TREATMENT OF SOFTDRINK INDUSTRY WASTEWATER USING AN INTEGRATED ANAEROBIC/AEROBIC MEMBRANE BIOREACTOR

by

INNOCENTIA GUGULETHU ERDOGAN

Thesis submitted in fulfilment of the requirements for the degree

Master of Technologae: Chemical Engineering

in the Faculty of

Engineering

at the

CAPE PENINSULA UNIVERSITY OF TECHNOLOGY

Supervisor: Ass. Prof. Marshall Sheldon

CAPE TOWN

October 2014

DECLARATION

I, Innocentia Gugulethu Erdogan, declare that the contents of this thesis represent my own unaided work, and that this thesis has not previously been submitted for academic examination towards any qualification. Furthermore, it represents my own opinions and not necessarily those of the Cape Peninsula University of Technology.

Erdogan

Signed **Date**

ABSTRACT

Most softdrink industries in developing countries are moving towards wastewater reuse or recycling. Water and wastewater reutilization, costs of treatment and disposal guidelines, remain the most critical factors for the development of sustainable water use for softdrink industries. Wastewater reuse or recycle has potential in the softdrink industry, depending on the wastewater characteristics' concentration and volume.

During this study, an integrated laboratory scale anaerobic/aerobic sidestream membrane bioreactor (MBR) system was used for treating softdrink industry wastewater (SDIW). The aim was to evaluate the system's performance, and identify potential opportunities to recycle the water, and therefore reduce freshwater intake and minimise wastewater production. The objectives were to: evaluate: 1) treatment efficiencies for the individual stages; 2) biogas production in the anaerobic stage; and 3) the overall performance of the integrated system under different operating conditions.

The SDIW used in this study was classified as medium to high strength wastewater with a total chemical oxygen demand (COD_t) ranging between 2 242 and 11 717 mg/L and a biological oxygen demand (BOD) of up to 1 150 mg/L. The major pollutants in the SDIW were caustic soda; dissolved sugars, namely fructose (1 071 mg/L) and sucrose (6 900 mg/L); with the pH ranging between 6.1 and 12. The SDIW was characterized by total suspended solids (TSS) of 66 mg/L, as well as fats, oils and greases (FOG) of 40 mg/L. The maximum turbidity and colour was 65.3 NTU and 42 mg Pt/L, respectively. All the physiochemical properties and inorganic parameters were within the within the City of Cape Town's (CCT's) industrial wastewater quality discharge standards by-law (South Africa, 2006). Excluding the total dissolved solids (TDS) and electrical conductivity (EC) with maximum values were 1 050 mg/L and 1 483 µS/cm, respectively.

Anaerobic pre-treatment of this SDIW was studied using a laboratory-scale expanded granular sludge bed (EGSB) reactor maintained at mesophilic temperature of between 35 to 37˚C. An organic loading rate (OLR), upflow velocity (V_{up}) and hydraulic retention time (HRT) of 10.9 kg COD/m^{3}d, 0.85 m/h and ~11.8 h, respectively, resulting in COD treatment efficiencies of up to 93% COD_t. An increase in nitrate (NO_3^-) in the EGSB product stream was an indication of an anaerobic ammonium (NH_4^+) oxidation (ANAMMOX) process.

Anaerobic digestion (AD) of SDIW in the EGSB resulted in biogas production with methane (CH_4), carbon dioxide (CO_2), nitrogen (N_2), and oxygen (O_2), concentrations of up to 70%, 11%, 14.8%, and 4.1%, respectively. At the OLR and V_{up} of 10.9 kg COD/m^{3}d and 0.85 m/h, respectively, the EGSB produced 16.7 L/d of biogas. The EGSB anaerobic pre-treatment

resulted in stable treatment efficiencies for the removal of organic constituents, as well as biogas production without adding an external carbon source.

The MBR post-treatment satisfactorily operated at a feed flowrate of up to 33.7 L/d, OLR of 2.3 and 3.1 kg COD/m^{3}d for the anoxic and aerobic zones, respectively, and an HRT of approximately 0.41 h for both zones. The average COD$_t$ removal achieved was 86%. The dissolved oxygen (DO) concentration of 2.1 mg/L in the anoxic zone combined with an aeration rate and DO concentration of 11.8 L/min and 5.7 mg/L, in the aerobic zone resulted in NH_4^+; NO_3^-; and orthophosphate (PO_4^{3-}), removal rates up to 90%; 55% and 39%, respectively. However, the MBR post-treatment did not decrease the orthophosphate concentration to within the SANS 241:2011 drinking water standards.

The integrated EGSB-MBR treatment for SDIW was able to achieve an overall COD$_t$ removal efficiency of up to 94%. Although the MBR performance was successful the EC, TDS, PO_4^{3-}, and colour concentrations in the ultrafiltration (UF) permeate did not meet the CCT's industrial wastewater standards by-law (2006) as well as the SANS' drinking water standards 241:2011 and required further treatment for reuse.

ACKNOWLEDGEMENTS

I wish to thank:

- The Almighty, for blessing me and giving me strength to complete this study.
- My sincere gratitude to my supervisor, Associate Professor Marshall Sheldon, for exceptional supervision on this project.
- Dr. Debbie de Jager, for her assistance and support, which I appreciate. “Ngiyabonga kakhulu dadewethu”.
- Our industrial partner, for agreeing to collaborate with us during this study. Without them this research would not have been possible.
- CPUT Transport Department for helping me to make successful the trips to obtain wastewater samples from our industrial partner.
- Biocatalysis and Technical Biology Research laboratory (BTB) at the Cape Peninsula University (CPUT), for allowing me to use their HPLC machine for fructose and sucrose analysis.
- Mr Lukhanyo Mekuto and Mr Nkosikho Dlangamandla for their moral support and confidence.
- Chemical Engineering: BTech students, Mr Jason August, and Luis de Sousa, for assisting me with analysis, running of the plant and collecting wastewater from the industrial partner; Ms Mantsha Maphoto and Ms Bianca Walbrugh, for assisting me with analysis and running of the plant.
- Biotechnology: In-service training students for assisting me to collect wastewater, perform analysis, and for the biogas samples collection.
- Chemical engineering staff; Mr Alwyn Bester, Mrs Hannelene Small and Mrs Elizma Alberts, for their contributions to this study.
- CPUT and the Engineering Faculty, for their financial assistance towards this project.
- I cannot begin to express my unfailing gratitude and love to my mother, Mrs Membry Mkhize, who has supported me throughout this process and has constantly encouraged me when the tasks seemed arduous and insurmountable.
- To my husband, Mr Naci Erdogan, who brought me back to life; I am everything I am because you loved me. Your understanding, support and encouragement during this study are appreciated.

DEDICATION

To my loving and caring daughter, Céline

All that I am or hope to be, I owe to you.
No one will ever know the strength of my love for you.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

GLOSSARY

TERM	DEFINITION
Aerobic	Conditions where oxygen acts as an electron acceptor for biochemical reactions (Judd, 2011).
Anaerobic	Conditions where biochemical reactions occur in the absence of oxygen (Judd, 2011).
Anammox	Defined as the biological process in which autotrophic bacteria convert ammonium directly into nitrogen gas under anaerobic conditions (Chen *et al.,* 2013).
Anoxic	Conditions where some species other than oxygen acts as the electron acceptor for biochemical reactions (Judd, 2011).
Activated Sludge	Suspended solids in the wastewater that undergo digestion by high concentrations of aerobic bacteria (Maczulak, 2010).
Air pollution	It is any change in the environment, which is caused by any substance emitted into the atmosphere from any activity, where that change has an adverse effect on human health or well-being or on the composition, resilience and productivity of natural or managed ecosystems, or on materials that are useful to people, or will have such an effect in the future (City of Cape Town, 2010).
Air scouring	Defined as a cleaning practice by passing air through a filtration medium during filter backwash (Logsdon, 2011).
Alkalinity	Refers to the capability of a solution to resist variations in pH (Turkdogan-Aydınol & Yetilmezsoy, 2010).
Beverage industry	Enterprises that manufacture beverages with an alcohol content of less than 0.5% (Chen, 2013).
Biochemical oxygen demand (BOD_5)	The amount of dissolved oxygen, which is required by aerobic biological organisms in a body of water to break down organic material that is present in given water sample at a certain temperature over a specific period (Manyele *et al.,* 2008).
Biodegradation	Defined as the breakdown of compounds to its chemical components by means of living organisms (Madsen, 2011).
Biofilm	A mixed deposit of microbes that attach to a surface that is generally immersed in moving liquor (Maczulak, 2010).

