# The Anaerobic Baffled Reactor for Sanitation in Dense Peri-Urban Settlements

by

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### Abstract

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Human consumption of water contaminated with faecal pollutants is the source of most sanitation related diseases. Excreta related diseases can be controlled by improvements in excreta disposal. The primary consideration is to remove contact between the people and the faecal matter. The conventional waterborne sewage system is not an achievable minimum standard in dense peri-urban areas in the short term, due to its high cost. A need for a cost effective system that is easily maintained and does not require electricity or highly skilled labour for developing communities in South Africa was identified. The objective of this investigation was to assess the suitability of the Anaerobic Baffled reactor (ABR) as a primary onsite treatment system for low-income communities.

The ABR is a high-rate compartmentalised anaerobic bioreactor, the design of which promotes the spatial separation of microorganisms. The trials were conducted on a 3200 L pilot-scale reactor placed at Kingsburgh wastewater treatment works, which receives only domestic wastewater. The ABR proved to be stable and consistent in its performance. Operating at a hydraulic retention time of 22.5 h, the reactor effluent was ca. 200 mgCOD/L. The 0.45µm filtered (soluble) COD was 100 mg/L, indicating there was approximately 100 mg/L of COD in the effluent that was in particulate form. The ABR achieved 60%VSS and 50%TSS removal with effluent TSS content of about 225 mg/L. The system was hydraulically overloaded and organically under loaded. The Biochemical Methane Potential tests showed that 60% of the COD in the effluent was biodegradable, and the effluent COD could be reduced to less than 100 mgCOD/L if the HRT is increased giving a possible removal of 80%. The analytical campaign revealed that we were sampling at peak flow, when COD was high. The average COD fed to the reactor was much lower than that showed by routine analysis and the ABR had a "true" COD removal of 42%. The reactor was able to handle the daily variation of the wastewater.

Settling tests were done to measure how much of the suspended solids in the ABR are retained at the operating upflow velocity. The method selected was shown to have an error that ranged from 5 to 42%, and the ABR was retaining between 60 and 90% of solids in the reactor at an upflow velocity of 0.5m/h.

The preliminary work with the fabric membrane showed great potential benefits that can be gained if it had to be included. It showed good ability to remove indicator organism and solids that contributed a lot to the effluent COD. The membrane had 5 log removal of indicator organism and 80% reduction of COD. The membrane was operated for a short time before clogging; its operational lifespan needs to be greatly extended before it can be used with the reactor in a community. Since there is no nutrient removal in the ABR, the effluent can be used for food production provided sufficient pathogens removal is achieved.

Provided that the first compartment can be modified and the concentration of pathogens in the effluent is sufficiently reduced, the ABR can be considered for use in a community.

628.354 MTE

### **Declaration**

I hereby declare that all the work submitted within this thesis except where specifically acknowledged is mine. This dissertation has not been submitted in whole or in part for a degree at another university or institution.

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## **ABBREVIATIONS**

AA	Amino Acids
ABR	Anaerobic Baffled Reactor
BOD	Biochemical Oxygen Demand
BPD	<b>Business Partners for Development</b>
c.f.	carried forward/ refer to
ca.	circa/about
CFD	Computational Fluid Dynamics
COD	Chemical Oxygen Demand
CSTR	Continuously Stirred Tank Reactor
DWAF	Department of Water Affairs and Forestry
FISH	Fluorescent in situ hybridisation
GC	Gas Chromatograph
HAc	Acetic acid
HBu	Butyric acid
HPr	Propionic acid
HRT	Hydraulic Retention Time
HVa	Valeric acid
LCFA	Long chain fatty acids
MS	Monosaccharides
MW	Molecular Weight
NGO	Non-Governmental Organisation
<b>OFN</b>	Oxygen Free Nitrogen
OLR	Organic Loading Rate
RBCOD	Readily Biodegradable COD (mg/l)
RHCOD	Readily Hydrolysable COD (mg/l)
RTD	Residence Time Distribution

SMP	Soluble Microbial Products
Soln	Solution
SRB	Sulphate Reducing Bacteria
SRT	Solids Retention Time
STP	Standard Temperature and Pressure
SVI	Sludge Volume Index
TC	Total Carbon
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TS	Total Solids
TSS	Total Suspended Solids
UASB	Upflow Anaerobic Sludge Blanket Reactor
VFA	Volatile Fatty Acids
VIP	Ventilated Improved Pit-latrine
VS	Volatile Solids
VSS	Volatile Suspended Solids
WRC	Water Research Commission

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## **GLOSSARY**

Acclimation	The adaptation of a microbial community to degrade a <i>recalcitrant</i> compound through prior exposure to that compound				
Adaptation	A change in the microbial community that increases the rate of transformation of a test compound as a result of acclimatisation to that test compound				
Adenosine triphosphate	Energy rich molecule				
Aerobic bacteria	Bacteria that use $O_2$ as the terminal electron acceptor				
Agglomerate	Solids or particles collecting into a coherent body of matter or mass				
Anabolism	The biosynthesis of new cellular material				
Anaerobic bacteria	Microorganisms capable of growing or metabolising in the absence of oxygen. The microorganisms may be facultative or obligate; the latter will perish in the presence of free oxygen.				
Anoxic	An environment where oxygen is present in the form of a chemical compound (e.g. bacteria use $NO_3$ as the terminal electron acceptor)				
Batch reactor	a reactor in which there is no flow of substrate or bacteria in or out of the reactor, and the concentration of substrate and bacteria vary with time				
Biodegradable	Capable of being decomposed by bacteria or other living organisms into simpler organic compounds or molecules				
Biodegradation	Breakdown of compounds by biologically mediated reactions				
Biomass	Bacterial cells				
Blanket (sludge)	a separate-more or less-fluid phase with its own specific characteristics				

COD (chemical oxygen demand)	A measure of the total amount of oxidisable material in that sample.
Fermentation	Breakdown of amino acids and sugars by microorganisms to alcohol, acetic acid, propionic acid and other intermediary products in the absence of oxygen
Flocculation	Occurs when particles aggregate resulting in change of size and settling rate
Grit	Heavy mineral matter associated with wastewater e.g. sand
Hybrid reactor	Combines properties of both the sludge blanket and the upflow anaerobic filter configurations
Inhibition	An impairment of the bacterial function
Medium	A mixture of nutrient substances required by cells for growth and metabolism.
Organic Loading rate	Measure of the organic content of the feed in relation to
	reactor volume (mass load per unit time per reactor-volume)
Pollution	reactor volume (mass load per unit time per reactor-volume) An adverse alteration of the environment
Pollution Retention time	reactor volume (mass load per unit time per reactor-volume) An adverse alteration of the environment Average period of time that the incoming matter is retained in the reactor for completion of the biological reactions, calculated by dividing the reactor volume by the incoming flow
Pollution Retention time Sanitation	reactor volume (mass load per unit time per reactor-volume) An adverse alteration of the environment Average period of time that the incoming matter is retained in the reactor for completion of the biological reactions, calculated by dividing the reactor volume by the incoming flow Is the maintenance or improvement of disposal of sewage and refuse from households
Pollution Retention time Sanitation Screen	reactor volume (mass load per unit time per reactor-volume) An adverse alteration of the environment Average period of time that the incoming matter is retained in the reactor for completion of the biological reactions, calculated by dividing the reactor volume by the incoming flow Is the maintenance or improvement of disposal of sewage and refuse from households Device for the removal of large solids from the wastewater
Pollution Retention time Sanitation Screen Scum	<ul> <li>reactor volume (mass load per unit time per reactor-volume)</li> <li>An adverse alteration of the environment</li> <li>Average period of time that the incoming matter is retained in the reactor for completion of the biological reactions, calculated by dividing the reactor volume by the incoming flow</li> <li>Is the maintenance or improvement of disposal of sewage and refuse from households</li> <li>Device for the removal of large solids from the wastewater</li> <li>Layer of fat and oils and gas bubbles which floats on a liquid surface</li> </ul>

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Suspended solids

Veld

Volatile fatty acids

Volatile solids

Wastewater

Un-dissolved non-settleable solids present in wastewater or sludge

open grassland or country

Short-chain organic acids produced in the anaerobic digestion process

Organic solids which are lost on ignition at 550 °C

General term to denote a combination or mixture of domestic sewage and industrial effluents

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## NOMENCLATURE

Ks	Substrate saturation constant (mol/L)
K <sub>m</sub>	Monod constant(mol/L)
V	Volume (m <sup>3</sup> )
$X_f$	Mass fraction of component in faeces (g/L)
Xu	Mass fraction of component in urine(g/L)
OLR	Organic Loading Rate (KgCOD/m <sup>3</sup> .d)
$\mu_{max}$	Maximum specific growth constant ( $d^{I}$ )
V <sub>s</sub>	Settling velocity (m/h)

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# Chapter 1 Introduction

In the World Health Organisation's 2002 report, *Reducing Risk Promoting Healthy Life*, diseases related to water and sanitation are in the top three causes of death and disability for developing countries. The diseases are associated with ingestion of unsafe water, lack of access to water (linked to inadequate hygiene), lack of access to sanitation, and inadequate management of water resources and systems, including in agriculture. Infectious diarrhoea makes the largest single contribution to the burden of disease associated with unsafe water and hygiene. In addition, schistosomiasis, trachoma, ascariasis, trichuriasis and hookworm disease were fully attributed to unsafe water. Approximately 1.7 million deaths and 54.2 million cases of illness worldwide are attributable to the consumption of unsafe water (WHO, 2002). Overall, 99.8 % of deaths associated with this risk factor are in developing countries, and 90 % are deaths of children.

Sanitation is the maintenance or improvement of disposal of sewage and refuse from households. Major advances in public health in the developed countries involved the reduction or the elimination of risk associated with drinking poor quality water. Improvements in drinking-water supplies and sanitation during the nineteenth and twentieth centuries were directly related to the control of the organisms that cause these illnesses.

### 1.1. Environmental health engineering

Environmental health engineering uses engineering methods for the improvement of the health of the community. In practice, it has come to focus upon domestic water supply and excreta disposal (Cairncross and Feachem, 1996). An infectious disease is one that can be transmitted from one person to another, and some times, transmitted to or from an animal. Organisms such as bacteria, viruses, and parasitic worms cause infectious diseases. The diseases are transmitted by the passing of the organisms, from one person to another. During the transmission process the organism may be exposed to the environment. So the *safe* passage of the organism to a new victim is vulnerable to environmental changes. Environmental engineering therefore seeks to modify the human environment in such a way as to prevent or reduce the transmission of infectious diseases.

All diseases in the faecal-oral category, as well as most of the water-based diseases, and several others not related to water, are caused by pathogens found in human excreta. The excreta-related diseases, which are also water-related, can be controlled at least partially by improvements to water supply and hygiene. The excreta related diseases might be controlled by improvements in excreta disposal. These range from the construction or improvements of toilets, choice of excreta transport, treatment, re-use and final disposal.

A water-related disease is one, which in some gross way is related to inadequate water supply, limited or inadequate water quantity, poor water quality or impurities in the water (Falkenmark, 1980). Water-bornediseases associated with the contamination of drinking water are mainly caused by excreta related pathogens such as *Entamoeba histolytica* and *Salmonella typhi* (Appendix I). It is necessary to distinguish the infectious water-related diseases from those related to some chemical property in the water. Human consumption of contaminated water, especially water with faecal pollutants, is the source of most sanitation related problems. For an environmental health engineer, it is convenient to classify the relevant infectious diseases into categories that relate to the various aspects of the environment that the engineer can alter (Table 1-1).

Category	Description	Preventative strategies
Water-borne	Transmission occurs when the pathogen is	Improve quality of drinking water.
disease	contained in water which is consumed	Prevent casual use of unprotected
		sources
Water-washed	Transmission from person to person in a	Improve hygiene, accessibility,
disease	domestic environment which might be	reliability and quantity of domestic
	reduced if more water was available	water supply
	· ·	
Water-based	Transmission of a pathogen with an	Reduce need or reasons to get in
disease	obligatory aquatic intermediate host or hosts	contact with infested water
Water-related	Transmission by insects which breed in water	Destroy insect breeding habitat
insect-vectored	or live and bite near water	
disease		

Table	1-1:	Water-related	diseases:	description	and	preventative	strategies
(Falkernamrk, 1980)							

### **1.2.** Waterborne sanitation

The conventional wastewater treatment process is a four-step process (Gray, 1989) consisting of:

- (1) Preliminary treatment: Involves the separating of floating material and the heavy inorganic solids.
- (2) Primary treatment: Sedimentation tanks are used to separate the suspended organic solids from the effluent.
- (3) Secondary treatment: Biological decomposition of the organic matter in the effluent coming from the sedimentation tanks.

(4) Final Treatment: The stage in which pathogenic bacteria is destroyed usually by chlorination. This stage is left out in many cases.

The conventional system requires cistern-flush latrines, a network of underground pipes, pump stations and a treatment works. The high cost of installing such a system is not the only obstacle to providing sanitation in developing communities. It is almost impossible to dig and lay pipes in an unplanned residential area. The scarcity of water in developing countries is another obstacle to the conventional system. South Africa is a water scarce country, and the management of water demand requires a critical analysis on the use of water in the removal and transport of human waste to a place of treatment and final disposal. (Water Rationalisation and Amendment Act No. 32, 1994).

### **1.3.** The state of sanitation in South Africa

In South Africa 3 million house holds or 18 million people do not have adequate sanitation facilities. These people may be using the bucket system, pit-latrines or the *veld*. Nearly half of all schools use ordinary pit latrines and these are often insufficient in number leading to unhygienic and unsafe conditions. An estimated 15 % of all community health clinics are without sanitation and water facilities (DWAF, 2001).

Progress has been hindered by the following factors:

- Sanitation has been a low priority at household and government level
- Limited human capacity and funding have been supplied to address the shortage
- Sanitation is still seen as a programme to provide infrastructure, which limits its full potential impact
- Inadequate understanding and acceptance of the various technical options to solve the problem
- Limited programme management for large-scale community-based implementation
- Inadequate coordination and integration of planning on all levels
- Grant funding programmes are fragmented

In addition to this there is an increase in poorly designed or operated waterborne sewerage systems, especially in urban areas (White Paper on Basic Sanitation, 2001). The Health Department recognised that diseases associated with poor sanitation are diarrhoea, dysentery, typhoid, bilharzia, malaria, cholera, worms, eye infections, skin diseases and increased risk of infection to people living with HIV and AIDS.

Significant investments have been made and are still being made in the provision of safe water for all. However the health benefits of this investment are limited to where adequate attention has been paid to sanitation, health and hygiene promotion. International experience shows that once basic water needs are met, sanitation improvements together with hygiene promotion result in the most significant impact on health (Falkenmark, 1980).

### 1.4. eThekwini pilot shallow-sewer study

eThekwini Water Services (EWS), in a joint venture with Water and Sanitation Services (South Africa) (WSSA) and the Water Research commission (WRC), investigated through a pilot project whether shallow sewers would provide a viable alternative waterborne sanitation system to the urban poor in dense settlements. The shallow sewer has been successfully implemented in Brazil, Greece, Australia, USA, Bolivia, India and Pakistan. The eThekwini Municipality provides three levels of water service. The first is the conventional full-pressure service with no physical restrictions. The second level is the semi-pressure supply system, which is provided at a reduced cost for connection and tariff. The house however, must be fitted with a 200 L roof tank in order to maintain a reservoir of water and the operating pressure. The last level is the 200 L ground tank that is filled once daily thus limiting consumption to 200 L/d (Eslick and Harrison, 2004).

The installation of Ventilated Improved Pit (VIPs) latrines, recommended as the basic sanitation system according to legislation, has been met with very limited success in communities where water supplied is greater than the evapotranspiration rate; removal of wastewater becomes a critical issue. The shallow depth sewer reduces considerably the amount of excavated material that needs to be moved. Smaller diameter pipes and flatter gradients are used, thus allowing access to areas that are not accessible to conventional sewerage. In this system maintenance is greatly reduced. The main advantage is that shallow-sewer systems are appropriate where water use is between 30 and 60 L per capita per day (i.e. pour flush toilets with yard tanks or yard taps), which may be too high for VIPs and too low for conventional waterborne sewage (Eslick and Harrison, 2004).

The joint EWS/WSSA study is an ongoing study, but the houses in the community will be arranged into condominiums (house grouped according to a geographical parameter i.e. slope, roads or topography). The ABR could be seen as a possible treatment system to communities that receive the shallow sewer. Each condominium could have its own ABR.

### **1.5. Project aims**

The aim of this project was to evaluate operability, and performance of the ABR in terms of:

- COD removal,
- Solids retention,
- Resistance to shock loading and,

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• The effect of diurnal flow variation on the performance.

Biochemical methane potential (BMP) tests were employed to determine the biodegradability of the residual COD in treated effluent from the ABR. This information can be used to decide whether the residual COD could have been further treated; ideally no biodegradable COD should leave the reactor.

The ABR uses a series of baffles to force the wastewater to flow under and over the baffles as it passes from the inlet to the outlet Inherent to the design of the reactor, is the decoupling of solids retention time from the hydraulic retention time. The extent of solids retention dependent on the velocity of the liquid as it moves from one compartment to the next. Sludge settling tests were undertaken to measure the retention of solids at different liquid up-flow velocities within the reactor.

The anaerobic digestion process is unable to remove nutrients and pathogens. The effluent will need to undergo secondary treatment before discharge to a natural water source. Because of its nutrient content, the effluent can be used for irrigation but the pathogen load has to be reduced. Post-treatment aims at mainly removing suspended solids, particulate COD and, reduce the pathogen load. Scoping trials were undertaken with an immersed fabric membrane to determine the removal of pathogen indicator organisms.

### **1.6.** Thesis outline

The thesis makes a start with a review of wastewater (Chapter 2). The role of microorganisms and the subsequent rates of treatment of water are discussed. Black water is described then the characterisation of wastewater is discussed, especially the parameters pertinent to this study. The Department of Water Affairs and Forestry (DWAF) water discharge standards to a natural water source and irrigation standards are included, as they will act as guides for the effluent water quality. Chapter 3 gives a brief overview of the digestion process based on the International Water Association (IWA) Anaerobic Digestion Model. This section discusses the principles that are crucial when operating an anaerobic reactor; the most common reactor types are discussed prior to describing the ABR.

Design of the pilot reactor and its feeding system to control flow to the reactor are discussed in **Chapter 4** with the further modifications that were made to improve performance. Results of the performance of the reactor with results of the analytical campaign are reported and discussed including the ability of the rector to recover from shock loads. Biochemical Methane Potential (BMP) tests were used to measure the anaerobically-biodegradable COD fraction in the influent and effluent, the results is reported in **Chapter 5**.

Chapter 6 describes the investigations into the suitability of a fabric membrane for post-treatment in the ABR. Chapter 7 reports on the settling tests that measure the amount of solids being retained in the reactor.

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The thesis is concluded with **Chapter 8**, a summary of the experimental work, conclusions and recommendations for future research are made.

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# Chapter 2 Domestic Wastewater

In biological water treatment the most widely occurring and abundant group of microorganism are the bacteria, and it is this group that is most important in terms of utilising the organic matter present in wastewater.

### **2.1.** The role of microorganisms

The organic matter is utilised by the microorganisms in a series of enzymatic reactions. Enzymes are proteins, or proteins combined with either an inorganic or low molecular weight organic molecule. They act as catalysts forming complexes with the organic substrate which they convert to a specified product. Enzymes have a high degree of substrate specificity and a bacterial cell must produce different enzyme for each substrate utilised (Gray, 1989). Although enzymes can increase the rate at which chemical reactions proceed, enzymes cannot carry out reactions which are thermodynamically unfavourable (Sundstrom and Klei, 1979). A portion of the absorbed material in the bacterial is oxidised to provide energy while the remainder used for cell synthesis

#### **2.1.1. Enzyme kinetics**

The overall rate of biological reactions within a reactor is dependent on the catalytic activity and concentration of the enzymes in the prominent reaction even if the enzyme is not consumed and undergoes no change. Michaelis and Menten formulated the Michaelis-Menten model for enzyme kinetics. If it is assumed that the enzyme catalysed reaction involves the reversible combination of an enzyme (E) and substrate (S) and form an enzyme-substrate complex (ES) with irreversible dissociation of complex to a product (P) and free enzyme (E). The overall reaction can be expressed as:

$$E + S \underset{k_2}{\overset{k_1}{\Leftrightarrow}} ES \overset{k_3}{\Longrightarrow} E + P$$
 [2-1]

 $k_1, k_2$  and  $k_3$  are the rates of reactions

$$[ES] = \frac{k_1[E][S]}{k_2 + k_3}$$
 [2-2]

If [E] and [S] are concentrations of the free enzyme and free substrate, and if  $[E]_0$  is the total concentration of the enzyme and substrate:

$$[E] + [ES] = [E] \circ$$
 [2-3]

Since there is little enzyme present, the free substrate concentration is almost the same as the total substrate concentration, then equation [2-2] develops into:

$$[ES] = \frac{k_1([E]_0 - [ES])[S]}{k_2 + k_3}$$
 [2-4]

This rearranges to:

$$[ES] = \frac{k_1[E] \circ [S]}{k_1 + k_2 + k_3[S]}$$
[2-5]

Under steady-state conditions the various rate constants  $k_1, k_2$  and  $k_3$  can be expressed as the Michaelis constant,  $k_m$  [2-6]

$$k_m = \frac{k_{2+}k_3}{k_1}$$
 [2-6]

km saturation/Michaelis constant



Figure 6-1: The rate of enzyme-catalysed reactions as presented by the Michaeli-Menten equation (Gray, 1989).

### 2.1.2. Bacterial growth

The rate of substrate removal depends on the rate of microbial growth. Monod studied the development of bacteriological cultures using *batch reactors*. When a small inoculum of viable bacterial cells is placed in closed vessel with excess substrate and ideal environmental conditions, unrestricted growth occurs (Gray,

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1989). Monod plotted the resultant microbial growth from which six distinct phases of development can be defined (Figure 2-2).

Figure 2-2: The microbial growth curve showing bacterial density and specific growth rate at various growth phases

- (1) Initially the bacterial population remains constant during a *lag phase* during which the cells become accustomed to the new media and conditions.
- (2) The bacteria begin to grow and multiply during an *acceleration phase*. During this phase the many intermediates involved in metabolic reactions chain build up to steady levels.
- (3) Then the organisms multiply very rapidly according to first order reaction rate, dX/dt = kX where X is the dry weight of cells/volume and k is the specific growth rate. The integral is a logarithmic expression; this growth is called the *log or exponential phase*. During this phase there is high substrate to microorganism ratio with a fraction of the cells being viable and cell deaths are not important.
- (4) Eventually the food is consume to a point where there are too many organisms and not enough substrate and essential nutrients left to sustain the rapid growth rate in the *declining growth phase*.
- (5) As the substrate concentration becomes limiting the death rate of organisms increases until *stationary phase* is reached where the death rate is nearly equal to the rate of cell synthesis.
- (6) Eventually the substrate concentration will be low enough to cause the death rate to exceed the cell synthesis rate and decrease the number of viable cells. During the *endogenous phase* the cells use the stored ATP respiration, when it is depleted the cell die. The walls of the dead cells rupture releasing carbon containing compounds as food for the reaming viable cells (Sundstrom and Klei, 1979).

The different phases of bacterial growth can be represented quantitatively. The growth rate of microorganisms is proportional to the rate of substrate utilisation.

$$\frac{\mathrm{dX}}{\mathrm{dt}} = \mu \mathrm{X} \qquad [2-7]$$

X is the concentration of microorganisms (mg/L)  $\mu$  is the specific growth rate (1/d)

Monod observed that the growth rate was not only a function of organism concentration but also of some limiting substrate or nutrient concentration. He described the relationship between the residual concentration of the growth-limiting substrate or nutrient and the specific growth-rate of the biomass ( $\mu$ ):

$$\mu = \mu_{\rm m} \frac{\rm S}{\rm k_s + S} \quad [2-8]$$

 $\mu_m$  is the maximum specific growth-rate at saturation concentration of growth limiting substrate (1/d) S is the concentration of the growth-limiting substrate (mg/L)

K<sub>s</sub> is the concentration of limiting substrate at which the specific growth-rate is  $\frac{1}{2}$  of the maximum specific growth-rate ( $\mu = \mu_m/2$ ).

In the Monod equation, microbial growth increases as the availability of substrate increases until the maximum specific growth is reached, at this point a factor other than substrate become growth limiting. The specific growth-rate under exponential growth conditions [2-7] can be replaced by the Monod kinetic equation for biological synthesis:

$$\frac{\mathrm{dX}}{\mathrm{dt}} = \mu_{\mathrm{m}} \frac{\mathrm{SX}}{\mathrm{k_s} + \mathrm{S}} \qquad [2-9]$$

At high substrate concentration (S »  $K_s$ ), the biomass reaction kinetics is are independent of substrate concentration and the equation reduces to [2-7], typically a zero-order process. Physically the surface of the bacteria is completely saturated with substrate and all the internal enzymes are in a complexed state, at this state the rate of biomass synthesis is at maximum. At low substrate concentrations (S «  $K_s$ ), the Monod ratio approaches S/Ks and the growth rate is directly proportional to the substrate concentration [2-10] characteristic of a first-order process (Sundstrom and Klei, 1979).

$$\frac{\mathrm{dX}}{\mathrm{dt}} = \mu_{\mathrm{m}} \frac{\mathrm{SX}}{\mathrm{ks}} \quad [2\text{-}10]$$

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The growth curve is a characteristic of the bacterial cells but rather a response of the cells to their environment and energy requirements. The cells react differently according to availability of substrate and nutrients. For a microbial community to survive there must be a constant input of substrate and environment conditions must be kept favourable. In biological treatment systems the microbial growth curve is manipulated by having continuous feeding that maintains the culture at a particular growth phase. This is done by controlling the food to microorganism (F/M) ratio (Gray, 1989).

### 2.1.3. Temperature effects

Temperature governs the rate of reactions. Although we treat  $\mu_m$  and  $k_m$  as constants, they are actually functions of state variables such temperature and pH. The effect of these secondary variables on the process may explain some of the variability in the reported kinetic constants (Sundstrom and Klei, 1979). Most enzymes and bacteria have optimum temperatures of activity in the range of 20 to 45 °C, above which they rapidly die or become inactive. The majority of biological treatment systems operate in the mesophilic range 20 to 40 °C. The increased temperature results in increased biological activity that in turn increases substrate removal. Van't Hoff's rule states that biological activity doubles for every 10 °C rise in temperature within the range of 5 to 35 °C. The variation in reaction rate with temperature is represented by the modified Arrhenius equation:

$$k_{\rm T} = k_{20} \theta^{(\rm T-20)}$$
 [2-11]

T is the temperature in  ${}^{O}C$ k is the reaction constant at temperature of interest  $k_{20}$  is the reaction rate constant at 20 OC, and  $\theta$  is the temperature coefficient

### 2.2. Wastewater characterisation

To comply with more stringent effluent legislation, treatment of wastewater has evolved from simple systems removing carbon, to more complex systems for carbon and nutrient removal (nitrogen and phosphorous). Not only has these expansions increased the complexity system configuration and its operation; concomitantly the number of biological processes influencing the effluent quality has increased. Thus the knowledge of the wastewater characteristics is an important step towards the successful design and operation of treatment plants (Wentzel and Ekama, 1996).

### 2.2.1. Domestic wastewater

Domestic wastewater is made up of toilet wastewater (black water) and sullage from the kitchen, and bathroom water (grey water). The quantity and concentration of the flow will depend on the socioeconomic behaviour of the population (van Haandel *et al.*, 1994).

Wastewater and its content is mainly characterised on the processes and operations that will occur during biological treatment. As the sewage enters the bioreactor, the wastewater consists of soluble and particulate material; the latter is subdivided into settleable and suspended (non-settleable) solids. Organic and inorganic materials are enmeshed (a biologically mediated flocculation), and become part of the sludge liquor. The soluble materials, both organic and inorganic remain in solution. The microorganisms present in the reactor will act on the *biodegradable* material. Whether organic or inorganic, soluble or particulate, these are transformed into other compounds. The products could be gaseous, soluble or particulate. The gaseous products escape into the atmosphere, the particulate products become part of the mixed liquor, and soluble products remain in solution. The unbiodegradable material is not transformed, and will remain in either soluble or particulate form.

The first major division of the influent is based on whether the material is *biodegradable* or *unbiodegradable*. If the material is unbiodegradable and particulate it is termed *unbiodegradable particulate*. The unbiodegradable soluble constituents are called *unbiodegradable soluble*, they do not settle out and will leave with the effluent.



Figure 2-3: Partial subdivision for steady-state design procedure for total organic material (COD, TKN and Total P). The biodegradable organic material is not subdivided further because it is accepted that for steady state purposes it is all degraded

The subdivision of biodegradable material is based on rates of transformation in the reactor. Soluble organic constituents are readily utilisable than the particulate ones. Physical size of the constituents plays an important role on utilisability. For this reason physical separation is used to assist in the identification of the readily biodegradable and the slowly biodegradable organic fractions. In the assessment of the

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performance of an activated sludge system, the wastewater carbon (C), nitrogen (N) and phosphorous (P) constituents are characterised biologically as:

- Biodegradable
- Unbiodegradable soluble
- Unbiodegradable particulate.



