inlet COD was the highest recorded during the course of this study (Table 4.1). This was due to high variations between inlet COD values. The analysis of Figure 4.1 shows that FTE COD values are close to the effluent COD than to the inlet COD values. It is an indication that most digestion is taking place in the feed tank. This implies that most COD removal occurs in the feed tank through digestion and solids retention. Therefore, it can be deduced that most COD had disappeared before the flow reached the first compartment. The significance of this situation is validated by COD mass balance in section 4.1.2.

- ***Change in the method of feeding the reactor***

During run 1, inlet CODs were obtained by measuring the COD per gram of VIP sludge. These measurements showed large variances, and the assessment of the reactor performance could be compromised due to high inlet COD deviations (see Table 4.1). Therefore, for subsequent runs it was decided to control the feed concentration by varying amounts of tap water added to VIP sludge in the reactor to obtain the desired COD value by dilution (section 3.2.3).

- ***Performance of the reactor during run 2***

Run 2 lasted 36 days at an estimated HRT of 3 days and a constant OLR of 0.096 kg COD/d. During the course of this run, the feed COD was made up to 1000 mg COD/l (Figure 4.1). An average COD removal of 69 % was recorded (see Table 4.3). Most COD removal was still occurring in the feed tank. However, the compartment train removed slightly more COD compared to run 1. This is shown in Figure 4.1 by a small gap between FTE COD values and effluent COD values from the first to the last day of run 2. Desludging was performed on day 138 but it did not seem to impact on effluent COD (see Figure 4.1). However, FTE COD increased slightly, possibly as a result of desludging. This implies that a fraction of active microorganisms was removed from the feed tank during desludging.

Mixing of the feed (in the feed tank) was implemented moderately a few times during the feeding of the reactor. The aim of this mixing was to prevent entrapment of biodegradable COD in non-biodegradable sediment in the feed tank.

- ***Performance of the reactor during run 3***

Run 3 was completed in 29 days at an estimated HRT of 3 days and constant OLR of 0.144 kg COD/d. During this run, inlet COD was made up to 1500 mg COD/l. The reactor achieved an average COD removal of 78%. Despite the fact that OLR was increased (compared to run 2), a large portion of COD removal occurred in the feed tank even after manual mixing of the feed (see Figure 4.1).

- ***Performance of the reactor during run 4***

Run 4 was completed in 21 days, with the same estimated HRT of 3 days and OLR of 0.192 kg COD/d (Table 3.5). During this run, the inlet COD was made up to 2000 mg COD/l. An average COD removal of 77% was recorded. As the inlet COD was greater, the FTE COD values were also higher compared to run 3. Solid concentrations increased in the reactor because less water was added to the reactor to reach the desired inlet COD. The accumulation of solids in the feed tank (as well as in compartments) due to the increasing OLR, gave rise to a sudden increase in FTE and effluent COD from day 208 to day 210 (Figure 4.1). This sudden increase of FTE and effluent COD was due to solids wash-through occurring within the reactor, more particularly in the feed tank. In this context, wash-through means the carry-over of solids (either biodegradable or non-biodegradable) into the flow as a result of displacement.

- ***Performance of the reactor during run 5***

Run 5 lasted 33 days with an estimated HRT of 3 days and 52 % average COD removal. The OLR was 0.288 kg COD/d. The inlet COD was made up to 3000 mg COD/l. The higher OLR impacted on the COD removal efficiency by decreasing it significantly. It was hypothesized that large mass of solids added to the feed tank in this run (estimated at 1.25 kg of solids per day) resulted in filling up of the reactor volume and an increase in solid concentrations of FTE and effluent samples.

Wash-through took place from day 218 to day 243 and reached its highest value on day 223 where effluent COD was the same as FTE COD (see Figure 4.1). This situation had a decreasing effect on COD removal efficiency which was the lowest for the entire operation period (see Table 4.3).

- ***Performance of the reactor during run 6***

Run 6 was an additional run completed from 26 February to 31 March 2009 (see Table 4.2). This run aimed to confirm the data generated in previous runs, more specially during run 4. The inlet COD was the same as at run 6 (see Tables 4.1 and 4.2). No major change was recorded during run 6 compared to run 4. However, an average COD removal of 74% was recorded for run 6 at constant OLR (0.192 kg COD/d) and an estimated HRT of 3 days.

Overall, it can be deduced that the fate of COD in this study depends on:

- The type of COD: inert COD is retained and accumulates within the reactor and eventually it overflows because of the increasing load of solids (wash-through during run 4 and 5) while biodegradable COD is virtually all consumed in the feed tank. Later, the feed tank effluent (FTE) COD started to increase and more biodegradable COD entered the system (see Figure 4.1).
- The conditions and concentrations of microorganisms: the amount of substrate present in the reactor, as well as the pH and the temperature, may have had an influence on population of microorganisms and COD removal. This is an assumption based on the data, however, microbial studies necessary to support this assumption were not part of this study.

4.1.2 COD mass balance

Table 4.4 presents the COD mass balance completed on the laboratory-scale ABR. This mass balance was performed over all COD measurements from the feed, the effluent and sludge accumulated in the reactor during the operation. Although there is a large degree of uncertainty in all the quantities, the result may be regarded as a good indication of the average characteristics of the VIP sludge and its fate in the system. Details of calculations are presented in Appendix 4.

Table 4.4: Summary of COD mass balance

Influent COD	46 kg COD	100 %	
Accumulated sludge COD (Compartments+ Feed tank)	21kg COD	46%	100%
Biogas COD	13kg COD	28%	
Effluent COD	12 kg COD	26%	

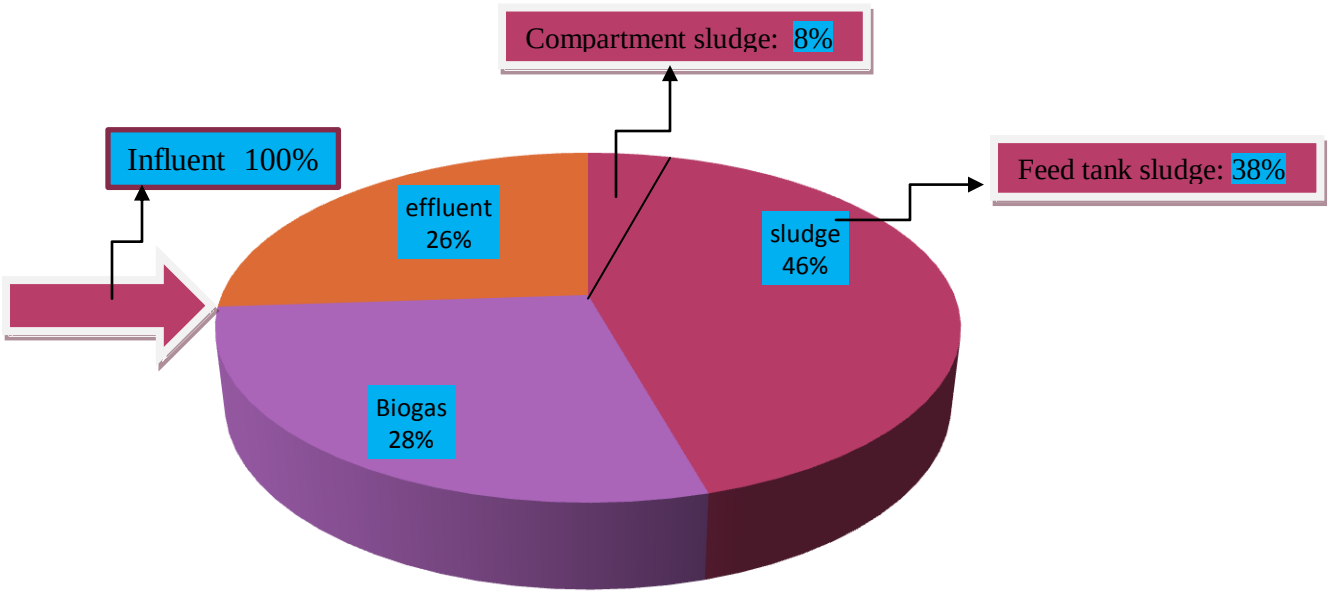


Figure 4.2: COD distribution in ABR treating wastewater from VIP sludge

The analysis of Figure 4.2 indicates that the sludge accounts for 46% of COD consumed in the system with 8% of COD taken by sludge in compartments and 38% taken by sludge in the feed tank. Biogas representing the biodegradability of VIP sludge takes only 28% of COD in the system. Consequently, there is no biodegradable COD in the effluent because it was completely taken by biogas. The biodegradability of VIP sludge (28%) found in this study is close to the one obtained in a parallel study completed by Bakare (2010) which was 28.9%. In his study, Bakare (2010) used an aerobic method to determine the biodegradability of VIP sludge. Nevertheless, biogas data to support the COD mass balance results could not be obtained because the serum

bottle method (used to determine Biochemical Methane Potential) was proven to be unsuccessful for VIP sludge (Nwaneri, 2009). However, the analysis of COD mass balance shows that a small fraction of COD is destroyed by digestion (production of biogas) and the remaining big portion of COD was retained by inert solids and biomass present in the reactor. This happened mostly in the feed tank where large amount of sludge was found. Therefore, the fact that a small portion of COD is taken by biogas, is an indication that biodegradability of VIP sludge is very low compared to the average biodegradability of a feed material during an anaerobic process (between 80 and 90%) (Speece, 1996).

Furthermore, VIP sludge was already fairly well stabilized before being added to the system. This stabilization is due to a permanent degradation of VIP sludge that would have occurred in the pit latrine aerobically (top layer) and anaerobically (bottom layer). As a result, there is a low biological activity taking place in the reactor.

4.1.2.1 COD load of solids and estimated methane production

- **COD load of solids**

The COD load of solids for each run presented in Figure 4.2a was calculated with the following expression:

$$\text{COD load of solids} = \text{COD}_{in} \times \text{Time} \times 0.46 \quad (4\text{-}2)$$

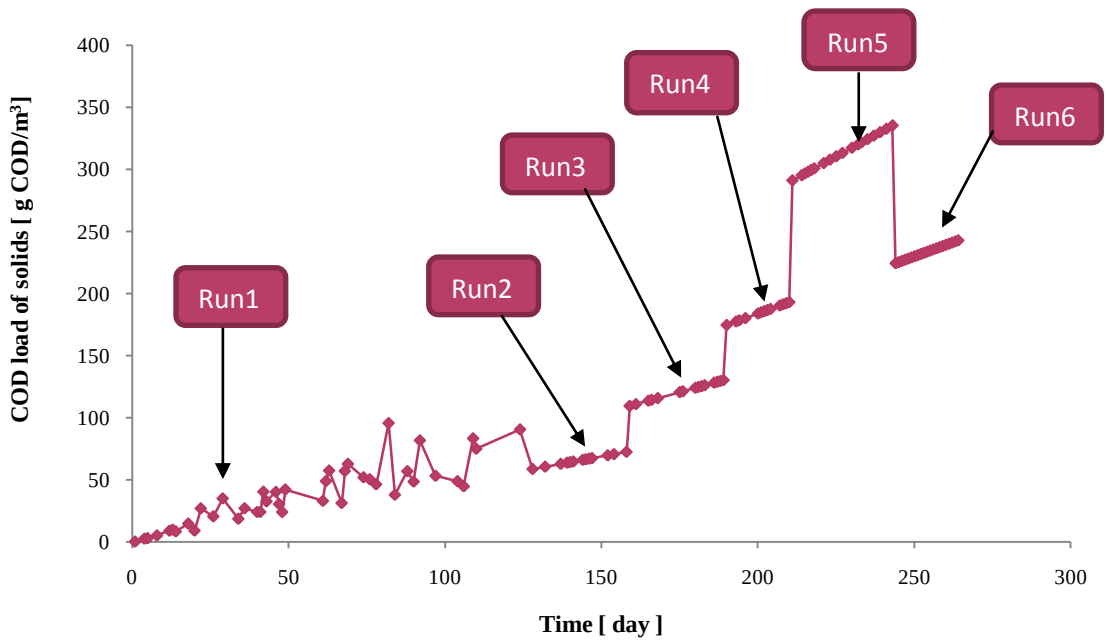


Figure 4.2a: COD load of solids in the reactor

Figure 4.2a shows the evolution of COD load of solids in the reactor for all 6 runs. Run 5 recorded the highest COD load of solids probably due to high organic loading rate applied during this run. From run 2 to run 6, it was observed that the COD load of solids was increasing from the beginning to the end of each run. This was due to the accumulation of solids from the feed in the reactor, mostly in the feed tank.