Biomass	Organic matter that can be used as fuel, which is derived from wastewater treatment, plants and animal waste (Maczulak, 2010).
Chemical oxygen demand (COD)	Determines the amount of organic pollutants found in surface water (for examples, lakes and rivers) or wastewater, making COD a useful measure of water quality (Manyele *et al.,* 2008).
Dissolved Oxygen (DO)	Is a relative measure of the quantity of oxygen that is dissolved or carried within a given medium (Wang *et al.,* 2009).
Fermentation Industry	Enterprises that use some or all of the fermentation and distillation processes to manufacture alcoholic beverages, alcohol, or vinegar from raw materials such as wheat, rice, or other grains, starch, or molasses (Chen, 2013).
Filtration	A process of purifying wastewater by passing it through a membrane with small pores (Maczulak, 2010).
Greenhouse gas	Signifies gaseous elements of the atmosphere, both anthropogenic and environment, which attract and re-emits infrared radioactivity, and include nitrous oxide (N_2O) CO_2 and, CH_4 (Peng *et al.,* 2013).
Hydraulic retention time (HRT)	A measure of the average length of time that a soluble compound remains within a system (Judd, 2011).
Industrial wastewater	When any fluid, whether or not it is a restrain substance in solution as a result of any industrial operation such as agricultural activity, chemical process, manufacturing, mining, laboratories, research, service and material, is discharged from a waste crusher (City of Cape Town, 2006).
Membrane	A semi-permeable barrier (Judd, 2011).
Membrane bioreactor (MBR)	Defined as a combination of membrane units, which is responsible for physical separation, and biological reactor systems for biodegradation of the waste compounds (Lin *et al.,* 2012).
Membrane fouling	A process where particles deposit onto a membrane surface area or into pores in a way that it reduces the performance of the membrane (Judd, 2011).
Membrane permeate flux	Defined as the product of water flow, which is divided by the surface area of the membrane (Le-Clech *et al.,* 2006).
Municipal sewer	Any pipe under the power of the Council which, may be used for the transportation of wastewater (City of Cape Town, 2006).

pH	Determines the alkalinity or acidity of a solution (Logsdon, 2011).
Potable water	Is water, which has been attained from the municipality and has some form of treatment to make it appropriate for human consumption (Gleick, 2014).
Sludge	A semi solid mixture, which is left over from wastewater treatment, containing infectious material and toxic chemicals (Maczulak, 2010).
Simultaneous nitrification and denitrification (SND)	Defined as a process from incomplete nitrification of ammonium to nitrite, which afterwards carries on with a direct nitrite reduction to nitrogen gas (Jenicek *et al.,* 2004; Lai *et al.,* 2004).
Softdrink	A non-alcoholic beverage that is habitually consumed, and distinguished by industry as bottled water, carbonates, juices and still drinks (Shachman, 2005).
Softdrink industry	The beverage sector that produces, markets and distributes non-alcoholic beverages, which are usually carbonated, water based, flavoured and sweetened (Allen & Albala, 2007).
Stress period	Defined as the stability of an anaerobic treatment system due to changes in environmental conditions such as pH, temperature and heavy metals (Mudhoo & Kumar, 2013).
Total dissolved solids (TDS)	Refers to the total quantity of all organic and inorganic constituents including cations or anions, minerals, metals and salts that are distributed within a volume of water or wastewater (Hayes, 2004).
Total suspended solid (TSS)	Gives the quantity of the turbidity of water or wastewater (Akpor & Muchie, 2013).
Trans membrane pressure (TMP)	It is the pressure difference from the average feed concentration to permeate transversely to the membrane wall (Ramaswamy *et al.,* 2013).
Turbidity	A measure of the degree to which the water or wastewater loses its transparency due to the presence of suspended solids (Rügner *et al.,* 2013).
Wastewater	Water that contains waste or water that has been in contact with waste matter (City of Cape Town, 2006; SA National Water Act, 1998).
Wastewater recycling	Utilization of untreated or treated wastewater for the similar purpose that produced it (recycling the wastewater of the industry) (Cisneros, 2008).

Wastewater reuse	Utilization of untreated or treated wastewater for a different purpose than for what it was produced (agricultural irrigation) (Cisneros, 2008).
Water treatment plants	Enterprises that employ physical, chemical, or biological methods to treat raw water, which is obtained from its source and supplied as public water (Chen, 2013).

ABBREVIATIONS

Abbreviation	Explanation
AD	Anaerobic digestion
AD-MBR	Anaerobic digestion & membrane bioreactor
AD-MCAB	Anaerobic digestion & membrane-coupled anaerobic bioreactor
AF	Anaerobic filter
AH	Anaerobic hybrid
AH-SBR	Anaerobic hybrid sequence batch reactor
AJLR	Aerobic jet-loop activated sludge reactor
AMBB	Air micro-bubble bioreactor
AMBR	Aerobic membrane bioreactor
AnCSBR	Anaerobic contact sequence batch reactor
AnMBR	Anaerobic membrane bioreactor
AnEMBR	Anaerobic external membrane bioreactor
AnFBR	Anaerobic fixed-bed reactor
AnIFBR	Anaerobic inverse fluidized bed reactors
AnRBC	Anaerobic rotating biological contactor
AnSBBR	Anaerobic sequencing batch biofilm reactor
AnSBR	Anaerobic sequencing batch reactor
AO&MBR	Anoxic/aerobic membrane bioreactor
AS	Activated sludge
ASBR	Aerobic sequence batch reactor
BOD	Biological oxygen demand
CA	CO_2 Absorber
CA-UASB	CO_2 Absorber & upflow anaerobic sludge bed
CF	Cartridge filter
CF-RO	Cartridge filter & reverse osmosis
CF-RO-IE	Cartridge filter, reverse osmosis and ion exchange
CIP	Cleaning in place
cm	Centimetre
COD	Chemical oxygen demand
CPUT	Cape Peninsula University of Technology
CSIR	Council for Scientific and Industrial Research
CSTR	Continuous stirred zone reactor
CT	Capillary tube

d	Day
DO	Dissolved oxygen
DSR	Double-shaft rotary
DSR-AnMBR	Double-shaft rotary anaerobic membrane bioreactor
DWAF	Department of Water Affairs and Forestry
DWEA	Department of Water and Environment
EC	Conductivity
EGSB	Expanded granular sludge bed
EGSB-AF	Expanded granular sludge bed & anaerobic filter
EMBR	External membrane bioreactor
EMS	Environmental management systems
FC	Filter cartridge
FBBR	Fluidised bed bioreactor
FBR	Fluidized bed reactor
FS	Pleated flat sheet
GRABBR	Granular-bed anaerobic baffled reactor
h	Hour
HF	Hollow fibre
HPLC	High-pressure liquid chromatography
HR	Hybrid reactor
HRT	Hydraulic retention time
HT	High-throughput
HT-MBR	High-throughput and MBR
IE	Ion exchange
iMBR	Immersed membrane bioreactor
ISO	International Organization for Standardization
kPa	Kilo Pascal's
KSAMBR	Kubota's submerged anaerobic membrane biological reactor
L	Litre
m	Metre
mg	Milligram per litre
min	Minute
MJ/hL	Mega joule per hectolitre
m^3	Metre cubed
mmol	Millimoles
NTU	Nephelometric Turbidity Unit
MBR	Membrane bioreactor
MBR-UV-NF	Membrane bioreactor and ultraviolet and nanofiltration

MCAB	Membrane-coupled anaerobic bioreactor
MF	Microfiltration
MT	Multi-tubular
NF	Nanofiltration
NF-RO	Nanofiltration and reverse osmosis
OLR	Organic loading rate
PAC	Powdered activated carbon
PBUAR	Packed-bed upflow anaerobic reactor
pH	Potential hydrogen
PFFR	Polyurethane frickling filter reactor
ppm	Parts per million
PTF	Polyurethane trickling filter
PTF-AH	Polyurethane trickling filter and anaerobic hybrid reactor
RBC	Rotating biological contactor
RO	Reverse osmosis
s	Second
SA	South Africa
SANAS	South African National Accreditation System
SANS	South African National Standard
SBR	Sequence batch reactor
SMBR	Submerged membrane bioreactor
SRT	Solid retention time
SS	Suspended solids
SW	Spiral wound
TAnMBR	Thermophilic anaerobic membrane bioreactor
TD	Typical digester
TDS	Total dissolved solids
TKN	Total Kjeldahl nitrogen
TMP	Transmembrane pressure
TN	Total nitrogen
TOC	Total organic carbon
TP	Total phosphorus
TSS	Total suspended solids
UAF	Upflow anaerobic filter
UAFFR	Anaerobic upflow fixed film glass column reactors
UASB	Upflow aerobic sludge bed