### Figure 2-4: Major divisions of chemical characterisation of wastewater

The quantity of each constituent fraction is assessed chemically. Chemical oxygen demand (COD) test is used as a basis for specifying the various fractions of organic materials (C). The Total Kjeldahl nitrogen (TKN) test forms the basis for specifying the various nitrogen (N) constituents. Total phosphorous (TP) tests forms the basis for specifying the phosphorous fraction (Henze *et al.*, 1997).

### 2.2.2. Characterisation of solids in wastewater

Solids affect the effluent water quality in a number of ways. The analysis thereof is important in the control of biological and physical wastewater treatment processes, and assessing compliance with regulatory wastewater effluent standards. *Total solids*, is a term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a specified temperature. Total solids include *total suspended solids*; this is a portion of total solids that is retained in 0.45 µm acetate filter. The portion that passes through the filter is called *total dissolved solids*. *Fixed solids*, is a term applied to the residue of total, suspended or dissolved solids after ignition for a specified time at a specified temperature. The weight loss on ignition is called *volatile solids* (Figure 2-3). Determination of fixed and volatile solids does not distinguish precisely between inorganic and organic matter. The weight loss on ignition is not confined to cellular matter; it includes losses due to decomposition or volatilisation of some mineral salts. Volatile solids are roughly correlated to cellular matter, but this does offer a rough approximation of the amount of organic matter present in the sludge. Better characterisation of organic matter can be made by such tests as total carbon (TC) and COD (Standard Methods, 1985).

Typically, volatile suspended solids (VSS) are used as a measure of viable (living) microorganisms in a culture. For a growing culture of microorganisms under conditions of excess substrate, total suspended solids (TSS) can also be used as an approximation of the number of viable cells in the culture. The
discrepancy between TSS and VSS measurements becomes more pronounced when the culture spends more time under limited substrate conditions. The death and lysis of cells under conditions of starvation contribute to the increase of suspended solids without any growth, hence the difference between the two measurements. VSS is a better measure than TSS because it does not include the inert solids. However for many cultures involved in wastewater treatment where substrate concentrations are low relative to the microbial mass, VSS will not be indicative of the number of viable microorganisms which are the active mass responsible for biodegradation (Droste, 1997).



Figure 2-5: Classification of solids in wastewater

#### 2.2.3. Chemical characterisation

Chemical characterisation involves the measuring the chemical constituents in the wastewater that are relevant (aim to treat or take part) when treating the water.

#### 2.2.3.1 Organic fraction

The chemical oxygen demand (COD) is used as a measure of the chemical oxygen demand matter in a sample that is susceptible to oxidation by a strong chemical oxidant (Henze *et al.*, 1997). The biodegradable COD is subdivided into two fractions. The first fraction is the readily-biodegradable COD fraction (RBCOD). The RBCOD fraction consists of small molecules that can pass directly through the cell wall of the organisms for metabolism and its utilisation is very rapid. The second fraction is the slowly-biodegradable COD fraction (SBCOD), which consists of larger complex molecules that cannot pass directly through the cell wall of the microorganism. The constituents that belong to the SBCOD fraction require several hydrolytic steps before they can be taken up and utilized by the bacteria in sludge, and its degradation occurs at a much slower rate.

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Figure 2-6: Division of influent COD into its constituent fractions

The biodegradable COD is subdivided into two fractions. The first fraction is the readily-biodegradable COD fraction (RBCOD). The RBCOD fraction consists of small molecules that can pass directly through the cell wall of the organisms for metabolism and its utilisation is very rapid. The second fraction is the slowly-biodegradable COD fraction (SBCOD), which consists of larger complex molecules that cannot pass directly through the cell wall of the microorganism. The constituents that belong to the SBCOD fraction require several hydrolytic steps before they can be taken up and utilized by the bacteria in sludge, and its degradation occurs at a much slower rate.

#### 2.2.3.2 Nitrogen

In water and wastewater, forms of nitrogen of greatest interest in order of decreasing oxidation state are, nitrate (NO<sub>3</sub>), nitrite (NO<sub>2</sub>), ammonia (NH<sub>3</sub>), and organic nitrogen (N<sub>2</sub>). All these forms of nitrogen are biochemically interconvertible and are components of the nitrogen cycle. Organic nitrogen is functionally defined as organically bound nitrogen in the tri-negative oxidation state. It does not include all organic nitrogen compounds. Organic nitrogen includes natural materials such as protein, peptides, nucleic acids, urea and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per litre in some lakes to more than 20 mg/L in raw sewage. Characterisation of nitrogenous material in the influent is in terms of the total Kjeldahl nitrogen (Figure 2-7).



Figure 2-7: Division of TKN into its constituent fractions

#### 2.2.3.3 Phosphorus

Phosphorus occurs in natural waters and in wastewaters solely as phosphates. These are classified into orthophosphates, condensed phosphate (polyphosphates) and organically bound phosphates. The phosphates occur in solution, particles, organisms or detritus material. The different forms of phosphates arise from a variety of sources. Some condensed phosphates are added to water during treatment, larger quantities are added when the water is used for laundry or cleaning.

Phosphorus analysis embodies two general procedural steps (a) conversion of phosphorus to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphates. Because phosphates may occur in combination with organic matter, a digestion method is used to determine total phosphorus. Acid hydrolysis at boiling water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphates. Typical wastewater characteristics and strengths are tabulated in **Appendix I**.

## 2.3. Discharge water standards

It is envisaged that the effluent from the ABR would be discharged to the closest water course. The option of using the effluent for irrigation is being considered but the pathogen content of the water will have to reach acceptable levels and even lower levels for discharge to a water source. Any effluent being discharged into a watercourse has to meet standards set by the Department of Water Affairs and Forestry (DWAF).

Substance	Unit	Discharge standard	Irrigation standard
COD	mgCOD/L	75	400
pH		5.5-9.5	6-9
Ammonia	mg N/L	3	No limit
Phosphorus	mg P/L	10	No limit
TSS	mg TSS/L	25	No limit
VSS	mg VSS/L	No limit	No limit
Total coliforms (cfu)	cfu/100 mL	1 000	100 000

# Table 2-1: Discharge standards for water being released into water sources for500 kL/d discharge and for irrigation (National Water Act No.36, 1998)

The total suspended solids (TSS) in the wastewater increase the turbidity of the water causing a decrease in photosynthesis in water plants due to reduced sunlight. The solids also tend to clog up fish gills and increase silting. The chemical oxygen demand (COD) is an indication of the organics found in the wastewater. Organisms use dissolved oxygen in the water to breakdown these organics, and in so doing, reduce the amount of oxygen available for the aquatic life resulting in fish kills and odours.

The nutrients, nitrogen and phosphorus, result in an increase in algal growth, which also results in the depletion of oxygen in the water. Nitrogen in drinking water may contribute to miscarriages and a serious disease in infants called methemoglobinemia or "blue baby syndrome".

## 2.4. Peri-urban communities and their wastewater

There has been a movement of people from rural areas to urban areas as people search for greater economic opportunities and a more sophisticated lifestyle. Urban centres are unable to keep up with urban growth and this has resulted in an increase in the number of people living in informal or unplanned settlements. Most poor urban residents in the eThekwini Municipality purchase or obtain water from kiosks, tankers or stand-free pipes and, do not have access to wastewater or sanitation services. Communities in dense peri-urban areas generally have a limited water access. The water consumption is very low, and the sewage is concentrated.

According to the national census carried out in 2001, the average household size in eThekwini is 4.00 persons per dwelling (StatsSA, 2003). In the eThekwini municipality, each serviced household in the periurban areas receives 200 L of water per day. The quantity and composition of human faeces and urine (**Table 2-2**) were used to calculate the composition of the wastewater (black water) that would be produced by one of household in the community. The following equation was used:  $Y = \frac{(X_f + X_u) \times 4}{160L}$  [2-12]

The 160 L is the amount of water assumed to go to drain (i.e. 40 L used for a purpose which renders it not being put in the sewer such as gardening), and:

Y is the concentration of the chemical constituent in the water in g/L,

X<sub>f</sub> is the dry mass components in the faeces in grams and,

 $X_u$  is the dry mass components in the urine in grams.

The community domestic wastewater (black water) is expected to contain 7 times the nitrogen, 6 times the total phosphorus, 2½ times the total COD and 2 times as much total solid compared to the wastewater received by Kingsburgh WWTW. Since anaerobic process is unable to remove nutrients the effluent from the ABR will be very rich with nutrients, which will make it a rich nutrient source suitable for irrigation.

In Table 2-2 within the given range, only the upper value was used for the calculation; \*dry matter is 30 to 60 g/person.day for faeces and 50 to 70 g/person.day for urine with a water content of 77 and 94% for faeces and urine respectively.

Approximate quantity	Units	Faeces	Urine	Calculated peri-urban	Kingsburgh wastewater
				composition (mg/L)	composition (mg/L)
Quantity (wet solids/person.day)	g	70-520	1000-1500		
Quantity (dry solids/ person.day)	g	30-70	50-70		
Approximate composition (%dry	%	88 <b>-</b> 97	65-85		
weight/matter*)					
Moisture content	%	66-85	93-99.5		
Organic matter	%	88-97	65-85	2 125	
Nitrogen (TKN)	mg	1 400-2 460	5 290	190	25
Total phosphorous	mg	690-2 500	1 080-2 200	120	20
Potassium (K)	mg	800-2 100	2 500-3 700	180	
Carbon (C)	mg	44-55	11 000-17 000	1 800	
Calcium (CaO)	mg	4.5	4.5-6.0	260	
COD <sub>total</sub>	mg/L	46 230-78 310	12.79	2 280	875
COD <sub>soluble</sub>	mg/L		11 330	280	
COD <sub>particulate</sub>	mg/L		1 460	2 000	
TS	%	33		1 710	810
Protein	mg	4 000-12 000		310	
Total lipids	mg	4 000-6 000		150	
Polysaccharides	mg	4 000-10 000	680	280	

# Table 2-2: Quantity and composition of human faeces and urine (Chaggu, 2003), calculated peri-urban wastewater; measured values for Kingsburgh water

## **Chapter 3** Anaerobic digestion and bioreactors

The processes involved in anaerobic digestion are many and complex. In 1997, a concept evolved amongst the International Water Association (IWA) Anaerobic Digestion Specialist Group members to consolidate the knowledge and, create a consensus model for the biochemical processes that occur in anaerobic digestion. The IWA Anaerobic Digestion Model (ADM1) written by Bastone *et al.* (2001) was used as a major guide in the following literature review.

Anaerobic digestion involves the breakdown of organic molecules to methane and carbon dioxide gas in the absence of molecular oxygen. The biochemical processes involved could be divided into four categories, as the bacteria (microorganisms) sequentially degrade the organic matter: (1) hydrolysis and disintegration; (2) acidogenesis; (3) acetogenesis and (4) methanogenesis. The microorganisms involved can be grouped into three categories; hydrolytic microorganisms which degrade the polymer-type material such as polysaccharides and proteins to monomers. The monomers are then converted to volatile fatty acids (VFA) with a small amount of hydrogen (Eckenfelder jr., 1989). All fatty acids with a molecular mass greater than acetic acid are converted to acetate and hydrogen by (second group of microorganisms) acetogenic microorganisms. The principal acids produced are acetic (HAc), propionic (HPr), and butyric acid (HBr) with small amounts of valeric acid (HVa) respectively. The organic acid, acetic acid and hydrogen are converted to methanogens.

The removal of COD is accomplished by the final conversion of organics into methane, which is a relatively insoluble gas. There is also a significant production of  $CO_2$ . The net production of other gases such as hydrogen is very small. Anaerobic COD treatment is realised in the final conversion of metabolic intermediates to methane. If the process is stopped short of this step, the effluent will contain soluble products from intermediate stages of metabolism with the COD of the initial material. The quantity of organic matter converted to gas during the anaerobic digestion varies between 80 and 90% (Droste, 1997). The yield of anaerobic fermentation is only about one seventh of the yield of aerobes, the relative rate of growth is slow and the yield of organisms is low. However this does not mean that their rate of processing substrate is low.

Sewage contains thousands of different organic molecules that contain carbon (C), hydrogen (H), oxygen (O) and, nitrogen (N). These can be represented by the formula  $C_xH_yO_zN_a$ , the stoichiometric coefficients are empirically determined. The oxidation products can be calculated using the following equation

$$C_{x}H_{y}O_{z}N_{a} \Rightarrow \left(\frac{2z-y+3a}{4}\right)H_{2}CO_{3} + aNH_{3} + \left(\frac{4x+y-2z-3a}{24}\right)C_{6}H_{12}O_{6} + \left(\frac{y-2x-3a}{2}\right)H_{2}O_{6}$$
[3-1].

The electron demand for complete oxidation of one mole of  $C_x H_y O_z N_a$ , e = 4x + y - 2z - 3a.

$$C_{x}H_{y}O_{z}N_{a} \Longrightarrow \left(x - \frac{e}{4}\right)H_{2}CO_{3} + aNH_{3} + \frac{e}{24}C_{6}H_{12}O_{6} + \left(z - 3x + \frac{e}{2}\right)H_{2}O$$
[3-2]

The chemical oxygen demand of  $C_xH_yO_zN_a$  is e/2 moles of O, or 8e grams of molecular oxygen. In the model  $C_xH_yO_zN_a$  is represented as kg/m<sup>3</sup> COD and all the other species (except H<sub>2</sub>O) as kmol/m<sup>3</sup>.

The utilisation of the organic fraction in wastewater is directly related to the methane production, and vice versa. Where the exact chemical nature of the substrate is known, the quantity of methane produced can be estimated by the following equation (Gray, 1989):

$$C_{n}H_{a}O_{b} + \left(n - \frac{a}{4} - \frac{b}{2}\right)H_{2}O \rightarrow \left(\frac{n}{2} - \frac{a}{8} + \frac{b}{4}\right)CO_{2} + \left(\frac{n}{2} - \frac{a}{8} + \frac{b}{4}\right)CH_{4} \quad [3-3]$$

McCarthy estimates that 1 Kg COD is stabilised as  $0.348 \text{ m}^3$  of methane at standard pressure and temperature. The volume of bacterial mass and methane produced was calculated for anaerobic sewage sludge. For each mole of sewage sludge 0.195 mol of new cells are produced and 5.75 mol methane is released (Gray, 1989),and the Water Research Group from the University of Cape Town measured CHON composition of primary sludge to be  $C_{3.5}H_7O_2N_{0.196}$  (Sötemann *et al.*, to be published).

The COD flow chart used in the ADM1 model (**Appendix II**), shows the COD flow through intermediates for a hypothetical composite particulate material that is 10% inerts, with the remainder split equally between carbohydrates, proteins and lipids. The COD flux would change considerably for different primary components.



Figure 3-1: Major step in anaerobic digestion (Gray, 1989)

## 3.1. Disintegration and hydrolysis

Large molecules and suspended matter cannot be directly assimilated and metabolised by anaerobic bacteria. Disintegration and hydrolysis is the breakdown of large, complex soluble and insoluble molecules into smaller molecules that can be transported into the cell and be metabolised. Extracellular enzymes secreted by the primary fermentative microorganisms are used to accomplish this task. Extracellular solubilisation steps are divided into disintegration and hydrolysis. Disintegration is not biologically mediated, e.g. lysis of dead cells causing the release of composite particulate substrates, inerts, particulate carbohydrates, protein and lipids. Hydrolysis is the enzymatic degradation of particulate carbohydrates protein and lipids, to monosaccharides (MS) amino acids (AA) and long-chain fatty acids (LCFA) and glycerine respectively. Disintegration is mainly included to describe the degradation of particulate material with lumped characteristics such as sludge by shearing and phase separation, while hydrolysis is used to describe the degradation of well-defined and relatively pure substrates such as cellulose, starch, protein etc. (Bastone *et al.*, 2002).

In practice, the hydrolysis step can be rate-limiting for the overall rate of anaerobic digestion, in particular the rate of lipid hydrolysis at below 20 OC (Haandel and Lettinga, 1994). When macromolecules concentration is significant, hydrolysis reactions become the rate limiting stage of anaerobic metabolism. All disintegration and hydrolysis processes are represented by first order kinetics. The microorganisms responsible for hydrolysis do not form methane.

Two conceptual models can be used to represent hydrolysis:

- (1) The organisms secrete enzymes in to the bulk liquid where they adsorb onto a particle or react with a soluble substrate.
- (2) The organism attaches to a particle, produces enzymes in close proximity to the particle and in return it benefits directly from the products released by the reactions.

It has been shown by Vavillin and Sanders, (Bastone *et al.*, 2002) that type (2) is the dominant mechanism in mixed cultures. Therefore the organism growing on the particle surface rather than the enzymes produced should be regarded as the effective catalyst.

## 3.2. Acidogenesis

The same organisms that carry out hydrolysis also perform acidogenesis; the soluble compounds generated in the hydrolysis step are taken up in the cell of the bacteria. Acidogenesis (fermentation) is usually defined as an anaerobic acid-producing microbial process without an additional (external) electron acceptor or donor. This includes the degradation of soluble sugars and amino acids to their simpler products. The products of acidogenesis diffuse out of the cells and sometimes are secreted as waste. This waste consists of volatile fatty acids (VFAs), alcohols, lactic acid and mineral compounds such as carbon dioxide,

ammonia, hydrogen and hydrogen sulphide gas. The degradation of LCFAs is an oxidation reaction with an external electron acceptor is thus included under acetogenesis (Bastone *et al.*, 2002).

Glucose is the common monomer used in illustrating the reactions that occur in the fermentation of saccharides. The most important products and their stoichiometric reactions from glucose with approximate ATP yields are shown in **Table 3-1**. All organisms producing propionate or succinate (the key intermediate to propionate) also produce acetate with carbon dioxide as a by-product.

There are two main pathways for amino acid fermentation:

- (1) Stickland oxidation-reduction paired fermentation.
- (2) Oxidation of a single amino acid with hydrogen ion or carbon dioxide as the external electron acceptor.

The relative yields of the 20 common amino acids from the hydrolysis of protein are dependent on the protein primary-structure. Characteristics of Stickland oxidation are listed below:

- Different amino acids can act as donors or acceptors, or both
- The electron donor lose one carbon atom to CO<sub>2</sub> and forms a carboxylic acid with one carbon less than the original amino acid (i.e. alanine C<sub>3</sub> → acetate C<sub>2</sub>)
- The electron acceptor retains carbon atoms to form a carboxylic acid with the same chain length as the original amino acid (i.e. glycine C<sub>2</sub> → acetate C<sub>2</sub>)
- Only histidine cannot be degraded via Stickland oxidation.

Products	Reactions	ATP /mol	Conditions	Note
		glucose		
(I) Acetate	$C_6H_{12}O_6 + H_2O \rightarrow 2CH_3COOH + 2CO_2 + 4H_2$	4	Low H <sub>2</sub>	1
(II) Propionate	$C_6H_{12}O_6 + 2H_2 \rightarrow 2CH_3CH_2COOH + 2H_2O$	Low	Not observed	2
(II') Acetate/	$3C_6H_{12}O_6 \rightarrow 4CH_3CH_2COOH + 2CH_3COOH +$	4/3	Any H <sub>2</sub>	
propionate	$2CO_2 + 2H_2O$			
(III) Butyrate	$\mathrm{C_6H_{12}O_6} \rightarrow \mathrm{CH_3CH_2COOH} + 2\mathrm{CO_2} + 2\mathrm{H_2}$	3	Low H <sub>2</sub>	1
(IV) Lactate	$C_6H_{12}O_6 \rightarrow 2CH_3CHOHCOOH$	2	Any H <sub>2</sub>	
(V) Ethanol	$C_6H_{12}O_6 \rightarrow 2CH_3CH_2OH + 2CO_2$	2	Low pH	3

#### Table 3-1: Products from glucose degradation (Bastone et al., 2002) Image: Comparison of the second sec

1. While thermodynamically possible at high  $H_2$  partial pressure, may be limited by the energetics of substrate level phosphorylation.

2. Not yet observed in cultured environmental samples. Coupling with substrate level oxidation is more common as in reaction (II).

3. Energy yield taken from yeast pathway. Bacterial pathway may have 0 ATP/mol ethanol.

Stickland oxidation occurs very rapidly compared to uncoupled oxidation. In a typically mixed protein system there is normally a 10% decrease in electron acceptors, and correspondingly about 10% of amino acids degraded by uncoupled oxidation. Uncoupled oxidation is also limited by the shortfall in electron acceptor capacity, which results in hydrogen or formate production (Bastone *et al.*; 2002).



Figure 3-2: Coupled Stickland digestion of alanine and glycine used as an example above (Bastone *et al.*, 2002)

The majority of the anaerobic processes with single amino acids will produce ammonia through deamination. Deamination can follow a couple of pathways each dependant on the enzymatic complement of the organism and its environmental conditions.

### **3.3.** Acetogenesis

Syntrophic acetogenesis is the degradation of higher organic acids to acetate in an oxidation step with an external electron acceptor. The organisms oxidising the organic acids are required to utilise an additional electron acceptor such as hydrogen ions or carbon dioxide to produce hydrogen or formate respectively (Bastone *et al.*; 2002). Hydrogen must be maintained at a low concentration (below  $10^{-4}$  atmospheres) for the oxidation reaction to be thermodynamically possible (Speece, 1996). Whether hydrogen ions or carbon dioxide is used as an acceptor depends on the oxidation state of the original organic matter (Droste, 1997). This can be seen in the following reaction (where NEL is the net electron number):

Where Y < 2Z (NEL < 4):

$$C_{xH_{Y}O_{Z}} + \frac{1}{4}(4X - 2Z) H_{2}O \rightarrow \frac{1}{8}(4X + y + 2Z)CH_{3}COOH + \frac{1}{4}(2Z - Y)CO_{2}$$
 [3-4]

Where Y > 2Z (NEL > 4):

$$C_{x}H_{Y}O_{z} + (X - Z)H_{2}O \rightarrow \frac{X}{2}CH_{3}COOH + \frac{1}{2}(Y - 2Z)H_{2}$$
 [3-5]

In a mixture of different organic substrates such as in sewage, it is likely that both processes occur simultaneously but generally more hydrogen is formed than carbon dioxide because the average number of electrons that are available in the organic matter is greater than four per carbon atom. Consequently, the conversion of influent organic matter into acetic acid is accompanied by the formation of hydrogen (Droste, 1997).

Homoacetogenesis is the conversion of  $H_2$  with carbon dioxide to acetate. Growth on carbon dioxide and hydrogen has been reported on all homoacetogens.

$$4H_{2} + 2CO_{2} \rightarrow CH_{3}COO^{-} + H^{+} + 2H_{2}O \ \Delta G = -95KJ$$
[3-6]  
$$4HCOO^{-} + 3H^{+} \rightarrow CH_{3}COO^{-} + 2CO_{2} + 2H_{2}O$$
[3-7]

*Clostridium thermoaceticum* and *Acetobacterium woodii* are able to reduce carbon dioxide with elemental hydrogen; therefore they compete for substrate with hydrogenotrophic methanogens. Homoacetogens are the most versatile group amongst anaerobic bacteria. They can also carry out incomplete oxidation of reduced fermentation products produced by other fermenting bacteria (Droste, 1997).

## 3.4. Methanogenesis

The formation of methane, which is the ultimate product of anaerobic digestion, is often the rate-limiting step in an anaerobic process occurring on soluble substrate. The formation of methane is carried out by two routes that are facilitated by two different groups of bacteria (genera). The major route is the fermentation of acetic acid to methane and carbon dioxide (Bastone *et al.*, 2002). Bacteria that utilise acetic acid are called acetoclastic bacteria and facilitate acetotrophic methanogenesis. The overall reaction, for biological production of methane from acetate, is given by:

$$CH_3COOH \rightarrow CH_4 + CO_2$$
 [3-8]

The most common acetoclastic methanogens in reactors treating wastewater with a high content of volatile acids are from the genera *Methanosarcina* and *Methanosaeta*. *Methanosarcina* are coccoid bacteria with doubling time of nearly 1.5 d, they dominate where concentration of acetate is greater than 10<sup>-3</sup> mol/L.

*Methanosaeta* are sheathed rods sometimes growing as long filaments with a doubling time of 4 d, and dominate at acetate concentration below  $10^{-3}$  mol/L. These doubling times were measured at optimum conditions. *Methanosaeta* has a lower K<sub>s</sub> value, a higher K<sub>m</sub> value and a lower yield compared to *Methanosaeta*. *Methanosaeta* uses 2 moles of ATP to assist activation of 1 mole of acetate at lower concentrations, while *Methanosaeta* uses 1 mole of ATP at higher concentrations of acetate. Even though *Methanosaeta* grows more slowly, they are frequently the dominant genus. The presence of the two organisms is normally mutually exclusive with *Methanosarcina* dominating in the high rate systems (Barber and Stuckey, 1999).

The secondary route is the conversion of carbon dioxide and hydrogen to methane. This is sometimes called hydrogenotrophic methanogenesis (reductive methanogenesis). Hydrogenotrophic bacteria grow much faster than those utilising acetate so that the acetoclastic methanogens are rate-limiting with respect to the transformation of complex macromolecules in sewage to biogas. Based on thermodynamic consideration and experimental data, Zikus in 1975 proposed the following reaction (Droste, 1997):

 $CH_{3}COOH + 4H_{2} \rightarrow 2CH_{4} + 2H_{2}O$  [3-8]

Subtle changes in the partial pressure of hydrogen can change the end-product of acetogenesis. As the hydrogen partial pressure rises, hydrogen oxidation becomes more favourable than acetate degradation and acetate production is increased. The synergistic relation between hydrogen producers and scavengers helps to keep the hydrogen partial-pressure in the reactor under favourable conditions for acetate degradation. Therefore it seems then convenient that obligatory  $H_2$  producing bacteria grow in close proximity to the methanogenic bacteria because the latter remove the  $H_2$  (Speece, 1997).

## 3.5. Operating an anaerobic process

For the proper functioning and performance of an anaerobic process, particular care has to be taken in the areas which have caused failure in other processes in the past. The main disadvantage of an anaerobic process is the relatively slow reaction rate of the of the methane production step, which is the rate-limiting step in the overall process. If the rate of methanogenesis does not keep-up with the rate of acid formation, the pH will drop below 6.5 and methanogenesis will cease. Since the digestion process is complex, there is no single parameter which can be used to predict failure; several parameters must be monitored for good control. Knowing whether a parameter is decreasing or increasing is often of more importance than knowing its absolute value (Sundstrom and Klei, 1979). The operation of an aerobic plant is done by means of a number of measurements. The number of parameters that need to be measured depends on the environmental conditions the plant is subjected to. **Table 3-2** gives a summary of common chemical parameters used to monitor an anaerobic process.

parameter	level
pH	6.5-8 (Speece, 1996)
Bicarbonate alkalinity	1 000-5 000 mg/L
Ammonia	< 1 000 mgNH4-N/L
Temperature	25-38 (Henze et al., 1996)
Methane in gas produced	65-70%

 

 Table 3-2: Common parameters for monitoring the anaerobic process (Sundstrom and Klei, 1979)

The alkalinity of water is a measure of its capacity to neutralise acids and is due primarily to the salts of weak acids. Since alkalinity controls the pH, it is used as a measure of the capacity of an aquatic system to resist acid/base influences. The anaerobic process influences alkalinity, acid formation reduces it and methane formation increases it. The overall result is a small reduction in alkalinity (Henze *et al.*, 1996).

Low pH conditions may be caused by two sources of acidity, carbonic acid  $(H_2CO_3)$  and VFA. In a welloperating anaerobic process, the major requirement for alkalinity is for the neutralisation of carbonic acid, which is formed at high partial pressures of carbon dioxide in the reactor; not the VFA which are normally low.

$$H_2O + CO_2 \leftrightarrow H_2CO_3$$
 [3-9]

When the pH drops below 6.5 it will inhibit microbial activity especially the methanogens. When methanogenesis ceases the VFA may continue to accumulate, exacerbating the situation. The high concentration of VFA is its self not toxic to the bacteria it is low pH. Normal VFA concentration in sewage sludge is between 250 and 1 000 mg/L, but values in excess of 1 800 or 2 000 mg/L indicate problems (Gray, 1989)

The more dilute a wastewater, the lower will be its inherent alkalinity generation potential and vice versa (Speece, 1996). The dilute wastewater contains little organics which can be converted to VFA which are further converted to methane and carbon dioxide. The carbon dioxide then forms carbonic acid. For a dilute wastewater, the digester can maintain stability at a lower alkalinity.

Ammonia (NH<sub>3</sub>) and ammonium ions (NH<sub>4</sub><sup>+</sup>) are an essential nitrogen source for anaerobic digestion, but can be inhibitory at concentrations above 150 mgN/L and 3 000 mgN/L respectively. However these concentrations are occasionally found in concentrated sewage sludge. However the system is largely self regulating in that the inhibition causes an accumulation of volatile acids, which in turn depress the pH value. This converts the dissolved ammonia to the less toxic ammonium ions, thus alleviating inhibition (Gray, 1989).