- **Estimated methane production**

The fraction of methane produced during the operation can be estimated by extending the general equation for COD mass balance described as follows:

$$COD_{in} - COD_{out} - COD_{sludge} = COD_{total\ methane} \qquad (4\text{-}3)$$

The COD mass balance around the feed tank is:

$$COD_{methane\ feed\ tank} = COD_{in} - COD_{FTE} - COD_{feed\ tank\ sludge} \qquad (4\text{-}4)$$

Also, COD mass balance for the compartment train is:

$$COD_{\text{methane in compartment train}} = COD_{\text{total methane}} - COD_{\text{methane feed tank}} \quad (4\text{-}5)$$

Methane production in the feed tank as well as in compartments presented in Figure 4.2b was calculated from equations 4-4 and 4-5.

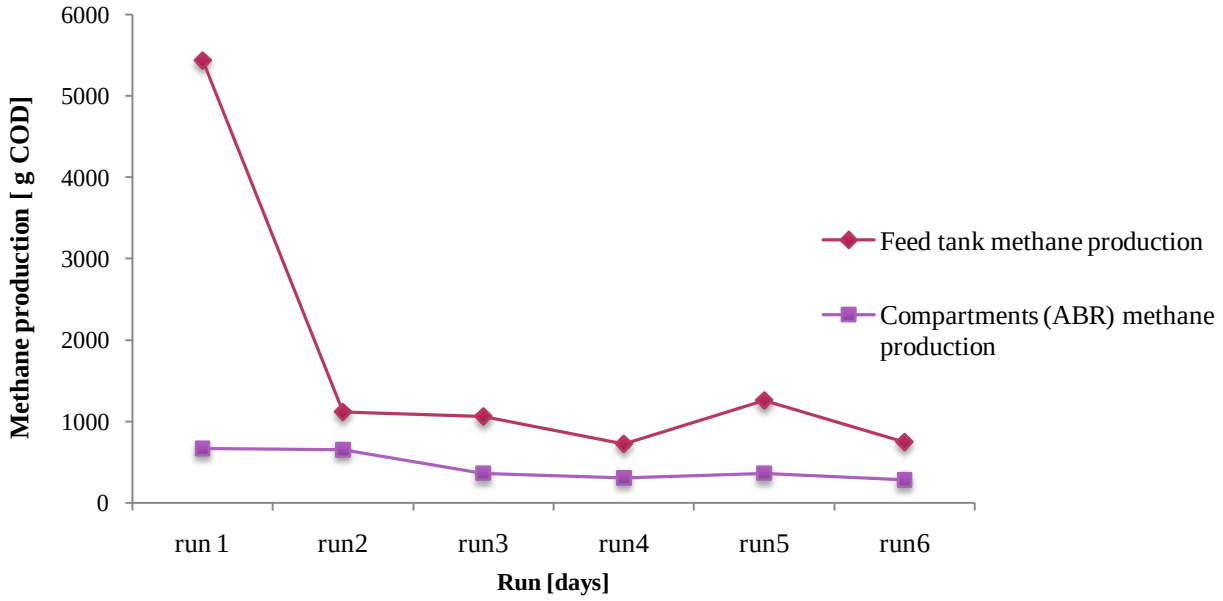


Figure 4.2b Estimated methane production in the reactor

Figure 4.2b indicates that high production of methane in the feed tank occurred during run 1. Run 5 recorded high production of methane in the feed tank between run 2 and run 6 where the loading rates were increased systematically. This is probably due to high OLR applied during this run. In compartments, the production of methane was not strongly affected despite the increase in loading rates. The estimated production of methane in compartments was low as indicated in Figure 4.2b. This was due to the low biodegradability of the feed material. It can be deduced that this low methane production in the system is an indication that biological stress within the system is ineffective or very low.

4.1.3 Total COD in compartments

- **Supernatant samples COD**

Figure 4.3 shows the variations of supernatant samples COD withdrawn between day 97 and 154. There does not appear to be a systematic trend in supernatant COD concentration. However, between day 201 and 218 (run 4 and run 5), COD values from earlier compartments (1 and 2) were higher than those recorded between day 97 and 154. This was probably due to solids wash-through taking place in the reactor. The increase of solids in the reactor (especially in the feed tank) at high organic loading rate was the main cause of this situation as most COD is retained by solids. It can be deduced that the loading rate can affect the COD in compartments. Therefore, the increased COD concentration could be understood as a result of solids displacement and therefore, probably does not indicate that the higher loading rate resulted in a biological stress.

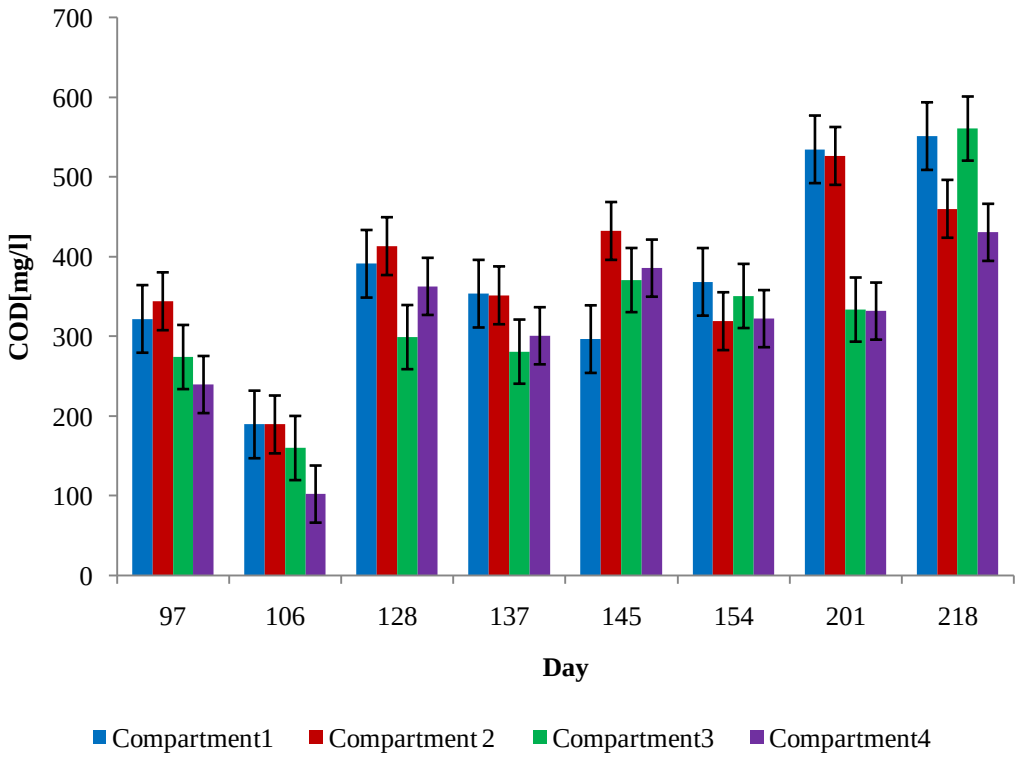


Figure 4.3: COD profile of supernatant samples in compartments

4.1.4 Relationship between effluent soluble COD and effluent total COD

Figure 4.4 indicates that there is a presence of soluble organics in the effluent due to the amount of soluble COD recorded in the effluent. According to Fieller's theorem dealing with the ratio of two means (appendix 2), the range of the ratio of mean soluble COD and mean total COD should be between 0.22 and 0.76. For this study this ratio is 0.43 and it is included in the range mentioned before. The average slope of linear regression for total COD is 8.442 ± 1.04 with R^2= 0.82 and the average slope for soluble COD is 2.345±1.3 with R^2= 0.18. From a statistical point of view it can be assumed that the increase of total COD with time is highly significant ($P = 1.26.10^{-6}$) while for soluble COD the decrease is not significant (P = 0.1).

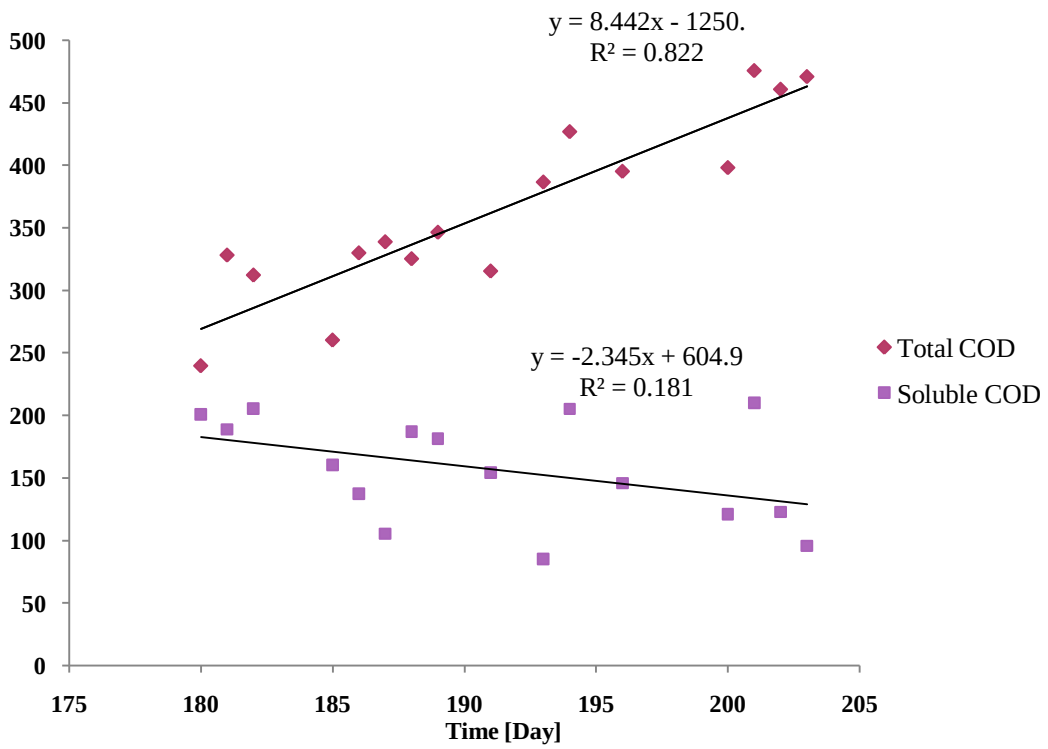


Figure 4.4: Soluble and Total COD in effluent samples

It is observed that although the slope of soluble COD seems to be changing (Figure 4.4), the significance of the slope is low. This suggests that soluble COD is not changing substantially with time. Since organic overload and related biological stress is usually observed by an increase in soluble and volatile components, this result indicates that there is no biological stress and the increased total COD is due to wash-through and washout of poorly or non-biodegradable COD.

In conclusion, there is no net change in the behavior of the reactor in terms of biodegradation since there is no change for soluble COD.

4.2 REACTOR pH

4.2.1 pH for the inlet, the feed tank and the outlet flow

Table 4.8 shows the pH data collected on the inlet (feed), the feed tank (FTE) and the outlet flow (effluent) of the laboratory-scale ABR from run 1 to run 6. The significance of all data recorded on pH is discussed in section 4.2.3.