UASB-AF	Upflow anaerobic sludge bed and anaerobic filter
UASB-ASBR	Upflow anaerobic sludge bed and aerobic sequence batch reactor
UASB-HR	Upflow anaerobic sludge bed and hybrid reactor
UASB-SBR	Upflow anaerobic sludge bed and sequence batch reactor
UASR	Upflow anaerobic sponge reactor
UASR-AS	Upflow anaerobic sponge reactor and activated sludge
UCT	University of Cape Town
UF	Ultrafiltration
UFFR	Upflow fixed film reactor
VFA	Volatile fatty acid
VOC	Volatile organic compound
V_{up}	Upflow velocity (m/h)
WWTP	Wastewater treatment plants

LIST OF SYMBOLS

Unit	**Definition**
A	Cross sectional area (m^2)
˚C	Degrees Celsius
ΔG	Gibbs free energy $(kmol^{-1})$
H	Height (m)
J	Flux (L/h.m^2)
P_{f-}	Feed pressure (kPa)
P_p	Permeate pressure (kPa)
%	Percentage (dimensionless)
Q	Wastewater widrawal rate (m$^{3/}$h)
V	Fluid volume in reactor (m^3)

Greek Symbols

Unit	**Definition**
ɲ	Efficiency (%)
μS/cm	Micro Siemens per centimetre

Chemical formulas

Element/Compound	Description
Al_2O_3	Aluminium oxide
C	Carbon
CH_3COONH_4	Ammonium acetate
CH_4	Methane
CO_2	Carbon dioxide
C:N:P	Carbon:Nitrogen:Phosphate
H_2	Hydrogen
HCO_3^-	Hydrogen carbonate ion
H_3PO_4	Phosphoric acid
H_2S	Hydrogen sulphide
KH_2PO_4	Potassium dihydrogen phosphate
NaClO	Sodium hypochlorite
NaOH	Sodium hydroxide
Na_2CO_3	Sodium bicarbonate
N_2	Nitrogen
NH_4^+	Ammonium
NO_3^-	Nitrate
O_2	Oxygen
PO_4^{3-}	Orthophosphate

CHAPTER ONE
INTRODUCTION

1.1 Background

In most industrial processes, water is the most extensively used raw material in the production of high value products (Wijekoon *et al.,* 2011; Melamane, 2007). Water quality and its scarcity, has been identified as a future global threat to human health, marine organisms, livestock and the environment (Skouteris *et al.,* 2012; España-Gamboa *et al.,* 2011; Agarwal *et al.,* 2010; Méndez-Acosta *et al.,* 2010; Acharya *et al.,* 2008; Alam *et al.,* 2007; Fritzmann *et al.,* 2007; Fewtrell & Bartram, 2001). South Africa‘s (SA’s) water resources are limited due to climate changes, population growth and a developing economy. Hence, awareness campaigns for reasonable water consumption have increased (Haroon *et al.,* 2013; Hsine *et al.,* 2010; Melamane, 2007; Van Schoor, 2005; DWAF, 2004). An increase in industrial activities, along with the discharge of high strength wastewater from various industries such as beverage industries (distilleries, wineries, breweries, and softdrink manufactures), result in challenges with regard to methods that are used to remediate contaminants in the water in order to limit its environmental impact (Wijekoon *et al.,* 2011; Melamane, 2007). A hindrance for developing countries is how to improve their wastewater treatment capabilities, which at times are based on obsolete technology (Haroon *et al.,* 2013).

Water and wastewater reutilization, costs of treatment and disposal guidelines remain the most critical factors for the development of sustainable water use for the food and beverage industries (Alam *et al.,* 2007; Fillaudeau *et al.,* 2006; Blöcher *et al.,* 2003). Regulatory bodies, therefore, have imposed restrictions on the quality of the water to be discharged into natural resources, In SA wastewater should have a potential hydrogen (pH) value between 5.5 and 7.5; and a total chemical oxygen demand (COD_t) below 75 mg/L [SA, National Water Act, 1998-Act No. 36 of 1998]. Hence, due to these regulatory guidelines, process water usage and wastewater management constitute a practical challenge for beverage industries in SA.

The beverage industry is a subdivision of the food industry (Guimarães *et al.,* 2012), and supplies a range of products from alcoholic (winery, vinasses, molasses and spirits) and brewery to non-alcoholic (fruit juices, vegetable juice, mineral water, sparkling water, flavoured water and softdrinks) beverages (Gonzalez-Garcia *et al.,* 2013; Haroon *et al.,* 2013; Satyawali, 2013; Guimarães *et al.,* 2012). According to Haroon *et al.* (2013) and Hsine *et al.* (2010), the beverage industry globally and in Africa requires large quantities (an average 7 000 m^3 to 25 000 m^3 per month) of freshwater, and do not reuse or recycle wastewater; hence they consume large volumes of freshwater. The beverage industry’s wastewater originates from different individual processes such as bottle washing, product filling, heating or cooling and cleaning-in-place (CIP) systems, beverage manufacturing, sanitising floors including work cells, cleaning of zones and piping networks (Agana *et al.,* 2013; Haroon *et al.,* 2013; Alam *et al.,* 2007; Noronha *et al.,* 2002). There has been an increased demand for more efficient methods to treat beverage industry wastewaters due to progressively stringent international discharge standards (Amuda *et al.,* 2006; Akunna & Clark, 2000). According to Lin *et al.* (2012); Judd (2011); and Yang *et al.* (2006), the membrane bioreactor (MBR) technology is one of the technologies that are touted internationally both in research studies and for industrial applications for its smaller footprint and effectiveness in rapidly treating large quantities of water on a continuous basis.

Despite the increasing number of research studies and full-scale applications of anaerobic digestion (AD) and MBR technologies, the use of MBR technology for the treatment of softdrink industry wastewater (SDIW) is an application area in which there is limited information and research studies. Hence, the aim of this study was to evaluate the performance of an expanded granulated sludge bed (EGSB) reactor, coupled with a sidestream/external MBR system for the treatment of wastewater from a softdrink industry partner, which produces carbonated softdrinks and carbonated flavoured water in the Western Cape, South Africa, with the purpose to recycle treated water and thus reduce freshwater intake by the industrial partner.

1.2 Research problem statement

SDIW presents potential environmental contamination problems due to its composition and characteristics. The wastewater that is generated by some softdrink industries does not meet the wastewater discharge standards, which, if not appropriately treated, can cause severe environmental problems when discharged into the municipal sewers.

1.3 Hypotheses or research questions

- What current wastewater treatment methods are used in the softdrink industry?
- How capable and efficient is an integrated anaerobic/aerobic sidestream/external MBR in treating SDIW?
- To what degree do the individual stages (anaerobic, anoxic and aerobic) achieve treatment when operated under different operating conditions?
- What is the biogas production volume rates and composition under the different operating conditions?
- Can the SDIW used in this study be treated to within the relevant wastewater discharged standards and drinking water standards for water recycling or reuse?

- What are the possibilities of reduction of potable water usage and wastewater production?

1.4 Aims and objectives

The main purpose of this study was to render the SDIW safer for discharge into the municipal sewer so that it does not cause environmental degradation and/or deterioration, and to treat the water for the purpose of recycling.

The objectives of the study were to:

- Evaluate the treatment efficiencies of the anaerobic, anoxic, aerobic and membrane treatment stages under different operating conditions; and evaluate the extent of contaminant removal in the individual stages, as well as the overall process;
- Evaluate biogas production and composition under different hydraulic retention times (HRT); and upflow velocities and organic loading rates (OLR) in the anaerobic stage;
- Evaluate the performance of an integrated anaerobic EGSB pre-treatment combined with an MBR post-treatment system to treat SDIW.

1.5 Significance of the research

The significance of performing the research project was to successfully treat the SDIW using an EGSB-sidestream/external MBR technology design. The study aimed to assist softdrink processors to meet the SA government industrial discharge standards, thus reducing penalties and levies that are associated with discharging such wastewater into municipal discharge points. Treated water could be available for recycle or reuse thereby: 1) reducing their potable water consumption; 2) reducing their costs; 3) providing a solution to industry specific needs; and 4) improving water and wastewater management.

1.6 Delineation of the research

The following were not included in this research project:

- Isolation, comparison, and evaluation of the effects of different microbial consortia on the treatment performance,
- The scrubbing and heat generation of the biogas,
- The study of any other food and beverages industries' (distilleries, breweries, fruit juices, vegetable juices, ciders) wastewater treatments,
- Design and modelling of the MBR system hydrodynamics, and
- Testing different membrane types, materials and sizes.

CHAPTER TWO
LITERATURE REVIEW

2.1 Wastewater quality management and legislations

It is necessary to understand the importance of environmental impacts on the community, and then to contemplate the advantages and disadvantages related to several ranks of environmental control. Most wastewater treatment plants (WWTP) will have on-site specific established requirements for the quality of wastewater that can be discharged into the natural environment and municipal sewers. in South Africa (SA) the water quality requirements stipulated in discharge permits/licenses are set and regulated by the Department of Water and Environmental Affairs (DWEA), and are issued as General Authorisations in terms of Section 39 of the National Water Act, 1998 (Act No. 36 of 1998). The General Authorisation offers additional guidance in respect of wastewater management, requirements and quality.