 $NH_3 + H_2O \leftrightarrow NH_4^+ + HCO_3^-$  [3-10]

## 3.6. Reactor types and technology

In recent decades, several developments have greatly increased energy efficiency and attractiveness of anaerobic waste treatment. Research groups throughout the world have developed anaerobic reactors that can treat the waste more quickly, more reliably and with the greatest net production of methane gas. Full-scale implementations of the developments have been met with success (Droste, 1997).

Several reactor types are utilised for waste treatment. On their biological means, they are broadly divided into two groups:

- (1) Non-attached biomass systems and
- (2) Fixed-film reactors

The biomass of the latter is attached as a film on inert supportive media. Non-attached biomass systems depend on the metabolic activity of microorganisms suspended as flocs or granules in the reactor vessel. The suspended bacteria have to form flocs to remain in the reactor. The efficiency of the biomass is to a great extent depended upon the flock forming and settling abilities of the sludge (Stronach *et al.*, 1986).

#### 3.6.1. The continuously stirred tank reactor (CSTR)

In common with many of the anaerobic bioreactor systems, the continuously stirred tank reactor was developed from its aerobic counterpart (Figure 3-4). This reactor design requires an extended hydraulic retention time (HRT) because it has no specific means of biomass retention. Consequently the solids retention time (SRT) must be sufficiently high to permit biological conversion reactions to occur. The HRT of the system treating is depended on the organic loading rate. For systems treating sewage it varies from 10 d for heated systems, to 60 d for cold digesters. The SRT can range from 90 to 200 d. The conventional single-stage CSTR comprises of a vessel of steel, concrete or brick.

Anaerobic Digestion and Reactors





Figure 3-3: The continuously stirred tank reactor (Stronach et al., 1986)

The anaerobic digester is technically a continuous microbial culture and as such, requires a continuous feed of medium and outflow. In cases where the volume of wastewater feeding the reactor is too large to permit continuous feeding, then the reactor is fed intermittently.

Mixing of contents in CSTR is important and is generally achieved by paddle or screw systems, and is normally intermittent. Mechanical agitation is frequently used in smaller digesters. Free rising bubbles of biogas within the system may be recirculated in large digesters. Some digesters depend entirely on gas mixing; insufficient agitation can result in a serious problem of scum formation.

CSTRs have been successfully used for the stabilisation of sewage sludge and the treatment of industrial wastewaters that contain high solids concentration such as crop residue. Efficiencies of digesters that were investigated (some with the stirred tank configuration) had loading rates of 0.7 to 3.2 kgVS/m<sup>3</sup>.d (Stronach *et al.*, 1986). Removal rates of 27 to 44% of volatile solids (VS), and 90 to 95% of total solids (TS) were achieved. Since the biomass in CSTR is not retained, COD removal tends to be limited. In the temperature range of 30 to 35 °C retention times of 10 to 20 d can be achieved, but can vary with waste composition and degree of agitation. CSTR systems have shown to be susceptible to shock loading and toxic substances.

#### 3.6.2. High-rate systems

The concept of high rate anaerobic reactors is based on three fundamental aspects:

- (1) Accumulation within the rector by means of settling, attachment to solids (fixed or mobile) or by recirculation. Such systems allow retention of slowly growing microorganisms by ensuring that the mean solids retention time becomes much longer than the mean hydraulic retention time.
- (2) Improved contact between biomass and wastewater, overcoming problems of diffusion of substrates and products from the bulk liquid to the biofilms or granules.

(3) Enhanced activity of the biomass, due to adaptation and growth. (J. Iza et al., 1991)

## 3.6.3. The upflow anaerobic sludge blanket reactor

The upflow anaerobic sludge blanket (UASB) reactor concept was developed on the recognition that inert support material for biomass attachment was not necessary to retain high levels of active sludge in the reactor. Instead the UASB concept relies on high levels of biomass retention through the formation of sludge granules (Stronach *et al.*, 1986). The bacteria develop as a flocculant mass in an upward flowing waste stream. Baffles or screens forming a setter unit at the top of the reactor retain the microbial blanket. The gas and effluent escape at the top of the vessel. Dissociation of the bacterial mass does occur to some degree. Bacteria are lost in the outflow but the mean retention time is extended to allow growth of a dense mass of methanogenic bacteria although the HRT is low.



Figure 3-4: The upflow anaerobic sludge blanket (Stronach et al., 1986)

One of the fundamental design principles for maintenance of high sludge retention is founded on sludge with good sedimentation properties. Under the correct conditions, biomass in the reactor forms compact grains or granules of 3 to 4 mm in diameter. Larger granules form the sludge bed or lower portion of reactor. The bed develops after a few months of operation; settleability is improved if minimal agitation is used. The bed can reach a density of 40 to 50 gVSS/L, and particle-settling rates can reach 50 m/h (Stronach *et al.*, 1986). Above the sludge bed is the sludge blanket. The blanket consists of smaller grains, flocs and gas bubbles. The reactor ranges from dense and granular particles with high settling velocities near the base, to less dense grains in the blanket (Figure 3-4). The granules that have caused the success of the UASB can only be developed in a staged process with pseudo plug-flow. According to researchers at the University of Cape Town, one of the essential conditions for granule formation is the existence of a zone of high hydrogen partial pressure, which occurs within a plug flow reactor (Speece *et al.*, 1997)

The second main design feature of the UASB is the installation of the gas/solids separating device in the upper part of the reactor. The smaller particle size and flocculation characteristic of the blanket gives rise to a settling rate inferior to that found in the bed. To permit retention the applied liquid velocity in the settler should be relatively low, higher velocities tend to produce unacceptable sludge losses. The UASB is

therefore, not an effective treatment system for wastewater high in suspended matter. One of the advantages of the UASB is that it can maintain pH values near neutrality.

A glucose/sucrose feed loaded at a rate of 45 kgCOD/m<sup>3</sup>.d was treated with an efficiency of 80% within 3 months of start-up. Increased organic loading rates are tolerated with little loss of stability in the reactor, but COD removal rates are not always consistent and erratic TSS removal rates have been observed. Raw sewage of 500 mgCOD/L was applied at loading rates of 0.25 to 0.47 kgCOD/m<sup>3</sup>.d to a laboratory-scale UASB followed by an aerobic filter. COD removals of 78 to 90% were obtained, and a pilot plant was constructed on the basis of these results. Superior results were achieved without mixing; an observation that was confirmed by Heertjies and van der Meer that mechanical mixing did not improve performance of the UASB (Stronach *et al.*, 1986).

#### 3.6.4. Multistage and multiphase operations

Staging is defined as the recycle of a common biomass between two reactors. Phasing refers to the development of unique biomass in each reactor. In configuration for multistage or multiphase, the reactors are in series. In the multiple parallel single-stage reactor configurations, each parallel stream has one reactor treating it (Figure 3-5). A continuous process having a second stage operates in series at a different retention-time although performing the same conversions as the initial stage. The microorganisms can catabolise the primary stage's residual substrate. The two-stage process has greater substrate utilisation with a lower overall retention time.



Figure 3-5; Basic concept of multistage operation (Stronach et al., 1986)

The two-phase anaerobic digester is structurally similar to the two-stage system, but is based on the premise that the environmental conditions in most digesters are not optimal for both fermentative and methanogenic microorganisms. The sequential biochemical conversions occurring during digestion are

attributed to discrete microbial populations that must exist symbiotically to ensure maximum system efficiency. A fragile balance exists between VFA production and utilisation (section 3.5.). If the biphasic reaction process can be physically separated by dialysis or kinetic control, so that both phases can operate under optimal conditions. Advantages of two-phase digestion include:

- Optimised environmental conditions for both acidogenesis and methanogenesis
- Altered intermediate product formation (acetate is the principal VFA formed)
- Minimised hydrogen concentration and maximised free energy for propionate conversion
- Minimised residual VFAs in the effluent
- Increased potential for organic loading rates (OLR)
- Enhance methane yields , and
- Substantial increase in solids reduction or retention (Speece et al., 1997)

## 3.6.5. The Anaerobic Baffled Reactor

The anaerobic baffled reactor (ABR) is a reactor design which uses a series of baffles to force the waste water containing organic pollutants to flow under and over (or through) the baffles as it passes from the inlet to the outlet (Figure 3-6). Bacteria within the reactor gently rise and settle due to flow characteristics and gas production but move down the reactor at a slow rate (Nachaiyasit and Stuckey, 1997). The main driving force behind the reactor design has been to enhance the solids retention capacity and treat difficult wastewaters. The ABR is simple and inexpensive to construct because there are no moving parts or mechanical mixing (Polprasert *et al.*, 1992).



Figure 3-6: The most common design of the ABR (Boopathy et al., 1988)

Probably the most significant advantage of the ABR is its ability to separate acidogenesis and methanogenesis longitudinally down the reactor. It behaves as a two-phase system but without the associated control problems and high cost (Barber and Stuckey, 1999). Two-phase operation can increase acidogenic and methanogenic activity by a factor of up to four, as different bacterial groups develop under more favourable conditions. Having a continuous gas space above the chambers enhances reactor stability

by shielding syntrophic bacteria from elevated levels of hydrogen, which are found in the front compartments of the reactor.

#### 3.6.5.1. Bacterial populations under phase separation

In the ABR various profiles of microbial communities may develop within each compartment. The ecology of each chamber will depend on the substrate and the amount of it present. Other factors such as pH and temperature also have an effect. The most common observation in the population shift is that of the acetoclastic methanogens *Methanosarcina* sp. *Methanosaeta* sp.; *Methanosarcina* has a doubling time of 1.5 days compared to 4 for *Methanosaeta*. At high acetate concentrations *Methanosarcina* outgrows *Methanosaeta*; however at low concentrations *Methanosaeta* is dominant because of its scavenging capability ( $K_s$ = 30 mg/L compared with 400 mg/L for *Methanosarcina*) (Barber and Stuckey, 1999). Other observations that have been made are summarised in **Appendix II**.

#### 3.6.5.2. Hydrodynamics

In 1992, Grobicki and Stuckey conducted a series of hydrodynamic studies on the ABR. They found low levels of dead space, less than 8% for an empty reactor; other designs have between 50 to 90% in an anaerobic filter and 80% for a CSTR. The presence of biomass had no significant effect on hydraulic dead-space, which was found to be function of flowrate and the number of baffles. Biological dead-space was established as the major contributor to the overall dead space at high HRT. Its effect decreased at low HRT because gas production prevented channelling within the biomass bed (Grobicki A. and Stuckey D.C., 1992)

#### 3.6.5.3. Solids retention

The main driving force behind the ABR design has been to enhance the solids retention capacity. The longer the solids stay in the reactor the longer the time available for biodegradation to occur. Boopathy and Sievers managed to measure the solids retention time for two hybrid reactors running at a retention time of 15 d (Barber and Stuckey, 1999). The three-compartment reactor had a solids retention-time of 25 d compared to 22 d for a two-compartment reactor. If the reactor manages to develop a sludge blanket its capacity to trap particles increases.

## 3.6.5.4. Treating low strength wastewater

Low strength wastewater can be described as those wastewaters with COD less than 2 000mg/L, which contain a variety of biodegradable compounds such as short chain fatty acids, alcohols, VFA, carbohydrates, lipids and proteins. Low strength wastewaters inherently provide a low mass transfer driving force between biomass and substrate (Kato *et al.*, 1997). As a result these waters encourage the dominance of scavenging bacteria such as *Methanosaeta*. No substantial change occurs in biomass along the length of the reactor, indicating the lack of population selection at low COD concentration (Barber and Stuckey, 1999). Decreased overall gas production has been noticed with increase in HRT, which suggests starvation

of biomass in the later compartments. Data on the performance of the ABR on low strength wastewaters is shown in **Table 3-3**.

Wastewater	HRT	COD (mg/L)		COD Removal	OLR	Gas
	(h)			(%)	(Kg m <sup>3</sup> /d)	Produced
		IN	OUT			(v/v.d)
Greywater	84	438	109	75	0.13	0.025
Greywater	48	492	143	71	0.25	0.05
Greywater <sup>a</sup>	84	445	72	84	0.13	0.025
Sucrose <sup>b</sup>	6.8	47	74	74	1.67	0.49
Sucrose <sup>b</sup>	8	473	66	86	1.42	0.43
Sucrose <sup>b</sup>	11	441	33	93	0.96	0.31
Slaughterhouse	26.4	730	80	89	0.67	0.72
Slaughterhouse	7.2	550	110	80	1.82	0.33
Slaughterhouse	2.5	510	130	75	4.73	0.43

 Table 3-3: Performance of the ABR on low strength wastewater (Barber and Stuckey, 1999)

<sup>a</sup>Temperature at 25 °C. bTemperature lower than 16 °C. All other work at performed at mesophilic temperature range.

The results show that the amount of gas produced is proportional to the organic loading rate, COD removal and hydraulic retention-time. The hydraulic retention-time is dependent on the temperature and type of substrate. Sucrose had the shortest retention time because it soluble and readily hydrolysable. Greywater had the longest retention time because it is a complex substrate, a mixture of soluble, readily-hydrolysable, slowly hydrolysable and particulate substrate. The particulate and the slowly-hydrolysable substrates need more time to be treated.

#### 3.6.5.5. Recovery of reactor from shock loads

At high loading rates, imbalances between acidogens and methanogens may lead to the accumulation of intermediate acid products thereby exceeding the buffering capacity of the environment and causing the pH to drop to a level that inhibits methanogens (Cohen *et al.*, 1981).

The variable nature of wastewaters requires the reactor to be stable to shock loads. Shock loads can manifest themselves in two ways: either as a short term transient slug which lasts a few hours, or as a long term step change lasting for days or weeks before reversing back to the original operating condition. The microbial response to both these shock loads are identical, however the long-term shock leads to a new steady state. Performance of the reactor in the new steady state may not be the same as the previous one (Nachaiyasit and Stuckey, 1997).

The hydraulic flow pattern in the ABR causes the bacteria to move horizontally down the reactor very slowly giving rise to cell retention time (CRT) of 100 d at 20 h HRT (Nachaiyasit and Stuckey, 1997).

Systems with high CRT such as the ABR in contrast to CSTR require a considerably longer time to establish a new steady-state. The accepted norm is three HRTs for a CSTR (Nachaiyasit and Stuckey, 1997).



Figure 3-8: COD profile of each compartment after the shock load with a readily hydrolysable substrate at an HRT of 20h (Nachaiyasit and Stuckey, 1997)

As the shock wave moves down the reactor, the size of the COD peaks decreased. Two days after the shock the peaks flattened out but at a higher COD level than at time zero (Figure 3-8). It was concluded that the reactor was stable to high shock loads and responded quickly. The pH initially rose and dropped dramatically in compartment 1 and 2. It stayed constant in compartment 3 and increased in compartments 4 to 8 (Figure 3-9). The decrease in pH in compartments 1 and 2 was the result of increased VFA production leading to a build up. The increases in pH in compartments 4 to 8 were due to increased buffering capacity from increased feed.



Figure 3-9: pH profile of each compartment after the shock load with a readily hydrolysable substrate at an HRT of 20h (Nachaiyasit and Stuckey, 1997)

	ADVANTAGES						
	CONSTRUCTION		OPERATION				
1	Simple design and inexpensive to construct	I	Low HRT				
2	No moving parts	2	Intermittent operation possible				
3	No mechanical mixing	3	Extremely stable to hydraulic shock loads				
4	High void volume	4	Protection from toxins in influent				
5	Reduced clogging	5	Long operation times without de-sludging				
6	Reduced sludge bed expansion	6	High stability to organic shocks				
7	Low capital and operating costs						
	BIOM	ASS					
1	No requirements for biomass with unusual settling prop	perties					
2	Low sludge generation						
3	3 High solids retention times						
4	4 Retention of biomass without fixed media or solids settling chamber						

## Table 3-4: Table summarising advantages of the ABR (Barber and Stuckey, 1999)

## **Chapter 4** The pilot scale anaerobic baffled reactor

The purpose of study is to ascertain whether the ABR would be suitable for use in dense peri-urban areas. It is hoped the ABR could offer an immediate solution to the sanitation problem in dense peri-urban areas where it could be used to treat the domestic wastewater of small groups within a community. The wastewater treatment system that would be implemented in the dense peri-urban area should comply with the following:

- It must not require electricity
- Should be compact
- Require low maintenance
- It can be operated by a member of the community and
- Should be designed such that it can be mass-produced by unskilled labourers.

The primary consideration is to remove contact between the people and the faecal matter. The ABR will remove COD and reduce the solids content of the combined sewage and grey water. Ammonia, nitrates and phosphorous are generally not removed during anaerobic digestion. Therefore there will be no nitrogen and phosphorous removal, and very little pathogen removal at ambient temperature (Tilche *et al.*, 1996). If necessary then a separate treatment process will have to be considered for nutrient removal.

## 4.1. Design of the pilot reactor

Priyal Dama designed the pilot-scale reactor based on the research carried out by Mudunge and Bell on 10 L Perspex ABR at the university. The results obtained from the lab scale reactor were presented by Mudunge 2000, and Bell 2002.

Mudunge investigated the performance of the 8 compartment 10 L ABR operating at 20 h HRT at different organic loading rates. He used a sucrose-protein synthetic medium (section 5.1.3.). The ABR was able to handle OLRs of up to 39 kgCOD/m<sup>3</sup>.d. The reactor was fed with feed of 8, 16, 32 and 64 gCOD/L; it achieved a COD-removal of 66% throughout the trial. He concluded that the removal was lower than the reported values because loading rate was being increased before the reactor could reach steady-state. The alkalinity of the system increased with increased organic loading and so did the pH. Alkalinity increased from 200 to 14 000 mgCaCO<sub>3</sub>/L and pH increased from 6.9 to 7.8. This was due to the increased alkalinity entering with the stronger feed **c.f. section 3.5.** (Mudunge, 2000). Bell using the same medium obtained COD removal between 90 to 99% at each HRT when steady-state was reached (Bell, 2002). In her studies she concluded that changes in the HRT affected the operation of the reactor, however, recovery from these upsets was almost immediate, and operation of the reactor was stable. The compartmentalised design of ABR allows for the development of various profiles of microbial communities across the reactor. Fast

growing bacteria capable of growth at high substrate levels and reduced pH dominate in the earlier compartments; whilst scavenging bacteria grow better at high pH dominate in the latter compartments. Microbial characterisation studies carried out by Bell (2002) · showed high concentrations of *Methanosarcina* in the front of the rector and higher concentrations of *Methanosaeta* in the latter compartments.

Some of the success of the ABR design is attributed to the fluid flow pattern in the system. It results in the retention of solids and the development of specialised microbial communities in the various compartments across the reactor. It was necessary to optimise the design to reduce upflow velocities such that the settling velocity of the biomass is greater than the upflow velocity. It became important to investigate the effect the design changes would make to the flow in the reactor. The flow patterns were modelled on FLUENT, a computational fluid dynamic (CFD) programme. The resultant flow patterns were compared to those observed in dye-tracer studies.

Two grids were set-up on the program. The first grid had the baffle in the centre of the compartment, and in the second grid, the baffle was placed such that the upflow to downflow area ratio was 3:1. The velocity-contour profiles along a transverse plane for the two baffle positions are presented in **Figure 4-1**. The light-grey regions represent areas of higher flow. A uniform distribution of flow was attained with configuration **B**. As expected, a greater surface area for the upflow region resulted in lower upflow velocities. However, increasing the upflow surface area also resulted in greater volume of deadspace and channelling. The height of the baffle above the bottom of the reactor is another important factor to be considered in the design. Flow at this region has to be sufficiently high in order to reduce clogging. Higher velocities can be obtained by reducing the distance between the bottom of the baffle and the reactor bottom. Very low areas would, however, also promote clogging (Dama *et al.*, 2001).



Figure 4-1: Velocity contour plot for transverse section through a single compartment of the ABR.

The flow patterns in the 10 L reactor will differ from that in the 3 600 L reactor. One would expect a greater probability of deadspace in the larger reactor. A simple scale-up of the 10 L reactor can result in an unstable system as the height to width ratio would be too large. A decision was made to construct a shorter, wider reactor.

Due to the lack of sanitation in peri-urban communities, very little information was available on expected flow rates in these areas, calculated guess were made when determining the size of the pilot reactor. Dama assumed that the communities would use below the 6 000 L per month per household free water. Of this, between 60 and 70 % would go down the sewer. It was recommended that initially, communities would be divided into groups of fifty households. The expected flow to the reactor was thus calculated to be between 6 000 L per day. At a 12 h HRT, an ABR with a working volume of 3 500 L would be large enough for the community.

The pilot reactor was built as a trial reactor with an intended life-span of one to two years. Mild steel was selected as the material of construction. The sheets were laser cut and welded together from one end to end to form gas-tight compartments. The dimensions and other specifications can be found in **Appendix III**.



Figure 4-2: Photograph of the pilot-scale ABR at Kingsburgh WWTW

Several 25 mm sockets were added for sampling purposes. Galvanised ball valves were attached to the top and bottom socket of each compartment, for sampling. Galvanised plugs were used to plug the other sockets. A 75 mm socket was added at the bottom of each compartment to facilitate easy emptying of the compartment. These sockets were plugged using galvanised 75 mm plugs. PVC plugs were used on the 75 mm sockets at the top of the reactor. The 6 mm sockets at the top of the reactor were for gas measurements.

For the purposes of the research a pump was needed to pump the sewage from the inflow channel at the

works after the screening and de-gritting stages The submersible Dolmo 7 pump has a capacity of 100 L/min. at a 3 m head, which is much higher than the desired flowrate to the reactor (2.83 L/min for a 20 h HRT). A throttling valve could not be used due to the high risk of clogging therefore a splitter box (Figure 4-2) was built in order to divert the excess flow back into the channel.

The splitter box was divided into 3 chambers with the aid of baffle plates. The effluent is pumped into the middle chamber. Weirs are cut into the baffle plates to divide the flow such that a large percentage of the flow enters the return chamber and the rest enters the feed chamber. A 100 mm pipe leads from the return chamber back into the channel. The feed chamber contains 3 outlets. A butterfly control valve (FC1) was fitted on the lowest outlet. This valve is opened when the flow exiting the reactor is greater than the desired out-flow.



Figure 4-3: Schematic diagram of the splitter box to control flow into the reactor

The outlet flowrate was recorded using a magnetic flow meter and pulses from the magnetic flow meter were registered on the Programmable Logic Controller (PLC). The pulses obtained at the outlet are compared to a set point programmed on the PLC at fixed time intervals, and the valve (FC1) is opened when the pulses exceed the set point and closed when the counts are lower than the set point. When FC1 is opened, the effluent in the feed chamber is returned to the channel. When this valve is closed, the level rises and the feed enters the reactor. The third outlet is a safety measure in case of a blockage in the reactor (Figure 4-3). The feed rate was kept at constant rate with the aid of a PLC. The function of the PLC is to maintain a constant flow, to enable the implementation of diurnal flow patterns, and to log data.

The PLC was also used to facilitate gas measurement of the individual compartments of the ABR. A photograph of the gas measuring system for the ABR is presented in **Figure 4-4**. Solenoid valves were fitted to each gas line exiting the reactor. The valves are opened periodically with the aid of the PLC. These lines are fitted onto a manifold. The manifold is attached to a tee-piece with a solenoid valve attached to one end and a U-tube at the other. A fixed volume of acidified water was filled into the U-tube and the gas entering the U-tube displaces the acidified water. Level probes were set-up to record a fixed volume displacement. The second solenoid valve opens to vent the gas measured and the total volume of gas produced in each compartment is recorded by the PLC.



Figure 4-4: Gas measuring system for the ABR

Figure 4-5 shows the plant layout that was used by Dama at Umbilo WWTW; the same layout was used at Kingsburgh WWTW.



Figure 4-5: Flow diagram showing the pilot-plant layout

## 4.2. Previous studies operating the pilot-scale reactor

Umbilo sewage works was selected as the location for the first trial on the reactor. The treatment works received approximately 50% domestic waste and 50% industrial effluent. The reactor was fed with screened and de-gritted sewage by means of a submersible pump. The trial was to provide an opportunity to get the reactor operating and sort out operational problems before moving the reactor to a treatment works treating a higher proportion of domestic waste. The main objective of this study was to evaluate the minimum operational requirements of the reactor and improve the efficiency of the reactor to allow for a low maintenance system. The system of gradually increasing the loading by reducing the hydraulic retention time was used as the start-up strategy in order to speed up the process of biomass build-up in the reactor.

The influent COD at the WWTWs varied considerably, and the initial hydraulic retention time was 60 h, then the HRT was reduced to 32 h, and then to 20 h. The COD reduction at a 60 h HRT was below 60%. The reduction was increased to 80% when the HRT was increased to 32 h. Most of the COD reduction was taking place in the earlier compartments. COD reduction of between 70 and 90% was noted at a 20 h HRT (WRC report project No. 1248, 2001).

The pH in compartment 1 was lower than in compartment 8 ca. 6.5 and 6.9 respectively. The alkalinity results indicated that the reactor recovers very quickly from situations of low alkalinity, and low outlet alkalinity values coincided with a poor COD removal in the ABR. Phosphorous is not generally removed during anaerobic digestion, but phosphorous tests indicated there was phosphorous reduction in the ABR and it was possibly due corrosion in the reactor. Ammonia analyses are carried out on inlet and outlet samples showed there was an increase in the ammonia concentration in the outlet. This was due to the breakdown of proteins to release ammonia.

Gas measuring at Umbilo proved to be difficult and data was unreliable. The changing liquid levels within the reactor from clogging and intermittent feeding meant the gas pressure within the reactor was continually changing but not from the production of gas alone.

The anaerobic baffled reactor (ABR) was operated at Kingsburg Waste Water Treatment Works (WWTW) with the following aims:

- To obtain data required for the design of a full-scale plant.
- To evaluate the COD removal
- Resistance to shock loading
- To investigate the effect of diurnal flow variation on the performance of the reactor

## 4.3. Operational difficulties and modification to the reactor

The problems associated with gas measurement meant no gas was going to be measured for this trial. The main problems experienced during the operation of the plant at Umbilo WWTW were the clogging of the pump. A plate was fitted under the pump to stop rags from being sucked-up the pump and a system was placed to ensure that the pump was cleaned twice a day and this worked reasonably well.

## 4.3.1 Pump blockages

The problems of pump clogging continued at Kingsburgh WWTW in the 2002 trial period (Figure 4-6). A plate fitted around the suction end of the pump proved very ineffective at Kingsburgh in preventing rags from entering the pump.



Figure 4-6: The plate proved very ineffective in preventing rags from entering the pump

In 2003, the pump was housed in a meshed basket. The basket was effective in reducing the number of incidents caused by clogging of the pump (Figure 4-7).



Figure 4-7: Pump housed in a meshed basket

#### 4.3.2 Outlet blockages

Several incidents of blockages of the reactor outlet occurred in 2002. Because of the 30 mm bore in magnetic flow meter, a smaller diameter outlet pipe had to be used and resulted in regular blockages at outlet. Lumps of sludge and small rags were getting caught in the magnetic flow meter and in the pipe. A blockage results in the liquid levels rising within the reactor and may lead to biomass washout when the cause of the blockage is removed and excess liquid is discharged. The pipe before the magnetic flow meter was cut to accommodate a small open space section in which a sieve plate was placed to remove the lumps of sludge and rags from the effluent before entering the meter magnetic flow meter. In field trials the outlet need not be restricted by a measuring device.

## 4.4. Sample collection and storage

Grab samples were obtained from the reactor feed box, and at the outlet pipe. Samples of the sludge column in the upflow region of each compartment were obtained using a specially designed sampling column, then mixed in a bucket and sampled for analysis. For the complete sampling and analysis protocols, see **Appendix III**.

### 4.5. Operational results

Due to operational difficulties experienced in 2002, most of the results presented in this chapter will be from the work undertaken in 2003.

## 4.5.1 Feeding the reactor (total flow)

Following the outlined in section 4.3.2., and a rigid maintenance schedule described in Appendix III. The schedule was drawn from experiences gained in 2002. The number of incidents was reduced for the trial period of 2003.