Table 4.5: pH data for the feed, feed tank effluent and effluent flow from run1 to run 6

		Median	Minimum	Maximum	N
Run 1	**In**	8.35	7.7	8.9	42
	FTE	7.5	7	7.8	19
	Out	7.43	7.1	7.9	42
Run 2	**In**	8.28	8.14	8.6	14
	FTE	7.63	6.95	7.94	14
	Out	7.4	6.57	7.5	14
Run 3	**In**	8.13	7.92	8	15
	FTE	7.82	7.4	8	15
	Out	7.44	7.18	7.74	15
Run 4	**In**	8.1	7.84	8.38	13
	FTE	7.72	7.52	7.83	13
	Out	7.57	7.4	7.75	13
Run 5	**In**	8.1	7.8	8.45	18
	FTE	7.71	7.5	7.92	18
	Out	7.67	7.54	7.8	18
Run 6	**In**	8	7.8	8.2	22
	FTE	7.81	7.5	7.92	22
	Out	7.15	7.05	7.7	22

FTE: feed tank effluent

4.2.2 pH in compartments

- **Supernatant samples**

Table 4.6 presents the pH measurements taken on supernatant samples collected from the top center of each compartment between day 97 and 218 (from run1 to run 5).

Table 4.6: pH data for supernatant samples from each compartment (run 1 to run 5)

	Median	Minimum	Maximum	N
Compartment 1	7.19	6.92	7.65	8
Compartment 2	7.15	7.05	7.4	8
Compartment 3	7.1	6.83	7.72	8
Compartment 4	7.15	6.85	7.36	8

N: number of samples

Figure 4.5 is a chart of the data presented in Table 4.6; however, this figure includes the pH of the feed and the effluent.

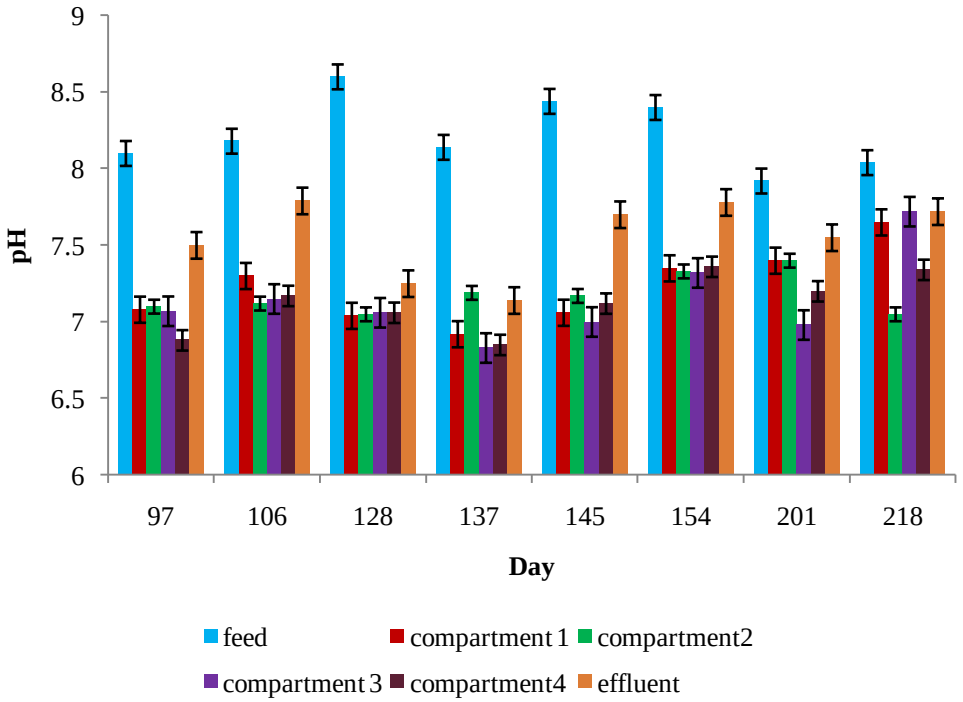


Figure 4.5 pH profiles for supernatant samples taken from each compartment

- **Samples taken from the bottom of each compartment (sludge)**

Table 4.7 shows the pH measurements of samples taken from the bottom of each compartment on days 137, 201, 243 and day 7 of run 6. (Run 2, 4, 5 and 6).

Table 4.7 pH data for samples taken from the bottom of each compartment (run 2, 4, 5, and 6)

	Median	Minimum	Maximum	N
Compartment 1	6.85	6.5	7.05	4
Compartment 2	7.01	6.6	7.2	4
Compartment 3	6.97	6.54	7.21	4
Compartment 4	7.07	6.64	7.07	4

N: number of samples

Figure 4.6 is a chart of the data presented in Table 4.7. Low pH values were recorded on day 7 of run 6.

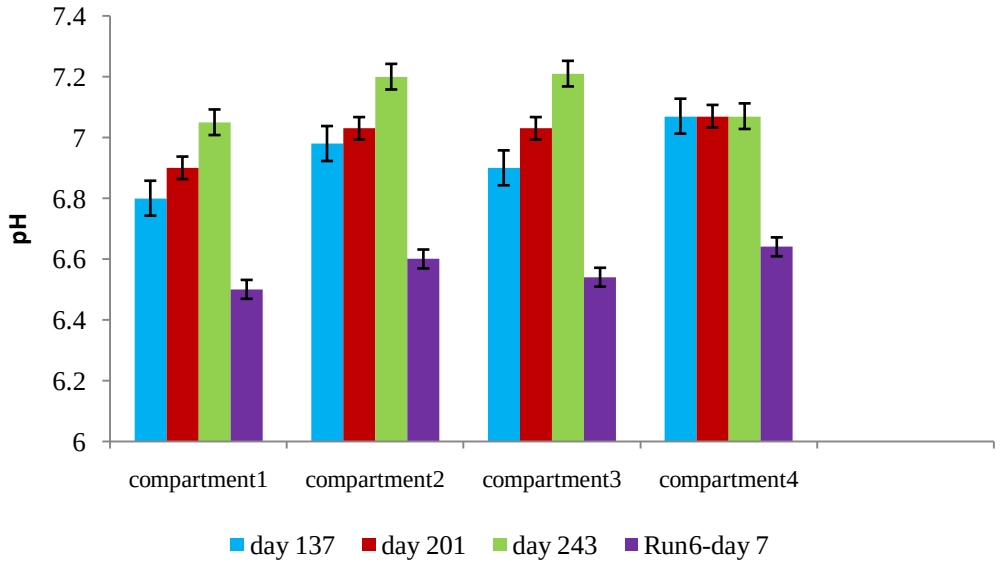


Figure 4.6 pH profiles for samples taken from the bottom of the compartments

4.2.3 Performance analysis and relationship between the feed and the effluent pH

- **Feed and effluent pH**

From the analysis of Table 4.5, it was observed that pH values (feed and the effluent) for all runs were within the appropriate range for a proper digestion (6.5 to 8) (see section 2.2.2.2). No pH inhibition was recorded during the operation. However, a drop in pH from the inlet (feed) to the outlet (effluent) was recorded for all measurements as indicated in Table 4.5. This implies a low buffering capacity of the system. It is an indication that low alkalinity production occurs in the system. This is validated by the fact that the pH of the effluent in an anaerobic digestion is mostly dependent on the mean oxidation state and alkalinity generation potential of the feed (Foxon, 2009; Speece, 1996).

- **Supernatant samples and samples from the bottom of each compartment**

From the analysis of Table 4.6 and Figure 4.5 (for supernatant samples) it was noticed that there are minor pH variations between compartments. The same observation was made on samples taken from the bottom of each compartment (Table 4.7 and Figure 4.6). However, samples from the bottom of compartments had low pH values compared to the supernatant samples. This suggests that a small amount of biological activity was occurring in the sludge bed. Figure 4.6 indicates that pH values for samples taken from the bottom of each compartment recorded on day 7 of run 6 were then lower than on other sampling days, this implies that conditions at the bottom of each compartment were significantly different during run 6. Also, it was observed that the feed and effluent pH values were higher than those in each compartment (Figure 4.5). This was due to the high pressure of CO_2 inside compartments which reduces the pH.

4.3 REACTOR ALKALINITY

This parameter was part of the analyses performed on the inlet, the outlet flow to determine the buffering capacity of the system. Table 4.8 and Figure 4.7 show the alkalinity data for the inlet and outlet flow during run 6 only. The significance of the results is discussed in section 4.3.1.

Table 4.8: Alkalinity data for the feed and effluent flow during run 6

	Mean [mg $CaCO_3$/l]	Minimum	Maximum	N
Feed	125± 20	101	178	23
Effluent	140± 21	114	188	23

N: number of samples

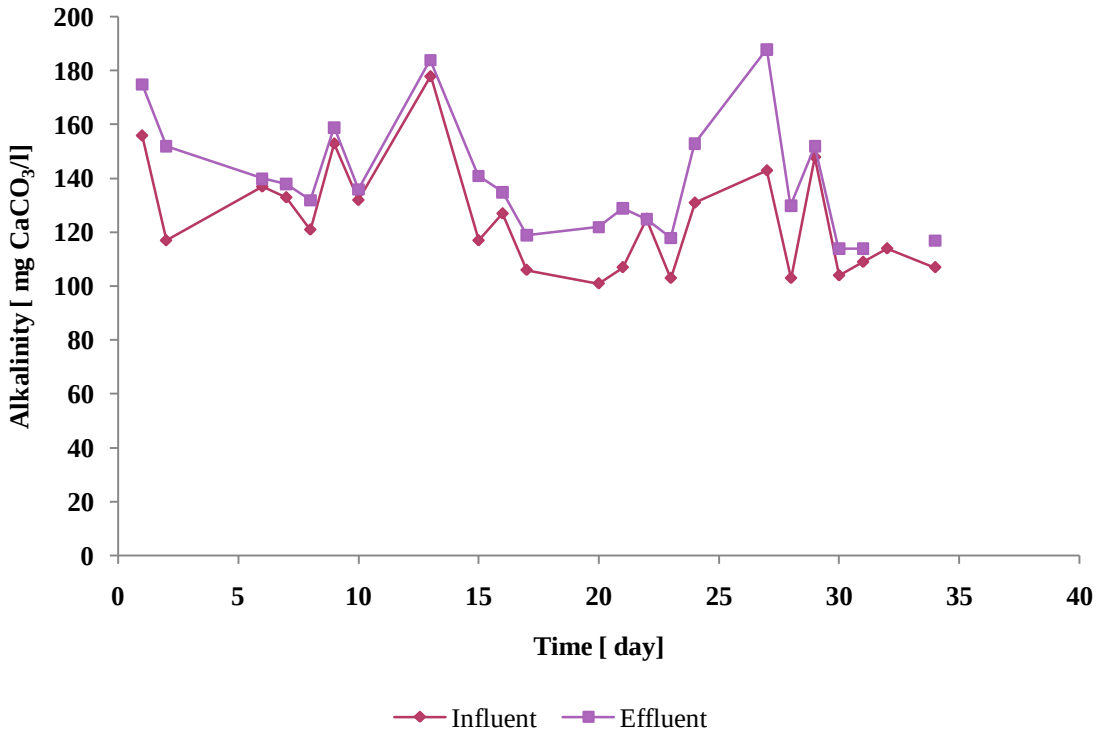


Figure 4.7: Alkalinity profile for the feed and the effluent during run 6

4.3.1 Performance analysis and relationship between the feed and the effluent alkalinity

From the analysis of Figure 4.7, it was observed that the feed alkalinity was lower than the effluent alkalinity. This increase was statistically significant (Student's T-test unequal variances

P= 0.012) with a coefficient of correlation between the inlet and outlet of 0.86. This coefficient value implies a strong relationship between the inlet and outlet for alkalinity measurements. However, the rate of alkalinity formation recorded during this study was very low, almost 15 times smaller than the average of 2000 mg $CaCO_3$/l recommended by Speece (1996) to ensure sufficient buffering. Nevertheless, it does not mean that the reactor is close to failure. The alkalinity generated for a stable operation depends on the type of treated wastewater and on the species contained in the wastewater. These species should be capable of generating bicarbonate or ammonia compounds required for alkalinity production.