The beverage industry should comply with several environmental protection acts and regulations (Simate *at al.,* 2011). Through environmental management systems (EMS) such as: 1) Municipal discharged standards and by-laws; and 2) the International Organization for Standardization (ISO) 14001. Therefore, the beverage industries have a duty to be able to proactively manage their impacts on the environment. EMS assists the beverage industries to pay attention to efficient and effective management of both existing and forthcoming environmental impacts.

Wastewater standards are specifications of the biological, chemical and physical quality of the wastewater that is produced by a treatment. These regularly comprise of allowable chemical oxygen demand (COD), suspended solids (SS), nitrogen (N), phosphorus (P) and other elements, which are expressed in concentrations as presented in Table 2.1, which indicates the municipal and industrial discharged standards, as well as the by-laws. Table 2.1 shows that the eThekwini Municipality is much stricter in COD discharge standard, as compared to the City of Cape Town (CCT) and Tshwane, and most of the other metros in SA. The WWTP designer should refer to the local wastewater discharge standards, and by-laws for the specific area in which the WWTP should be constructed and designed (DPW, 2012).

Section A: General	City of Cape Town	eThekwini Municipality	Tshwane
Temperature at point of entry (°C)	0 – 40	<40°C	-
Electrical Conductivity (mS/m)	500	400	300
pH Value at 25 °C	5.5 – 12	6,5 – 10	6 - 10
Chemical Oxygen Demand (mg/L)	5 000	Charge	5 000
Section B: Chemical substances other than heavy metals – maximum concentrations			
Settleable Solids (mg/L)	50	Charge	-
Suspended Solids (mg/L)	1 000	2 000	-
Total dissolved solids (mg/L)	4 000	1 000	-
Chloride as Cℓ (mg/L)	1 500	500 – 1 000	100
Total sulphates as SO_4 (mg/L)	1 500	250	1 800
Total phosphates as P (mg/L)	25	-	10
Total cyanides as CN (mg/L)	20	10 – 20	20
Total sulphides as S (mg/L)	50	1	50
Total phenols as C_6H_5OH (mg/L)	50	5	1
Total sugars and starches as glucose (mg/L)	1 500	1000	1 500
Oils, greases, waxes and fat (mg/L)	400	50	2 000
Sodium as Na (mg/L)	1 000	-	-
Section C: Metals and inorganic content – maximum concentrations Group 1: Total collective concentration of all metals in Group 1 shall not exceed 50 mg/L			
Iron as Fe (mg/L)	50	5 – 50	-
Chromium as Cr (mg/L)	10	-	< 20
Copper as Cu (mg/L)	20	5 – 50	< 20
Zinc as Zn (mg/L)	30	5 – 50	< 20
Section C: Metals and inorganic content – maximum concentrations Group 2: Total collective concentration of all metals and			
Arsenic as (mg/L)	5	5 – 20	20
Boron as B (mg/L)	5	5 – 50	20
Lead as Pb (mg/L)	5	5 – 20	-
Selenium as Se (mg/L)	5	5 – 50	-
Mercury as Hg (mg/L)	5	1	-
Titanium as Ti (mg/L)	5	5 – 20	-
Cadmium as Cd (mg/L)	5	5 – 20	-
Nickel as Ni (mg/L)	5	5 – 50	-

Table 2.1 Industrial wastewater municipal discharged standards and by-laws (City of Cape Town, 2006; Tshwane 2001; eThekwini Municipality, 1999)

2.1.1 The origin and constituents of wastewater

Wastewater is traditionally generated and largely classified as municipal wastewater and industrial wastewater (Henze *et al.,* 2008; Karia & Christian, 2006). Table 2.2 indicates the most significant parameters to determine the strength of both municipal and industrial wastewater. According to Adams (2004), the strength of industrial wastewater varies when compared to domestic or municipal wastewater.

2.1.1.1 Municipal wastewater

Municipal wastewater is also known as domestic wastewater or sewage wastewater, which is defined as “discharged used water from commercial, community institutions and residential sectors that discard it via the sewage system”. Domestic wastewater contains microorganisms, mainly bacteria, and inorganic and organic solids. However, its constituents will depend on the sector from which it is generated. Municipal wastewater comprises about 0.1% solids and 99.9% water (Karia & Christian, 2006).

2.1.1.2 Industrial wastewater

Industrial wastewater, including agro-industrial wastewater, results from human activities, which are associated with processing, manufacturing and raw material handlings, generated from medium to large scale industries. This wastewater arises from cooling, heating, extraction, reaction of by-products, washing and quality control as a result of specification products being rejected (Feroz *et al.,* 2012). The characteristic of wastewater depends on the quality of water that is used by the different types of industries, as well as the community and treatment of such wastewater (Karia & Christian, 2006). Industrial wastewater is difficult to characterize as it varies according to the processes, season and products that are produced (Henze *et al.,* 2008; Karia & Christian, 2006). The main contaminants in industrial wastewater are organic matter, which include microorganisms, biodegradable organic material, metals, nutrients, odour, organic and inorganic materials (Karia & Christian, 2006).

2.1.1.3 The strength of industrial wastewater

Beverage industries produce wastewater with a diverse quantity of COD, which determines the strength of the wastewater (Chmiel *et al.,* 2003). Industrial manufactures usually generate high strength wastewater [COD: <700 mg/L (low); 700 to 3 000 mg/L (medium) and > 3000 mg/L (high)]. From mixing and equalizing tanks, COD ranges from: 2 500 to 6 500 mg/L; electrical conductivity (EC) from 2 300 to 4 700 µS/cm, which varies in constituents depending on the type of industry and the products that are being manufactured (Feroz *et al.,* 2012; Karia & Christian, 2006; Chmiel *et al.,* 2003). The strength of wastewater is generally indicated by the contaminants load, which is determined by the concentration of major biological, chemical and physical contents of the wastewater (Karia & Christian, 2006).

Table 2.2: The significant parameters used to determine the biological, chemical and physical characteristics of municipal and industrial wastewater (Karia & Christian, 2006)

Biological	Chemical	Physical
Animal Plants Protista Pathogenic Organisms Viruses	**Organic Contents** BOD, COD, phenols, oil and grease, surfactants and fats **Inorganic Contents** Alkalinity, chlorides, nitrogen, sulphur, heavy metals, pH, phosphorus and carbohydrates **Gases** Hydrogen sulphide, methane and oxygen	Colour Fixed or volatile mineral solids Odour Temperature Total suspended solids Dissolved solids

2.2 Beverage industry wastewater

The beverage industry wastewater originates from different individual processes such as bottle washing, product filling, heating or cooling and cleaning-in-place (CIP) systems, beverage manufacturing, and sanitising floors, including work cells, cleaning of zones and piping networks (Agana *et al.,* 2013; Haroon *et al.,* 2013; Alam *et al.,* 2007; Noronha *et al.,* 2002).

2.2.1 Typical composition and the strength of beverage industry wastewater

Biodegradable industrial wastewater is wastewater, which contains organic waste that arise from industrial activities, including manufacture and bottling of softdrinks, alcohol production for alcoholic beverages in breweries, wineries and malting facilities (SA, National Water Act, 1998 (Act No. 36 of 1998)). The raw materials (phosphates, sugars and agricultural products), which are used in the manufacture of the beverages enhance the organic load of the wastewater (Haroon *et al.,* 2013; Hsine *et al.,* 2010; Amuda & Amoo, 2007; Amuda *et al.,* 2006). The concentration of beverage industry wastewater parameters such as EC, COD, BOD, total suspended solids (TSS), total dissolved solids (TDS), and total Kjeldahl nitrogen (TKN), are normally high (Agana *et al.,* 2013; Sangave & Pandit, 2008; Shao *et al.,* 2008; Bustamate *et al.,* 2005; Chmiel *et al.,* 2003; Akunna & Clark, 2000). The amount of total nitrogen (TN), total phosphorus (TP) and pH vary, depending on the chemicals that are used (nitric acid, phosphoric acid and caustic soda) (Nyilimbabazi *et al.,* 2011; Dai *et al.,* 2010; El-Kamah *et al.,* 2010; Chmiel *et al.,* 2003; Noronha *et al.,* 2002). Table 2.3 shows some of the typical composition of wastewater from various alcoholic and non-alcoholic beverage production facilities.