Figure 4-8(a): Graphical illustration of the incidents that caused interruptions in the feeding of the reactor in 2002

Log of eve	Log of events and incidents during operation of the ABR at Kingsburgh WWTW in 2002					
Date	Day of operation	Event / Incident				
2-Jul	0	PLC Control – set to 20 h HRT				
3-Jul	1	Blockage of outlet by fat/scum before magnetic flow-meter. Reactor overfilled, and then washed out. Some biomass lost.				
8-Jul	6	Once pumped stopped, outlet seemed to block - reactor overfilled. Possible loss of biomass during emptying.				
22-Jul	20	No flow (the high flow of water into the WWTW after rains caused flexible pump discharge hose to be twisted, pump slightly blocked, plastic in magnetic flow-meter). All blockages cleared, but reactor overfilled, loss of biomass during emptying. Rain wetted inside of PLC box. Entry point unknown.				
24-Jul	22	PLC confirmed damaged - reverted to timer control				
25-Jul	23	Set the flow rate to close as possible to 20 h HRT using timer control only				
29-Jul	27	Outlet blocked, reactor overfilled. Possible biomass loss during emptying				
l-Aug	29	Outlet blocked, reactor overfilled. Possible biomass loss during emptying				
19-Aug	47	No power; rain entered control box and tripped PLC				
26-Aug	54	No power; rain entered control box, but pump tripped circuit				
10-Sep	68	New enclosure installed, wires connected, pump power restored - reactor on: timer control				
13-Sep	71	PLC Control - 20 h HRT				
19-Sep	77 .	Pump capacitor damage identified (causing tripping) pump removed for repair				
30-Sep	88	New pump installed - 20 h HRT				
<u>5-Oct</u>	93	Loose power wire in enclosure-no power for 2 days (3/4 Oct)				
9-Oct	97	Pump overload manual reset installed				
10-Oct	98	Reset not working, pump stopped since 9 Oct pm				
24-Oct	112	Outlet blocked, reactor overfilled. Possible biomass loss during emptying, pump earth leakage, changed pumps				
4-Nov	122	Blockage of outlet reported and cleared by operator. Some biomass lost when connection opened, and probably washout during emptying of reactor.				
8-Nov	126	Effluent sour - checked and found consistently low pH (<5) across reactor. Flow rate dropped (halved). Worker reported coloured wastewater the previous day, but it appears to not have affected the activated sludge plant.				
11-Nov	129	Earth leakage problem-not associated with ABR. Power off on arrival - not certain when trip occurred. Reactor overfilled. Possible sludge loss during emptying.				
13-Nov	131	Overload tripped on previous afternoon. Removed for repairs and bypassed overload relay switch				
18-Nov	136	Pump not properly submerged. Possible damage to pump				

## Table 4-1: Details of incidents that occurred in 2002

The incidents in 2002 represented by the vertical lines in Figure 4-8 (a) were so numerous that the target HRT of 20 hrs could not have been reached. The souring incident (highlighted) is not shown in Figure 4-8(a) because it did not cause any downtime but the incident presented an opportunity to study the reactor recovery from organic overloading or shock loads (section 4.7.).



Figure 4-8(b): Graphical illustration of the incidents that caused interruptions in the feeding of the reactor in 2003

Log of events and incidents during operation of the ABR at Kingsburgh WWTW in 2003					
Date	Day of operation	Event / Incident			
17-Feb	0	Started 2003 trial – set PLC 20 h HRT			
3-Mar	14	Pump jammed by strings and tripped the overload switch			
22-Mar	33	Pump blocked and overload switch tripped			
14-Apr	56	Heavy during the previous nigh, the heavy water flows into the WWTW shifted the pump from its position in the channel and tripped the overload switch			
26-Apr	68	String caught in the pump chamber, the pump did not trip the switch but the pump was not generating enough head to feed the reactor			
28-Apr	70	Overload switch tripped			
12-May	84	Pump was not generating enough head to feed the reactor. Pump was cleaned and reset (pump possibly damaged).			

<b>Table 4-2: D</b>	etails of the	e incidents th	hat occurred	in 2003
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For 2003, the amount of effluent coming out of the reactor was recorded and corrections were made i.e. if there was downtime feeding was increased and this was done with the aid of the PLC. The PLC was able to maintain a constant flow into the ABR but it did not reach the target HRT of 20 h. The system for setting and tuning the set points relies on the number of pulses the PLC receives form the magnetic flow-meter which was 240 pulses per minute. This proved to be a coarse method for tuning the set point; the set points could not be set any closer without exceeding the target flowrate of 2.66 L/min for a 20 h HRT. The average flow to the ABR was 3015 L/d and gave an average HRT of 22.5 hrs (Figure 4-9).





Figure 4-9: Cumulative flow of effluent treated by the ABR between February and June 2003

### 4.5.2 Alkalinity and pH

The basic function of alkalinity in anaerobic processes is to buffer the carbon dioxide acidity, not the volatile acids. Alkalinity was tested on a weekly basis since it provides an indication of potential reactor failure; a decrease in outlet alkalinity could be a warning that the reactor may be approaching failure c.f. section 3.5. The average alkalinity of influent was  $194 \pm 47 \text{ mg/L}$ , and the effluent was  $248 \pm 37 \text{ mg/L}$ . From influent to effluent there was an average generation of 54 mg/L of alkalinity in the reactor (Figure 4-10) leading to effluent alkalinity being greater than the influent alkalinity. The drop of alkalinity in both influent and effluent happened on Day 56, the same day the WWTW was flooded after a heavy downpour from the previous night's storm (Table 4-2).



Figure 4-10: Alkalinity profiles of the influent and effluent from the ABR

The alkalinity profiles of compartments were taken on Day 44 of operation, this was midway through the trial and there had been no incidents close to the day of sampling. The results show higher alkalinities within the reactor, and they increase to 529 mg/L in compartment 7 and 8 (Figure 4-11). This is due to the degradation of cation-releasing organic species such as proteins and soaps. Some of this alkalinity is lost in the discharge pipe and this occurs very quickly suggesting stripping of or carbon dioxide (section 3.5.).



Figure 4-11: Alkalinity profiles within the reactor on Day 44 of operation

In a properly operating anaerobic process there should be a slight decrease in alkalinity from influent to effluent (section 3.5.); the results indicate that there was low production of carbon dioxide in the reactor in latter compartments. The low production of carbon dioxide suggests there is no or not sufficient acetotrophic methanogenesis taking place (section 3.4.). Figure 4-11 shows that even in the last compartment there very little acetotrophic methanogenesis happening or reduction of alkalinity. This parallels with a poor COD removal in the ABR (section 4.5.3.).

At least 2000 mg/L of alkalinity are required to buffer a liquid to pH = 7 for a high strength wastewater (Speece, 1996). However, the alkalinity concentration of both the influent and effluent are around 200 mg CaCO<sub>3</sub>/L, 10 times less than the value recommended by Speece for a mixed reactor. This does not mean the reactor is close to failure as the amount of alkalinity required and generated for stable operation is proportional to the strength of the wastewater being treated.

Methanogens are sensitive to pH and prefer nearly neutral pH conditions with a generally accepted optimum range of 6.5 to 8.2. The pH values in the different compartments provide an indication of the performance of the reactor. The earlier compartments generally have a lower pH value as acidogenesis and acetogenesis start proceeding in these compartments (Figure 4-12). The pH drops in compartment 1 and recovers slightly in compartment 2 and is almost constant to the outlet.





Figure 4-12: pH profiles by compartments

The pH profiles within the reactor and of the effluent from the ABR are dependant on the pH of the influent. (Figure 4-13). There is an approximate decrease of 0.3 pH units from influent to effluent. The drop in pH on Day 46 was not connected with any incident except the pH into the wastewater was low and the pH within the reactor responded accordingly, on the same day there was a slight drop in effluent alkalinity.



Figure 4-13: pH curves of influent and effluent from the ABR

Table 4-3 gives the major chemical species involved in wastewater pH chemistry and their K<sub>a</sub> values.
Chemical Species	Chemical formula	K <sub>a</sub> value	pK <sub>a</sub> value
Phosphoric acid	H <sub>2</sub> PO <sub>4</sub>	6.2 × 10 <sup>-8</sup>	7.2
Ammonium	NH₄⁺	1.8 × 10 <sup>-5</sup>	4.8
Carbonic acid	H <sub>2</sub> CO <sub>3</sub>	$4.2 \times 10^{-7}$	6.3
Bicarbonate	HCO <sub>3</sub> -	$2.4 \times 10^{-8}$	7.62

Table 4-3: Major chemical species involved pH chemistry of wastewater

The analysis of pKa values suggest that the combination phosphate, carbonic acid and bicarbonate play the biggest role in keeping the pH within the range of 6.5 to 7.5 within the reaction (Figure 4-12). If the quantity of the species increases but the ratios remain the same, then pH will essentially remain the same but alkalinity will increase.

The profiles of compartment 3 to 8 indicate high stability of pH in the reactor within a pH range from 6.2 to 6.8. The average pH in the eighth compartment was  $6.46 \pm 0.18$  indicating the presence of organic acids to the last compartment. The pH in the reactor was low and was on the boundary limit of the permissible pH for methanogenesis to occur. The low pH is probably the reason why methanogenesis could not occur.

#### 4.5.3 Chemical oxygen demand

Figure 4-14 is a graph of grab influent and effluent samples for chemical oxygen demand. Two effluent measurements are presented; whole effluent and filtered effluent. The latter is filtered through 0.45  $\mu$ m acetate filters. The filtrate COD is measured to provide an indication of the COD of the effluent that would be obtained after passing through a secondary filtration unit; this is the residual COD. It has been pointed out that influent and effluent samples taken at the same time do not give an accurate calculation of COD removal due to the reactor residence time. Consequently, in an attempt to record "true" COD removal, effluent samples were taken 20 hours after the influent samples were taken approximately one retention time apart.



Figure 4-14: COD values of influent, effluent, filtered samples, COD removal and incident lines

#### Chapter 4

The average influent COD was  $721 \pm 147 \text{ mg/L}$  and average effluent COD was  $196 \pm 61 \text{ mg/L}$  with an average COD removal of 71%. The average residual soluble COD in the effluent was 82 mg/L. COD removal curves mirrors the influent curve because the effluent from the reactor was stable at **ca**. 200 mg/L. On Day 56 the wastewater into the plant diluted after a storm and COD removal decreased because the COD in the effluent increased. The effluent COD was greater than that of the influent. This was hard to explain as there was no solids washout (**Figure 4-18**).

The influent, effluent and all the compartments were analysed for soluble COD. This fraction is sometimes called the readily biodegradable COD (RBCOD). This is the fraction that is readily available to the biomass to use (section 2.2.3.1.). Soluble COD increases from influent to compartment 1, and then decrease to compartment 5 and thereafter it remains constant to discharge (Figure 4-15). Hydrolysis increases soluble COD in compartment 1; hydrolysis exceeds consumption in this compartment. From compartment 2 to 5, consumption exceeds hydrolysis hence the decrease in soluble COD. From compartment 6 to effluent there is no decrease in soluble COD suggesting hydrolysis and consumption are equal but in the latter compartments we expect hydrolysis to have been completed. The COD consumed is consumed mainly by the acidogens for their own respiration and growth since methanogenesis was not happening (section 4.5.2.).

It interesting to note that from compartment 6 there is no decrease in soluble COD occurring, and comparing with alkalinity profiles (Figure4-11), there was a dip in alkalinity in compartment 6 which also went against the trend. This could be indicative of some activity in that compartment but cannot be certain of that.



Figure 4-15: Profile of soluble COD in the reactor from influent to effluent on Day 36 and 40

The concentration of soluble COD is leaving the reactor 100 mg/L; ideally no readily degradable COD should leave the reactor, unless this soluble COD is not biodegradable. This result indicated biochemical methane potential (BMP) tests should be conducted to examine the biodegradability of the COD in the effluent. BMP tests were performed and results are presented in **Chapter 5**.

#### 4.5.4 Solids in the reactor

The sludge levels were measured using the Coring test, which is described in **Appendix III**. Figure 4-16 shows the average liquid and sludge levels for 2002 and 2003. There was more liquid in the reactor in 2002 than in 2003. The numerous blockages and partial blockages in 2002 in the discharge pipe caused the liquid levels within the reactor to increase, because of the unequal rate of feeding to that of discharging. There was twice the amount of sludge in the reactor for 2003 than in 2002. Better feeding allowed solids to accumulate especially in the first and last compartments.



Figure 4-16: Average liquid and sludge levels in the reactor for 2002 and 2003

The sludge levels in the compartments 1 and 2 were highly variable in 2002 and 2003. Sometimes the sludge levels would rise because of the presence of a fatty scum. The scum was thick and floats, and was present in compartment 1 for most of the trial period in 2003. It presented no operational difficulty, except when the liquid levels were high and it blocked the inlet hole. In 2003 there was more sludge in compartments 6, 7 and 8 than the previous year. There is possible slow but significant movement of sludge down the reactor since acidogenesis is the only phase of anaerobic digestion occurring in the reactor.



Figure 4-17: Characterisation of solids for each compartment on Day 50 of operation in 2003

Characterisation of solids for each compartment showed that there was increasing biomass down the reactor with the exception of compartment 6 (Figure 4-17). Compartment 6 has been the exception in a number of results (Figure 4-15 and 4-11). The results also show that the solids in compartment 1 and 2 although occupying a large volume of the compartments were light because they had low TSS and VSS. This was indicative of the presence of the fatty scum in the compartments 1 and 2.

### Table 4-4: Ratios of TSS to VSS for each compartment

Compartment	1	2	3	4	5	6	7	8
Ratio	3.1	3.0	3.3	3.3	3.0	1.3	2.4	2.2

Table 4-4 shows that the ratio of TSS: VSS is ca. 3 for compartment 1 to 5, for compartment 6 the ratio decreases to 1.3, and increases to above 2 for compartment 7 and 8. The amount of VSS in compartment 6 is close to that of TSS suggesting the compartment consists mainly of biomass, and this happens in conditions of excess substrate (section 2.2.2.). This links in with the results in section 4.5.3., where soluble COD was present throughout the reactor.



Figure 4-18: Total suspended solids in and out of the ABR

Figure 4-18 shows total suspended solids concentrations of the influent and effluent streams of the ABR. All effluent TSS values were below 400 mg/L with an average of  $225 \pm 96$  mg/L. The removal curve follows the influent curve, because the amount of solids coming out of the ABR does not vary with the influent. On Day 56 the influent TSS decreased with the dilution of storm water but this did not decrease the amount of TSS in the effluent. This suggests that the amount of TSS in the effluent is above all related to the liquid upflow velocity within the reactor. The average TSS removal was ca. 50%.



Figure 4-19: Volatile suspended solids in and out of the ABR

Figure 4-19 shows the volatile suspended solids in the effluent like TSS were they were not dependent on the amount coming into the reactor. The average VSS of the effluent was ca. 100mg/L and the removal was about 60%.

#### 4.5.5 Discussion

The number of incidents was reduced for the 2003 trial period. With the aid of a PLC the feeding to the reactor was kept constant and an average HRT of 22.5 h was achieved. This allowed the solids levels increase to about twice the amount obtained in 2002. The alkalinity in the effluent was greater than that in the influent indicating low production of carbon dioxide which is produced in acetotrophic methanogenesis, indicating a low rate of methanogenesis. The 100me/L of soluble COD leaving the reactor confirmed the incomplete degradation of COD in the reactor. The pH inside was stable and almost constant from compartment 2 to discharge at low values that prevented methanogenesis. The actual pH profile within the reactor depended only the on the pH of the feed but the pattern was always the same. The amount solids in the effluent depended mainly on the upflow velocity within the reactor. The ratio of TSS: VSS is **ca**. 3 for compartment 1 to 5, for compartment 6 the ratio decreases to 1.3, and increases to above 2 for compartment 7 and 8. The amount of VSS in compartment 6 is close to that of TSS suggesting the compartment consists mainly of biomass, and this happens in conditions of excess substrate **c.f. 2.2.2**. The reactor is operating as a single phase digester performing primarily acidogenesis because methanogenesis was not occurring.

## 4.6. Analytical campaign

An analytical campaign was undertaken to obtain hourly variations in ABR influent and effluent characteristics. The campaign was carried out on the  $29^{th}$  of May, a week after the reactor trial period. The reactor was kept running while preparing for the campaign. Hourly samples were taken for 24 h on the influent, and after 20 h (roughly one retention time) hourly samples of the effluent were take for a further 24 h in order that corresponding profiles could be obtained. pH and 0.45 µm filtered COD analyses were performed during the campaign. Due to the large number of samples taken, the measurement was performed in duplicate only by the closed reflux method so that samples could be analysed before significant changes in composition could occur. Samples were homogenised using a hand-held blender to reduce random sampling error before acidification and storing below 4 °C.



Figure 4-20: Influent COD, effluent and filtered effluent COD profiles translated back by 20 h

The effluent curve has been transposed 20 h back in time so that effluent characteristics can be directly compared with influent characteristics. It should have been transposed back 22 h, but the calculation of the retention time at the time was done after the campaign. The influent COD profile shows an increase in COD of the wastewater coming into the plant between 10:00 and 14:00. There is no corresponding peak in the effluent COD profile indicating the reactor is able to digest the peak. The influent COD profile shows a cyclical trend with period of 1 d where the COD starts to increase at 06:00 going to 10:00. The effluent COD concentration was on average under 200 mg/L, basically remaining the same compared to the routine analysis results. The filtered effluent COD concentration were constant at  $64 \pm 6$  mg COD/L, this has decreased from 82 mg/L from the routine analysis results. The slight improvement could be due possibly to better environmental conditions on the day such as temperature. The 02:00 whole effluent sample had a COD concentration of 1 200 mg/L; the sample was black and had high suspended solid content, which caused the high COD value. However, the filtered COD values remained low indicating biomass washout. This incident is not associated with any other observed change in operating conditions or

performance and is attributed to an overflow of anaerobic sludge from the last compartment as a result of gas release or fluid dynamics in the last compartment.

The average COD obtained from the routine analysis was 721 mg/L, from the campaign the average daily influent COD is about 564 mg/L. The influent daily variation shows that the routine analysis sampling time (09:00 to 12:00) coincided with the daily influent COD peak. The average daily effluent COD from routine analysis results was 198 mg/L, but from the campaign it was 234 mg/L, it was increased by the biomass washout which occurs as a peak or slug outside the routine analysis sampling time . If COD removal is calculated from the campaign data a value of 42% is obtained as opposed to 71% from using routine analysis results.



Figure 4-21: The pH profile of the reactor with effluent pH translated back by 20 h

Figure 4-21 shows the pH profiles of the influent and effluent. The pH of the influent increases from 6.8 to 7.5 from 06: to 12:00 just as the COD profile did, but there is another peak at 17:00. The average pH of the influent was  $6.96 \pm 0.32$  with the routine analysis pH average at 7.2. The pH of the effluent was lower than that of the influent, just the results of routine analysis had. The effluent pH was constant throughout the day at  $6.36 \pm 0.07$ . The routine analysis results average pH was 6.5 but from Day 46 to the end of the trial the average pH of routine analysis was 6.3. (Figure 4-13), the effluent pH has not changed the same since the end of the trial.

The peak between 06:00 to 09:00 is between the time when people wakeup, wash, eat breakfast and go to work or school. Then thereafter house cleaning is carried out until midday (12:00), hence the increase in COD and pH of the water coming into the WWTW. It is interesting to note that the small COD peak in the late afternoon (17:00) coincides with the preparation of dinner, and 2 h later (19:00) there is a pH peak presumably from the washing of dishes. The ABR handles these daily variations very well.

## 4.7. Reactor recovery from organic shock loads

On 8 November 2002, the reactor was found to be sour. Worker reported a dark coloured wastewater the previous day, but it appears to not have affected the activated sludge plant. On discovering the incident the flow into the reactor was halved to 1.3 L/min. It was decided that the reactor should be monitored and its recovery recorded. Anaerobic digester souring occurs when the rate of acidogenesis exceeds the rate of methanogenesis, causing a build-up of acids. The pH values in the reactor drop, inhibiting the activity of the methanogenic biomass causing a greater difference in the rates of acidogenesis and methanogenesis, until the pH drops sufficiently to inhibit most of the anaerobic activity.

Causes of anaerobic digester souring are varied, but fall into three major categories. Firstly toxic inhibition: toxic components in the wastewater inhibit the activity of the methanogens, which tend to be more sensitive than the acidogens. Secondly hydraulic overload: the hydraulic flows are sufficiently high to wash out the slower growing methanogens, but not the more rapidly growing acidogens. Thirdly organic overload: a high concentration of readily biodegradable organic compounds in the wastewater causes the growth rate of the acidogenesis to exceed that of the methanogenesis.

The worker responsible for the head of works reported that on the previous day (Thursday 7 November 2002) a sour smelling black water like septic tank sludge was observed in the wastewater entering the wastewater treatment works. The wastewater treatment works composite COD for both 7 and 8 November were unusually high (1 081 and 1 166 mgCOD/L) compared to the usually observed concentration of 750 mgCOD/L. The aforementioned values were obtained from the WWTW's own results whose analysis is performed by the eThekwini Metro. No problems were observed in the plant activated sludge processes, implying that there were no toxicants in the wastewater. On enquiry, it was discovered that septic tank sludge is often illegally dumped into the sewers, which feeds Kingsburgh wastewater treatment works, but no action is taken since it seems to have little effect on the plant operation. Based on the above, it is suspected that a slug of concentrated septic tank sludge was fed to the ABR causing organic overloading, and subsequent souring. The incident has provided valuable insight into the behaviour of the ABR under organic overloading.

Figure 4-22 shows the pH across the compartments for normal operation, souring and recovery. The reactor had substantially recovered after 3 days, with the pH value above 6 for most compartments. By day 10 after the souring incident, the effluent COD concentration was around 300 mg/L (Table 4-5) and the pH was almost normal.

Table 4-5: The recovery of effluent COD numbered from the day the incident was found

Day	0	4	7	10
Effluent COD (mg/L)	2630	1347	365	310



Figure 4-22: pH Profiles of souring showing the recovery phase ending with the pH curve of normal operation for each compartment for 2002, with curve numbered relative to the day souring was first observed

The ability of the ABR to have a cell retention time of 100 d at 20 h HRT causes the reactor to require a considerably longer time to reach a new steady when organic loading changes (section 3.6.5.5.). In this case the shock load was transient and the recovery of the reactor should depend on two factors. Firstly how long does the high COD feed (peak) lasts or is fed into the reactor. Secondly the duration of the resultant drop in pH within the reactor will depend on the HRT because the low pH can be washed out much quicker than the cell can respond. Dropping the flow into the reactor while the COD in the feed was high was the correct response. But soon after the COD had returned to normal in the feed the flow should have been increased to the original HRT. This would have assisted in washing out the acids or the low pH and return the reactor to normal operation as soon as possible.

Some conclusions as to how the reactor would behave in a community under these circumstances can be drawn: In general, the ABR seems to recover quickly from this kind of souring incident (within 10 d), with day-by-day improvement in pH values. No chemical treatment was required to assist in the recovery of the reactor. However, overloading should be attempted again so that a more thorough investigation into the dynamics of recovery can be made and can measure how long it takes for the reactor to sour after the organic load was introduced.

#### 4.8. Discussion

The alkalinity in the effluent was greater than that in the influent indicating low production of carbon dioxide which is produced in acetotrophic methanogenesis, indicating a low rate of methanogenesis. Lalbahadur *et al.* concluded from the observed low pH that there is insufficient breakdown of particulate biodegradable matter is being obtained. Barely any gas-production was detected contrasting the 2.78 mol CH<sub>4</sub>/h that should have been produces to close the mass balance that was calculated from the average COD removal. This was supposed to be the minimum possible gas production rate that should be observed if the assumption of being near steady state was correct (Foxon *et al.*, 2004). COD measurements per compartment suggest that up to 95 mg COD/L in compartment 1 and 20 mg COD/L in compartment 8 may be potentially available as short chain fatty acids for acetogenesis and methanogenesis, but are currently not being released as available substrate due to incomplete hydrolysis and fermentation of particulate organic. (Lalbahadur *et al.*, 2004). The soluble COD that was detected throughout the reactor (Figure 4-11) was not in the form that is utilisable to the methanogens. This implies that the solids retention time is insufficient for the complete degradation of biodegradable particulates that are leaving the reactor in the effluent.

In a fluorescent *in-situ* hybridisation study, Lalbahadur *et al.* found no *Methanosaeta* in any compartment of the reactor for any of the months tested, while *Methanosarcina* represented 100% of the archeae in each compartment. *Methanosarcina* were found only in the early compartments, and decreased in number with each subsequent compartment. This indicated that short chain fatty acid (SCFA) precursors to methanogenesis are being scavenged at low concentrations, preventing the establishment of a healthy methanogenic population.

The average alkalinity within the reactor was about 300 mg/L  $Ca_2CO_3$ , much less than the recommended value. However this does not mean the reactor is close to failure as the amount of alkalinity required and generated for stable operation is proportional to the strength of the wastewater being treated which in this study was a low strength wastewater. The pH profiles of the system were not dependently on alkalinity because the pH of any buffering system is a function of the chemical species present.

The ABR recovers quickly from organic shock loads. The duration of the recovery for a shock that enters as a slug is depended on the HRT, but a change in organic loading induce the reactor to reach a new steadystate and this is dependent on the cell retention time. The duration of the recovery for a shock that enters as a slug is depended on the HRT, but a change in organic loading induce the reactor to reach a new steadystate and this is dependent on the cell retention time. The duration of the recovery for a shock that enters as a slug is depended on the HRT, but a change in organic loading induce the reactor to reach a new steadystate and this is dependent on the cell retention time. The reactor is susceptible to souring if the organic load is readily biodegradable, however the expected wastewater from the community will be in particulate form. Because the hydrolysis of particulate organic compounds is rate limiting in the system, then the increase of acids will be slow and prolonged, then the system can have time to adjust.

## 4.9. Conclusions

No methanogenesis was occurring in the reactor because there was slow hydrolysis of particulate matter to make available the short chain fatty acids that are required for methanogenesis. The slow hydrolysis of particulates leads to acidogenesis occurring further down the reactor causing low pH that suppress methanogenesis. The retention time of 20 h is short for complete hydrolysis of solids, the reactor should be operated at longer retention times and its performance be reassessed.

The ABR can handle the daily variations in the incoming feed very well even though it was not operating optimally. The ability of the ABR to maintain stable pH within is dependent on the pH of the incoming wastewater, the strength of the water and the ratio of the phosphate, carbonic acid and bicarbonate that are major role players in the buffering system.

The ABR recovers quickly from organic shock loads (within 10 d), with day-by-day improvement in pH values without any chemical treatment being used to assist in the recovery of the reactor. In the community the ABR is unlikely to go sour because the bulk of organic compound will in the particulate form. The slow hydrolysis of these will probably allow the system to adjust.

The amount of total biodegradable COD in effluent needs to be measured. The result can be used calculate the minimum effluent COD that the ABR can achieve.

## **Chapter 5** Biochemical methane potential tests

The ABR should operate under conditions that allow it to degrade all biodegradable constituents in the feed. The effluent should contain only un-degradable material. The biochemical methane potential (BMP) test was used to ascertain ultimate biodegradability of sewage feed and effluent from the reactor.

A simple and relatively inexpensive procedure was needed to monitor biodegradability and possible toxicity of constitutes in feed sources to an anaerobic process. There is no suitable chemical procedure available that can determine biodegradability of organic compounds, and measure the presence of toxic substances in the feed to avoid inhibiting digestion. An anaerobic assay procedure was developed. Continuous and batch techniques have been used to evaluate biodegradability. The continuous procedure closely simulates full-scale operation; however they are costly in terms of facilities and equipment, time and personnel. Batch assays techniques do not have these limitations permitting the evaluation of a wide range of substrates and variables (Owen *et al.*, 1979).

The general procedure of Owen *et al.* can be modified to estimate either biochemical methane potential or anaerobic toxicity assay (ATA). The anaerobic serum bottle containing sample, defined media and seed inoculum is incubated at the desired temperature. Gas production is monitored volumetrically using the syringe method described by Nottingham and Hungate in 1969. The liquid and gas phases can be sampled periodically for analyses or at completion of the test.

## 5.1. Experimental procedure

Experiments were performed in 125 ml glass serum bottles (Supelco, Inc.), which were sealed with a butyl rubber septum and an aluminium crimp seal.

#### 5.1.1. Preparation of nutrient medium

A defined solution containing trace elements, minerals and vitamins was prepared according to Owen *et al.* (1979). The stock solutions for the preparation of the nutrient medium are presented in **Table 5-1** and the method for preparation of medium is in **Table 5-2**.

.

Stock solution	Composition	Concentration (g/l)
S2	Resazurin	 1
\$3	(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	26.7
S4	CaCl <sub>2</sub> .2H <sub>2</sub> O	16.7
	NH4Cl	26.6
	MgCl <sub>2</sub> .6H <sub>2</sub> O	120
	KCl	86.7
	MnCl <sub>2</sub> .4H <sub>2</sub> O	1.33
	CoCl <sub>2</sub> .6H <sub>2</sub> O	2
	H <sub>3</sub> BO <sub>3</sub>	0.38
	CuCl <sub>2</sub> .2H <sub>2</sub> O	0.18
	Na2MoO4. 2H2O	0.17
	ZnCl <sub>2</sub>	0.14
S5	FeCl2. 4H <sub>2</sub> O	370
<u>S6</u>	Na <sub>2</sub> S.9H <sub>2</sub> O	500
S7	Biotin	0.002
	Folic acid	0.002
	Pyridoxine Hydrochloride	0.01
	Riboflavin	0.005
	Thiamin	0.005
	Nicotinic acid	0.005
	Panthothenic acid	0.005
	p-aminobenzoic acid	0.005
	Thioctic acid	0.005

## Table 5-1: Stock solutions for preparation of defined mineral salt solution

STEP	METHOD	Volume	Mass (g)
		(ml)	
1	1 L of deionised water was added to 2 L Pyrex bottle		
2	The following was added:		
	Stock solution S2		
	Stock solution S3	1.8	
	Stock solution S4	5.4	
3	Deionised water was added to make 1.8 L	27.0	
4	Boiled for 15 min whilst flushing with OFN gas		
5	Cooled to room temperature		
6	The following was added:		
	Stock solution S7	18.0	
	Stock solution S5	1.8	
	Stock solution S6	1.8	
7	NaHCO3 was added as powder		8.4
8	Flushed with OFN until pH around 7.1		
9	Autoclaved (30 min at 121 °C)		
10	Stored at 4 <sup>o</sup> C until use		

### Table 5-2: Preparation of the defined mineral salt solution

## 5.1.2. Preparation of biomass

Equal volumes of biomass were collected from each compartment of the ABR and mixed together. Digester sludge was collected from Umbilo WWTW digester. The sludge was dewatered by centrifuging at 5 000 rpm for 10 min, and the supernatant was discarded.