Generally, low proteinaceous concentrations in the feed result in the metabolic generation of low alkalinity in the effluent (Speece, 1996). According to Lettinga et al., (1997), not all processes produce alkalinity: if no cation is released from the organic compounds, no alkalinity is generated. Therefore, the low alkalinity generated in this study is an indication of low concentrations of organic compounds in the feed (such as proteins and carbohydrates). This implies that less cations such as bicarbonate and ammonia are released from organic compounds during the treatment of complex particulate wastewater in the ABR.

In general, the small pH and alkalinity changes support the conclusion that the amount of biodegradation occurring during the treatment process is small as a result of the low biodegradability of the feed material. Furthermore, the fact that the pH and alkalinity vary between the feed and effluent does not appear to change with increases in organic loading rates supports the conclusion that there is no biological stress.

4.4 REACTOR TOTAL AND VOLATILE SOLIDS

The aim of these measurements on the feed and effluent samples was to assess the reactor solids retention capacity under increasing organic loading rates. Also, measurements were taken on supernatant samples from compartments a day after run 6 (1 st April 2009). The results are presented in Tables 4.9 and 4.10. Their significance is discussed in section 4.4.1. Table 4.9 summarizes the data for total (TS) and volatile (VS) solids achieved on both feed and effluent during run 1, 2, 4 and 6.

Table 4.9: Summary of data for total (TS) and volatile (VS) solids

Run		Feed				Effluent			
		Mean	**Min**	**Max**	**N**	**Mean**	**Min**	**Max**	**N**
Run 1	**TS**	779±330	340	1319	12	442±14	285	660	7
[mg/l]	**VS**	586±277	209	988	12	190±73	85	308	7
Run 2	**TS**	482±196	224	954	12	338 ±102	343	637	6
[mg/l]	**VS**	340±133	158	613	12	146±79	70	115	6
Run 4	**TS**	505±213	235	720	13	208±64	116	302	9
[mg/l]	**VS**	104±80	80	818	13	40±14	19	60	9
Run 6	**TS**	276±130	35	831	23	29±8	15	47	23
[mg/l]	**VS**	196±66	64	619	23	12±5	4	26	23

Min: minimum Max: maximum N: number of samples

Table 4.10: Total and volatile solids in compartments (mean values) from supernatant sample taken on 1st April 2009

	Compartment 1	**Compartment 2**	**Compartment 3**	**Compartment 4**
TS [mg/l]	40±5.5	44±8.7	39± 1.5	50± 10
VS [mg/l]	15±1	15±4.3	16±3.3	21±5.3

4.4.1 Performance analysis and relationship between the feed and the effluent based on solids concentrations

The analysis of Table 4.9 reports that run 1 recorded an average of 43 % for TS removal and 68% for VS removal. During run 2 an average of 70 % for TS removal was recorded and the average VS removal was 53%. Run 4 recorded on average 59 % and 60 % for VS and TS removal respectively. Run 6 recorded an average of 90 and 89% for VS and TS removal respectively. From these solids removal efficiencies, it can be deduced that the reactor has the capacity to retain large amount of solids. Consequently, there is an accumulation of solids in the reactor during the operation. The accumulation of solids in the reactor may be due to the build-up of biomass and the overflowing of the sludge from one compartment to another. The overflowing may be due to the entrainment of solids in the liquid flow or growth in the previous compartment displacing extra sludge over the intermediate standing baffle. Table 4.10 shows that the supernatant in the last compartment had a high solids content compared to earlier compartments. However, these differences are not statistically significant. This suggests that most solid particles in compartments are subjected to settling.

Settling depends very much on the type of solids within the reactor. Generally, in the ABR, solids particles with high density will settle (mainly in the first compartment) and those with lower density will float mostly on top of compartments (Lettinga et al., 1992). Solids with density similar to wastewater will be carried by the flow of wastewater from the first compartment to the effluent collecting unit. However, the flow rate of wastewater through the reactor is very important because at a very high flow, more solids will flush out if they are prevented from settling. Also, it was noticed from the detailed data summarized in Table 4.9 that while the difference TS_{in} -TS_{out} was often not significant, VS_{in}- VS_{out} was significant, hence, biodegradation occurred but did not have a great effect on the solids because the biodegradation fraction is relatively small.

4.5 REACTOR CONDUCTIVITY

Table 4.11 shows the conductivity data recorded from run1 to run 5 for the feed, feed tank and the effluent flow. The results are discussed in section 4.5.1.

Table 4.11: summary of conductivity data for the feed, feed tank effluent and effluent flow.

Run		Median [µs /Cm]	Confidence (95%)	Minimum	Maximum	N
Run 1	**In**	543	[500,586]	266	841	33
	FTE	661	[628,694]	506	837	20
	Out	703	[665,741]	362	910	33
Run 2	**In**	366	[343,389]	297	459	14
	FTE	437	[384,490]	303	677	14
	Out	462	[427,497]	368	598	14
Run 3	**In**	442	[394,490]	447	698	15
	FTE	478	[440,516]	385	410	15
	Out	526	[482,570]	410	749	15
Run 4	**In**	537	[476,598]	370	720	13
	FTE	628	[576,680]	453	758	13
	Out	667	[628,706]	554	771	13
Run 5	**In**	671	[635,706]	441	738	18
	FTE	881	[796,906]	518	1110	18
	Out	897	[835,959]	652	1116	18

FTE: Feed tank effluent N: number of samples

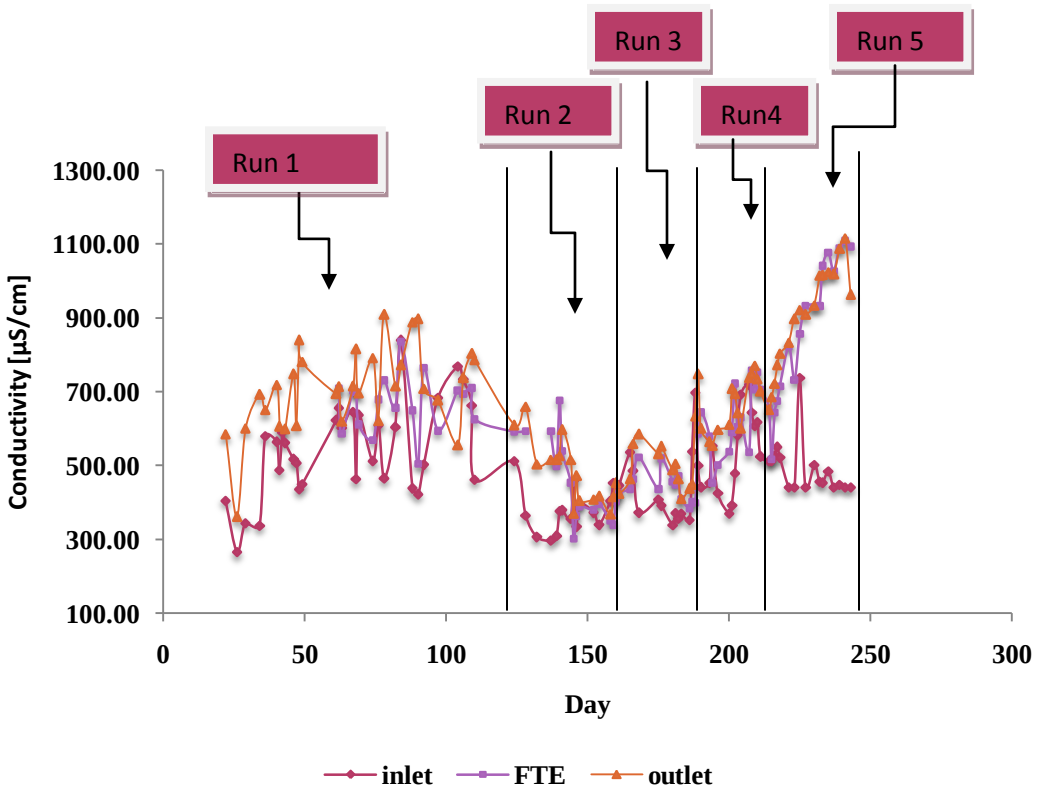


Figure 4.8: Plot of conductivity measurements of the feed, feed tank effluent and effluent flow

Figure 4.8 shows the profile of conductivity measurements taken on the feed, feed tank effluent and effluent for run 1 to run 5.

4.5.1 Performance analysis and relationship between the inlet (feed) and the outlet (effluent) conductivity

The analysis of Figure 4.8 and Table 4.11 shows that outlet conductivities are higher than inlet and FTE conductivities for all measurements. This increase in conductivity from the inlet to the outlet implies that there is a production of dissolved ionic solids during digestion in the reactor. Also, it was observed that run 5 recorded high conductivity values (for inlet, outlet and FTE) compared to other runs (see median values in Table 4.11), this is probably due to high organic loading rate applied during this run. This suggests that at high organic loading rate more dissolved solids were certainly produced to increase the conductivity values.

Furthermore, Figure 4.8 indicates that conductivity followed a similar pattern to the inlet COD. As the inlet COD were increased from 1000 to 3000 mg COD/l, the outlet conductivity increased from 660 to 1116 μs/ cm. Statistically, there is a significant increase of conductivity between the inlet and the outlet (Student T-test with unequal variances $P \leq 0.0103$). The correlation coefficient between the inlet and the outlet conductivities for all runs is 0.64. This implies a strong relationship between the inlet and the outlet conductivities.

4.6 REACTOR WORKING TEMPERATURES

Figure 4.9 presents the measurements of temperature at the inlet and outlet flow of the ABR. The reactor operated at room temperature and all recorded temperatures were below 25 ^{O}C. During run 1 the reactor operated under summer and winter temperatures. From run 2 to run 5 the reactor operated under summer temperatures. The results are discussed in section 4.6.1.

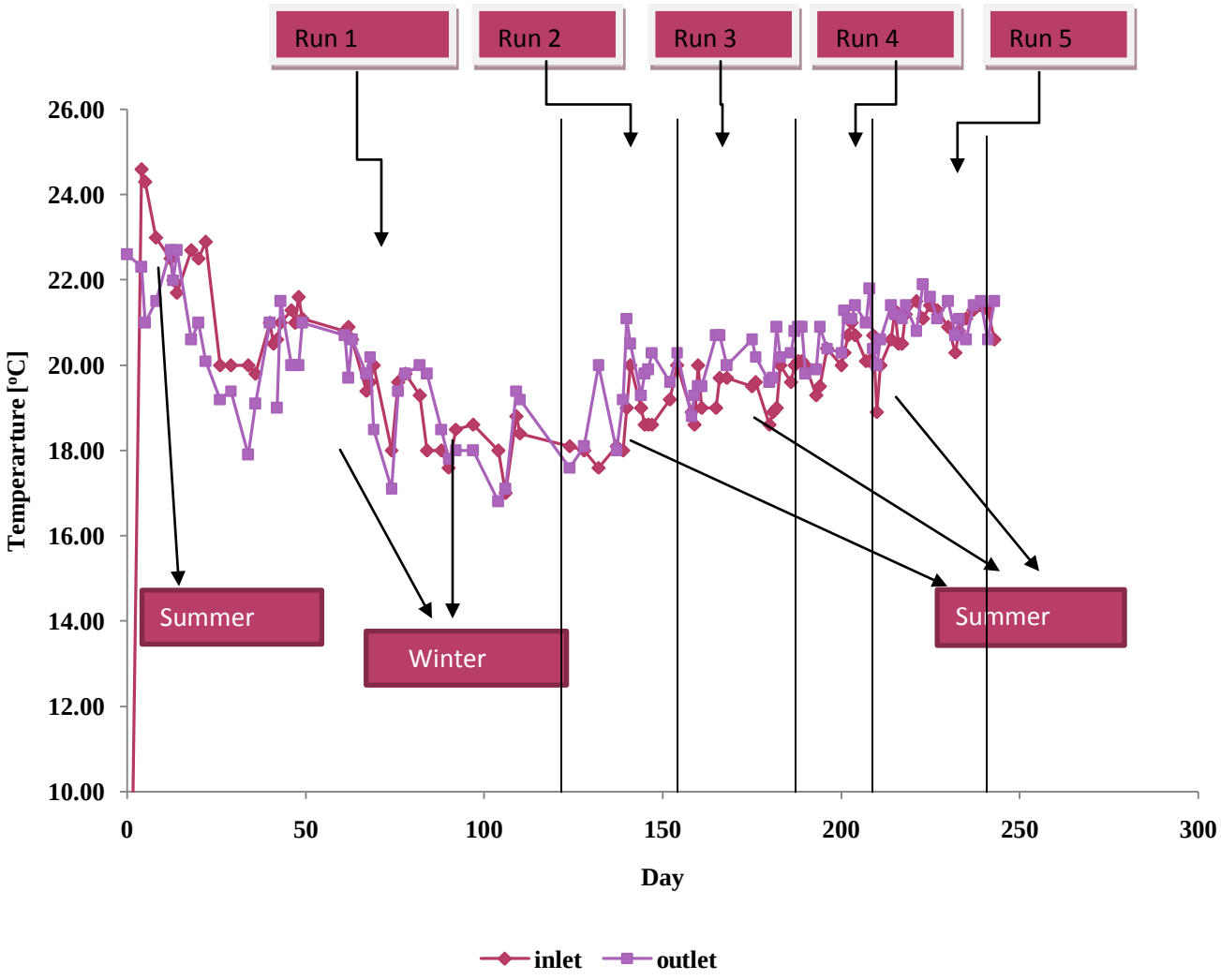


Figure 4.9: Temperature data of the feed and effluent flow

Figure 4.10 presents the data for temperature in compartments between day 97 and day 218.