Table 2.3: Characteristics of various alcoholic and non-alcoholic beverage industries wastewater

Wastewater Type	pH	COD (mg/L)	BOD (mg/L)	TSS (mg/L)	TDS (mg/L)	TKN (mg/L)	TN (mg/L)	TP (mg/L)	References
Distillery: *Sochu* Sillage	3.60 - 4.76	57 000 - 182 400	-	-	-	-	1 530 - 5 130	-	Kanai *et al.,* 2010.
Distillery	3.8 - 6.4	2 433 - 44 100	5 445 - 22 700	255	-	-	-	-	Agarwal *et al.,* 2010; Zhang *et al.,* 2009.
Distillery	4.01 - 8.2	54 000 - 125 000	18 600	-	11 600 - 38 140	-	-	-	Murthy & Chaudhari, 2009; Venkata Mohan *et al.,* 2008.
Distillery: Molasses	5.7	106 500	31 600	-	74 000	-	1 900	300	Sreethawong & Chavadej, 2008.
Distillery	3 - 4.5	110 000 - 190 000	50 000 - 60 000	13 000 - 15 000	90 000 - 150 000	-	5 000 - 7 000	2 500 - 2 700	Mohana *et al.,* 2009; Acharya *et al.,* 2008.
Distillery: Grain	3.3 - 4.3	16 500 - 22 520	-	-	-	-	120 - 150	15 - 18	Gao *et al.,* 2007.
Distillery	3 - 4	100 000 - 110 000	30 000 - 45 000	3000 - 5000	51 500 - 110 000	-	-	-	Sangave & Pandit, 2008; Nataraj *et al.,* 2006.
Distillery: Whisky	3.8	16 600 - 58 000	8 900 - 30 000	-	6 080 - 17 750	500 - 1 200	-	150 - 600	Akunna & Clark, 2000.
Winery: Vinasse	3.3 - 8.7	11 815 - 111 520	4 767 - 32 500	271	-	21.3 - 252.5	41	1.9 - 472.7	Valderrama *et al.,* 2012; Bustamate *et al.,* 2005.
Winery: White & Red	6 - 6.2	3 112 - 3 997	1 740 - 1 970	-	-	67 - 71	-	7 - 8.5	Vlyssides *et al.,* 2005.
Winery	3.6 - 11.8	738 - 296 119	125 - 130 000	-	-	142.8	-	3.3 - 188.3	Bustamate *et al.,* 2005.
Winery	4 - 11	3 100 - 27 200	2 100 - 8 000	150 - 490	-	21.3 - 64	-	16.6 - 65.7	Ganesh *et al.,* 2010; Eusébio *et al.,* 2004.
Winery	-	800 - 11 000	500- 6 900	200 - 1 300	-	-	-	5 - 77	Petruccioli *et al.,* 2002; Petruccioli *et al.,* 2000.
Winery-Distillery	5 - 5.4	9 000 - 17 400	-	2 400 - 5000	-	-	-	-	Genovesi *et al.,* 2000.
Brewery	6.5	1 250 - 2 250	1 350	480	-	-	16	50	Wen *et al.,* 2010; Wang *et al.,* 2008.
Brewery	3.2 - 4.3	22 500 - 125 000	-	1 550 - 1 750	-	320 - 450	-	144 - 216	Shao *et al.,* 2008; Baloch *et al.,* 2007.
Brewery: Opaque Beer	3.3 - 6.3	8 240 - 20 000	-	2 901 - 3 000	-	-	0.034	16 - 24	Parawira *et al.* 2005.
Brewery	6.3 - 7	500 - 1900	197 - 1 470	60 - 380	-	16.4 - 36.4	3 - 41.1	5.3 - 12.5	Nyilimbabazi *et al.,* 2011; Dai *et al.,* 2010; Ahn & Speece, 2004.
Brewery	3 - 12	2 000 - 6 000	1 200 - 3 600	200 - 3 000	2 020 - 5 940	25 - 116	25 - 80	10 - 50	Alvarado-Lassman *et al.,* 2008; Rao *et al.,* 2007; Driessen & Vereijken, 2003.
Fruit Juice	5.9	3 210	1 650 - 6 900	112 - 1 534	2 304 - 17 918	38 - 252	-	4.6 - 20.8	El-Kamah *et al.,* 2010; Drogui *et al.,* 2008.
Fruit Juice: Sour cherry	-	1 000 - 8 000	-	-	-	3.5 - 55	-	0.104 - 10	Ozbas *et al*., 2006.
Fruit Juice: Apple	-	1 600 - 2 500	-	-	-	73 - 114	-	0.63 - 0.98	Ozbas *et al*., 2006.
Fruit Juice	-	3 650	1 581	-	-	-	12	2.4	Chmiel et *al.,* 2003; Noronha *et al.,* 2002.
Fruit Juice	7 - 9.5	620 - 6 600	729 - 1 745	367 - 2 940	-	-	-	-	Amuda & Amoo, 2007; Blöcher *et*
Softdrink-Bottle-Washing	9 - 11	25 - 125	-	26 - 90	750 - 1 200	-	-	-	Haroon *et al.,* 2013.
Softdrink	5.3 - 9.9	228 - 2 990	130 - 350	-	-	-	-	-	Matošić *et al.,* 2009.
Softdrink-Energy Drink	5.4	33 000	-	250	-	54	-	2.5	Oktay *et al.,* 2007.
Softdrink	3.4 - 3.5	72 900 - 145 000	-	5 700 - 38 000	-	0.05 - 31	20 - 1 180	130 - 250	Guven, 2001.

According to Henze *et al.* (2008), organic matter is normally measured as COD and BOD, which are the major contaminants in wastewater.

2.2.2 Alcoholic beverages industry wastewater composition

Distilleries, wineries and breweries produce alcoholic beverages. The characteristics of alcoholic beverages are summarized in Table 2.3. They have strong similarities in terms of their manufacturing processes, fermentation and separation operations (Fillaudeau *et al.,* 2006). As a result, they are high consumers of freshwater and thus produce high volumes of wastewater worldwide (Simate *et al.,* 2011; Wen *et al.,* 2010; Mohana *et al.,* 2009; Strong & Burgess, 2008; Pant & Adholeya, 2007; Fillaudeau *et al.,* 2006; Coetzee *et al.,* 2004; Mosse *et al.,* 2004). The main raw materials, which are used by distilleries, are barley, wheat, corn, rice, potatoes, sugar beets, sugar cane and molasses (Tomczak-Wandzel *et al.,* 2009; Blonskaja *et al.,* 2003). According to Brito *et al.* (2007), wine is the product that is obtained from total or fractional alcoholic fermentation of fresh grapes, whether crushed or not crushed. Distillery and winery wastewater is also referred to as spent wash, which contains residual unfermented carbohydrates (Sreethawong & Chavadej, 2008; Melamane *et al.,* 2007; Pant & Adholeya, 2007; Wilkie *et al.,* 2000). Beer brewing involves the blending of sugar-based raw materials, wort, barley, maize grits and sorghum malt, followed by alcoholic fermentation with yeast and large volumes of freshwater (Brito *et al.,* 2007; Parawira *et al.* 2005).

According to Mohana *et al.* (2009) and Acharya *et al.* (2008), alcoholic beverage facilities produce wastewater with a high nitrogen and phosphorus content due to the chemicals that are used in the CIP units. Therefore, the disposal of the untreated wastewater from distillery, winery and brewery industries is considered an environmental hazard, since it can result in salination and eutrophication of fresh water resources, should the wastewater be discharged into the environment without treatment (Gonzalez-Garcia *et al.,* 2013; Oliveira & Duarte, 2011; Kanai *et al.,* 2010; Mohana *et al.,* 2009; Brito *et al.,* 2007; Van Schoor, 2005).

2.2.2.1 Distillery wastewater background

Distillery wastewater refers to wastewater, which is generated from alcohol distilleries. On average 8 to 15 litres of wastewater is generated for every litre of alcohol that is produced. Alcohol manufacturing in distilleries consists of four steps, namely; 1) feed-stock preparation; 2) fermentation; 3) distillation; and 4) packaging (Mohana *et al.,* 2009; Satyawali & Balakrishnan, 2008; Nataraj *et al.,* 2006; Saha *et al.,* 2005).

Distillery wastewater, which is generated from the distillation of fermented mash is dark brown in colour, contains acidic high organic matter, and has an unpleasant odour. The amount of pollution that is produced from the distillery wastewater depends on the quality of the molasses, feedstock, location, characteristics of the distillery manufacturing process and the distillation process that is used to produce the ethanol (Mohana *et al.,* 2009; Murthy & Chaudhari, 2009; Sangave & Pandit, 2008; Pant & Adholeya, 2007; Nataraj *et al.,* 2006; Saha *et al.,* 2005; Pandey *et al.,* 2003).The BOD:COD ratio of distillery wastewater is considered to be high at above 0.6 (Fillaudeau *et al.,* 2006). Distillery wastewater is mildly acidic, with a pH of 3 to 5.7; COD of 2 433 to 190 000 mg/L; BOD of 5 445 to 60 000 mg/L; TSS of 255 to 15 000 mg/L; total organic carbon (TOC) of 2 500 to 56 000 mg/L; TDS of 11 600 to 150 000 mg/L; TN of 7000 mg/L; and TP of 2 700 mg/L (Agarwal *et al.,* 2010; Mohana *et al.,* 2009; Acharya *et al.,* 2008; Venkata Mohan *et al.,* 2008; Sangave & Pandit, 2008; Nataraj *et al.,* 2006; Genovesi *et al.,* 2000).