The sludge was incubated at 37 °C, a week before it was to be used in the test. This was for the biomass to acclimatise to the temperature, and to metabolise any residual substrate present in the sludge. This ensures that the methane produced during the test is from the substrate being tested.

#### 5.1.3. Substrates

- Sewage was collected from the head of the plant.
- Sucrose-protein synthetic medium was prepared in the laboratory.
- Effluent was collected from the anaerobic baffled reactor.
- Scum from the reactor, normally observed in winter

Umbilo digester sludge was used because it is the sludge normally used in our laboratory to test the biodegradability of other substances. This was to observe if there was a difference in the activity of the two sludges. Synthetic feed was included in the test because it is readily biodegradable, and its COD concentration was diluted to be the same as that of the influent wastewater being tested. This was to be a reference substrate for comparison.

#### Table 5-3: Preparation of sucrose-protein synthetic medium

Chemical	Mass (g)	
Sugar	53.34	
Peptone	16.00	
Meat Extract	5.34	
Di-potassium hydrogen orthophosphate	1.60	
Sodium hydrogen carbonate	65.00	
	in a cominal flamb	

The above chemicals were dissolved in 2 L of distilled water and sealed in a conical flask.

A mineral solution containing trace elements that bacteria require is also prepared. The mineral solution has the following chemicals:

Chemical	Mass (g)
Iron chloride 4 hydrate (FeCl <sub>2</sub> .4H <sub>2</sub> O)	15.70
Cobalt chloride 6 hydrate (CoCl <sub>2</sub> .6H <sub>2</sub> O)	2.38
Manganese chloride 4 hydrate (MnCl <sub>2</sub> .4H <sub>2</sub> O)	0.75
Sodium molybdenate 2 hydrate (Na <sub>2</sub> MoO <sub>4</sub> .2H <sub>2</sub> O)	0.75
Nickel chloride 6 hydrate (NiCl <sub>2</sub> .6H <sub>2</sub> O)	0.90

The above chemicals are dissolved in 2 L of distilled water and left for about 2 days on a magnetic stirrer to dissolve.

A volume of 40 mL of mineral solution is placed in a conical flask. The mineral solution and substrate are autoclaved separately for 30 minutes. Once the feed and the mineral solution have cooled to room temperature, they are then added together (2L of sugar-peptone soln. + 40mL mineral soln.).

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#### 5.1.4. Preparation of serum bottles

The 125 mL bottle serum bottle used for the test has a working volume of 100 mL. The bottles, biomass, nutrient medium and substrate were equilibrated at the incubation temperature of  $37 \,^{\circ}$ C. A 30 mL inoculum of biomass was used per serum bottle. The biomass was mixed with 30 mL of the defined nutrient medium. Then 40 mL of substrate was added. Each bottle was flushed with oxygen free nitrogen (OFN) for 15 min. The bottles were then sealed with a butyl rubber septum and an aluminium crimp seal prior to incubation at 37  $^{\circ}$ C. The serum bottles were prepared in duplicate.

After equilibration for 1 h, the gas volumes were zeroed to ambient pressure with a glass syringe. The excess gas at ambient pressure is withdrawn and the bottle is said to have zero volume. The serum bottles were shaken manually once a day to facilitate contact between the microorganisms and substrate (Sacks J, 1997).

#### 5.1.5. Analytical methods

The core information in the BMP is obtained by measuring and analysing the gas produced in the bottle.

#### 5.1.5.1. Gas measurement

Gas measurements were taken at the incubation temperature. Gas volume sampling and removal during incubation were performed with a graduated 50 mL syringe. The syringe was first lubricated with distilled water. The syringe needle was inserted through the rubber septum into the headspace. The syringe was held vertically for measurement. Volume determination was made by allowing the syringe plunger to move and equilibrate between bottle and atmospheric pressure. The reading was verified by drawing the plunger past the equilibrium point and releasing to ensure that plunger returned to original equilibration volume .Gas was only vented to atmosphere if the difference between internal and atmospheric pressure was greater than 0.5 atm (12 mL). After wasting, 12 mL of gas was injected back to the bottle (Sacks J, 1997).

#### 5.1.5.2. Gas composition

The composition of the gas was analysed with a GOWMAC-350 gas chromatograph equipped with a thermal conductivity detector (TCD), which could detect methane, carbon dioxide and nitrogen peaks.

A biogas sample was drawn from the serum bottle with a 100  $\mu$ L precision syringe. 20  $\mu$ L of gas were injected to the chromatograph. The gas was sampled just after wasting so the gas pressure in the bottle during sampling was at 0.5 atm. The carrier gas was Helium at a flow rate of 5 mL/min. The residence time for nitrogen, carbon dioxide and methane were 0.8, 1.27 and 2.4 sec respectively. The peak area was recorded with a Varian integrator model 4270 with the attenuation set at 1.

Settings for GOWMAC-350

Column oven temperature	: 25°
Detector temperature	: 30°C
Carrier gas	: Helium
Detector current	: 100 mA
Attenuation	: 1

## 5.1.6. Gas chromatograph calibration

In the analysis of gas from anaerobic digestion, 3 gases are normally of interest as they are the major gases produced. These are nitrogen, carbon dioxide and methane. In this calibration nitrogen was omitted.

The calibration curves were prepared by injecting increasing volumes of high purity carbon dioxide or methane at 1.5 atm as this was the sampling pressure (ambient pressure, 1 atm, + 0.5 atm due to 12 mL of gas injected back into the bottle). The gases used were of technical grade which has a purity of 99%. The settings for the GC were as stated previously (section 5.1.5.2.). The injections were done in quadruplicate.



Figure 5-1: Calibration of GC using 4 replicates of increasing volume of methane

There was little scatter in data for methane. The  $R^2$  value was 0.9978. The methane peak area (pa) is directly proportional to the volume of methane injected, and can be seen in the following equation:

$$pa = 213.81 \times V_{CH4}$$

The volume of methane can be calculated from the peak area.

$$V_{CH4} = \frac{pa}{213.81}$$



Figure 5-2: Calibration of GC using 4 replicates of increasing volume of carbon dioxide

The calibration of carbon dioxide had an R<sup>2</sup> value of 0.9935.

$$pa = 296.48 \times V_{CO2}$$

The volume of carbon dioxide can be calculated from peak area.

$$V_{co2} = \frac{pa}{296.48}$$

### 5.1.7. Calculation of biodegradability

The volume measured daily with the syringe is the total volume of gas produced by the test bottle for that day. This gas will have nitrogen, carbon dioxide and methane.

$$V_{\text{TOTAL}} = V_{\text{N2}} + V_{\text{CH4}} + V_{\text{CO2}}$$

The methane produced for that day was calculated using the following equation:

## Methane produced = $Y_{CH4} \times V_{MEASURED}$

 $Y_{CH4}$  is the mass fraction of methane in the gas produced obtained from gas composition analysis (section 5.1.6.). The daily volumes are then summed at the end of the test. The gas is sampled at 37 OC and at 1.5 atm and the calculated methane needs to be corrected to standard temperature and pressure (STP). In the equation there was no need to correct for pressure as the gas in the measuring syringe was at atmospheric pressure

$$V_{\rm STP} = V_{\rm CH4} \times \left(\frac{273\rm K}{310\rm K}\right)$$

1 g of COD is removed per 0.350L of methane gas produced (Speece, 1996). Similarly 1 mg of COD is removed per 0.350 mL of methane produced. The amount of COD removed as.gas can be calculated using the following equation:

$$mgCOD = \frac{V_{STP}}{0.350}$$

The COD removed as methane represents the portion of COD in the sample that is biodegradable.

%Biodegradability =  $\frac{\text{COD}_{\text{REMOVED}}}{\text{COD}_{\text{INITIAL}}} \times 100$ 

## 5.2. Gas loss from serum bottles

The gas produced increases the pressure within the serum bottle over and above the 1.5 atm initial pressure. Depending on the amount of gas produced the pressure can rise to a point it can leak from the bottles. The gas loss from the bottles is commonly given as the reason for the discrepancy of COD in the COD balance. In some cases the discrepancy is very large. Before proceeding with the BMP test, we wanted to ascertain how fast the gas is lost and under what conditions.

Two sets of bottles were prepared. In the first set, the 125 mL serum bottles were closed with a butyl rubber septum and were crimped tightly with a crimp seal then 50 mL of air was injected into the bottles. In the second set, the bottles were filled with 100 mL of water, which is the working volume of the serum test. 50 mL of gas was injected into the bottles after they were closed. This was noted as day zero. The pressure inside the first set of bottles was 1.4 atm and the second set 3 atm. The syringe was inserted into the serum bottles everyday to check how much of the initial 50 mL of air injected had been lost. After measurement

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the gas was re-injected back into the bottle.

The PREM bottle was done as a quick test to see whether it was worth pursuing a further investigation. The experiments showed large amounts of gas were lost quickly at pressures much less than in the actual BMP test (Figure 5-3). Bottles EM-1 and EM-2 show little gas loss over the duration of the experiment compared to bottle PREM. Both bottles had slow and steady gas loss. EM-1 had no further gas loss until the end of the experiment; EM-2 shows a second phase of gas loss. Bottles 100-1 and 100-2 showed much higher rate of gas loss than the other bottles. 100-1 lost most of the gas in the first two days. 100-2 had the same trend as 100-1 but less pronounced.



Figure 5-3: Results of gas loss experiment

The results indicated that there was no gas loss due to the crimp seal. This was supported by the fact that EM-2 measured 43 mL on day 4, 44 mL on day 7 and was 35 mL on day 9. If the crimp seal were leaking one would expect continuous gas loss. Whilst gas loss does occur during the insertion of the needle especially if the bottle is under high pressure, this does not fully explain the different patterns of gas loss or the sudden losses of gas as in EM-2 after day 6. Probably the hole caused by inserting the needle into the septum sometimes does not seal properly after the needle is removed. So one gets what is similar to a slow-leak. The results suggest this type of leak occurs more often if the bottle is under high pressure. The curve of 100-1 indicates that this leak does close because no gas was lost after day 2, but gas started being lost after day 7.

Whilst the loss of gas during the insertion of the needle cannot be stopped it is small compared to a slowleak. To minimise gas loss, pressure must not be allowed to reach high levels. After taking out the needle one should rub the septum with a thumb to try to close any holes that can lead to a slow-leak.

# 5.3. Results of biodegradability of Kingsburgh wastewater and reactor effluent

The two tests did not end just when it seems to be another phase of gas production, but the last readings include the 12 mL of gas that was injected back into the bottle.



Figure 5-4: Graph of cumulative gas production for bottles with Umbilo digester sludge



Figure 5-5: Graph of cumulative gas production for bottles with ABR sludge

The samples with Umbilo digester sludge had similar gas production curves. The bottles with synthetic medium produced the most gas, 165 and 174 mL for bottles 1 and 2 respectively. They were followed by the bottles with sewage from Kingsburg WWTW 153 and 160 mL for bottles 1 and 2 respectively. The samples with ABR sludge also had similar gas production curves. The bottles with synthetic medium produced the most gas, 99 and 121 mL for bottles 1 and 2 respectively. They were followed by the bottles with sewage from Kingsburg WWTW 87 and 99 mL for bottles 1 and 2 respectively, results in **Appendix V**.

The gas production for digester sludge follows the typical Michaeil-Menten curve. High production rate as there is plenty substrate. As substrate levels drop so will the gas production rate, hence the flattening of the curve at the end. The bottles with ABR sludge had a lower gas production rate and, the curves are almost linear (linear for most part of the test before flattening) indicating constant rate of gas production. This ABR sludge was deficient in methanogens (Lalbahadur *et al.*; 2004). Since the production of methane depends on this group gas production will be slow. The linear curve further confirms the lack of methanogens in the sludge, the few methanogens present were saturated with substrate (section 2.1.2.), and the gas could not be produced any faster.

		ABR Sludge	ABR Sludge		dge
	Initial	COD	%Biodegradability	COD	%Biodegradability
	COD	removed as		removed	
	(mg/L)	CH₄		as CH₄	
Influent	748	349	46.6	354	47.3
Effluent	232	136	58.4	149	64.4
Synthetic	989	758	76.6	601	60.2
Feed					
Scum	817	538	65.5	144	17.6

 Table 5-4: Table showing the biodegradability of different substrates with ABR and

 Umbilo digester sludge

## 5.4. Discussion and conclusion

Synthetic feed had the highest biodegradability with both sludges at 76.6% for ABR sludge and 60.2% for digester sludge. The biodegradability of the effluent was higher than that of the influent. There is a large amount of biodegradable material coming out of the reactor; most probably they are the slowly biodegradable organics. This suggests the ABR should be operated at a longer retention time to give more time for these slowly degradable organics to be digested. The ABR sludge proved successful at degrading

the scum contrasting digester sludge. This shows that ABR sludge has adapted to treating the scum as it exposed to it in the reactor. Degradation of this scum requires an acclimatised sludge. The scum itself is highly biodegradable, this probably the reason why it is not observe in summer when it is warmer and the rates of degradation are accelerated compared to winter.

Sewage was 46% and 47% biodegradable with ABR and Umbilo digester sludge respectively. This is very low compared to the 74% COD biodegradability on domestic wastewater (Elmitwalli *et al.*, 2001), and the test took 80 days at 30 °C. We incubated for 55 days and this seems to have been a short period to allow for complete digestion of the particulate COD. The slow hydrolysis of particulates is the rate-limiting step, and when the tests were stopped methanogenesis had probably not stopped but was happening very slowly.

The biodegradability of the effluent was 58.4% with ABR sludge and 64.4% with Umbilo digester sludge. The effluent had 232 mgCOD/L, of that 58% was biodegradable. The minimum amount of COD the effluent can be treated to (232-136) is 96 mgCOD/L. The Kingsburgh wastewater can be treated by the ABR to below 100mgCOD/L at a longer retention time.

## Chapter6 Membrane Tests

Due to the intrinsic characteristics of wastewater, anaerobic digestion alone is not able to produce the effluent quality that can meet the discharge standards in South Africa. From a public health point of view, the effluent is highly contaminated with pathogens as removal thereof during short retention times is insufficient (Tilche *et al.*, 1996). Post-treatment aims at mainly removing suspended solids, particulate and dissolved COD, and reduce the contents of pathogens. Odegaard prepared a review paper in 1988 on the important parameters related to post-treatment. His conclusions were that:

- Significant improvement of effluent quality may be achieved by improved solid-liquid separation
- Membrane filtration in conjunction with anaerobic digestion will have a great future
- Aerobic BOD-COD removal does not seem to be a problematic process

Sedimentation is normally used for solid-liquid separation in wastewater treatment, and this seems to be the case with anaerobic treatment. In some reactor design, a lamella sedimentation unit is used. The settling properties of anaerobic sludge particles are not very good, with typical settling velocities of 0.5 m/h. Single bacteria and small flocs stay in suspension and even float. "Although gravity separation techniques dominate the most exciting developments lie in the use of membrane filtration techniques" (Odegaard, 1988). There has been a substantial decrease in membrane costs due to technological advancements, thus promoting their use as alternatives to long established conventional water and wastewater treatment methods. A major advantage of membranes is their capability to provide high levels of microbial removal from water, wastewater and industrial wastes. The high removals ultimately lead to a reduction in health risk attributed to exposure to environmental media containing pathogens.

The effluent from the ABR has a high pathogen load and will pose a health risk if the water is discharged to a storm water system or water course. Since anaerobic digestion cannot remove nutrients, the effluent from the ABR is a water source rich in nutrients for urban agriculture. The limiting factor in the use of the effluent for agricultural purposes is the inherent pathogen load which poses a health risk for the farmers and the consumers of the produce. Pathogen tests were carried out on the influent and effluent of the ABR. Filtered and unfiltered samples were analysed by the eThekwini Metro Council in 2002 (**Table 6-1**). The results showed there is some pathogen removal in the reactor. The removal is probably due to retention of solids in the reactor. Post-treatment is necessary to improve the quality of the effluent before discharge to the environment. The results showed further improvements can be achieved with the use of a membrane.

	Preparation	Total Coliform	E.coli
		(Cfu/100mL)	(Cfu/100mL)
IN	Unfiltered	$> 4.0 \times 10^7$	5000
	Filtered	$> 4.0 \times 10^7$	Not Detected
OUT	Unfiltered	$2.0 \times 10^{6}$	Not Detected
	Filtered	$1.4 \times 10^{6}$	Not Detected

#### Table 6 -1: Pathogen removal in the ABR

## 6.1. Membrane filtration

A membrane can be defined as a thin film separating two phases and acts as a selective barrier to the transport of matter. It is very important to note that a membrane is not defined as a passive material but better as a functional material. The structure of the membrane should be discussed when considering how best to adopt and improve separation. Membrane operation can be defined as an operation where a feed stream is divided into two streams: permeate containing material that has passed through the membrane, and a retentate containing non-permeating species.



Figure 6-1: Principles of membrane operation (three-end module)

A distinction has to be made between *dead-end filtration* and *cross-flow filtration* depending on the hydrodynamics of the feed flow. Dead-end filtration systems results in a rapid build up of solids and resistance to permeate. Dead-end filtration is suitable only for dealing with suspensions with low solids content. In cross-flow filtration, the liquid shear tends to sweep away-accumulated solids thereby improving the rate of filtration. Cross-flow filtration on the other hand can be used for higher concentrations, as deposits on the membrane are swept away. The accumulation of materials in the membrane results in an increase in the resistance of the membrane over time. Permeate flux decline can be described mathematically for the case where resistance is produced by both the membrane and solids accumulated near, or on the membrane. A layer or *cake* of materials deposited on the membrane surface and more loosely associated materials in the concentration-polarization layer, present additional resistance to permeate. Resistance varies as a function of the composition and thickness of each layer. These are in turn determined by the water quality and characteristics of mass transfer in the membrane module. Cake formation, pore blockage, and adsorptive fouling appear to be the dominant causes of decreased permeate

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flux over time in the UF and MF membranes. In most instances encountered in water and wastewater treatment, it appears that the concentration-polarisation layer, if it is formed, contributes very little resistance to permeate flux and is often neglected. None the less concentration polarisation plays a key role in the formation of cake and gel layers (Mallevialle *et al.*, 1996).

Even if the membrane is characterised by its structure, its performance in terms of flux and selectivity will depend mainly on the elements contained in the two phases, and on the driving force applied. This has led to membranes being classified according to the separation they can achieve. As for all transport phenomena, the transmebrane flux for each element can be written by the following simple expression:

 $Flux = force \times concentration \times mobility$  [6-1]

Membranes are classified according to the size of the particles they can retain:

(1)	Microfiltration (MF)	$0.1-1 \ \mu m$
(2)	Ultrafiltration (UF)	0.001 – 0.1 μm
(3)	Reverse Osmosis (RO)	0.001 µm

In general microfiltration membranes can be used for the retention of particulates, microorganisms, viruses and colloids. Ultrafiltration membranes are commonly used to recover macromolecules in solution as well as colloids. Reverse osmosis is capable of rejecting ionic species down to the size of a water molecule. The MF membranes had nominal pore sizes of 0.1 to 0.2  $\mu$ m and the UF membranes, molecular weight cut-offs ranging from 100,000 to 500,000 daltons. 7 to 8 logs of two common water-related bacteria, *Escherichia coli* and *Pseudomonas aeruginosa*, were removed by UF and MF to the detection limits of the respective assays (Jacangelo, 1991).

This chapter outlines a short study of the possible use of a flat-sheet fabric membrane for post treatment of the ABR effluent. It is envisage that if successful the filter or filters would be placed in the last or after the last compartment of the ABR. The study was undertaken to find out the efficiency of the membrane to remove solids, pathogens, and optimum operating conditions.

One of the requirements of the ABR is that it should use no electricity. The driving force or liquid head normally encountered where no pump is used is between 20 to 100 cm, and flux under these operating conditions can be as low as  $1 \text{ L/m}^2$ .h. Operating a membrane at low head and flux has been shown to have many advantages. The formation of the cake is slow; this serves to extend the operational life of the membrane. The cake itself is not compact so it improves separation without substantially increasing the pressure drop across the membrane. It is impossible to routinely examine all water samples for the presence of all the pathogens. This has led to the use of indicator organism to indicate the likelihood of contamination by faeces (Gray, 1989). Total coliform-bacteria are the indicator organisms commonly used by at number of countries, followed by faecal coliform-bacteria, *Escherichia coli* and faecal streptococci.

## 6.2. Method

The membrane tests were performed in the university laboratory in a simple membrane plant described below. The pilot ABR could not be easily modified to test the membranes in the reactor without adversely affecting its operation as further operational tests had to be done. A one compartment small scale plant was constructed to test the flat-sheet membrane.

The membrane was placed vertically in a 50 L plastic tank with a stirrer for mixing. The stirrer speed was set low as we wanted gentle mixing so as to not break up the solids and strip away the cake layer that will form on the membrane. A peristaltic pump was included in the system to control the flux and maintain a constant liquid level in the tank, and the filtrate was recycled back to the tank. The concentration of solids in the tank does not change significantly as the system is closed. The U-tube was chosen to measure pressure changes because of its sensitivity at low pressure (Figure 6-2).

Effluent from the ABR was collected and analysed for solids, COD and indicator organisms before it was fed to the membrane plant the following day. Flux and  $\Delta P$  were measured, 30 mL samples were taken from the return line and were analysed for COD, TSS and Total indicator organisms. For determination of the number of indicator organisms, Fluka 70137 ENDO Agar (Base) was used. This is selective medium for growing and differentiating lactose fermenting and lactose non-fermenting intestinal organisms. The samples were incubated at  $37^{\circ}$ C for 2 days before counting the colonies.



Figure 6-2: The lab scale membrane plant and the fabric membrane used for the tests

## 6.3. Results

The first trial-run was a clean water test of the system. On **Figure 6-3** are the results of the first run which was a short run. The curve of cumulative volume shows there was constant flow through the system at the same time there was a slight decrease in the flux. The results also show that the pump can deliver a constant flow, and any changes in flow and flux should be due to the change in resistance at the membrane.



Figure 6-3: Results of the first (short) trial-run

The second run was also carried with clean water but the run was longer, 18.5 h. On **Figure 6-4**, the graph on the left is flux plotted against time; it shows logarithmic decrease of flux with time. The curve on the right is a plot of flux versus filtrate volume. The decrease in flux is due to the presence of solids in the clean water such as dust, and in time growth of bacteria will occur in the water. The accumulation of solids on the membrane is directly related to volume filtered. Comparing the two curves, for analytical purposes it is better to plot the parameter of interest against volume filtered. The curve is smoother and the data points have a better distribution.



Figure 6-4: Curves of the second run with clean water (extended run). Fist curve is Flux vs. Time; second curve is Flux vs. Volume filtered.



Figure 6-5: Experimental run with ABR effluent

The experimental run with ABR effluent was a short (less than one hour). The experiment was stopped because the membrane was completely blocked. The flux rose quickly to  $122 \text{ L/m}^2$ .h before slowly decreasing to a steady flux of 30 L/m<sup>2</sup>.h, which lasted for a short duration before the membrane was clogged. The differential pressure rose immediately to 69 cm, it remained steady for short period of time before rising linearly to 192 cm. The differential pressure exceeded the 100 cm limit but we continued the run because we wanted to operate as long as possible. Changes in COD, total solids and total indicator organisms are listed in **Table 6-2**.

	Initial conc. (mg/L)	Final conc. (mg/L)	% Removal	Log Removal
COD	333	76	77	
Total Solids	755	555	27	
Indicator Organisms	$1.24 \ge 10^{16}$	$6.8 \times 10^{10}$		5.26

Table 6-2: Performance results for the membrane test

## 6.4. Discussion and conclusion

The membrane tests could not be repeated due to time constraints and the results cannot be taken as the true performance of the membrane. The results can only be interpreted qualitatively.

The large decrease in COD within the short duration of the experiment indicates that most of the COD in the effluent is in particulate form and the membrane can remove it. Another interesting observation is that there is a strong correlation between total solids and indicator organisms as the curves follow the same shape. The indicator organism removal achieved in the experiment was high (log removal 5.26) especially since the filter was operated for a short period and only a small cake layer could have formed. This compares very well to the 7 to 8 logs of removal of *Escherichia coli* and *Pseudomonas aeruginosa*, using UF and MF (Jacangelo, 1991).

In the experiment we reached very high differential pressures, above one meter of head. The flux reached a high of  $122 \text{ L/m}^2$ .h. The lowest flux reached was  $30 \text{ L/m}^2$ .h, which is very high compare to  $1 \text{ L/m}^2$ .h at natural head. This probably contributed to the membrane fouling very fast. If the membrane can be operated at a lower flux the duration of the test can be extended and this will lead to a slower increase in the differential pressure. The trials should be repeated at lower differential pressure (less than 1 m) and flux (about 1  $\text{ L/m}^2$ h.).

## Chapter 7 Settling Tests

In order to design a treatment facility with improved solids retention, there is a need to know the settling characteristics of the solids. The interest in this parameter lies in the fact that it encompasses other parameters at the same time, like particle size, density and shape. Hence devices and methods were developed to test and measure settling velocities of solids in sewage (Aiguier *et al.*, 1996). Solids in wastewater include floating matter, solids in suspension, colloidal solids and matter in solution. Suspended solids can range in size from sub-micron sized particles to solids in the order of 100's of millimetres.

The design of sedimentation devices can be improved based on the knowledge of the wastewater characteristics. The reactor must be designed such that the up-flow velocity is less than the settling velocity of the biomass on the up-flow section of the compartment to prevent solid entrainment in the liquid flow. Knowledge of the settling rates of the biomass is necessary to design for sufficient solids retention in order to prevent the biomass from washing out of the reactor. Sedimentation theory indicates that for discrete particles, sedimentation efficiency is a function of the overflow rate in the device. Settling tests can be done to calculate the settling velocities of the solids in the compartments. Since the characteristics of solids will vary from compartment to compartment, settling tests should be done for each compartment of the reactor.

## 7.1. Sedimentation and settling

Sedimentation can be described as the removal of solid particles from a suspension by settling under gravity. A wide range of solids is encountered in wastewater, and the solids exhibit a range of particle size and densities. Some of these particles will not change their properties during sedimentation (discrete particles), whereas others will agglomerate and flocculate, therefore undergoing changes in settling properties. Stoke's law quantifies the factors affecting the velocity of a spherical particle under quiescent conditions. In the equation below, V is the settling velocity, r is the particle radius, g is the gravitational acceleration, n is the kinematic viscosity of the fluid,  $d_1$  is the density of the fluid and  $d_2$  is the density of the particle (Horan, 1996).

$$V = \frac{2g}{9} \cdot \frac{r^2}{n} \cdot (d_2 - d_1)$$
 [8-1]

However, it is not possible to apply this equation to wastewater because it is not practical to determine size or density, since the particles are irregular in shape. Things are complicated further because particles tend to agglomerate and flocculate (Horan, 1996). Agglomeration causes the formation of larger particles and heavier particles with increased settling velocities. For the purposes of design, four distinct modes of settling have been described. These modes are a function of particle size and interaction. In wastewater the four modes of settling normally occur simultaneously.

## 7.1.1 Discrete settling

This is encountered in dilute suspensions of discrete particles. Discrete particles undergo no change in shape, size or density during settling. The unhindered settling of discrete particles such as sand is best described by Stokes' law.

#### 7.1.2 Flocculent settling

Flocculation is encountered in the settling of colloidal  $(0.1 \ \mu m)$  and larger particles in a dilute suspension. Flocculating particles are continually changing in size and shape, leading to particle velocity increasing with depth. So many factors contribute to the flocculation process that it has been impossible to develop a general formula for determining settling velocities (Peavy *et al.*, 1985).



Figure 7-1: Settling velocities exhibited by discrete and flocculent particles (Horan, 1996)

In a flocculated suspension, the flocs are the basic structural units and in a low shear rate process such as gravity sedimentation, the rates and sediment volumes depend largely on the volumetric concentration of flocs and on inter-particle forces. The type of settling behaviour exhibited by flocculated suspensions depends largely on the initial solids concentration and chemical environment. When the solids concentration is very high the maximum settling rate is not immediately reached and may increase with increasing initial height of suspension (Horan, 1996).