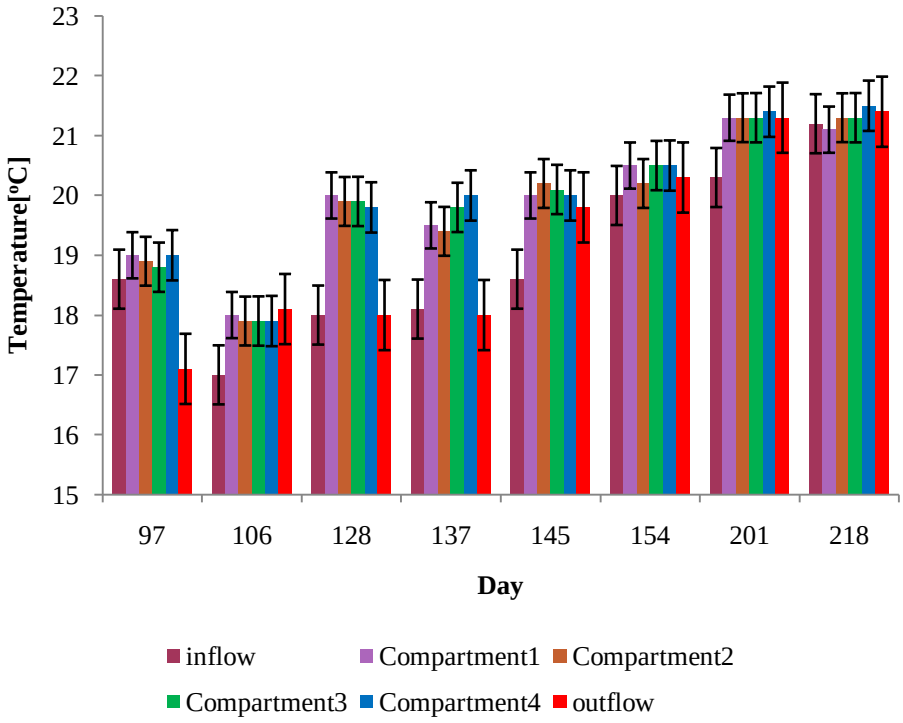


Figure 4.10: Temperature data of the flow in compartments

Very little variation was observed between temperatures in compartments; they increased from day 97 to 218 (Figure 4.10).

4.6.1 Analysis for temperature in ABR

According to Henze and Harremoes (1997), the optimum conversion rate of anaerobic digestion takes place between 30 and 40°C. At temperatures below this range, the digestion rate decreases by about 11% for each Celsius degree temperature decrease. It well known that temperature has an influence on the rate of biological reactions that occur within the ABR (Barber and Stuckey, 1998). Therefore, it was observed that all temperature measurements recorded during the operation were below the theoretical range for the optimum conversion in an anaerobic system. The reactor was operated at room temperatures (17 to 22.8 °C) from run 2 to 6 and from 16 to 24 ºC for run 1, these temperatures were at the low end of the mesophilic range.

Seasonal temperatures or the change in temperature due to biological activity could be the main cause of low these low temperatures. As the biological activity in the reactor was found to be low, the first reason (seasonal temperatures) is more likely to be acceptable because the reactor operated during winter and summer periods (see Figure 4.9). As a result, the conversion rate was low because of low biodegradability of the feed digested at low temperatures.

4.7 REACTOR EPS (Proteins and carbohydrates)

One of the aims of this study was to analyze EPS characteristics of the effluent since it well known that this affects membrane operation. Soluble proteins and carbohydrates content were analyzed by Pillay (2009) on feed and effluent flow while the reactor was operating from run 2 to run 5. They are very important for this study; they provide information on the biological activity in the reactor while organic loading rates are increasing. These results are shown in Table 4.12 (with permission) and discussed in section 4.7.1.

Table 4.12: Summary of data for proteins and carbohydrates taken on the feed and effluent flow from run 2 to run5 (Pillay, 2009).

Run		Feed				Effluent			
		Mean	Min	Max	N	Mean	Min	Max	N
Run2	Proteins	n.d	n.d	n.d	n.d	71.6±25	30	115	5
[mg/l]	Carbohydrates	n.d	n.d	n.d	n.d	1.1±0.5	0.3	1.6	5
Run 3	Proteins	78±0.5	78	79	2	47.2±14.5	30	75	8
[mg/l]	Carbohydrates	2.3±0.3	2.1	2.6	2	1.4±0.6	0.4	2.3	8
Run 4	Proteins	54.9±16.9	16	80	11	31.2±6.1	20	38	12
[mg/l]	Carbohydrates	2.1±0.6	0.8	2.8	11	1.9±0.3	1.5	2.4	12
Run 5	Proteins	47.5±16.5	27.8	73.9	6	21.4±4.2	16.8	29.6	7
[mg/l]	Carbohydrates	2.4±0.6	1.6	3.3	6	1.9±0.3	1.7	2.4	7

Min: minimum Max: maximum N: number of samples n.d= not determined

4.7.1 Performance analysis and relationship between feed and effluent for proteins and carbohydrates

During run 2, proteins and carbohydrates were measured in effluent samples only. Run 3 recorded 40% and 39% respectively for protein and carbohydrate removals. Run 4 recorded 43% and 10% respectively for protein and carbohydrate removals. During run 5, the recorded protein and carbohydrate removals were 55% and 21% respectively.

The analysis of Table 4.12 shows that despite the increasing loading rates from run 2 to run 5, effluent EPS concentrations are smaller than feed EPS concentrations. However, the effluent EPS concentrations were expected to be greater than the feed EPS concentrations because of the increasing OLR applied from one run to another. This situation was unexpected because it is different to what occurs to EPS during an anaerobic process as mentioned in literature (see section 2.2.4.1). The understanding of this unexpected situation suggests that the biodegradability of the feed material (VIP sludge) was very low and there was not enough biological activity to produce EPS through metabolism. The low biodegradability of the feed used in this study has validated the fact that the feed material was fairly well stabilized as mentioned previously.

Furthermore, despite the fact that there was removal of EPS from the influent to the effluent, it was evaluated from the data presented in Table 4.12 that more than 60% of EPS remained in the effluent, membrane fouling was observed with rapid decrease in effluent flux from the membrane post-treatment unit (Pillay et al., 2009). Therefore, the EPS present in the effluent were the major cause of membrane fouling in ABR treating wastewater from VIP sludge (Pillay et al., 2009).

4.8 OVERALL PERFORMANCE OF THE ABR TREATING VIP SLUDGE

Generally, the main objective in wastewater treatment is the stabilization of pollutants and separation of solids to produce a clarified effluent with reduced toxic and dangerous substances (BORDA, 2008 p. 44). In this study, organic loading rates were increased from run 2 to 6 under a constant HRT. Anh et al., (2003 and 2007) reported similar removal efficiencies (for COD and solids) in ABR reactors treating similar type of wastewater in Vietnam. For a baffled reactor, 70 to 90% of COD removal is expected in a properly functioning system though the effluent

quality may depend also on the nature of the influent (BORDA, 2008 p.11). Removal efficiencies recorded in this study are key indicators of the reactor performance. These COD and solid removals reveal the effluent type produced in ABR treating wastewater from VIP sludge with increasing organic loading rates. However, the anaerobic digestion of VIP sludge has shown that the biodegradability of the VIP sludge is too low; it was validated by COD mass balance. This implies that the anaerobic digestion is not the most appropriate choice regarding VIP sludge management strategies.

In terms of reuse/discharge implications, it was noticed that the average COD of the effluent from each run was higher than the discharge limit acceptable (limit to surface water) by the Department of Water and Environmental Affairs which is 75 mg/l. However, the same average COD for the effluent was within the permissible limit for irrigation (in agriculture) which is 400 mg/l (for a discharge of 500000 l/d) (DWAF, 1996) from run 1 to run 3, whilst run 4 recorded an effluent COD value slightly higher than 400 mg/l. In addition, a sample of analysis for nutrient concentration, trace elements and heavy metals achieved on ABR effluent from this study taken in July 2009 by Bame (2009) revealed that these compounds were within agricultural re-use limits (Appendix 3).

4.9 DATA CORRELATION BETWEEN THE ORGANIC LOADING RATES (OLR) AND EFFLUENT CHARATERISTICS

It was hypothesized that the increases of the organic loading rates may affect the effluent characteristics (see section 1.4). In this section, the effect of the increasing loading rates on the effluent characteristics is analyzed to determine the relationship between both parameters.

4.9.1 Correlation between OLR and effluent COD

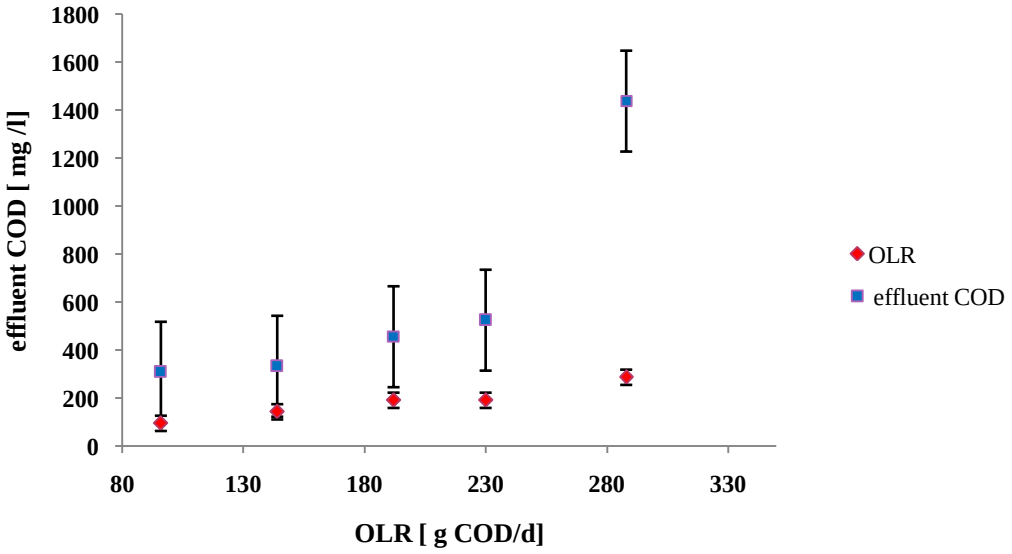


Figure 4.11: Effect of OLR on effluent COD

Figure 4.11 shows that as the OLR were increasing, the effluent COD were also increasing. A significant increase of the effluent COD was recorded during run 5 which had the highest OLR. Solids washout and wash-through occurring in the reactor is the main cause of this situation. This implies that the effluent total COD is linked to solids dynamics within the reactor. In conclusion, OLR have a direct effect on effluent COD through solid retention and digestion. This is supported by COD mass balance results (section 4.1.4).