2.2.2.2 Winery wastewater background

The wine industry can be separated into two sub-divisions based on activity: 1) the winemaking industry that creates winery wastewater and by-products; and 2) recycling of winery by-products within wine distilleries (alcohol distillation, which results in the generation of wastewater consist mostly of stillage that is normally non-toxic) (Fillaudeau *et al.,* 2006). Winery wastewater primarily originates from various washing operations during the crushing and pressing of grapes, as well as rinsing of the fermentation zones, barrels, washing of equipment, bottles and from the cooling processes (Strong & Burgess, 2008; Van Schoor, 2005; Petruccioli *et al.,* 2002). The degree of pollution varies, depending on the wine making process and the technology, which is applied for red and/or white wine production (Coetzee *et al.,* 2004; Euse´bio *et al.,* 2004; Petruccioli *et al.,* 2002).

Winery wastewater contains large amounts of biodegradable organics, polyphenols, organic acids, salts and sugars with a BOD:COD ratio of less than 0.67 (Valderrama *et al.,* 2012; Fillaudeau *et al.,* 2006; Mosse *et al.,* 2004; Petruccioli *et al.,* 2002). The quality of winery wastewater varies due to the high organic loads; average COD of 296 119 mg/L; BOD of 125 to 130 000 mg/L; TKN of 142.8 mg/L, and TP of 188 mg/L. Cleaning chemicals, which consist of caustic biocides results in high alkalinity of above 10 (Fillaudeau *et al.,* 2006), and low pH values of 3.3 to 6.2 (Bustamate *et al.,* 2005; Vlyssides *et al.,* 2005). A strong relationship exists between ethanol and the COD of winery wastewater. However, the generation of winery wastewater is seasonal (Fillaudeau *et al.,* 2006).

2.2.2.3 Brewery wastewater background

Brewery wastewater is categorized as a medium to high strength organic wastewater (Huei, 2005; Fillaudeau *et al.,* 2006). The beer manufacturing process, which is called brewing, converts the sugars that are present in starch into ethanol through the fermentation by yeast

(Guimarães *et al.,* 2012). The composition of brewing wastewater also depends on the various different processes that occur within the brewery (raw material handling, wort preparation, fermentation filtration, CIP, and packing) (Brito *et al.,* 2007; Fillaudeau *et al.,* 2006; Driessen & Vereijken, 2003).

Brewery wastewater consists of high strength biodegradable organic components and primarily contains organic solids such as spent grains, soluble starch, sugars, waste yeast, ethanol, and volatile fatty acids (VFA) with a BOD:COD ratio of 0.5 to 0.7 (Guimarães *et al.,* 2012; Simate *et al.,* 2011; Wen *et al.,* 2010; Alvarado-Lassman *et al.,* 2008; Shao *et al.,* 2008; Brito *et al.,* 2007; Fillaudeau *et al.,* 2006). Brewery wastewater normally has a high COD, ranging from 1 250 to 125 000 mg/L; TSS of 3 000 mg/L; TDS of 2 020 to 5 940 mg/L; TOC of 970 mg/L; and TP of 216 mg/L (Simate *et al.,* 2011; Wen *et al.,* 2010; Shao *et al.,* 2008; Wang *et al.,* 2008; Baloch *et al.,* 2007; Rao *et al.,* 2007; Parawira *et al.* 2005). The pH values are irregular ranging from 3 to 12 depending on the types of chemicals used (Rao *et al.,* 2007; Driessen & Vereijken, 2003).

2.2.3 Non-alcoholic beverages wastewater composition

Table 2.3 summarises typical characteristics of non-alcoholic beverage wastewater. The non-alcoholic beverage industry’s wastewater consists of pollutants including cleaning chemicals, product mixes, concentrates, dirt, carbohydrates, sugars, pectin’s, flavourings and colouring additives (Agana *et al.,* 2013; Haroon, 2013; Hsine *et al.,* 2010; Amuda *et al.,* 2006; Chmiel *et al.,* 2003). According to Hsine *et al.* (2010), Matošić *et al.* (2009) and Noronha *et al.* (2002), syrups are the largest pollutants in the non-alcoholic beverage industry wastewater as it generates pollutants, which are rich in sucrose. In most non-alcoholic beverage industry’s, wastewater arises from different operations (juice production, and cleaning of zones and pipes), which are stored in equalizing mixing zones prior to municipal discharge.

2.2.3.1 Fruit juice wastewater background

Wastewater from fruit juice manufacturing industries contains high concentrations of organic materials, and an uneven amount of nutrients with low pH values (Ozbas *et al.,* 2006). The raw materials such as oranges, grapes, guavas, sugars and colorants that are used for production of fruit juices may enhance the organic load of this wastewater (Amuda & Amoo, 2007). Fruit juice wastewater contains high biodegradable organic matter with a BOD:COD ratio of 0.6 (El-Kamah *et al.,* 2010).

Fruit juice wastewater also contains sugars (large non-polar organic molecules) and fine colloidal particles, therefore, possessing lower conductivity when compared to other types of beverage wastewater (Drogui *et al.,* 2008; Ozbas *et al.,* 2006). Fruit juice wastewater is characterized by high COD values of 8 000 mg/L; BOD of 6 900 mg/L; TSS of 2 940 mg/L, indicating a high organic content and irregular pH values of 5.4 to 9.5 (El-Kamah *et al.,* 2010; Amuda & Amoo, 2007; Amuda *et al.,* 2006; Blöcher *et al.,* 2002).

2.2.3.2 Softdrink industry wastewater (SDIW) background

The wastewater from bottle washing constitutes almost 50% of the total wastewater, which is generated by the softdrink industry (Hsine *et al.,* 2010; Ramirez *et al.,* 2004; Miyaki *et al.,* 2000). According to Hsine *et al.* (2010); Guven (2001) and Ghosh & Henry (1981), wastewater from the softdrink industry is highly biodegradable and constitutes various blends of chemicals, including fructose, glutose, sucrose, lactose, artificial sweetener, fruit juice concentrates, flavouring agents, dissolved carbon dioxide/carbonic acid, bicarbonates, colouring agents (caramel and synthetic dye-stuff), preservatives (phosphoric acid and tartaric acid) and mineral salts that are used during production.

The BOD:COD ratio of wastewater from washing equipment during the manufacturing of softdrinks is generally 0.05:1 (Hsine *et al.,* 2010). Waste sugar is the main contributor to high COD values of 25 to 145 000 mg/L, BOD of 130 to 350 mg/L, TSS of 26 to 38 000 mg/L, TDS of 750 to 1 200 mg/L, TN of 20 to 1 180 mg/L, and TP of 130 to 250 mg/L. The pH could be acidic or alkaline, ranging between 3.35 and 11 (Haroon *et al.,* 2013; Hsine *et al.,* 2010; Matošić *et al.,* 2009; Oktay *et al.,* 2007; Guven, 2001). Wastewater from the production of softdrinks is classified as high strength waste, since the BOD and SS concentrations are high and, therefore; direct discharge into municipal sewers without treatment causes environmental pollution (Matošić *et al.,* 2009; Manyele, *et al.,* 2008; Cheremisinoff, 2001). The characteristics of SDIW have been reported by some researchers and are presented in Table 2.3 (Haroon *et al.,* 2013; Matošić *et al.,* 2009; Oktay *et al.,* 2007; Guven, 2001). However, based on the available documented literature, there is little evidence and/or research reports focusing on the treatment of SDIW.

2.3 Wastewater treatment methods

Wastewater treatment methods are essential for community development to remove pollutants and to recover water for reuse (Libralato, 2013). There are four main types of wastewater treatment systems, namely: 1) preliminary, which is selected for the removal of large materials and coarse solids regularly found in the wastewater; 2) primary, which is designed for the removal of inorganic and organic floating material and the physical processes of sedimentation; 3) secondary, where biological processes are employed to remove the remaining organic material and colloids (activated sludge, tickling filter and secondary settling zone lagoons, ponds, expanded granular sludge bed (EGSB), rotating biological contactor (RBC), upflow anaerobic filter (UAF), and upflow anaerobic sludge bed (UASB); and 4) tertiary or advanced to further treat secondary wastewater to eliminate or decrease remaining contamination by utilizing different methods such as biological and chemical processes, membrane filtration (MF), ion exchange (IE) and reverse osmosis (RO) (Feroz *et al.,* 2012; Bitton, 2011; Karia & Christian, 2006; Sonune & Ghate, 2004).