#### 7.1.3 Zone settling

Zone settling is characterised by activated sludge and flocculent chemical suspensions when the concentration of solids exceed 500 mg/L (Eckenfelder, 1989). The concentration of flocculent particles is

sufficiently high to allow the inter-particle forces to bind them together in a lattice structure. The particles no longer settle independently, but as a mass with a visible solid-liquid interface between the flocs and the supernatant. Zones of liquid are displaced by the settling particles and this is known as hindered settling. The rate of settling is controlled by the rate at which liquid passes upward through the mass (Horan, 1996).

#### 7.1.4 Compressive settling

At the bottom of the column the concentration of solids is so high that the particles are in contact with each other. A layer of particles below supports each layer of particles. Further settling only occurs by the forcing water from the compressing particles and this requires an adjustment in the matrix that forms the sludge blanket. The settling rate is determined by compressive properties of the sludge, settling in this region is very slow (Horan, 1996).



Figure 7-2: Diagram showing conditions for each type of settling (Eckenfelder, 1989)

### 7.2. Settling column tests

Most of the suspended material in municipal sludge other than grit is organic in nature and tends to flocculate and aggregate rather easily. There is a distribution of different particle sizes, contact results in particles that are larger and settle faster than either of the parent particles, which then catch up with smaller particles that were ahead in terms of settling (Schroeder, 1977). Since a mathematical analysis is not possible, a laboratory settling analysis is required to establish the necessary parameters. The settling characteristics of a flocculent suspension under quiescent conditions can be established in a laboratory by carrying out settling column tests.

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Figure 7-3: Schematic of settling column (Horan, 1996)

A batch-settling column with a number of sampling ports is used (Figure 7-2). The solids are allowed to settle under quiescent conditions, samples are removed at intervals from the sampling ports fixed at different depths. The suspended solids concentration of the samples is determined. When sampling, one should not see a change in solids concentration until the fast settling particles have moved past the sampling point. Further change will occur when the medium-settling fraction moves past the sampling point and so forth. Considering the mechanism of flocculent settling, it is important to allow adequate time for settling to occur. The results obtained are normally expressed in the form of depth-time grid (Peavy *et al.*, 1985). The curves are constructed by determining the percentage solids removal of each sample analysed and then plotting curves of equal percentage removal (Figure 7-4).



Figure 7-4: Iso-removal from settling analysis (Peavy et al., 1985)

Data typically collected using this method is highly scattered and a fitted curve is required. Subjective judgment is involved, as the end points of the curve should be asymptotic (Pisano, 1996). Settling velocity distribution curves can be produced as cumulative graphs, which show the proportion of material by weight . with settling velocities less than a given velocity (Andoh and Smisson, 1996). We modified Pisano's assumption; the curve should intersect the Y-axis at 100% as all settleable solids settle or have settling velocity greater than zero.

Fraction of suspended solids after settling has been initiated:

$$SS = \frac{C_t}{C_{t=0}} \times 100$$
 [8-2]

where  $C_t$  is the concentration of the suspended solids at time t and ,  $C_{t=0}$  is the initial concentration of solids.

$$V_{s} \cdot (m/h) = \frac{\left[h_0 - \left(\frac{S_{n-1} \times S_V}{C_{SA}}\right) - P_h\right]}{\left(S_{t \min} - 60\right)} [8-3]$$

where  $h_0$  - is the starting liquid height (m)

P<sub>h</sub> – sampling port height (m)

 $S_n - n^{th}$  sample taken

 $C_{SA}$  – column surface area (m<sup>2</sup>)

$$S_V - sample volume (m^3)$$

St<sub>min</sub> – time sample was taken in minutes

In practice this type of column is difficult to use, as it requires large volume of sample, and is prone to structural failure and leaking seals. The mechanical mixing can shear larger organic and flocculent materials and entrain air (Pisano, 1996). Obtaining a stable "time zero" TSS concentration is both difficult and essential since all subsequent values are referenced to this estimate. Obtaining samples having particles with settling velocities in the 36 m/h - 360 m/h range is difficult as the faster settleable solids will fall before the first set of samples can be taken. Taking large number of samples depresses the column sample height and this change must be considered (Pisano, 1996).

#### 7.3. Data analysis

To observe a change in the concentration of the solids when sampling, the fastest fraction of solids has to settle or move past the sampling point. Since the column is perfectly mixed the *tail* of the fastest settling solids will be the solids starting from the top of the column. For the next change in concentration the

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second fastest fraction must pass the sampling point with its tail. The velocities of particles are calculated on the solids starting from the surface to the level of the port where they are sampled. The time taken to travel that distance is represented by the time the sample was taken.

### 7.3.1 Data selection

Settling tests and calculations that follow are based on the assumption that the solids should settle. The plot of %-unsettled solids vs. time for the 3 ports or sample heights for each compartment showed that for port 2 and 3 settling was not occurring (**Figure 7-5**). Hindered settling can start occurring at concentrations above 500mg/L (Schroeder, 1977), the sludge in the reactor compartments was between 8000 and 26000 mg/L. It is also possible the concentration was close to the zone where hindered settling ends and compressive settling begins. All the calculations and results that follow were based on the measurements made on port 1.



Figure 7-5: Settling occurring in port1, ports 2 and 3 oscillating around the initial concentration

The exclusion of port 2 and 3 also meant excluding data at high settling velocities. Little information would have been extracted from these points as the operating upflow velocity in the reactor was 0.5m/h.

#### 7.3.2 Error analysis

Due to time constraints, the settling tests could only be done once. The method had not been attempted previously. We analysed for errors that could be associated with the method in an attempt to ascertain the reliability of the results.

Two possible sources of error were analysed for the test: sampling and the measuring of TSS. The error associated with measurement of solid was calculated from results of our routine measurement of solids for the ABR. The other important factor in the test was the mixing of solids in the column before each port was sampled. The %-standard deviation was for each compartment was calculated from the zero samples taken
from each port when testing the compartments. Error associated with the weighing balance was ignored because they were negligent.

Compartment	Initial density measurement (g/L)			Avg.	Standard	% Standard
-	port 1	port 2	port 3	density (g/L)	deviation	deviation
· · · · · · · · · · · · · · · · · · ·	11.67	15.2	16.09	14 42	2 43	16.85
compartment 1	11.0/	15.5	10.20	14.42	2.45	10.85
compartment 2	24.97	27.2	27.75	26.64	1.47	5.53
compartment 3	11.55	20.78	28.45	20.26	8.46	41.77
compartment 4	9.33	10.98	14.18	11.50	2.47	21.45
compartment 5	9.95	9.33	16.43	11.90	3.93	33.04
compartment 6	8.77	9.37	12.80	10.31	2.17	21.08
compartment 7	8.24	8.81	12.03	9.69	2.04	21.08
compartment 8	10.07	16.755	17.305	14.71	4.03	27.38

Table 7-1: Error analysis of mixing of solids during the settling tests

The %-standard deviation from the routine measurement of ABR solids was 40%; this figure includes deviations caused by the variation in the incoming wastewater and the process. The %-standard deviation calculated from the zero-samples ranges from 5% for compartment 2 to 42% for compartment 3. Good mixing was not being achieved in the column; Pisano mentioned this weakness of the test (Pisano, 1996). The maximum error that can be associated with the method is 42% This could be due to the fast settling solids settling very quickly before the zero sample could be taken, and port 3 (bottom port) always had most solids.

#### 7.3.3 Statistical analysis (R-squared value)

We are interested in a relationship between two sets of data (variables) that are thought to vary together. The measure of the degree of relationship between two sets of data is called a correlation. Sometimes we want to mathematically describe the correlation using a model. By definition, R-squared represents the fraction of the total variation accounted for by the fitted equation or model (Daniel and Wood, 1971). Thus values approaching one are desirable, while zero means that the model does not explain the relationship between the x and y-values.

$$R^{2} = 1 - \left[\frac{rr}{yy}\right]$$

$$rr = \sum (measurement - mod el)^2$$

where

$$yy = \sum (measurement - measument'sAverage)^2$$

and

Data from Peavy *et al.*, 1985 pg121 was used to test the procedure (Appendix VI) and examine whether results obtained make sense. The initial concentration of the sludge was 300mg/L, which implies that flocculent settling should occur. A two-parameter model was used to fit the best curve to the data.

$$Y = (100 - c)e^{-ax} + c$$
 [8-4]

The parameters **a** and **c** have no physical meaning relating to the property of the sludge. The parameters allow for manipulation of the equation to fit the best curve to the data. Equation [8-4] was used to fit the best curve on the data. The Excel solver function was used to maximise  $R^2$  by varying the model parameters **a** and **c**. To test the procedure of fitting the best curve to the data, we used data from settling tests used as an example in Peavy *et al.* the actual data for our tests can be found in **Appendix VI**.



Figure 7-6: (a) Distribution curve of suspended solids vs. settling velocity showing curve a and b; (b) shows the curves of the individual ports labelled with the depth of the port in meters (reproduced using data from Peavy *et al.*, 1985 pg121)

Curve (b) is the curve with the highest  $R^2$ -value of 0.25, but through visual inspection this was not the best curve through the data. Curve (a) was visually the best curve with an  $R^2$ - value of -0.11. This prompted us to analyse the results for each sampling height or port. When the results were separated and analysed for each port we were able to use  $R^2$  to fit the best curve for all the ports. The  $R^2$  values are reported in **Table 7-2**.

Table 7-2: Best  $R^2$  values when fitting the best curve to data for each port

Port and	Port	Port	Port	Port	Port	Port
Height	(0.5m)	(1.0m)	(1.5m)	(2.0m)	2.5m)	(3.0m)
R-squared	0.992	0.994	0.995	0.996	0.997	0.999

This exercise showed that  $R^2$  is best used where data has linear distribution as when one port is analysed. In the case where the data is pooled together, most of the data points are grouped in the "middle range", so fitting is weighted to where the data points are concentrated and this change's mainly the tail of the curve.



Figure 7-7: Analysis of individual ports labelled with the depth of the sampling port showing curves (a) and (b) (reproduced using data from Peavy *et al.*, 1985 pg121)

Figure 7-7 illustrates that the trend followed by the curves of the individual ports is the same as that of Curve (a), but Curve (b) deviates from this pattern.

#### 7.4. Results discussion and conclusion

 Table7-3: Highest R-squared values, and values for parameters a and c for the model
 fitted to settling test measurements

Compartment	1	2	3	4	5	6	7	8	_
R-squared	0.98	0.98	0.93	0.98	0.99	0.99	0.99	0.97	
a	0.91	3.13	0.30	0.54	0.61	0.37	0.37	1.03	
c	3.39	5.32	16.08	8.46	4.96	8.12	10.65	5.80	

Comparing magnitudes of the parameter **a** for each compartment, compartments 1, 2, 3, 4, 5 and 8 were significantly different from each other, but 4 and 5 were closer to each other. Compartments 6 and 7 had the same magnitude of  $\mathbf{a}$ .

Figure 7-8 shows the fitted curves for each of the reactor compartments. The results of each individual compartment and its fitted model are presented in Appendix VI. The results show that the reactor is retaining between 60 and 90% of the solids at the operating upflow velocity of 0.5 m/h. Lettinga and Hulshoff Pol found for even voluminous flocculent types of sludge with poor settling properties, has the admissible superficial velocities for a UASB are 0.5 m/h with temporary admissible peaks up to 2 m/h (Lettinga and Hulshoff Pol, 1991). The test indicates that the sludge has poor settling properties but the reactor still is able to retain the sludge at the up-flow velocity of 0.5 m/h. Error analysis showed the result

could deviate by up to 40%. We believe the calculated solids retention is reasonable because there was more sludge in 2003 in the compartments of the reactor than the in the previous year indicating growth and retention of the produced biomass. The reactor obtained 50% TSS removal and 60% VSS removal of the solids in the feed and these figures excludes the large quantities of sludge within the reactor (sectio4.5.4.). A quick calculation was performed to test whether the retention of up to 90% is reasonable by methods used to obtain it. The average TSS leaving the reactor throughout the trial was below 400 mg/L and the amount of sludge in compartment 8 when settling test were performed was 14.71 g/L. 400mg/L of solids were leaving the last compartment out of 14.71 g/L which gives a retention of 97%.



Figure 7-8: shows the best-fit curves for the compartments. In this case, for all the curves the highest R-squared value corresponded to the best curves.

The tests should be repeated and more data should be collected see how reliable the test is. A mass balance on solids should be done, and the results should be compared to those obtained from the settling tests.

## Chapter 8 Conclusions and recommendations

The anaerobic baffled reactor experienced numerous problems with regard to continuous feeding (pump and outlet blockages). In the community the reactor will have no pump since gravity feeding would be used, eliminating the need for a pump; and in community the outlet need not be restricted by a measuring device and the outlet pipe can be enlarged to eliminate blockages (c.f. section 4.3.2.). Difficulties that could be experienced in the community with the outlet will depend on the post treatment option chosen and its configuration.

Methanogenesis was not occurring in the reactor because the slow hydrolysis of particulate matter to make available the short chain fatty acids that are required for methanogenesis was rate limiting. The slow hydrolysis of particulate matter led to acidogenesis occurring throughout the reactor, which created conditions of low pH in the reactor that can suppress methanogenesis. The retention time of 20 h is short for complete hydrolysis of solids and should be increased so that more time is available for acidogenesis can occur completely thus allowing methanogenesis to begin, and the increase in HRT should take in to consideration that methanogenesis should occur complete as well.

A high rate reactor can be loaded between 1.5 and 3 kgCOD/m<sup>3</sup>.day (Stronach *et al.*, 1986). Mudunge managed to load up to 40 Kg/m<sup>3</sup>.d of soluble feed in the laboratory scale ABR, but the pilot ABR was being loaded at 0.525 kgCOD/m<sup>3</sup>.day. In contrasts the ABR was organically under-loaded. The analytical campaign showed that the average daily COD of wastewater at Kingsburgh was 565 mgCOD/L, and the effluent from ABR had 234 mgCOD/L with a COD removal of 42%. Attempting to load at the rates achieved by others using weak wastewater leads to the reactor being hydraulically overloaded and. The hydraulic overloading results in the particulate COD not being completely hydrolysed and being carried over. The BMP tests showed that biodegradability of the effluent was **ca**. 60%. The ABR can treat the wastewater at Kingsburgh to less than 100 mgCOD/L, giving a COD removal of 80%. The limiting factor to higher loading will be the slow hydrolysis of particulate COD.

Alkalinity is a measure of the system to buffer acids when they occur and the average alkalinity within the reactor was low. This did not present a problem as the amount of alkalinity required and generated for stable operation is proportional to the strength of the wastewater being treated which in this study was a low strength wastewater. The pH of the system depends on the chemical species present. The analysis of pKa values suggested that the combination phosphate, carbonic acid and bicarbonate play the biggest role in the buffering system, and the pH should be within the range of 6.5 to 7.2 when the organic acid have been completely degraded. The ABR recovers quickly from organic shock loads with day-by-day

#### Chapter 8

improvement in pH values with decreasing effluent COD. In the community the ABR is unlikely to go sour because the bulk of the COD will be in particulate form. The slow hydrolysis of particulate COD will probably allow the system to adjust.

The reactor obtained 50% TSS removal and 60% VSS removal of the solids in the feed. The results of settling tests showed that the reactor was retaining between 60 and 90% of the solids at the operating upflow velocity of 0.5 m/h. The test indicated that the sludge had poor settling properties but the reactor was still able to retain the sludge at the current operating upflow velocity. The TSS and VSS results show good retention of solids within the reactor but they exclude the large quantities of sludge within the reactor so the ability of the reactor retain solids is above 95% which is very good and if a membrane is included at outlet it will further improve the ability of the reactor to retain solids.

The preliminary work with the fabric membrane showed enormous benefits can be gained if it had to be included. The membrane removed **ca**. 75% of the COD and 25% of TSS in the effluent. The membrane achieved 5 log removal of indicator organisms. Given that the filter was operated for a short period and only a small cake layer could have formed, the membrane can obtain better results if it operated at low fluxes of  $1 \text{ L/m}^2$ .h and operating at such fluxes will extend the operational life of the membrane. The membrane showed good ability to remove indicator organism and solids that contributed a great deal to the effluent COD. Since there is no nutrient removal in the ABR makes the effluent a rich nutrient source for irrigation. The standard for irrigation is that its COD must below 400 mg/L and the pH must be between 6 and 9. The effluent from the ABR can be used for food production if pathogens are removed.

The analytical campaign showed that there were variations in COD and of the pH during the course of the day. The variation can be connected to the functions that take place within a household. The peak between 06:00 to 09:00 was between the time when people wake up, wash, eat breakfast and go to work or school. Thereafter house cleaning is carried out until midday (12:00) hence COD and pH of the water coming into the WWTW remains high. The small COD peak in the late afternoon (17:00) coincides with the preparation of dinner, but 2 h later (19:00) there is a pH peak presumably because of the washing of dishes. The ABR handled these daily variations very well.

The following was concluded:

- ABR proved to be stable and consistent in its performance
- Solubilisation of particulate COD was the rate liming step in degradation of COD
- COD can be treated to below 100mgCOD/L and will give a COD removal of 80%
- The ABR retains between 60 and 90% of solids

- The ability of the reactor to control and maintain pH and the alkalinity for stable operation is dependent on the strength of the wastewater being treated and the pH of the feed
- ABR is able to recover from shock loads very quickly and this is independent of the buffering capacity
- The reactor was organically under loaded; the reactor has a large capacity to receive wastewater with very high COD as is expected in the target communities but will need to operate at higher HRT
- The ABR can be successfully used in a community to remove primarily COD, and with the aid of a membrane pathogens can be removed and the effluent used for irrigation
- The effluent from the ABR can be used for food production if pathogens are removed.

Based on the above conclusions, the following work is recommended

- The hydraulic retention time should be increased to allow more time for the degradation of particulate COD
- The first compartment should be modified and increased in size to trap as much of the solids as possible
- Membrane tests should be continued at low flux and differential pressure (1m), paying particular attention on increasing the operational life of the membrane
- The method for settling tests should be improved, the test repeated for the new HRT and the results confirmed with a solids mass balance on the reactor

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## **APPENDIX I** Wastewater and its constituents

## Table I-1: Diseases affecting potable water and wastewater

Etiological Agent	Illness/Disease	Primary sources/ Major Reservoirs
Viral		
Hepatitis A & B virus	Infectious hepatitis	Human faeces
Poliovirus	Poliomvelitis	Human faeces
Norwalk virus	Gastroenteritis	Human faeces
Coxsackie A & B virus	Meningitis, respiratory & cardiovascular disease	Human faeces
Rotavirus	Gastroenteritis	Human faeces
Echovirus	Aseptic meningitis	Human faeces
Adenovirus	Respiratory & gastrointestinal illness	Human faeces
Reovirus	Respiratory & gastrointestinal illness	Human & animal faeces
Bacterial		
Pathogenic Eschericia coli	Gastroenteritis	Human faeces
Salmonella typhi	Typhoid fever	Human faeces
Vibrio cholera	Cholera	Human faeces
Pseudomonas sp.	Gastroenteritis	Soil & water
Campylobacter sp.	Gastroenteritis	Human & animal faeces
Legionella sp.	Pneumonia	Thermally enriched water
Leptospria sp.	Leptospirosis	Human & animal faeces
Shigella	Bacillary dysentery	Human faeces
Mycobacteria	Pulmonary illness	Soil & water
Fungal		
Aspergillus sp.	Allergic & respiratory diseases	Soil & water
CryptococcuS	Respiratory diseases	Soil & water
Histoplasmosis	Respiratory diseases	Soil & water
Candida albicans	Candidiasis	Soil & water
Various dermatophytes	Athletes foot, etc.	Soil & water
Protozoans		
Entamoeba histolytica	Amoebic dysentery	Human faeces
Giardia limblia	Giardiasis (gastroenteritis)	Human & animal faeces
Cryptospiridium	Gastroenteritis	Human & animal faeces
Acanthamoeba	Corneal lesions	Soil & water
Naegleria sp.	Meningoencephalitis	Soil & water

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Table I-2: Typical	wastewater	<i>characteristics</i>	
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Substance	Unit	Weak	Medium	Strong
Solids, total (TS)	mg/L	350	720	1200
Dissolved, total solids (TDS)	mg/L	250	500	850
Fixed	mg/L	145	300	525
Volatile	mg/L	105	200	325
Suspended solids (SS)	mg/L	100	220	350
Fixed	mg/L	20	55	75
Volatile	mg/L	80	165	275
Settleable solids	mg/L	5	10	20
Biochemical oxygen demand,:	mg/L			
5-day ,20°C (BOD5,20°C)	mg/L	110	220	400
Total organic carbon (TOC)	mg/L	80	160	290
Chemical oxygen demand (COD)	·mg/L	2500	5000	10000
Nitrogen (total as N)	mg/L	20	40	85
Organic	mg/L	8	15	35
Free ammonia	mg/L	12	25	50
Nitrites	mg/L	0	0	0
Nitrites	mg/L	0	0	0
Phosphorus (total as P)	mg/L	4	8	15
Organic	mg/L	1	3	5
Inorganic	mg/L	3	5	10
Chloridesa	mg/L	30	50	100
Sulphatea	mg/L	20	30	50
Alkalinity(as CaCO <sup>3</sup> )	mg/L	50	100	200
Grease	mg/L	50	100	150
Total coliformb	no/100mL	106-107	107-108	107-109
Volatile organic compounds (VOCs)	μg/L	<100	100-400	>400

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**APPENDIX II** Biochemical models and ABR microbial consortium



Figure II-1: COD flux for a particulate composite comprising of 10% inerts 30% each of carbohydrates, protein and lipids in terms of COD.

No.	Observation	Technique	Ref
1	Methanosarcina predominant at the front of the	SEM, TEM, LLM	Boopathy and Tilche. 1991, 1992
	reactor with Methanosaeta found towards the rear		Tilche and Yang. 1987; Garuti et
			al. 1992; Yang et al. 1988
2	Active methanogenic fraction within biomass;	ATA	Bachman et al.; 1985; Orozco.
	highest at the front of reactor and lowest in the		1988
	last chamber		
3	Bacteria resembling Propionibacterium,	TEM	Grobicki; 1989
	Syntrobacter and Methanobrevibacter found in		
	close proximity within granules. Methanosaeta and		
	colonies of Syntrophomonas also observed		
4	Large number of Methanobacterium at front of	EP	Tilche and Yang.; 1987
	ABR along with Methanosarcina covered granules;		
	subsequent chambers consisted of Methanosaeta		
	coated flocs		
5	Virtually all biomass activity (>85%) occurred in	ATPA	Xing et al.; 1991
	the bottom third of each compartment where		
	biomass was concentrated; highest activity (92%)		
	found in the bottom of the first chamber		D 1
6	Mainly Methanosaeta observed with some cocci;	SEM	Polpraset et al.;1992
	no Methanosarcina observed	05) (	U. h. et al. 1007
7	Irregular granules with gas vents covered by single	SEM	Holt et al.; 1997
	rod shaped bacteria; no predominant species		
	observed	ATDA SEM EP	Boonathy and Tiche <sup>,</sup> 1992
8	Bacteria resembling Methanobrevibacter,	ATTA. SEIVI. ET	Doputity and violity, 1992
0	Wide variet of bacteria observed at from of reactor	SEM TEM	Boopathy and Tiche; 1991
У			Barber and Stuckey; 1997
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## Table II-1: Bacterial observations in the ABR (Barber and Stuckey; 1999)

ATA = anaerobic toxicity assay. ATPA = ATP analysis, EP = epifluorescence microscopy, LLM = light level microscope, SEM = scanning electron microscope, TEM = transmission electron microscope

Substrate	Reactions	ΔG°	Δ <b>G</b> '
		(kJ/gCOD)	(kJ/gCOD)
H2, HCO <sub>3</sub> <sup>-</sup> -	$4H2 + CO2 \rightarrow CH4 + 2H2O$	-2.12	-0.19
Propionate	$CH_{3}CH_{2}COOH + 2H_{2}O \rightarrow CH_{3}COOH + 3H_{2} + CO_{2}$	0.68	-0.13
Butyrate	$CH_{3}CH_{2}CH_{2}COOH \rightarrow 2CH_{3}COOH + H_{2}$	0.30	-0.16
Palmitate	$CH_3 (CH_2)_{14}COOH + 14H_2O \rightarrow 8CH_3COOH + 14H_2$	0.55	-0.16

## Table II-2: Thermodynamic values for reactions of fatty acid oxidising organisms

 $\Delta$  G' calculated for T 298K, pH 7, pH<sub>2</sub> 1x10<sup>5</sup> bar, pCH<sub>4</sub> 0.7 bar, HCO<sub>3</sub><sup>-</sup> 0.1M and organic acids 1mM

# **Appendix III** Operation of the ABR and analytical methods

## III-1 Sampling

Each compartment of the pilot reactor has four sampling ports. Two of the ports are on the side of the reactor. The higher port is in line with the overflow weir in the compartment and the lower port is 20 cm from the bottom of the reactor. The other two ports are found at the top of the reactor. One port is on the down-flow side, and the other on the up-flow side of the compartment. The sampling was taken from the top of the ABR on the up-flow side, using a closable sampling column with a bottom stopper.



Figure III-1: Orthographic projection of the pilot-scale ABR

When doing the Coring sampling test (Appendix II), the top sampling port on the upflow side of each compartment was used. The column was found to be useful instrument in obtaining a good composite sample of solids in each compartment.

The sample for pH measurement was taken from the higher side port of the compartment. This sample has the least solids, and this made for good pH measurement.

The inlet COD sample was taken from the feed box and the effluent sample from the outlet pipe before entering channel back to the WWTW plant. The same samples were used for determination of alkalinity TSS and VSS. The sampling column was immersed into the compartment until it touched the bottom, then the stopper was closed and the captured liquid was collected in a bucket. This method of sampling gives a good composite sample of all the solids in each compartment.

## III-2 Checks and maintenance of the reactor

From past experiences, it was decided the following checks should be performed routinely:

- Pump blockages
- Gas vent blockages

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- Reactor outflow blockages
- Clean mash trapping solids to magnetic flow meter

#### To clear pump blockages

- Turn off pump at the pump power point)
- Lift the pump out of the channel
- Check for rags and strings stuck in rotor chamber
- Remove and clean the housing basket
- Lower pump back to the channel and swing it from side to side when immersed in the channel to push air trapped s in rotor chamber in to the delivery pipe

$1 u v v c 111 - 1 \cdot \Lambda v u v v c s u m v v m u u u u u v s s p v s u u u m u v c 1 v v m c 1 u v c 1 $	Table III-1.	: Routine	sampling and	l analysis p	rogramme	for the ABR
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Day	Check	Sample	Analyse
Monday	Pump	Influent I <sub>1</sub> (12:00 -12:30)	COD
	Flow meter	Effluent E <sub>1</sub>	
Tuesday	Pump	Influent	COD
	Flow meter	Effluent (8:30)	Alkalinity
	PLC		TSS & VSS
Friday	Pump	Influent	COD
	Flow meter	Effluent	
	PLC		
	pH		
	Levels		

### **III-3** Analytical Methods

#### III-3.1. Coring column test (levels and biomass heights)

The top sampling port on the up-flow side of each compartment was opened. Knocking the plunger down opened the column bottom stopper. The column was then inserted into the compartment down to the bottom of the reactor. The plunger was then pulled up to close the column. Once the column was pulled out the height of the liquid level was read. The sample was allowed to settle for 5min before biomass height was read.

#### *III-3.2*. рН

The sample was taken from the topside port of each compartment. The pH immediately read with Metro Ohm pH meter mode744.

#### III-3.3. Alkalinity

**PRINCIPLE:** Hydroxyl ions present in the sample because of dissociation or hydrolysis react with addition of standard acid. Alkalinity thus depends on the end-point pH used. For samples containing more than 150 mgCaCO<sub>3</sub>/L and f or samples known or suspected to contain phosphates or silicates, pH 4.5 is suggested as the equivalence point.

**SAMPLING:** The range of alkalinities found in wastewater is so large that a single sample size and normality cannot be used, as titrant cannot be specified. It is suggested that one uses a sample large enough to use 20 mL or more from a 50 mL burette. This allows for relatively good volumetric precision while keeping sample volume sufficiently small to permit sharp end-points.

**STORAGE:** Samples were collected in polyethylene or borosilicate glass bottles. Bottles were completely filled with sample (must have no pockets of air) and capped tightly. Samples were kept on ice for transportation. Because waste samples maybe subject to microbial action and to loss or gain of CO2 or other gases when exposed to air, samples were be analysed within 6hrs. Prolonged exposure to air and agitation was avoided.

#### REAGENTS

#### 0.02N Sulphuric Acid (M/100)

Dissolved 3mL conc. H<sub>2</sub>SO<sub>4</sub> in distilled water and diluted to 1L. This was approximately 0.1N.

Accurately weighed 1.325g of anhydrous  $Na_2CO_3$ , previously dried at 270°C. Dissolved in distilled water, and made up to 250mL in a volumetric flask. This is 0.10 Normal.

#### Mixed Bromocresol green - Methyl red Indicator solution

Mix 0.2g bromocresol green and 0.4g methyl red in 120mL 95% ethyl alcohol.