4.9.2 Correlation between OLR and effluent EPS

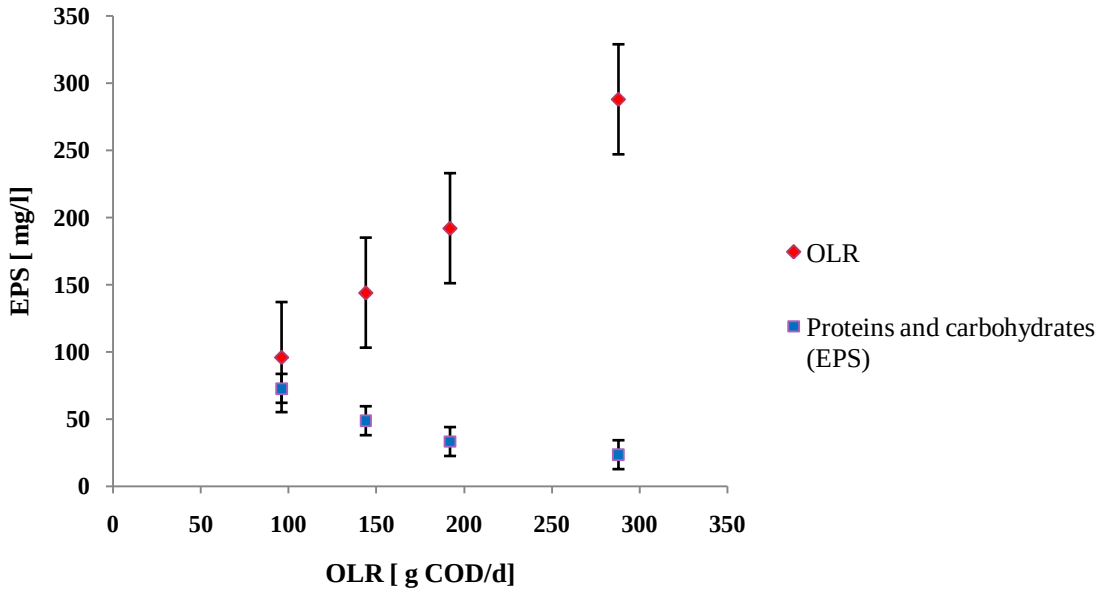


Figure 4.12: Effect of OLR on effluent EPS

The analysis of Figure 4.12 indicates that despite the increasing loading rates, the EPS did not follow a similar pattern with the organic loading rates. The increase of OLR did not have any effect on the EPS during the treatment process. Therefore, there was no relationship between the increasing OLR and the EPS in this particular case. This suggests that EPS are only produced in the pit latrine toilets and they are degraded in the reactor during the treatment of VIP sludge. This is supported by the data presented in Table 4.12 and discussion provided in section 4.7.1.

4.9.3 Correlation between OLR and effluent pH

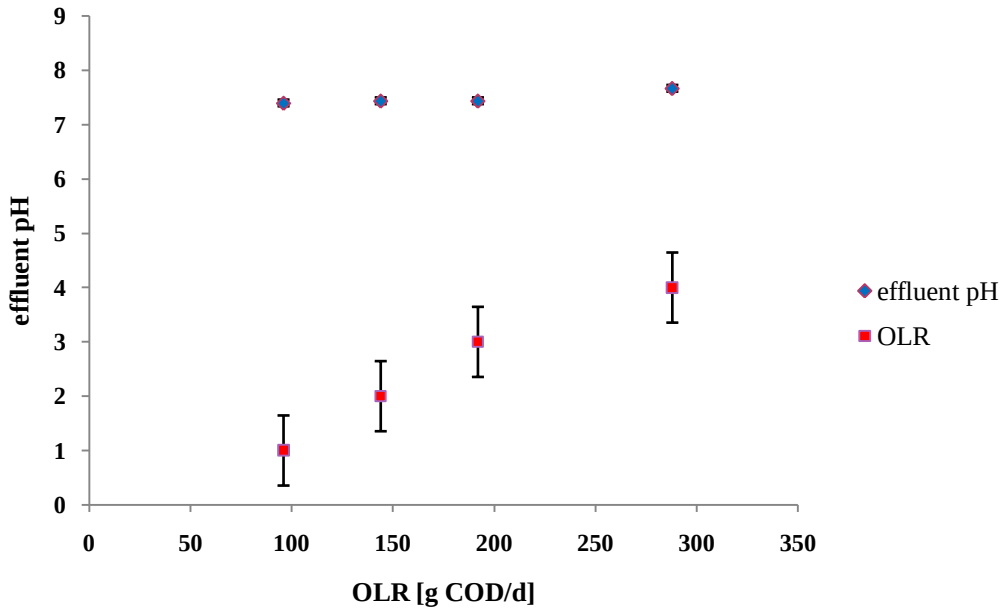


Figure 4.13: Effect of OLR on effluent pH

Figure 4.13 shows that the pH of the effluent was not affected by the increase of the organic loading rates. Median values of the effluent pH remained almost constant even though the organic loading rates were increasing from one run to another. In conclusion, increasing the organic loading rates did not have an effect on the effluent pH during the treatment process. There is no relationship between the loading rates and the pH. The only parameter that could have an effect on the pH is the alkalinity.

4.9.4 Correlation between OLR and effluent conductivity

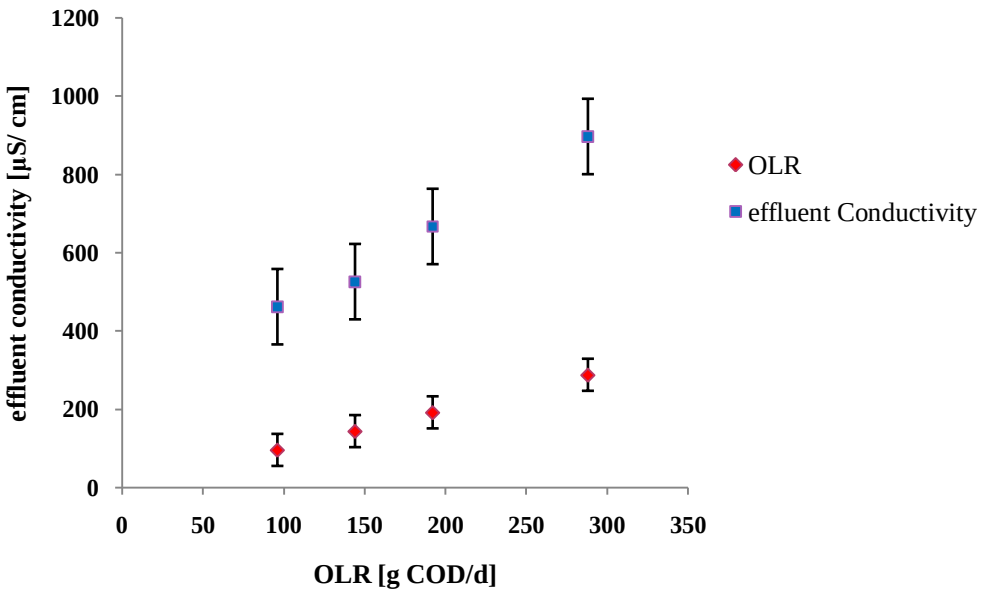


Figure 4.14: Effect of OLR on effluent conductivity

From the analysis of Figure 4.14, the increasing organic loading rates have a direct influence on the effluent conductivity. As the loading rates were increased from one run to another, total dissolved solids also were increasing in the system. As a result, the effluent conductivity was higher than the feed conductivity as mentioned before. It was observed that the effluent conductivity followed a similar pattern with the feed COD because there is relationship between the OLR and the feed COD (see equation 3-3). It can be concluded that there is a strong relationship between the loading rates and the effluent conductivity.

4.9.5 Correlation between OLR and effluent total solids (TS)

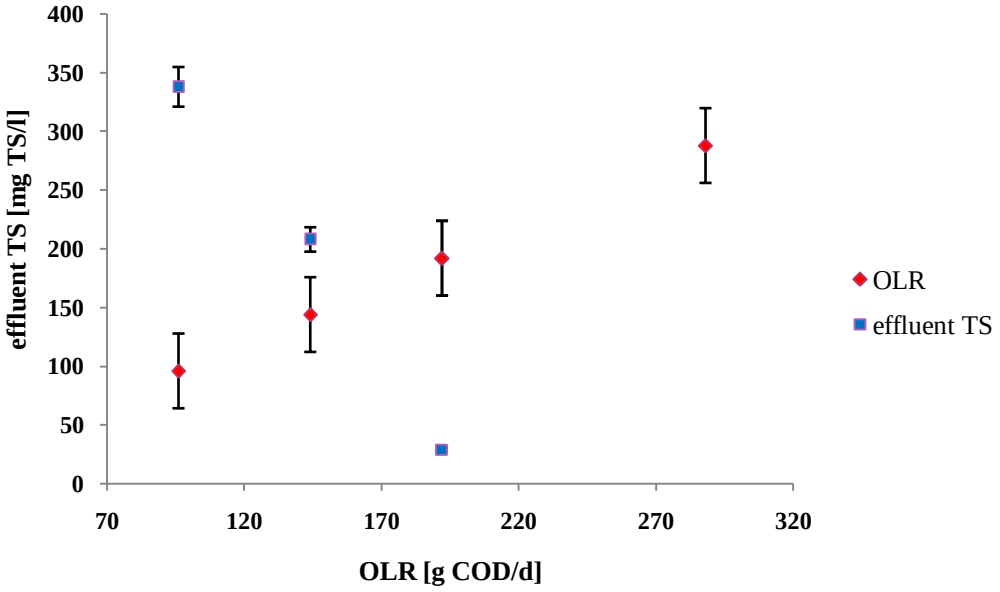


Figure 4.15: Effect of OLR on effluent total solids (TS)

Figure 4.15 indicates that despite the increasing loading rates, total solids are decreasing from the feed to the effluent. This is due to the high solids retention capacity of the ABR. This supports the fact that large amount of solids was retained (mostly by settling) in the reactor during the treatment process.

CHAPTER 5:

CONCLUSIONS AND RECOMMENDATIONS

A four - compartment laboratory ABR treating complex particulate wastewater from VIP sludge was investigated for a period of 264 days. The main focus of this study was to understand the effect of the increasing loading rates on the effluent characteristics. The reactor operated from run 1 to run 6, under a constant HRT of 3 days at room temperatures. The organic loading rates were increased from one run to another. With the inlet COD ranging from 1000 to 3000 mg COD/l, the ABR produced an effluent with an average COD ranging from 303 to 1439 mg COD/l. This equated to COD removal efficiencies between 52 and 80% for the system including the feed tank and the compartments. COD removal was achieved through solids retention and digestion mostly in the feed tank. The reactor retained large amount of solids during the operation with more than 80 % of solids removal efficiency.

The generated average alkalinity was very low; the type of treated wastewater did not generate high alkalinities. This was probably due to insufficient production of bicarbonate or ammonia cations indispensable for alkalinity production. This low alkalinity generated by the system caused a slight drop of pH from the inlet to the outlet ranging between 8.9 and 7.

Due to the production of dissolved ionic substances during digestion, the recorded effluent conductivities were higher than the feed conductivities. Therefore, with effluent COD recorded from run 1 to run 4 being below the standard for irrigation (400 mg COD/l and a pH above 7), this effluent can serve for irrigation once pathogens are removed by the membrane system in the ABR.