2.3.1 Biological wastewater treatment methods

Biological wastewater treatment can be separated into off-site treatment and on-site systems (Okpor, 2011). Biological treatment is an essential and significant constituent of several wastewater treatment plants that treat wastewater, either from industry or the municipality, containing soluble organic materials (Mittal, 2011). The biological treatment methods for both municipal and industrial wastewater are mostly carried out by activated sludge (AS) (Nielsen *et al.,* 2009). Wastewater, which is generated by agricultural and food-processing industries, is primarily comprised of readily biodegradable organic materials; therefore, anaerobic digestion (AD) is a suitable choice for wastewater treatment (Alvarado-Lassman *et al.,* 2008)

2.3.2 Anaerobic digestion treatment for wastewater and application

AD of wastewater has been applied in full-scale and pilot-scale operation and its use is increasing (Fillaudeau *et al.,* 2006). AD or treatment is referred to as a fermentation process during, which organic material is degraded and biogas is produced through a mixture of microbial community in the absence of oxygen. This process is applied for the treatment of industrial and domestic wastewater (Cioabla *et al.,* 2012; Romaro *et al.,* 2011; Wang & Ivanov, 2010; Henze *et al.,* 2008). AD is characterized by biological conversion of organic compounds (COD) into biogas, primarily methane (CH_4) and carbon dioxide (CO_2) with odour traces of hydrogen sulphide (H_2S) (Driessen & Vereijken, 2003; Guven 2001). Equations 2-1 represent the overall equation for AD (Driessen & Vereijken, 2003).

$$COD \rightarrow CH_4 + CO_2 + anaerobic\ biomass \qquad \text{Equation: 2-1}$$

2.3.3 Advantages and disadvantages of anaerobic digestion

There are two major groups for AD, which are classified as conventional and high-rate systems. The conventional systems are septic zones, anaerobic ponds and sludge reactors (Von Sperling *et al.,* 2005). Wastewater that contains high concentrations of soluble constituents are presently treated successfully with a variety of high-rate anaerobic reactor designs such as anaerobic filter (AF), anaerobic hybrid (AH), fluidised (FB), including EGSB and UASB reactors. These high-rate reactors are able to separate solid retention time (SRT) from hydraulic retention time (HRT), thus allowing the moderately slow growing anaerobic micro-organisms to remain within the reactor in parallel to the flow of wastewater. As a result, higher volumetric loads and high removal efficiencies are achieved (Akunna & Clark, 2000).

2.3.4 Highrate anaerobic reactor

According to Lim and Kim (2014), full-scale high-rate anaerobic reactors such as the UASB have been operating worldwide, which were developed by Lettinga (1995). Applications of these reactors include those of the treatment of brewery industry wastewater (Brito *et al.,* 2007; Rao *et al.,* 2007; Zupančič *et al.,* 2007; Connaughton *et al.,* 2006; Parawira *et al.,* 2005; Ahn & Speece, 2004); distillery wastewater (Blonskaja *et al.,* 2003; Wolmarans & De Villiers 2003); paper mill wastewater (Sheldon *et al.,* 2012); winery wastewater (Gao *et al.,* 2007; Laubscher *et al.,* 2001); fruit juice industry wastewater (El-Kamah *et al.,* 2010; Ozbas *et al.,* 2006) and SDIW (Kalyuzhnyi *et al.,* 1997). The EGSB reactor is operated by means of granular anaerobic sludge, which is similar to the UASB reactor. The EGSB and UASB reactors separate the biogas, biomass and wastewater in a one-step process by using a three-phase separator that is situated at the top of the reactor (Sheldon *et al.,* 2012; Brito *et al.* 2007). EGSB has been mainly developed to enhance substrate-biomass interaction within the treatment system by expanding the sludge bed and increasing hydraulic mixing, which normally builds up to 16 m (Lim & Kim, 2014; Zhang *et al.,* 2008). The EGSB reactor can be operated at a higher upflow velocity (V_{up}) of up to 6 m/h with a higher recycle ratio. Therefore, the EGSB reactor is capable of treating high strength organic wastewaters up to an OLR of 30 kg COD/m^3d. The EGSB can also treats low strength wastewaters where COD concentrations are less than 1 000 mg/L (Lim & Kim, 2014; Lettinga, 1995). A modified schematic diagram from Guo *et al.* (2008) and Zhang *et al.* (2008) for the EGSB reactor is presented in Figure 2.1.

There have been minimal studies reported on the treatment of beverage industry wastewater using the EGSB, especially SDIW. However, Connaughton *et al.* (2006) presented that there was slight variation between psychrophilic and mesophilic conditions for brewery wastewater. The wastewater was from a brewery industry, and the OLR was 4.5 kg COD/m^3d, where both reactor COD removal efficiencies ranged from 85 to 93%, and for methanogenic activities, the biogas production rates were comparable.

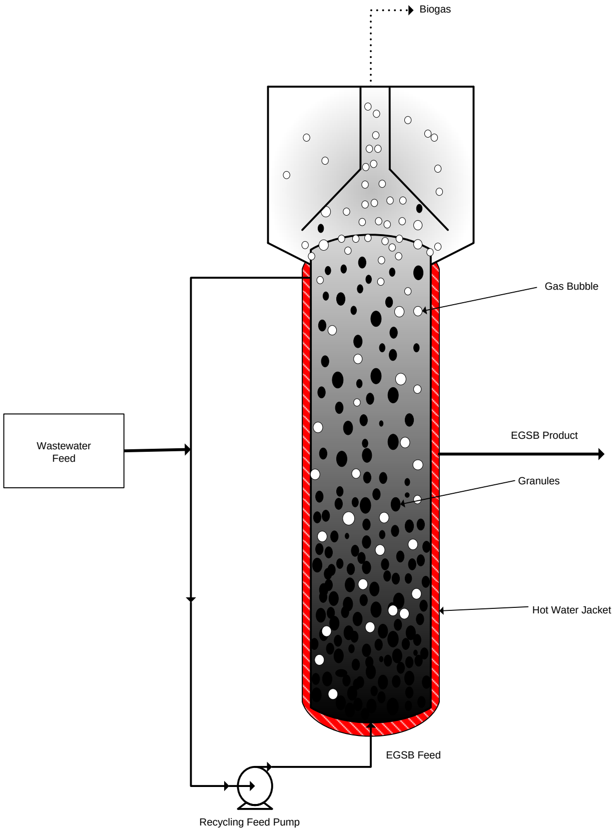


Figure 2.1: Schematic diagram for the EGSB [drawn using Microsoft Visio 2010] (Modified from Guo *et al.,* 2008; Zhang *et al.,* 2008)

2.3.5 The relationship between the EGSB and UASB

The Dutch sugar industry developed an UASB reactor, which was first utilized in the late 1970's (Driessen & Vereijken, 2003). The EGSB reactor is a modified version of an UASB (Zhang *et al.,* 2008; Mutombo, 2004; Driessen & Vereijken, 2003). The EGSB has been developed mainly to improve substrate-biomass contact within the treatment system by escalating hydraulic mixing and expanding the sludge bed to allow for high organic loading rates and COD removal efficiencies with a minimum of solids lost in the permeate. As a result, the EGSB reactor has a more improved reactor performance and stability than the UASB (Zhang *et al.,* 2008; Liao *et al.,* 2006; Dinsdale *et al.,* 2000). Thus, the EGSB reactor has a higher mixing capacity due to the higher V_{up} smaller footprint, increased biomass activity and improved mass transfer than the UASB reactor (Mutombo, 2004; Dinsdale *et al.,* 2000). According to Zhang *et al.* (2008), Liao *et al.* (2006), Connaughton *et al.* (2006), and Driessen & Vereijken (2003), several studies have been conducted on the EGSB to evaluate the impact of flow patterns, kinetics, by-product inhibition, as well as start-ups and operation characteristics on the overall efficiency of the bioreactor. In addition, EGSB reactors have been successfully applied to treat various types of wastewater, including municipal wastewater, molasses wastewater, brewery wastewater, starch wastewater and domestic wastewater (Cervantes, 2009, Connaughton *et al.*, 2006; Van Haandel *et al.,* 2006).

However, literature shows that EGSB has not been utilized or evaluated for the treatment of softdrink industry wastewater.