To calculate Normality, titrated the  $H_2SO_4$  against 25mL of  $Na_2CO_3$  solution using Bromocresol green and Methyl red mixed indicator. Calculate the normality using the following equation

Normality of  $H_2SO_4$  solution:  $N = 25 \times 0.1$ Vol of  $H_2SO_4$  used

Diluted the  $H_2SO_4$  solution 5 times to bring it to 0.02 N (N/5) 1 mL of 0.02N  $H_2SO_4 = 1mg CaCO_3$ 

#### PROCEDURE

Measured 50mL of the well mixed (unaltered) sample into an Erlenmeyer flak using a measuring cylinder. 2-3 drops were added of mixed indicator to sample. Titrateed with 0.02N Sulphuric acid and observed the colour change from greenish blue to dull grey. Prepare and titrated an indicator blank and subtract this volume from the sample titration. Checked whether endpoint was at pH4.5 with a pH meter.

> Alkalinity (mg/L as CaCO<sub>3</sub>) =  $\underline{A \times (N/5) \times 50\ 000}$ Vol of sample (mL)

Where A = mL of diluted acid used for titration.

**NOTE:** Silicates, phosphates and borates contribute to alkalinity. Soaps, oily matter, suspended solids coat the glass electrode and give sluggish response. The colorimetric method might be affected by turbid samples so a potentiometric titration can be used to titrate.

#### III-3.4. Solids determination

#### (a) Total Solids (TS)

A dish pre-dried in the oven at 103 °C to 105 °C was weighed and was used to evaporate a well mixed aliquot of sample. The dish was dried again in the oven. The increase in weight over that of the empty dish represents the total solids.

**SAMPLE HANDLING & STORAGE:** Resistant plastic or glass bottles were used. When analysis could not be done immediately, samples were stored at 4 °C to minimise microbial decomposition of solids.

**APPENDIXIII** 

#### CALCULATION

mg Total Suspended Solids/L =  $(A-B) \times 1000$ sample vol, mL

Where: A = weight of dried residue + dish (mg) B = weight of empty dish (mg)

#### (b) Total Volatile Solids (TVS)

The residue from the determination of Total Suspended Solids was ignited to constant weight at  $550 \pm 50$  °C. Normally an ignition period of 20 to 30 min was used. The remaining solids represent the fixed total dissolved solids, while weight loss is the volatile solids.

#### CALCULATION

mg Volatile Suspended Solids/L =  $(A-B) \times 1000$ 

sample vol, mL

Where: A = weight of dried residue + dish (mg) before ignition

B = weight of residue + dish (mg) after ignition

#### (c) Total Suspended Solids (TSS)

A well-mixed sample is filtered through a weighted standard glass fibre filter, and the residue is dried at 103 °C to 105 °C. The filter is washed with 3 successive 20 mL portions of distilled water. The increase in weight of the filter represents the total suspended solids (Standard Methods; 1995)

We used an alternative method, which is also by the University Of Cape Town Waste Water Engineering & Research Group. A measured volume (100 mL) of well-mixed sample was placed in a centrifuge tube and centrifuged at 15000 to 20 000 rpm for 20 min. The supernatant was decanted into and used for dissolved solids analysis. The pellet was then suspended in distilled water and transferred into pre-weighted crucible. This was dried in an oven at 105 °C.

#### CALCULATION

mg Total Suspended Solids/L =  $(A-B) \times 1000$ sample vol, mL

where: A = weight of dried residue + dish (mg) B = weight of empty dish (mg)

#### (d) Volatile Suspended Solids (VSS)

The residue from the determination of Total Suspended Solids is ignited to constant weight at  $550 \pm 50$  °C. Usually a 20 to 30 min ignition period is enough. The remaining solids represent the fixed total dissolved solids, while weight loss is the volatile solids.

#### CALCULATION

mg Volatile Suspended Solids/L =  $(A-B) \times 1000$ sample vol, mL

where: A = weight of dried residue + dish (mg) before ignition

B = weight of residue + dish (mg) after ignition

#### III-3.5. Chemical oxygen demand (COD)

The dichromate reflux method is preferred over procedures using other oxidants. This is because of its superior oxidizing ability, applicability to a wide variety of samples, and ease of manipulation. Oxidation of most organic compounds is 95 to 100% of the theoretical value. Pyridine and related compounds resist oxidation, and volatile organic compounds are oxidized only to the extent that they remain in contact with the oxidant. Ammonia present or liberated in the waste from nitrogen containing organic matter is not oxidized in the absence of significant concentration of free chloride ions.

**PRINCIPLE:** Boiling a mixture of chromic and sulphuric acid oxidizes the organic matter. The sample is refluxed in strongly acidic solution with a known excess of potassium dichromate ( $K_2Cr_2O_7$ ). After digestion, the remaining unreduced dichromate is titrated with ferrous ammonium sulphate to determine the chromate consumed. The oxidisable organic matter is calculated in terms of oxygen equivalent.

The half reaction of the reduction of dichromate is:

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

The reaction of the titration with standard ammonium (II) sulphate solution:

$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \rightarrow 6Fe^{3+} + 7H_2O + 2Cr^{3+}$$

The equivalence point is indicated by the sharp colour change from blue-green to red as the ferroin indicator undergoes reduction from the iron (III) to the iron (II) complex.

**INTERFERENCES & LIMITATIONS:** In the reflux method, the volatile straight chain-aliphatic compounds are not oxidized to an appreciable extent. This occurs partly because volatile organic compounds present in the vapour space are not exposed to the oxidizing liquid. The straight chain aliphatic

compounds are oxidized more effectively when  $Ag_2SO_4$  is added as a catalyst. However,  $Ag_2SO_4$  reacts with chlorides, bromides, and iodides to produce precipitates that oxidize only partially. The presence of halides can be overcome largely though not completely, by complexing with mercuric sulphate (Hg SO<sub>4</sub>) before refluxing. To eliminate interference due to NO<sub>2</sub><sup>-</sup>, 10mg of sulfamic acid is added for each mg NO<sub>2</sub><sup>-</sup> N in the sample. Add the same amount of sulfamic acid to the blank.

SAMPLE HANDLING & STORAGE: Collected samples in glass bottles. If storage was unavoidable, samples were preserved by acidification to  $pH \le 2$  using conc.  $H_2$  SO<sub>4</sub>. Samples containing settleable solids were shaken thoroughly to permit representative sampling. Preliminary dilutions were made for waste containing high COD, to reduce the error inherent in measuring small sample volumes.

#### REAGENTS

Standard Potassium Dichromate Solution (0.0417M) Dry primary standard grade K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> at 103 °C for 2 hrs Dissolve 12.259g K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in distilled water and dilute to 1L

Sulphuric Acid Reagent

Weigh out 1Kg of conc. sulphuric acid (550mL acid) Add 5.5g Ag<sub>2</sub>SO<sub>4</sub> (technical grade or crystal powder) Allow to dissolve for 2 days

Ferroin Indicator 1.485g 1,10-phenanthroline monohydrate and 695mg FeSO<sub>4</sub>.H<sub>2</sub>O Dissolve in 100mL of distilled water

Ferrous Ammonium Sulphate (FAS: 0.25M) Weigh 98g of Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>.6H<sub>2</sub>O Dissolve in distilled water Add 20 mL conc. Sulfuric acid Cool Dilute to 1L

#### PROCEDURE

50 mL of appropriately diluted sample 1g Hg<sub>2</sub>SO4 Glass beads 5 mL Sulfuric Acid Reagent 25 mL K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> Attach to reflux Add 70ml more Sulphuric Acid Reagent Boil for 2hrs Titrate with FAS

#### **STANDARDIAZATION**

10 mL K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> Dilute to 100 mL Add 30 mL conc. sulphuric acid Cool Titrate with FAS

Molarity of FAS solution = <u>volume of 0.0417M Chromate (mL) used</u>  $\times$  0.25

Volume FAS used to in titration (mL)

### CALCULATION

COD as mg O2/L =  $(A - B) \times M \times 8000$ 

mL sample

where : A = FAS mL used for blank

B = FAS mL used for sample M = molarity of FAS

## **APPENDIX IV** Standardisation of the COD test

Measuring COD accurately is important for the purpose of operating and monitoring of biological water treatment processes. Measuring accurately COD is not easy, especially in samples that containing solids such as sewage and sludge. In the following experiment we want to measure the accuracy of the method using an ideal solution such as Potassium Hydrogen Phthalate (KHP). For sewage we are more interested on the standard deviation.

## IV-1 Method

Three standard KHP solutions at different concentration were prepared; each solution was diluted into three concentrations. So from each solution different concentrations were prepared. Each concentration was analysed in duplicate. KHP has a theoretical COD of 1.176 mg COD/mg KHP in a litre.

800 mgCOD/L Solution; 340.1 mg KHP were dissolved in 500 mL distilled water.

600 mgCOD/L Solution; 255.1 mg KHP were dissolved in 500 mL distilled water.

400 mgCOD/L Solution; 170.1mg KHP were dissolved in 500 mL distilled water.

Further dilutions of the standard solutions were prepared according to Table IVI-1.

		SAMPLES		
SOLUTIONS	800 mgCOD/L	600 mgCOD/L	400 mgCOD/L	SEWAGE
DILUTIONS				
D <sub>0</sub>	800	600	400	1
D <sub>1</sub> (1/2 D <sub>0</sub> )	400	300	200	1/2
D <sub>2</sub> (1/3 D <sub>0</sub> )	266.7	200	133.3	1/3

Table IV-1: Table for the further dilution of the standard solutions and sewage

**IV-2** Calculations

Arithmeticmean 
$$X = \frac{\sum xi}{n}$$

APPENDIX IV

$$S \tan dardDeviation = \sqrt{\frac{\sum (xi - Xi)^2}{(n-1)}}$$

 $S \tan dardError = \frac{StdDeviation \times 100}{ArithmeticMean}$ 

## IV-3 Results

	Table IV-2	Experimental	results	using	KHP
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DILUTION	800 mgCOD/L	600 mgCOD/L	400 mgCOD/L
	779.52	555.82	383.04
D(1/1)x1	751.24	554.74	398.36
	672	495.04	372.92
D(1/2)x2	746.9	559.1	378.72
	806.4	534.12	387.18
D(1/3)x3	742.56	563.46	378.72
Average COD	749.77	543.71	383.16
Standard Deviation	45.17	25.91	8.85
Standard Error (%)	6.02	4.77	2.31

Table IIV-3: Experimental results on influent and effluent samples

	Influent		Effluent	
	Sample1	Sample2	Sample1	Sample2
	782.69	765.86	209.66	205.6
D(1/1)x1				
	705.86	745.61	197.78	193.53
D(1/2)x2				
	516.48	541.69	185.07	185.07
D(1/3)x3				
Avg STD Deviation		19.28		2.9
Avg STD Error (%)		2.85		1.48

## IV-4 Discussion and conclusion

The highest Standard Error for the experiment using KHP was 6% which is low, the test shows high precision. The difference between the measured COD and the calculated COD is probably due to the quality of the KHP. It does disintegrate while in storage.

For influent and effluent samples the Standard Error is also very low, but the measured COD for the diluted samples does not agree with the dilution factor. This indicated that diluting accurately samples that have solids is not easy. A lot of COD is contained in the solids; if they are not evenly distributed during dilution they can give inaccurate values.

The COD test has high precision but great care has to be taken when dealing with samples with solids. Samples with solids have to be kept well shaken whilst diluting as to have even distribution of solids.

# **Appendix** V Biochemical methane potential test results

DIGESTER SLUDGE										
Cumulative gas production (mL)										
SERIAL TIME	BLNK1	BLNK2	INF1	INF2	EFF1	EFF2	SYN1	SYN2	SCM1	SCM2
0.602222			0	0	0	0	0		0	0
0.583333	0	0	0	0	0	0	0	0	0	0
1.541667	12	12	16	18	16	14	22	22	15	16
2.53125	22	21	26	30	26	23	36	36	25	28
3.708333	28	29	36	40	34	33	46	46	33	36
4.489583	34	35	46	49	44	41	52	54	41	.44
5.447917	38	39	52	49	52	45	58	60	45	46
6.46875	42	43	56	59	58	49	64	66	49	52
7.416667	45	47	60	63	62	55	68	70	53	56
9.53125	52	54	68	70	71	61	76	80	59	64
12.46875	60	64	78	78	83	73	86	93	68	74
14.5	65	69	85	89	91	79	96	101	74	82
19.5	75	79	97	101	103	91	108	113	80	94
21.5	81	82 .	101	105	107	91	112	119	84	99
23.5	87	86	105	109	111	91	116	123	88	103
27.45833	93	92	111	115	116	93	121	129	92	107
34.4375	104	102	121	125	125	101	131	139	101	116
36.40625	106	106	124	129	128	105	135	143	103	119
40.4375	109	110	128	134	133	109	139	147	107	124
45.41667	112	113	128	136	137	113	141	150	109	127
54.48958	136	137	153	160	161	137	165	174	133	150

## Table V-1: Cumulative gas production for serum bottle with digester sludge

	ABR SLUDGE									
<b>.</b>	Cumulative gas production (mL)									
SERIAL TIME	SERIAL TIME BLNK1 BLNK2 INF1 INF2 EFF1 EFF2 SYN1 SYN2 SCM1 SCM2									SCM2
0 583333	0	0	0	0	0	0	0	0	0	0
1.541667	0	0	0	0	0	0	1	1	0	0
2.53125	0	0	0	0	0	0	2	2	0	0
3.708333	0	0	2	1	0	0	2	3	1	I
4.489583	0	0	2	2	0	0	4	5	2	1
5.447917	2	1	3	3	1	0	6	7	3	2
6.46875	3	3	4	4	1	0	8	8	4	3
7.416667	5	4	6	6	2	0	10	10	6	5
9.53125	7	4	8	10	6	0	14	15	10	7
12.46875	14	10	14	18	12	0	24	25	18	10
14.5	23	14	22	28	18	0	35	36	28	14
19.5	37	26	38	42	33	0	47	50	42	31
21.5	43	30	40	46	39	0	49	58	48	33
23.5	47	34	44	50	43	0	49	64	51	37
27.45833	53	37	49	56	49	0	53	69	57	43
34.4375	60	43	57	68	57	0	65	83	69	55
36.40625	64	45	61	72	61	0	67	86	73	58
40.4375	66	47	63	75	63	0	73	94	77	62
45.41667	68	47	65	77	65	0	75	98	79	64
54.48958	90	69	87	99	87	0	97	121	102	86

Table V-2: Cumulative gas production for serum bottle with anaerobic baffled reactor sludge

# APPENDIX VI Settling tests

## VI-1 Apparatus

A 1.8 m perspex column was used for the test. Sampling ports were fitted along the length of the column. The diameter of the tube was 10 cm, and the sampling heights are at 140 cm, 110 cm, 80 cm, 50 cm and 20 cm. The column is rotated about its axis for mixing, the idea is to sufficiently mix the column, stop and start the sampling as soon as possible. Each of the five sampling ports was designed to draw a sample from the centre of the column, as the walls of the column will have an effect on the settling particle close to the wall. The ports were 0.5 inch in diameter, the large size allowed fast sampling and were not prone to blocking during sampling.

#### VI-2 Method

Two sampling ports are found at the top of the reactor. One port is on the down-flow side, and the other on the up-flow side of the compartment. The sample was taken from the top of the ABR on the up-flow side, using a closable sampling column with a bottom stopper. The sampling column was immersed into the compartment until it touched the bottom, then the stopper was closed and the captured liquid was collected in a bucket. This method of sampling gives a good composite sample of all the solids in each compartment.

The settling column was then charged, until the height of 180 cm. The apparatus was sealed, and then shaken to completely to mix sample. Two samples were taken at  $t_0$ . Samples were collected at time intervals of  $t_{0.5}$ ,  $t_{1.0}$ ,  $t_{1.5}$ ,  $t_{2.0}$ ,  $t_{2.5}$ ,  $t_{3.0}$ ,  $t_{4.0}$ ,  $t_{.5.0}$ ,  $t_{6.0}$ ,  $t_{8.0}$  and  $t_{10}$  minutes. Each port is sampled individually. Before sampling the next port the apparatus is recharged with solids to the previous level. This is to keep the liquid height constant throughout the sampling.

Before analysing for solids, a blank is prepared by allowing a sample to settle for 24 hrs in the sampling bottle. The blank represent the fraction of solids that cannot settle out of the liquid phase eve after 24 hrs. The samples are analysed for TSS. The fraction of settled solids is plotted against the settling velocities. The velocity can easily be calculated as the height of the port and the time is known. The solids in the sample are an indication of the unsettled solids.

#### VI-3 Calculations

$$\frac{\text{TSS}_{n} - \text{TSS}_{b}}{\text{TSS}_{0} - \text{TSS}_{b}} \times 100 = \% \text{Unsettled solids(suspended)}$$
[VI-1]

$$100 - \%$$
 Unsettled solids = % Solids Retained [VI-2]

where :  $TSS_0$  - sample at t=0

TSS<sub>n</sub> - sample take at t=n

 $TSS_b$  – blank sample (settled for 24hrs)

$$\frac{\left[H_0 - \left(S_{n-1} \times \frac{S_v}{C_{SA}}\right)\right] - P_H}{t_{min}/60} \qquad [VI-3]$$

where :  $H_0 - starting liquid height (m)$ 

P<sub>H</sub> - height of port being sampled (m)

 $S_n - n^{th}$  sample taken whilst sampling that port

 $S_V$  – sample volume (m<sup>3</sup>)

 $C_{SA}$  – column surface area (m<sup>2</sup>)

 $t_{min}-\ensuremath{\text{time}}$  sample was taken in minutes

Table VI-1: %Suspended solids from settling tests (Peavy et al., 1985)

DEPTH	TIME (b)						
	0.5	1	1.5	2	2.5	3	
0.5	47	67	80	85	88	91	
1	28	50	63	74	78	83	
1.5	19	40	53	63	72	77	
2	15	33	46	56	64	72	
2.5	12	28	42	51	59	68	
3	10	25	38	47	55	62	