However, despite the increasing organic loading rates, it was recorded that most COD was retained by the system through solids retention and a smaller amount of COD was destroyed (through digestion), consequently, less biogas was produced. This is supported and validated by COD mass balance obtained by calculation: 46 % of COD was retained within the sludge while 24 % was taken by effluent, and only 28 % of COD was lost as biogas (which represents the

biodegradability of the VIP sludge): it suggests a low biodegradability of VIP sludge. This low biodegradability is an indication that the VIP sludge was already fairly well stabilized before the feeding of the reactor. Furthermore, extrapolymeric substances (EPS) were found to be the major cause of membranes fouling in a parallel project. The data showed that less EPS were recorded in the effluent than in the feed despite the increasing loading rates. Normally, effluent EPS were expected to be higher than feed EPS. It implies that the system was unable to produce EPS. Therefore, this was an indication that low biological activity was taking place in the reactor.

These outcomes suggest the following for this study:

- An incorrect experiment design: the research team suggested (in 2007) that by using VIP sludge because of the lack of domestic wastewater from the municipality, comparative results can be recorded. This was not realistic because VIP sludge is fundamentally different from domestic wastewater (in terms of physical appearance and biodegradability) and the outcomes should be different.
- A weakness in the equipment design: large amount of solids settled in the feed tank. Consequently, clogging and solids washout occurred during the operation. Desludging was the only option applied to avoid clogging and solids washout. Mixing was not part of the design.

This led to the conclusion that the equipment design did not fit the experiment design.

Based on this conclusion, the following is recommended:

- Because the experiment design did not fit the equipment design, it is suggested to use blackwater from waterborne sewage instead of VIP sludge as a feed. This approach is more practical and will provide more information on the ability of the technology to treat blackwater from waterborne sewage and the effluent type produced by the ABR under known operating conditions.
- Further analyses such as free and saline ammonia and total Kjeldahl nitrogen (TKN) tests should be undertaken on the feed and effluent flow. Both tests will be valuable for COD mass balance in providing more clarity and understanding on COD removal mechanisms.

- Additional measurements of sludge levels should be recorded for the COD mass balance.

Overall, despite the fact that the experimental concept was poorly designed by the team in 2007, the ABR could potentially be used as sanitation option in low income communities for the pre-treatment of sewage with higher solids retention and COD removal efficiencies as it was recorded in this study and evidenced in other studies [Foxon et al., (2005) and Anh et al., (2003 and 2007)]. Also, this work has provided an independent assessment of an overall biodegradability of VIP sludge which is of use for proposing sludge management strategies. In this regard, anaerobic digestion is not the most appropriate method for treating VIP sludge.

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APPENDIX 1

A1. ANALYTICAL METHODS

Analytical methods were carried out according to standard methods (APHA, 1988).

A 1.1 CHEMICAL OXYGEN DEMAND (COD)

Total COD for the influent and effluent was measured in line with the open reflux method (APHA 1988); soluble COD samples were prepared by centrifuging samples in a Centrifuge HERMLE model Z323 for 15 minutes at 15000 rpm. The COD measures the oxygen equivalent of that portion of the organic matter in a sample that is easily oxidized by a strong chemical oxidant. It is an important and rapidly measured parameter for stream an industrial waste studies and in operational control of wastewater treatment plants.

A1.1.1 Apparatus

- Heating block.
- Condensers.
- 250 ml Erlenmeyer flasks.
- 10 ml pipette.
- 15 ml and 5 ml automatic pipette.

A1.1.2 Reagents

Potassium Dichromate $K_2Cr_6O_7$

- Dry some standard potassium 0.250 N (0.0417 M) in the oven at 103 ^oC for 2 hours.
- Cool in desiccator.
- Weigh out 12.2588 g.
- Dissolve in 1000 ml volumetric flask.
- Mix thoroughly.

Sulphuric Acid H_2SO_4 and Silver Sulphate Ag_2SO_4

- Add 26 g of silver sulphate crystals or powder to 2.5 l of concentrated sulphuric acid using a magnetic stirrer.
- Shake well and leave for 2 days for dissolution.

Mercuric Sulphate $HgSO_4$

This reagent is used to remove chlorides which give a higher COD result, 0.04 g crystal or powder is used for the analysis.

Ferrous Ammonium Sulphate (FAS): $Fe(NH_4)_2(SO_4)_2.6H_2O$

- Dissolved 98 g of ferrous ammonium sulphate in distilled water.
- Add 20 ml concentrated sulphuric acid.
- Cool and dilute to 1000 ml (approximately 0.25 M).
- Dilute 25 ml standard $K_2Cr_2O_7$ to 100 ml.
- Add 30 ml of concentrated sulphuric acid and cool.
- Titrate with ferrous ammonium sulphate titrant using 3 drops of ferroin indicator.

Ferroin indicator

Dissolve 1.485 g of 1:10 phenentroline monohydrate and 0.695 g ferrous sulphate ($Fe\ SO_4.7H_2O$) in distilled water.

Standard preparation

- Pipette 5 ml potassium dichromate into an Erlenmeyer flask.
- Dilute to 5o ml with distilled water.
- Add 15 ml of concentrated sulphuric acid.
- Cool and titrate against ferrous ammonium sulphate (FAS) with 2-3 drops of ferroin indicator.

A1.1.3 Calibration

- Prepare a standard $K_2Cr_2O_7$ solution daily to correct any variation in the concentration of ferrous ammonium sulphate.
- Prepare a blank with each set of samples consisting of 10 ml distilled water in place of sample together with all the reagents and digest together with samples.
- Quality control : Potassium hydrogen Phthalate (KHP)

  -Light crush and then dry KHP to a constant weight at 120 ^{o}C

  -Dissolve 0.425 g in distilled water and dilute to 1000 ml. This solution has theoretical COD of 500 mg /l.

  The solution is stable when refrigerated up to 3 months in the absence of biological growth.

A1.1.4 Procedure

Sample preparation

- Add approximately 0.04 g 9 (2 match heads) of mercuric sulphate to a dry 250 ml Erlenmeyer flask.
- Add 5 glass beads.
- Add 10 ml sample (if 2 ml sample used add 8 ml distilled water).
- Add 10 ml distilled water to another flask (blank).
- Add 5ml sulphuric dichromate.
- Add 15 ml sulphuric acid reagent (with silver sulphate).
- Pour acid down the wall of the flask while flask is tilted (if sample is too concentrated then it turns green, a low volume must be used).

Heating block (Digestion)

- Switch the heating block on one hour prior to testing. The temperature of the solution in the boiling tube must be stable at 145 ^{o}C. Stability is checked during analysis by immersion of suitable thermometer into the sample, but not touching the sides of the tube. The digestion temperature setting is then adjusted as necessary.
- Carefully attach flask to the jacket condenser. Flasks must be leveled on heating pad.

- Digest samples for two hours .Ensure that the water flow rate in the condensers are swift.
- Cool samples with the condensers still in position.
- Pour approximately 80 ml distilled water through the top opening of each of the condensers into the sample mixture.

Titration

- Titrate the excess dichromate in the digest mixture with standard ferrous ammonium sulphate using 3 drops of ferroin indicator.
- Titrate from a sharp green / orange to red brown end point.
- Take reading.

Calculation

$$\text{COD as mg } O_2 \text{ / l} = \frac{(\text{Blank} - \text{Titration}) \text{ x molarity of FAS x } 8000}{\text{Sample volume[ml]}} \qquad \text{(A1- 1)}$$

Where:

8000 = milliequivalent weight of oxygen x 1000 ml /l

$$\text{Molarity of FAS} = \frac{Volume\ 0.04167\ M\ K2Cr2o7\ solution\ titrated\ [ml]}{Volume\ FAS\ used\ in\ titration\ [ml]} \qquad \text{(A1-2)}$$

A1.2 TOTAL SOLIDS (TS)

Total solids are determined in a wide variety of liquid and semi-liquid materials. These include potable waters, domestic and industrial waters, polluted waters and sludge produced from treatment processes. It is of particular importance for the efficient operation of a treatment plant. ‘’Total solids’’ is a term applied to material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature (103-105 ^{o}C).

A 1.2.1 Apparatus

- 50 ml capacity evaporating porcelain crucibles
- Desiccator
- Drying oven
- Analytical balance

A 1.2.2 Reagents

None

A1.2.3 Calibration

- Check the temperature throughout the oven area by placing a calibrated thermometer on each shelf, after 30 minutes, check temperature at each level against oven setting.
- Adjust oven setting if necessary.
- If temperatures are uneven on the shelves, check insulation.

A1.2.4 Sampling

- Mix the sample well to suspend solids uniformly.
- Remove the test portion rapidly before any settling of solid matter occurs.
- Use a measuring cylinder and not a pipette.
- Use a volume of sample to ensure a measurable residue.
- Suitable aliquots: liquid samples: 100 ml, sludge: 30ml.

A1.2.5 Procedure

Preparation of crucible

- Heat a porcelain crucible in an oven for 2 hours at 103- 105°C.
- Cool for 15 minutes in a desiccator
- Weigh the crucible : W1

Sample analysis

- Measure out appropriate volume (30 ml) of a well mixed sample using correct volume measuring cylinder. Transfer quantitatively to the weighed crucible, rinsing the cylinder with small volumes of distilled water to dislodge heavy particles. Add washings to the crucible.
- Place in hot oven at 103-105°C overnight.
- Remove the next day and cool for 15 minutes.
- Weigh the crucible with residue after cooling : W2

Calculation

$$\text{Total solids in a sample [mg /l]} = \frac{(W2-W1)g\ x\ 1000000}{Sample\ volume} \qquad \text{(A1-3)}$$

A1.3 VOLATILE SOLIDS (VS)

The residue from the total solids method is ignited to a constant weight at 550 ^{o}C. The remaining solids represents he fixed total, dissolved or suspended solids while the weight lost on the ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough estimate of the amount of organic matter present in the solid fraction of wastewater, activated sludge and industrial wastes.

A1.3 .1 Apparatus

- Muffle furnace
- Desiccator
- Analytical balance

A1.3.2 Reagents

None

A1.3.3 Procedure

- Ignite residue from the total solids to constant weight in a muffle furnace at a temperature of 550 ^{o}C.
- Have furnace up to temperature before inserting sample.
- Usually on hour for VIP and sludge samples.
- Let the crucible cool partially in air until most of the heat has dissipated
- Transfer to a desiccator for final cooling. Do not overload the desiccator.
- Weigh dish as soon as it has cooled to balance temperature.

Calculation

$$\text{Volatile solids [mg/l]} = \frac{(A-B)x\ 1000000}{Sample\ volume[\ ml]} \qquad \text{(A1-4)}$$

Where A: weight of residue + dish before ignition [g]

B: weight of residue + dish after ignition [g]

A1.4 ALKALINITY

Alkalinity test was using a method validated by eThekwini municipality water and sanitation laboratory. In this method, Hydroxyl ions present in the sample react with addition of standard sulphuric acid at 0.02 N. Alkalinity thus depends on the end-point pH used. For samples containing more than 150 mg $CaCO_3$/l and for samples known or suspected to contain phosphates or silicates, pH 4.5 is suggested as the equivalence point.

A1.4.1 Reagents

0.02N Sulphuric Acid (M/100)

- Dissolve 3ml of concentrated H_2SO_4 in distilled water and diluted to 1l. This is approximately 0.1**N**.
- Accurately weigh 1.325g of anhydrous Na_2CO_3, previously dried at 270^oC and dissolve it with distilled water up to 250 ml in a volumetric flask. This is 0.10 N.
- Dilute the H_2SO_4 solution 5 times to bring it to 0.02 **N** (**N**/5), 1 ml of 0.02N H_2SO_4 = 1mg of $CaCO_3$.

Mixed Bromocresol green – Methyl red Indicator solution

- Mix 0.2g of Bromocresol green and 0.4g of methyl red in 120 ml of 95% ethyl alcohol.
- To determine normality, titrate H_2SO_4 against 25ml of Na_2CO_3 solution using Bromocresol green and Methyl red mixed indicator.
- Calculate the normality of H_2SO_4 using the following equation:

$$N_{ACID} \times V_{ACID} = N_{BASE} \times V_{BASE} \qquad \text{(A1-5)}$$

A1.4.2 Procedure

- Add 50 ml of the sample in an Erlenmeyer using a measuring cylinder.
- Add 2-3 drops of mixed indicator to the sample.
- Titrate with 0.02N Sulphuric acid and observe the color change from greenish blue to pink.

$$\text{Alkalinity (mg/l as CaCO3)} = \frac{A\, x\, \frac{N}{5}\, x\, 50000}{\text{Sample volume[ml]}} \qquad \text{(A1-6)}$$

Where A = ml of diluted acid used for titration.