## VI-4 Results of the settling tests

Fine (min)For (min)For (min)Period (min)0270532728552011.673028105728.105728.20720.20111.65127.45427.605920.20111.951.528.60727.80520.20111.952.525.90227.21720.20110.9052.525.90227.80520.20110.9052.627.74820.40220.2010.005527.74920.40220.2016.35022.75626.42220.2016.35022.85626.32120.2016.35021.85920.20120.2016.35021.85920.20120.20112.541021.95722.03320.20112.541126.95227.95720.20120.20112.541528.96420.0120.20112.541528.95020.0120.20113.951528.95020.0120.20113.951528.95020.4020.40113.951528.95020.40120.40113.951528.95020.40120.40113.951528.95020.40120.40113.951528.95020.40120.40113.951529.295020.40120.40113.951629.295020.40120.40113.951529.295020		PORT1						
027.05327.28501.6750.528.109528.3427201.66127.45427.6952012.0951.528.6629.0866201.1952.52.6992827.217201.945328.016528.2405201.5552.774927.9603209.27627.85628.040201.05552.074929.693208.03102.527628.462208.03102.527628.162208.03102.527628.162208.03102.527628.162208.03102.527628.162201.5295102.649220.111.5295112.613528.093201.254112.613526.9759201.3121528.00629.4097201.95522.85829.4097201.85722.85828.1559201.85732.783228.648201.85742.6335201.85752.732727.558201.85752.732727.558201.662552.732727.6933201.45462.44142.6952201.662552.54142.6052201.85162.73272.7358 <td< th=""><th>Time (min)</th><th>Empty crucible (g)</th><th>Total solids (g)</th><th>Volume of sample (mL)</th><th>Density (g/L)</th></td<>	Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
0.5         28,105         28,1427         20         1.165           1         27,454         27,659         20         1.2095           1.5         28,86         290866         20         1.133           2         27,655         27,6604         20         1.195           2.5         26,9928         27,2317         20         1.1945           3         28,0165         27,2317         20         1.22           4         27,1855         27,3956         20         1.52           5         27,7749         27,9603         20         9,27           6         27,8564         28,042         20         8,33           10         28,1667         26,4282         20         8,33           10         28,1667         26,4282         20         8,33           10         28,1667         26,4282         20         12,24           110         28,1697         20,12         12,54         12,54           110         26,672         27,021         20,12         12,54           111         26,713         26,9759         20         13,12           15         28,064         20	0	27.053	27 2865	20	11 675			
non-1.000 2.74547.76559.2.000 2.0001.20051.52.846.02.90666.02.01.1.3322.7.636.52.7.860.42.01.1.94532.5.925.02.7.217.02.01.1.94532.8.0165.02.8.2405.02.01.0.9552.8.716.02.7.7492.7.9603.02.00.0.5762.7.7492.9.603.02.08.030.0102.8.8662.08.030.01.0.9782.6.26762.6.422.02.08.030.0102.8.8692.01.0.978.001.0.9782.6.9752.8.203.02.01.5.251.5.250.52.7.95752.8.203.02.01.1.211.52.8.9042.01.9.951.9.952.62.7.8582.9.40072.01.9.952.52.7.8582.9.40072.01.9.952.62.8.8042.01.9.953.62.8.8042.01.9.953.72.8.8042.01.9.953.82.8.2092.01.5.555.62.7.8272.9.6432.01.9.955.62.7.32872.5.812.01.9.9562.4.8142.6.9522.01.9.9562.4.8142.6.9522.01.9.957.7.52.7.27672.01.9.851.62.7.27772.01.3.351.52	0.5	28 1095	28 3427	20	11.66			
1.52.8.662.9.08662.011.3322.7.63652.7.6042011.1952.52.8.0452.8.24652.011.242.7.18552.7.39562.09.2752.7.77492.7.6032.00.3782.6.26762.6.42822.08.03102.8.1692.8.142.03.5PORTThre (min)Empty crucibe (g)Total solids (g)Volume of sample (mL)Pensity (g/L)12.6.59622.7.00112.015.2950.52.7.95752.8.20322.015.2950.52.7.95752.8.20322.015.2950.52.7.95752.8.20322.012.541.52.8.0662.9.40672.012.6052.52.7.95752.8.20322.013.121.52.8.0662.9.40672.012.6052.52.7.3582.8.13592.013.89532.7.8222.8.6482.013.89532.7.8222.8.6482.013.69552.7.2872.7.5582.016.69262.6.4142.6.5922.016.69262.7.9242.7.5582.016.692102.0.9942.7.93512.014.54552.7.2872.7.0312.014.54552.7.2972.7.5582.014.5456.52.6.7932.0 <td>1</td> <td>27.454</td> <td>27.6959</td> <td>20</td> <td>12.095</td>	1	27.454	27.6959	20	12.095			
22,863527,86042011.1952.626,92827,3172010.945326,016528,24052010.2427,185527,395620927527,74927,960320927627,856628,640208.031028,18628,2422208.031128,18628,314208.03PORTPORTPortole (g)Portole (g) <td>1.5</td> <td>28.86</td> <td>29.0866</td> <td>20</td> <td>11.33</td>	1.5	28.86	29.0866	20	11.33			
2.52.699282.723172.01.1.94532.801652.84052.01.242.718552.739562.09.2752.735662.80642.00.03782.626762.82422.08.03102.18692.81402.08.03PORTThre (min)Empty crucibe (g)Total solids (g)Volue of sample (mL)Density (g/L)02.65927.00212.015.2950.52.73752.82032.01.52950.52.737572.82032.01.52950.52.73552.82032.01.52951.52.80062.94072.01.6052.52.73582.94072.01.8953.42.73222.86482.01.8953.42.75272.75372.82031.15552.73572.82692.01.16562.73522.75582.01.16562.73522.75582.01.1551.02.73952.86482.01.15562.73952.84592.01.16562.73952.84592.01.16562.73952.73712.01.16562.75952.01.6251.5451.02.75952.01.4161.12.75952.01.3311.52.64142.85062.0	2	27.6365	27.8604	20	11.195			
328.016528.24052011.2427.185527.39662010.505527.774927.96032092.71627.856628.064208.031028.186928.314206.355PORT2Total solids (g)Volume of sample (mL)Density (g/L)026.696227.00212015.2950.527.957528.20832015.2951.627.357528.20832013.121.528.00629.40752013.121.528.00629.40752013.8952.527.35828.13592018.8953.627.482228.06482011.8953.727.88228.06922011.8953.627.482228.06482011.85426.059528.28692011.85626.481426.69522011.6652626.481426.69522011.851027.25727.32712015.85527.55728.0632014.181.127.55428.0632014.181.127.55728.0732014.181.127.55727.55726.0332014.181.127.55727.55720.55715.652527.55727.55720.55715.652627.55727.557 <td>2.5</td> <td>26.9928</td> <td>27.2317</td> <td>20</td> <td>11.945</td>	2.5	26.9928	27.2317	20	11.945			
427.185527.3956200.505527.74927.9603209.27627.385626.02020.30310.37826.26726.422206.3531028.186928.314206.355FORTTime (min)Empt crucibe (g)7.0021205.550.527.997528.20832012.54126.696227.00212013.2950.527.997528.20832013.2951.528.00629.40972012.605229.28629.40972013.895229.28629.40972015.695327.88228.46842015.55327.88228.64842015.55527.328727.5582015.55527.328727.5582015.55626.491426.69522011.813626.795427.97542015.65626.491426.69522016.6825627.38727.5582016.68257527.38427.003120.20115.657527.36427.003120.20113.31127.5582020.41133.31127.5592020.41133.312222236.6220.4111.522	3	28.0165	28.2405	20	11.2			
527.74927.903209.27627.856528.0642010.37826.267628.064208.031028.186928.102208.35FORT2FORT2Time (min)Zef.66627.002120 June of sample (mL)Density (g/L)026.69627.00212015.2950.527.957528.20832012.54126.713526.97592013.121.528.00629.04052011.995229.28629.04072012.6052.57.85828.13592013.895327.828228.05482011.8348.055928.28692011.85527.328727.5582011.85552.739526.69322011.8551026.379526.69322011.8551026.379526.6932011.8551126.379526.6932013.371525.5427.8582014.1661585.4428.0592013.331527.57720.9032014.181627.57727.9032014.3151525.5428.8062013.332527.5582013.331527.5592013.331528.5428.05920 <t< td=""><td>4</td><td>27.1855</td><td>27.3956</td><td>20</td><td>10.505</td></t<>	4	27.1855	27.3956	20	10.505			
627.856628.0642010.37826.267626.4282208.031028.186928.314206.355INTERISTING COLSPANSION OF COLSPANSIO	5	27.7749	27.9603	20	9.27			
826.267626.4282208.0.31028.186928.314206.355PORT2FORT2Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)026.696227.00212012.54126.713528.20832012.54126.713528.0062011.995229.238629.40072012.605229.238629.40072013.895327.828.228.06482011.83426.055928.28692011.465527.32725.582011.465626.481426.69522011.657527.32727.5752011.8551020.595420.20111.652626.481426.69522011.657626.481426.69522011.657720.595420.20111.9551020.595420.20111.851020.595420.0032014.6181127.56427.00932014.5181526.725720.00932014.5181526.725727.00932014.5151526.725720.00932013.331527.596428.0062013.332527.59728.0262013.332527.597	6	27.8566	28.064	20	10.37			
1028.186928.314206.355PORT2Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)026.696227.00212015.2950.527.957528.20832012.54126.713528.07992013.121.528.803629.40072012.6052.527.85828.13592013.8952.527.85828.06482011.83327.828228.06482011.465527.32712011.465626.481426.69522010.69827.095427.32712011.5851026.379526.60332011.951126.379526.6032011.95527.526427.00312014.181127.364426.9522014.181228.409928.743552014.181321.53428.0562013.332.526.5342013.3313.332.527.59720.6332013.332.527.5972013.3313.332.527.5972013.3313.332.527.5972013.3313.332.527.5972013.3313.551.528.53428.0562013.332.527.5972013.33 <td>8</td> <td>26.2676</td> <td>26.4282</td> <td>20</td> <td>8.03</td>	8	26.2676	26.4282	20	8.03			
FORT2           Time (min)         Empty crucible (g)         Total solids (g)         Volume of sample (mL)         Density (g/L)           0         26.6962         27.0021         20         15.295           0.5         27.9575         28.2083         20         12.54           1         26.7135         26.9759         20         13.12           1.5         28.8006         29.0405         20         13.995           2         29.2386         29.4907         20         18.895           3         27.8528         28.1359         20         18.895           3         27.8282         28.0648         20         11.83           4         28.0559         28.2869         20         11.655           5         27.3287         27.558         20         11.69           6         26.4814         26.6952         20         10.69           8         27.0954         27.3271         20         11.95           10         26.3795         26.6033         20         11.19           0         28.4099         28.74355         20         14.18           1         27.3684         27.6593         20	10	28.1869	28.314	20	6.355			
Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)026.696227.0021015.2950.527.957528.20832012.54126.713526.9759013.121.528.800629.04072019.957229.238629.49072013.8952.527.58328.1599018.895327.828228.06482011.83428.055928.28692011.65527.328727.5582011.65626.481426.69522010.69827.995426.0332011.921026.37526.0332011.92526.725727.00932014.18127.68427.69332014.18127.68427.69332014.3152.527.527627.00932014.3151.528.54428.80062013.33227.69426.257720.00114.3152.527.527620.00114.3152.527.527620.00114.3152.527.527620.00114.3152.527.527620.00114.3152.527.527620.00114.3152.527.527620.00114.3152.527.527620.00115.452.527.527620.00115.45 <th></th> <th></th> <th>PORT2</th> <th></th> <th></th>			PORT2					
0         26.6962         27.0021         20         15.295           0.5         27.9575         28.2083         20         12.54           1         26.7135         26.9759         20         13.12           1.5         28.8006         29.0405         20         11.995           2         29.2386         29.0405         20         12.605           2.5         7.7858         28.1359         20         13.895           3         77.8282         28.0648         20         11.83           4         28.0559         28.2869         20         11.655           5         27.3287         27.558         20         11.665           6         26.4814         26.6952         20         10.69           8         27.0954         27.3271         20         11.55           10         26.3795         26.6033         20         11.9           Volume of sample (mL)         Density (g/L)         Density (g/L)           0         28.4099         28.74355         20         14.18           1         27.3644         27.6593         20         14.545           1.5         28.534         28.2467	Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
026.696227.00212015.2950.527.957528.20832012.54126.713526.97592013.121.528.800629.04052011.995229.238629.49072012.6052.527.85828.13592013.895327.828228.06482011.83428.055928.28692011.55527.328727.5582011.65626.481426.69522010.69827.095427.32712011.5851026.379526.6032011.19PORT3Colspan="3">Colspan="3">Density (g/L)1.191.19O1.19Colspan="3">Colspan="3								
0.5 $27,975$ $28,2083$ $20$ $12,54$ $1$ $26,7135$ $26,9759$ $20$ $13,12$ $1.5$ $28,8006$ $29,0405$ $20$ $11.995$ $2$ $29,2386$ $29,4907$ $20$ $12,605$ $2,52$ $27,858$ $28,1359$ $20$ $13,895$ $3$ $27,8282$ $28,0648$ $20$ $11.83$ $4$ $28,0559$ $28,2869$ $20$ $11.65$ $5$ $27,3287$ $27,558$ $20$ $11.65$ $6$ $26,4814$ $26,6952$ $20$ $10.69$ $8$ $27,0954$ $27,3271$ $20$ $11.585$ $10$ $26,3795$ $26,6033$ $20$ $11.19$ <b>Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)</b> $0$ $28,4099$ $28,74355$ $20$ $14,18$ $1$ $27,3644$ $27,6593$ $20$ $14,315$ $1.5$ $28,534$ $28,8066$ $20$ $13,33$ $2$ $27,9604$ $28,2467$ $20$ $14,315$ $2,5$ $27,2597$ $27,5276$ $20$ $13,33$ $2$ $27,9577$ $28,2857$ $20$ $13,395$ $3$ $27,9767$ $28,2857$ $20$ $14,315$ $4$ $28,1551$ $28,6154$ $20$ $14,292$ $5$ $27,3535$ $27,6393$ $20$ $14,292$ $6$ $27,9594$ $28,8156$ $20$ $14,292$ $6$ $28,514$ $28,136$ $20$	0	26.6962	27.0021	20	15.295			
126.713526.97592013.121.528.800629.04052011.995229.238629.49072012.6052.527.82828.13592013.895327.828228.06482011.83428.055928.28692011.55527.328727.5582011.465626.481426.69522010.69827.095427.32712011.5851026.379526.60332011.99PORT3Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Pensity (g/L)028.409928.743552014.181.127.36427.65932014.181.528.53428.80062013.33227.35727.52762013.33227.959727.52762013.33227.59727.52762013.332.527.59728.8572013.332.527.59728.8572015.453.427.95728.8572015.453.427.95727.63932014.29428.15128.41152012.82527.333527.63932014.29626.733327.63932014.29626.733428.07692014.29627.3334	0.5	27.9575	28.2083	20	12.54			
1.5 $28,8006$ $29,005$ $20$ $11,995$ $2$ $29,2386$ $29,4907$ $20$ $12,605$ $2.5$ $27,828$ $28,1359$ $20$ $13,895$ $3$ $27,8282$ $28,0648$ $20$ $11.83$ $4$ $28,0559$ $28,2869$ $20$ $11.55$ $5$ $27,3287$ $27,558$ $20$ $11.465$ $6$ $26,4814$ $26,6952$ $20$ $10.69$ $8$ $27,0954$ $27,3271$ $20$ $11.585$ $10$ $26,3795$ $26,6033$ $20$ $11.19$ PORT3Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L) $0$ $28,4099$ $28,74355$ $20$ $14.18$ $1$ $27,364$ $27,6593$ $20$ $14,18$ $1$ $27,364$ $28,8006$ $20$ $13,33$ $2$ $27,257$ $27,276$ $20$ $14,315$ $2,5$ $27,257$ $27,5276$ $20$ $13,335$ $2$ $27,9767$ $28,2857$ $20$ $13,335$ $3$ $27,9767$ $28,2857$ $20$ $13,395$ $3$ $27,7576$ $20$ $15,45$ $4$ $28,1551$ $28,4115$ $20$ $14,212$ $5$ $27,3535$ $27,6393$ $20$ $14,29$ $6$ $27,7833$ $20$ $14,29$ $14,29$ $6$ $27,3534$ $28,0749$ $20$ $14,29$ $6$ $27,3534$ $28,0749$ $20$ </td <td>1</td> <td>26.7135</td> <td>26.9759</td> <td>20</td> <td>13.12</td>	1	26.7135	26.9759	20	13.12			
229,238629,49072012,6052.527,85828,13592013,895327,828228,06482011,83428,055928,28692011,55527,328727,5582010,69827,095427,32712011,5851026,379526,60332011,19PORT3Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)028,409928,743552014,18127,368427,65932014,18127,368427,65932014,352.527,259727,52762013,33227,960428,24672013,395327,976728,28572013,395327,976728,28572013,395327,976728,28572013,395327,976728,28572013,395327,976728,28572013,395327,976728,28572015,45428,15128,41152012,82527,353527,63932014,29626,705927,04112016,676828,531428,81362014,111028,131428,81362014,11	1.5	28.8006	29.0405	20	11.995			
2.5 $27.858$ $28.1359$ $20$ $13.895$ $3$ $27.8282$ $28.0648$ $20$ $11.83$ $4$ $28.0559$ $28.2869$ $20$ $11.55$ $5$ $27.3287$ $27.558$ $20$ $10.69$ $6$ $26.4814$ $26.6952$ $20$ $10.69$ $8$ $27.0954$ $27.3271$ $20$ $11.19$ $10$ $26.3795$ $26.6033$ $20$ $11.19$ PORT3Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L) $0$ $28.4099$ $28.74355$ $20$ $14.18$ $1$ $27.3684$ $27.0934$ $20$ $14.545$ $1.5$ $26.534$ $28.8006$ $20$ $13.33$ $2$ $27.9904$ $28.2467$ $20$ $14.315$ $2.5$ $27.2597$ $27.5276$ $20$ $13.395$ $3$ $27.9767$ $28.2857$ $20$ $15.457$ $4$ $8.1551$ $28.4115$ $20$ $14.292$ $5$ $27.3335$ $27.6393$ $20$ $14.292$ $5$ $27.3335$ $27.6393$ $20$ $14.292$ $6$ $27.5335$ $27.6393$ $20$ $14.292$ $6$ $28.314$ $28.036$ $20$ $14.292$ $6$ $27.3335$ $27.6393$ $20$ $14.292$ $6$ $28.314$ $28.036$ $20$ $14.292$ $6$ $28.314$ $28.036$ $20$ $14.111$	2	29.2386	29.4907	20	12.605			
327.828228.06482011.83428.055928.28692011.55527.328727.5582010.69626.481426.69522010.69827.095427.32712011.9851026.379526.60332011.19PORT3Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)028.409928.743552016.68250.526.725727.00932014.18127.368427.65932014.5451.528.53428.80062013.33227.960428.2672013.395327.976728.28572013.395327.976728.28572015.45428.155128.41152015.45527.333527.63932014.292527.333527.63932014.292527.333527.63932014.292527.333527.63932014.292628.331428.0362014.292628.331428.0362014.292628.331428.0362014.1111027.433327.63932014.292527.333527.63932014.292628.331428.0362014.111	2.5	27.858	28.1359	20	13.895			
4         28.0559         28.2869         20         11.55           5         27.3287         27.558         20         11.465           6         26.4814         26.6952         20         10.69           8         27.0954         27.3271         20         11.585           10         26.3795         26.6033         20         11.19           PORT3           Time (min)         Empty crucible (g)         Total solids (g)         Volume of sample (mL)         Density (g/L)           0         28.4099         28.74355         20         16.6825           0.5         26.7257         27.0093         20         14.18           1         27.3684         27.6593         20         14.545           1.5         28.534         28.8066         20         13.33           2         27.9604         28.2467         20         14.315           2.5         27.2597         27.5276         20         13.395           3         27.9767         28.2857         20         13.395           3         27.9767         28.2857         20         12.82           5         27.3355         27.6393 </td <td>3</td> <td>27.8282</td> <td>28.0648</td> <td>20</td> <td>11.83</td>	3	27.8282	28.0648	20	11.83			
5         27.3287         27.558         20         11.465           6         26.4814         26.6952         20         10.69           8         27.0954         27.3271         20         11.585           10         26.3795         26.6033         20         11.19           PORT3           Total solids (g)         Volume of sample (mL)         Density (g/L)           0         28.4099         28.74355         20         16.6825           0.5         26.7257         27.0093         20         14.18           1         27.3684         27.6593         20         13.33           2         2.7.9604         28.2467         20         13.33           2         27.9577         27.5276         20         13.33           3         27.9597         27.5276         20         13.395           3         27.9577         28.2857         20         15.45           4         28.1551         28.4115         20         12.82           5         27.3355         27.6393         20         14.29           6         26.7059         27.0411         20         14.29	4	28.0559	28.2869	20	11.55			
6       26.4814       26.6952       20       10.69         8       27.0954       27.3271       20       11.585         10       26.3795       26.6033       20       11.19         PORT3         Time (min)       Empty crucible (g)       Total solids (g)       Volume of sample (mL)       Density (g/L)         0       28.4099       28.74355       20       16.6825         0.5       26.7257       27.0093       20       14.18         1       27.3684       27.6593       20       13.33         2       27.9604       28.2467       20       13.33         2       27.9504       28.2857       20       13.395         3       27.9577       27.5276       20       13.395         3       27.9577       28.857       20       13.395         3       27.9577       28.2857       20       15.45         4       28.1551       28.4115       20       12.82         5       27.3535       27.6393       20       14.29         6       26.7059       27.0411       20       16.76         8       28.514       28.136       20	5	27.3287	27.558	20	11.465			
8         27.0954         27.3271         20         11.585           10         26.3795         26.6033         20         11.19           PORT3           Time (min)         Empty crucible (g)         Total solids (g)         Volume of sample (mL)         Density (g/L)           0         28.4099         28.74355         20         16.6825           0.5         26.7257         27.0093         20         14.18           1         27.3684         27.6593         20         14.545           1.5         28.534         28.8006         20         13.33           2         27.9604         28.2467         20         14.315           2.5         27.2597         27.5276         20         13.395           3         27.9767         28.2857         20         13.395           3         27.9767         28.2857         20         12.82           5         27.3535         27.6393         20         14.29           6         26.7059         27.0411         20         16.76           8         28.5314         28.8136         20         14.11           10         27.8133         28.0749<	6	26.4814	26.6952	20	10.69			
10         26.3795         26.6033         20         11.19           PORT3           Time (min)         Empty crucible (g)         Total solids (g)         Volume of sample (mL)         Density (g/L)           0         28.4099         28.74355         20         16.6825           0.5         26.7257         27.0093         20         14.18           1         27.3684         27.6593         20         14.545           1.5         28.534         28.8006         20         13.33           2         27.9604         28.2467         20         14.315           2.5         27.2597         27.5276         20         14.315           2.5         27.9767         28.2857         20         14.315           3         27.9767         28.2857         20         15.45           4         28.1551         28.4115         20         12.82           5         27.3535         27.6393         20         14.29           6         26.7059         27.0411         20         16.76           8         28.5314         28.8136         20         14.11           10         27.8133         28.0749	8	27.0954	27.3271	20	11.585			
PORT3           Time (min)         Empty crucible (g)         Total solids (g)         Volume of sample (mL)         Density (g/L)           0         28.4099         28.74355         20         16.6825           0.5         26.7257         27.0093         20         14.18           1         27.3684         27.6593         20         14.545           1.5         28.534         28.8006         20         13.33           2         27.9604         28.2467         20         14.315           2.5         27.2597         27.5276         20         13.395           3         27.9767         28.2857         20         15.45           4         28.1551         28.4115         20         12.82           5         27.3335         27.6393         20         14.29           6         26.7059         27.0411         20         14.29           6         26.7059         27.0411         20         16.76           8         28.5314         28.8136         20         14.11	10	26.3795	26.6033	20	11.19			
Time (min)Empty crucible (g)Total solids (g)Volume of sample (mL)Density (g/L)028.409928.743552016.68250.526.725727.00932014.18127.368427.65932014.5451.528.53428.80062013.33227.960428.24672013.3952.527.259727.52762013.395327.976728.28572015.45428.155128.41152012.82527.353527.63932014.29626.705927.04112016.76828.531428.81362014.111027.813328.07492016.76			PORT3					
028.409928.743552016.68250.526.725727.00932014.18127.368427.65932014.5451.528.53428.80062013.33227.960428.24672014.3152.527.259727.52762013.395327.976728.28572015.45428.155128.41152012.82527.353527.63932014.29626.705927.04112016.76828.531428.81362014.111027.813328.07492012.62	Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
028.74552016.68250.526.725727.00932014.18127.368427.65932014.5451.528.53428.80062013.33227.960428.24672014.3152.527.259727.52762013.395327.976728.28572015.45428.155128.41152012.82527.353527.63932014.29626.705927.04112016.76828.531428.81362014.111027.813328.07492013.00	0	28 4000	28 74255					
0.526.725727.00932014.18127.368427.65932014.5451.528.53428.80062013.33227.960428.24672014.3152.527.259727.52762013.395327.976728.28572015.45428.155128.41152012.82527.353527.63932014.29626.705927.04112016.76828.531428.81362014.111027.813328.07492013.00	0.5	26.4099	28.74355	20	16.6825			
121.50421.5032014.5451.528.53428.80062013.33227.960428.24672014.3152.527.259727.52762013.395327.976728.28572015.45428.155128.41152012.82527.353527.63932014.29626.705927.04112016.76828.531428.81362014.111027.813328.07492013.02	1	20.7237	27.0093	20	14.18			
13.33       20.504       20.600       20       13.33         2       27.9604       28.2467       20       14.315         2.5       27.2597       27.5276       20       13.395         3       27.9767       28.2857       20       15.45         4       28.1551       28.4115       20       12.82         5       27.3535       27.6393       20       14.29         6       26.7059       27.0411       20       16.76         8       28.5314       28.8136       20       14.11         10       27.8133       28.0749       20       13.02	15	28 534	27.0333	20	14.545			
2.5       27.2597       27.5276       20       13.395         3       27.9767       28.2857       20       15.45         4       28.1551       28.4115       20       12.82         5       27.3535       27.6393       20       14.29         6       26.7059       27.0411       20       16.76         8       28.5314       28.8136       20       14.11         10       27.8133       28.0749       20       13.395	2	27.9604	28.8000	20	13.33			
3       27.9767       28.2857       20       15.45         4       28.1551       28.4115       20       12.82         5       27.3535       27.6393       20       14.29         6       26.7059       27.0411       20       16.76         8       28.5314       28.8136       20       14.11         10       27.8133       28.0749       20       13.395	2.5	27.2597	27 5276	20	14.515			
4       28.1551       28.4115       20       12.82         5       27.3535       27.6393       20       14.29         6       26.7059       27.0411       20       16.76         8       28.5314       28.8136       20       14.11         10       27.8133       28.0749       20       13.02	3	27.9767	28 2857	20	15.393			
5     27.3535     27.6393     20     14.29       6     26.7059     27.0411     20     16.76       8     28.5314     28.8136     20     14.11       10     27.8133     28.0749     20     13.02	4	28.1551	28.4115	20	12.45			
6     26.7059     27.0411     20     16.76       8     28.5314     28.8136     20     14.11       10     27.8133     28.0749     20     12.00	5	27.3535	27 6393	20	12.02			
8         28.5314         28.8136         20         14.11           10         27.8133         28.0749         20         13.00	6	26.7059	27.0411	20	14.27			
10 27,8133 28,0749 20 13,00	8	28.5314	28.8136	20	10.70			
20.0777 ZV I I K IX	10	27.8133	28.0749	20	13.08			

Table VI-2	e: Results of	of settling test	measurements	for compartment 1

## <u>APPENDIX VI</u>

PORT1							
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
0	27.2865	27.7858	20	24.965			
0.5	28.3427	28.8682	20	26.275			
1	27.6959	28.2279	20	26.6			
1.5	29.0866	29.6261	20	26.975			
2	27.8604	28.3658	20	25.27			
2.5	27.2317	27.7869	20	27.76			
3	28.2405	28.7467	20	25.31			
4	27.3956	27.8997	20	25.205			
5	27.9603	28.4938	20	26.675			
6	28.064	28.6186	20	27.73			
8	26.4282	26.8944	20	23.31			
10	28.314	28.837	20	26.15			

## Table VI-3: Results of settling test measurements for compartment 2

PORT2							
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
0	27.0021	27.5461	20	27.2			
0.5	28.2083	28.7862	20	28.895			
1	26.9759	27.5457	20	28.49			
1.5	29.0405	29.6659	20	31.27			
2	29.4907	30.0495	20	27.94			
2.5	28.1359	28.7053	20	28.47			
3	28.0648	28.6415	20	28.835			
4	28.2869	28.6415	20	17.73			
5	27.558	28.1227	20	28.235			
6	26.6952	27.2602	20	28.25			
8	27.3271	27.8622	20	26.755			
10	26.6033	27.144	20	27.035			

PORT3								
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)				
0	28.74355	29.2985	20	27.7475				
0.5	27.0093	27.5626	20	27.665				
1	27.6593	28.2355	20	28.81				
1.5	28.8006	29.3502	20	27.48				
2 .	28.2467	28.781	20	26.715				
2.5	27.5276	28.1024	20	28.74				
3	28.2857	28.8495	20	28.19				
4	28.4115	28.9843	20	28.64				
5	27.6393	28,1929	20	27.68				
6	27.0411	27.6279	20	29.34				
8	28.8136	29.3984	20	29.24				
10	28.0749	28.6412	20	28.315				

## Table VI-4: Results of settling test measurements for compartment 3

PORT1							
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
0	27.7858	28.0167	20	11.545			
0.5	28.8682	29.0777	20	10.475			
1	28.2279	28.3916	20	8.185			
1.5	29.6261	29.7904	20	8.215			
2	28.3658	28.5307	20	8.245			
2.5	27.7869	27.9312	20	7.215			
3	28.7467	28.9045	20	7.89			
4	27.8997	28.0025	20	5.14			
5	28.4938	28.588	20	4.71			
6	28.6186	28.6983	20	3.985			
8	26.8944	26.9508	20	2.82			
10	28.837	28.8485	20	0.575			

PORT2							
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)			
0	27.5461	27.9616	20	20.775			
0.5	28.7862	29.243	20	22.84			
1	27.5457	27.996	20	22.515			
1.5	29.6659	30.1263	20	23.02			
2	30.0495	30.4661	20	20.83			
2.5	28.7053	29.1639	20	22.93			
3	28.6415	29.0773	20	21.79			
4	28.6415	29.2773	20	31.79			
5	28.1227	28.4949	20	18.61			
6	27.2602	27.6316	20	18.57			
8	27.8622	28.218	20	17.79			
10	27.144	27.4906	20	17.33			

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
		•		
0	29.2985	29.8675	20	28.45
0.5	27.5626	28.1766	20	30.7
1	28.2355	28.8161	20	29.03
1.5	29.3502	29.914	20	28,19
2	28.781	29.3919	20	30.545
2.5	28.1024	28.6575	20	27.755
3	28.8495	29.4757	20	31.31
4	28.9843	29.579	20	29.735
5	28.1929	28.8165	20	31.18
6	27.6279	28.1453	20	25.87
8	29.3984	29.9872	20	29.44
10	28.6412	29.3234	20	34.11

## <u>APPENDIX VI</u>

PORTI				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.0167	28.2033	20	9.33
0.5	29.0777	29.2613	20	9.18
1	28.3916	28.5615	20	8.495
1.5	29.7904	29.949	20	7.93
2	28.5307	28.6847	20	7.7
2.5	27.9312	28.09	20	7.94
3	28.9045	29.0528	20	7.415
4	28.0025	28.1285	20	6.3 .
5	28.588	28.6977	20	5.485
6	28.6983	28.8092	20	5.545
8	26.9508	27.0404	20	4.48
10	28.8485	28.9147	20	3.31

## Table VI-5: Results of settling test measurements for compartment 4

PORT2				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	27.9616	28.1812	20	10.98
0.5	29.243	29.4377	20	9.735
1	27.996	28.1862	20	9.51
1.5	30.1263	30.3048	20	8.925
2	30.4661	30.6608	20	9.735
2.5	29.1639	29.353	20	9.455
3	29.0773	29.2526	20	8.765
4	29.2773	29.4482	20	8.545
5	28.4949	28.6961	20	10.06
6	27.6316	27.8027	20	8.555
8	28.218	28.3915	20	8.675
10	27.4906	27.6706	20	9

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	29.8675	30.1511	20	14.18
0.5	28,1766	28.4104	20	11.69
1	28.8161	29.0763	20	13.01
1.5	29.914	30.1572	20	12.16
2	29.3919	29.6183	20	11.32
2.5	28.6575	28.8782	20	11.035
3	29.4757	29.7266	20	12.545
4	29.579	29.8106	20	11.58
5	28.8165	29.0618	20	12.265
6	28.1453	28.3863	20	12.05
8	29.9872	30.2516	20	13.22
10	29.3234	29.6015	20	13.905
## Table VI-6: Results of settling test measurements for compartment 5

PORTI				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.2033	28.4023	20	9.95
0.5	29.2613	29.4623	20	10.05
1	28.5615	28.7499	20	9.42
1.5	29.949	30.136	20	9.35
2	28.6847	28.8642	20	8.975
2.5	28.09	28.2626	20	8.63
3	29.0528	29.2196	20	8.34
4	28.1285	28.2927	20	8.21
5	28.6977	28.8373	20	6.98
6	28.8092	28.9418	20	6.63
8	27.0404	27.139	20	4.93
10	28.9147	28.9942	20	3.975

PORT2				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.1812	28.3677	20	9.325
0.5	29.4377	29.6996	20	13.095
1	28.1862	28.4318	20	12.28
1.5	30.3048	30.5429	20	11.905
2	30.6608	30.8921	20	11.565
2.5	29.353	29.594	20	12.05
3	29.2526	29.4868	20	11.71
4	29.4482	29.6952	20	12.35
5	28.6961	28.914	20	10.895
6	27.8027	28.0455	20	12.14
8	28.3915	28.6323	20	12.04
10	27.6706	27.8784	20	10,39

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	30.1511	30.4796	20	16.425
0.5	28.4104	28.7573	20	17.345
1	29.0763	29.4183	20	17.1
1.5	30.1572	30.4777	20	16.025
2	29.6183	29.9324	20	15.705
2.5	28.8782	29.1814	20	15.16
3	29.7266	30.0642	20	16.88
4	29.8106	30.1282	20	15.88
5	29.0618	29.3747	20	15.645
6	28.3863	28.7118	20	16.275
8	30.2516	30.5573	20	15.285
10	29.6015	29.9374	20	16.795

### APPENDIX VI

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PORTI				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.4023	28.5776	20	8.765
1	28.7499 30.136	28.91 30.2876	20 20 20	7.955 8.005 7.58
2	28.8642	29.0066	20	7.12
2.5	28.2626	28.3936	20	
3	29.2196	29.3342	20	5.73
	28.2927	28.3974	20	5.235
5	28.8373	28.9322	20	4.745
6	28.9418	29.0214	20	
8	27.139	27.196	20	2.85
10	28.9942	29.0328	20	1.93

Table VI-7: Result	s of settling test measurements for compartment	6
	PORTI	-

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PORT2				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.3677	28.5551	20	9.37
0.5	29.6996	29.879	20	8.97
1	28.4318	28.6158	20	9.2
1.5	30.5429	30.702	20	7.955
2	30.8921	31.0682	20	8.805
2.5	29.594	29.746	20	7.6
3	29.4868	29.6894	20	10.13
4	29.6952	29.8134	20	5.91
5	28.914	29.0872	20	8.66
6	28.0455	28.2018	20	7.815
8	28.6323	28.7945	20	8.11
10	27.8784	28.0442	20	8.29

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	30.4796	30.7356	20	12.8
0.5	28.7573	28.9949	20	11.88
1	29.4183	29.655	20	11.835
1.5	30.4777	30.705	20	11.365
2	29.9324	30.1695	20	11.855
2.5	29.1814	29.4319	20	12.525
3	30.0642	30.3056	20	12.07
4	30.1282	30.3748	20	12.33
5	29.3747	29.6308	20	12.805
6	28.7118	28.9574	20	12.28
8	30.5573	30.8224	20	13.255
10	29.9374	30.1995	20	13.105

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# Table VI-8: Results of settling test measurements for compartment 7

PORTI				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.5776	28.74238	20	8.2391
0.5	29.6214	29.77095	20	7.4777
1	28.91	29.06049	20	7.5247
1.5	30.2876	30.4023	20	5.735
2	29.0066	29.1404	20	6.69
2.5	28.3936	28.5167	20	6.155
3	29.3342	29.4419	20	5.385
4	28.3974	28.4858	20	4.42
5	28.9322	29.02141	20	4.4603
6	29.0214	29.09622	20	3.7412
8	27.196	27.2555	20	2.975
10	29.0328	29.069	20	1.81 .

PORT2				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.5551	28.7312	20	8.805
0.5	29.879	30.04763	20	8.4315
1	28.6158 .	28.78876	20	8.648
1.5	30.702	30.8515	20	7.475
2	31.0682	31.23373	20	8.2767
2.5	29.746	29.88	20	6.7
3	29.6894	29.87984	20	9.5222
4	29.8134	29.9245	20	5.555
5	29.0872	29.25091	20	8.1854
•6	28.2018	28.34872	20	7.3461
8	28.7945	28.95697	20	8.1234
10	28.0442	28.20005	20	7.7926

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	30.7356	30.97624	20	12.032
0.5	28.9949	29.2182	20	11.165
1	29.655	29.8765	20	11.0749
1.5	30.705	30.91846	20	10.6731
2	30.1695	30.3923	20	11.14
2.5	29.4319	29.66737	20	11.7735
3	30.3056	30.5325	20	11.345
4	30.3748	30.606	20	11.56
5	29.6308	29.87634	20	12.277
6	28.9574	29.18964	20	11.612
8	30.8224	31.0715	20	12.455
10	30.1995	30.445	20	12.275

### <u>APPENDLX VI</u>

PORT1				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.809	29.0104	20	10.07
0.5	29.8536	30.0547	20	10.055
1	29.134	29.3345	20	10.025
1.5	30.534	30.7389	20	10.245
2	29.4022	29.5976	20	9.77
2.5	28.652	28.8277	20	8.785
3	29.5784	29.754	20	8.78
4	28.6517	28.8177	20	8.3
5	29.1837	29.3539	20	8.51
6	29.2611	29.4285	20	8.37
8	27.4233	27.5779	20	7.73
10	29.2579	29.3746	20	5.835

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### Table VI-9: Results of settling test measurements for compartment 8

PORT2				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	28.9313	29.2664	20	16.755
0.5	30.244	30.5024	20	12.92
1	29.0076	29.2723	20	13.235
1.5	31.0981	31.3731	20	13.75
2	31.4809	31.7588	20	13.895
2.5	30.2008	30.4883	20	14.375
3	30.096	30.348	20	12.6
4	30.242	30.5124	20	13.52
5	29.5211	29.7847	20	13.18
6	28.6177	28.9063	20	14.43
8	29.1992	29.4905	20	14.565
10	28.473	28.7667	20	14.685

PORT3				
Time (min)	Empty crucible (g)	Total solids (g)	Volume of sample (mL)	Density (g/L)
0	31.2399	31.586	20	17.305
0.5	29.4814	29.7969	20	15.775
1	30.135	30.4603	20	16.265
1.5	31.1886	31.4484	20	12.99
2 .	30.6448	30.9463	20	15.075
2.5	29.9204	30.2222	20	15.09
3	30.8054	31.0974	20	14.6
4	30.8498	31.131	20	14.06
5	30.1309	30.426	20	14.755
6	29.5022	29.8147	20	15.625
8	31.2598	31.5679	20	15.405
10	30.6498	30.9562	20	15.32



## VI-5 Model curves for each compartment





Figure VI-2: Compartment 2

APPENDIX VI

Settling tests







Figure VI-4: Compartment 4



Figure VI-5: Compartment 5



Figure VI-6: Compartment 6

#### APPENDIX VI

Settling tests