$\frac{N}{5}$ = 0, 02 (acid normality)

APPENDIX 2

A 2. RATIO OF TWO MEANS: FIELLER'S THEOREM

This theorem is used for the establishment of the confidence interval where the ratio of two means a and b can be included at a significance level α with *n* φ degrees of freedom, and t the appropriate t-statistic (Davies and Goldsmith, 1977 p.236)

$$\frac{\frac{a}{b} - \frac{t^2 C(a,b)}{b^2} \pm \frac{t}{b}\sqrt{V(a) - \frac{2a}{b}C(a,b) + \frac{a^2}{b^2}\, v\,(b) - \frac{t^2}{b^2}\frac{V(b)}{b^2}[v(a) - \frac{C(a,b)^2}{V(b)}]}}{1 - \frac{t^2}{b^2}V(b)}$$

For C (a, b) = 0 and the above expression becomes:

$$\frac{\frac{a}{b} \pm \frac{t}{b}\sqrt{V\,(a) + \frac{a^2}{b^2}\,V\,(b) - \frac{t^2}{b^2}V(b).V(a)}}{1 - \frac{t^2}{b^2}V(b)} \qquad \text{(A2−1)}$$

APPENDIX 3

A 3. SAMPLE ANALYSIS OF AN ABR EFFLUENT

Table A3.1 presents a data from the analysis of an effluent sample taken during the operation of the laboratory -scale ABR in July 2009 by Bame (2009).

Table A 3.1: Characterization of an ABR effluent (Bame, 2009)

	Ist characterization	**2nd characterization**
Elements	**mg.l^{-1}**	**mg.l^{-1}**
Nitrate-Nitrogen	-0.271	-0.102
Ammonium- Nitrogen	14.35	14.13
Phosphorus	25.287	24.083
Potassium	8.551	8.150
Sulphur	6.6032	6.427
Calcium	18.9261	19.802
Magnesium	26.3094	24.791
Sodium	32.56447	30.834
Aluminium	0.0826	0.001476625
Cadmium	-0.0049	-0.009293978
Cobalt	-0.0580	-0.024565142
Chromium	0.0134	0.007073153
Copper	-0.0076	-0.001349286
Iron	0.2870	0.235977985
Manganese	0.0027	0.146378465
Molybdenum	0.0036	0.04119
Nickel	0.0087	-0.003018136
Lead	0.02751	-0.014378196
Selenium	0.06408	0.046676133
Vanadium	0.01188	0.01753394
Zinc	-0.01769	0.035130141
Boron	-1.29906	0.20767428

Table A3.1 provides a feedback on the quality of effluent generated by the ABR during its operation. The analyses of effluent indicate for both characterizations that nutrients such as ammonium-nitrogen, phosphorus are potentially useful for agricultural purposes. Compounds such as sodium, magnesium and calcium are also important in agricultural purpose as secondary elements while heavy metals are insignificant but also in normal range for agriculture purposes.

APPENDIX 4

A4. DETAILS OF SAMPLE CALCULATIONS FOR COD MASS BALANCE

The COD mass balance for the ABR section of ABR-MBR unit can be described as follows:

$$COD_{inflow} - COD_{outflow} - COD_{accum.sludge} - COD_{Biogas} = 0 \qquad \text{(A4-1)}$$

With COD_{inflow}: The total COD at the inlet.

$COD_{outflow}$: The total COD at the outlet.

$COD_{accum.sludge}$: The accumulated COD of the sludge.

COD_{biogas} : Biogas COD, the destroyed COD.

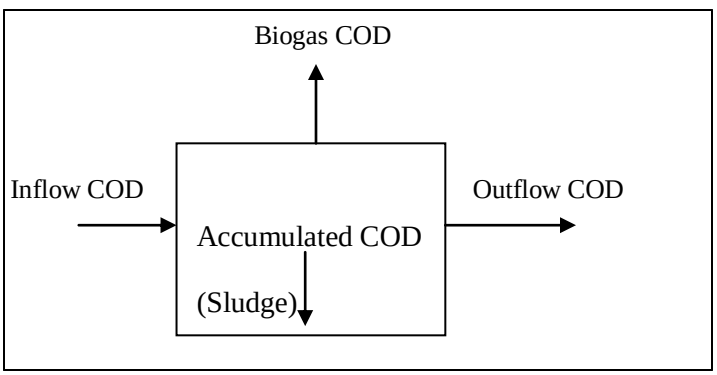


Figure A4.1: COD distribution in ABR

These CODs were determined as follows:

COD inflow= ∑ (average COD inflow x Flow rate x Time) [kg COD] (A4-2)

COD effluent =∑ (average COD outflow x Flow rate x Time) [kg COD**]** (A4-3**)**

COD accumulated sludge= estimated sludge COD x estimated total volume of sludge [kg COD] (A4-4)

COD biogas = COD inflow-COD effluent-COD sludge [kg COD] (A4-5)

The estimated inflow and outflow CODs are calculated from the data presented in Tables 4.2 and 4.4.

A4.1 INFLOW COD

- **Estimated inflow COD**= [average COD (run1) x average flow rate (run1) x time (run1)] + [average COD (run 2) x average flow rate (run 2) x time (run 2)] + [average COD (run 3) x average flow rate (run 3) x time (run 3)] + [average COD (run 4) x flow rate (run 4) x time (run 4)] + [average COD (run 5) x flow rate (run 5) x time (run 5)]+ [average COD (run 6) x flow rate (run 6) x time (run 6)]

- **Estimated inflow COD** = [1.561 g COD /l x 106 l/d x 124 d] + [1.033 g COD/l x 96 l/d x 36 d] + [1.5 g COD/l x 103 l/d x 29 d] + [2 g COD/l x 96 l/d x21 d] + [3 g COD/l x 91 l/d x 33 d] + [2 g COD/l x 96 l/d x 21 d] = 45641 g COD= **46 kg COD**

A4.2 OUTFLOW COD

- **Estimated effluent COD**= [average COD (run1) x average flow rate (run1) x time (run1)] + [average COD (run 2) x average flow rate (run 2) x time (run 2)] + [average COD (run 3) x average flow rate (run 3) x time (run 3)] + [average COD (run 4) x flow rate (run 4) x time (run 4)] + [average COD (run 5) x flow rate (run 5) x time (run 5)]+ [average COD(run 6) x flow rate (run 6) x time (run 6)]

- **Estimated outflow COD**= [0.303 g COD/l x 106 l/d x 124 d] + [0.309 g COD/l x 96 l/d x 36] + [0.334 g COD/l x 103 l/d x 29d] + [0.457 g COD/l x 96 l/d x 21 d] + [1.4 g COD/l x 91 l/d x 33 d] + [0.526 g COD/l x 96 l/d x 21 d] = 12230 g COD = **12 kg COD**

A4.3 ESTIMATED COD FOR ACCUMULATED SLUDGE

- **Estimated volume of sludge**

The breadth and length of each compartment are 150 mm and 445 mm respectively.

The sludge heights (or levels) recorded on the 1st April 2009 from each compartment are:

- Compartment 1: 210 mm
- Compartment 2 : 110 mm
- Compartment 3 : 60 mm
- Compartment 4: 40 mm

These sludge heights are used to calculate the volume of sludge in each compartment. The estimated volume in each compartment is expressed as follows:

Estimated volume in each compartment = height of sludge x breadth x length (A4-6)

Using the data from Table 4.3 and the equation (A4-6), the estimated volume in each compartment will be:

- Estimated volume of sludge in compartment 1= 0.210 m x 0.150 m x 0.445 m = 0.014 m^3= 14 l
- Estimated volume of sludge in compartment 2 = 0.110 m x 0.150 m x 0.445 m = 0.007 m^3 = 7 l
- Estimated volume of sludge in compartment 3 = 0.060 m x 0.150 m x 0.445 m = 0.004l m^3 = 4 l
- Estimated volume of sludge in compartment 4 = 0.040 m x 0.150 m x 0.445 m = 0.00267 m^3 = 2.7 l
- **Total Estimated volume of sludge within compartments** = 14 l+7 l+4 l+2.7 l = **27, 7 l**

Also, desludging was undertaken during the experiment to avoid solids washout in the reactor. 120 l of sludge was removed on day 138, 135 l of sludge on day 207 and 80 l of sludge on 31st March 2009 (last day of run 6). Therefore, the **total estimated volume of sludge** is: 27, 67 l + 120 l + 135 l + 80 l =**362.67 l of sludge.**

- **Estimated sludge COD**

On day 216 a sample of sludge taken from the bottom of the feed tank was analyzed. The total COD of the sample was **57370.03** mg COD/l (or 57 kg COD/m^3). This value indicates the average COD for sludge and is used to determine the estimated COD of accumulated sludge. Also, this value is closer to the conversion value found by Foxon (2009) which was 60 kg COD/m^3. Therefore, the estimated COD of the accumulated sludge is calculated according to the equation A4-4.

- **Estimated COD of accumulated sludge**= 57.37 g COD/l x 362.67 l = 20806 g = **21 kg COD**

From the equation A4-5 biogas COD is determined as follows:

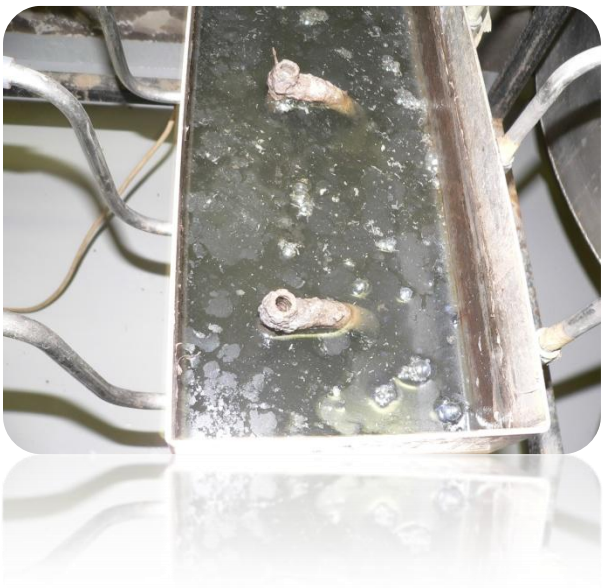
- **Estimated COD of biogas** = 46 kg- 12 kg -21 kg = **13 kg COD**

APPENDIX 5

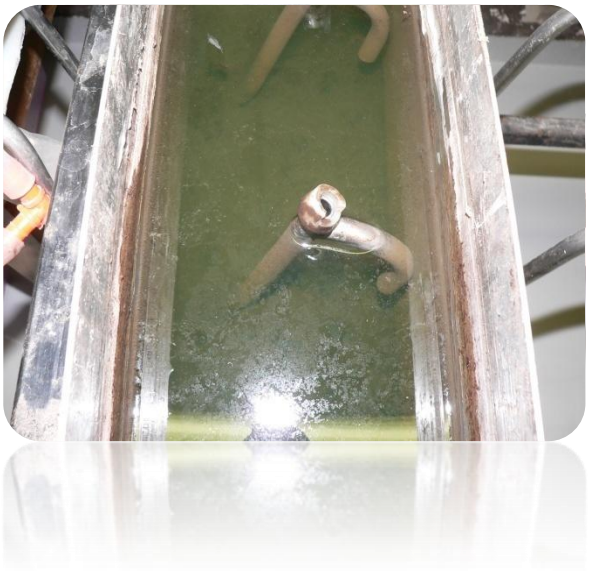
Photograph A 5.1: Feed tank during the feeding of the reactor

Photograph A5.2: ABR feed (left) and effluent (right)

Photograph A 5. 3: ABR feed (left) and effluent from MF and UF membranes (ABR-MBR)



Photograph A5.4: First compartment



Photograph A5.5: Last compartment