2.3.6 Anaerobic degradation of organic matter

There are a number of microbiological phases that outline the pathway for organic matter degradation in AD. The environment and the nutritional necessities of a huge quantity of microorganisms have to be met in order for the biogas production process to be successful (Cioabla *et al.,* 2012; Schnurer & Jarvis, 2010; Weiland, 2010; Poh & Chong, 2009, Henze *et al.,* 2008; Fillaudeau *et al.,* 2006). The different phases of the microorganisms and decomposition that are active are (i) hydrolysis; (ii) acidogenesis; (iii) acetogenesis; and (iv) methagenogenesis.

(i) **Hydrolysis:** Through hydrolysis, complex polymers such as carbohydrates, fats and proteins are being degraded into amino acids, long chain fatty acids and sugars. This degradation process occurs primarily through fermentative bacteria which excrete enzymes that change complex undissolved matter into smaller units and dissolved compounds that are capable of passing through the cell walls and membranes of the fermentation bacteria. The hydrolysis process is affected by several factors such as substrate composition, particle size, temperature, and residence time (Matovic, 2013; Manyi-Loh *et al.,* 2013; Song *et al.,* 2005).

(ii) **Acidogenesis:** Acidogenesis is the second phase of AD, where the fermentation of soluble substrate into more oxidized intermediates, mainly volatile fatty acids (VFAs), which take place by hydrogen generating acidogens. Dissolved compounds present in the cell fermenting bacteria are transformed into a number of simple compounds, which are then excreted. The compounds produced during this phase includes new cell material, hydrogen (H_2), CO_2, H_2S, ammonium (NH_4^+), alcohol and lactic acid, as well as trace amount of by-products, Equations: 2-2 to 2-4 demonstrates the conversion of glucose to acetate, ethanol and propionate, respectively (Matovic, 2013; Manyi-Loh *et al.,* 2013; Kangle *et al.,* 2012).

$$C_6H_{12}O_6 + 2H_2O \rightarrow 2CH_3COOH + 2CO_2 + 4H_2 \qquad \text{Equation: 2-2}$$

$$C_6H_{12}O_6 \rightarrow 2CH_3CH_2OH + 2CO_2 \qquad \text{Equation: 2-3}$$

$$C_6H_{12}O_6 + 2H_2 \rightarrow 2CH_3CH_2COOH + 2H_2O \qquad \text{Equation: 2-4}$$

(iii) **Acetogenesis:** In general, acetogenesis is the generation of acetate, which is derived from acetic acid and carbon with energy supplied by the acetogens. Acetogens break down the biomass to a point where the methanogens can utilize much of the remaining material to create methane as a biofuel and intermediary acid generation, where the digested product is transformed into new cells, acetic acid, H_2 and CO_2. Equation: 2-5 represents an acetogenesis reaction (Kangle *et al.,* 2012).

$$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2 \qquad \text{Equation: 2-5}$$

(iv) **Methanogenesis:** Methanogenesis represents the final phase of AD during, which methanogens produce methane (CH_4) from the ultimate products of acetogenesis, as well as from the intermediary products from hydrolysis and acidogenesis. There are two common pathways relating to the use of acetic acid and carbon dioxide; the two main products from the first three phases of AD, and to generate the CH_4 in methanogenesis:

The three main reactions responsible for generating biogas during AD are: Represented by Equations 2-6 to 2-8 (Matovic, 2013; Manyi-Loh *et al.,* 2013; Kangle *et al.,* 2012).

1) **Acetotrophic methanogenesis:**

$$4CH_3COOH \rightarrow 4CO_2 + 4CH_4 \qquad \text{Equation: 2-6}$$

2) **Hydrogenotrophic methanogenesis (CO_2 reduction):**

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O \qquad \text{Equation: 2-7}$$

3) **Methylotrophic methanogenesis:**

$$4CH_3OH + 6H_2 \rightarrow 3CH_4 + 2H \rightarrow O \qquad \text{Equation: 2-8}$$

While CO_2 can be converted into CH_4 and H_2O through the reaction, the main mechanism to create CH_4 in methanogenesis is the path involving acetic acid. This path creates CH_4 and CO_2, the two main products of AD, where H_2, carbonate, acetate and ethanol are transformed into new cell material (Matovic, 2013; Manyi-Loh *et al.,* 2013; Song *et al.,* 2005). Figure 2.2 indicates the metabolic pathway involved in AD. Information on biogas generation will be discussed in subsequent sections Section 2.5.

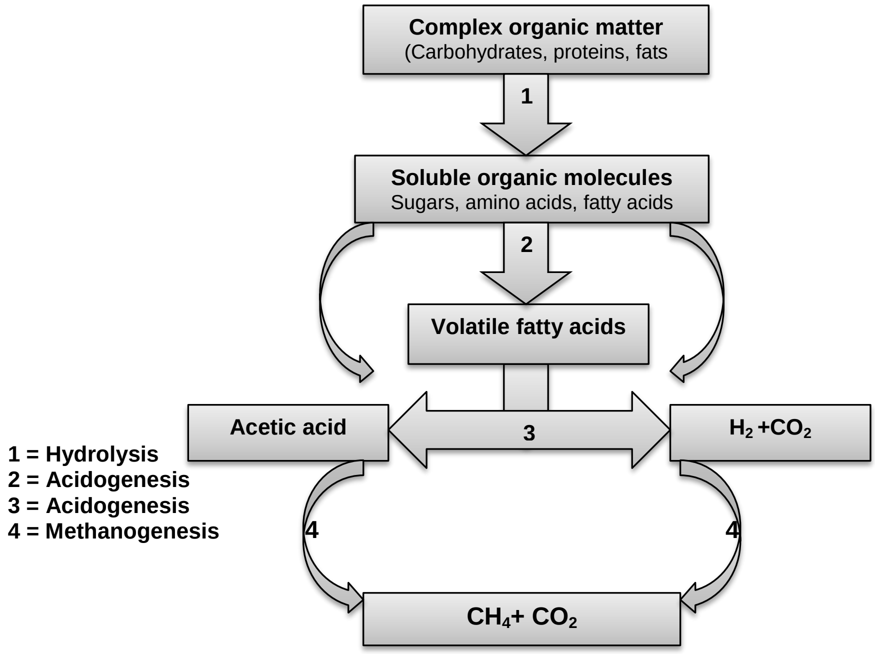


Figure 2.2: Microbial groups and metabolic pathway involved in anaerobic digestion (modified from Matovic, 2013)

2.3.7 Factors that influence the operation of anaerobic digestion (AD)

The complexity of bioconversion processes may result in the performance of AD to be affected by various aspects (Cioabla *et al.,* 2012; Surroop *et al.,* 2012; Ward *et al.,* 2008). These aspects can be divided in three major classes: 1) feedstock characteristics, 2) reactor design and 3) operational conditions. The operational conditions include temperature, pH and alkalinity, organic loading rate (OLR), inhibitory substances and inoculums to substance ratio (Matovic, 2013; Trzcinski & Stuckey, 2009; Monson *et al.,* 2007). The biological processes are fundamental for successful operation of AD for optimal environmental conditions. Operating parameters as indicated such as hydraulic retention time (HRT), and OLR have been used to operate AD for beverage industries, as shown in Table 2.4.

Table 2.4: Essential parameters for anaerobic digestion (Deublein & Steinhauser 2011)

Parameter	Hydrolysis/Acidogenesis	Methanogenesis
pH	5.2 - 6.3	6.7 - 7.5
Temperature	Psychophillic: 25 - 35°C	Mesophilic: 30-40˚C Thermophilic: 50 - 60˚C
C:N ratio	10 – 45	20 - 30
C:N:P:S ratio	500:15:5:3	600:15:5:3
Trace elements	No particular requirements	crucial: Ni, Co, Mo, Se

2.3.7.1 Temperature

Most of the anaerobic reactors were operated either at around 35 to 37˚C in the mesophilic range (Méndez-Acosta *et al.,* 2010; Acharya *et al.,* 2008; Akunna & Clark, 2000), or at around 50 to 60˚C in the thermophilic range (Banerjee & Biswas, 2004). The psychrophilic temperatures of around 21 and 25˚C were also tested (Bilad *et al.,* 2010; Nataraj e*t al.,* 2006). The temperature of the mixed-liquor affected the COD removal efficiencies; and higher temperatures led to better COD removal efficiencies. Increasing the temperature level to 37˚C, decreased the time required for AD (Cioabla *et al.,* 2012; Cha *et al.,* 2008; Choorit & Wisarnwan, 2007). In general, mesophilic AD is favoured over thermophilic AD, since the latter offers a smaller amount of CH_4 production, and is susceptible to environmental changes (Matovic, 2013; Manyi-Loh *et al.,* 2013; Trzcinski & Stuckey, 2009; Monson *et al.,* 2007).

2.3.7.2 Alkalinity and pH effect

The buffering ability of AD is determined by the amount of bicarbonate ions (HCO_3^-) supplied by the buffer that is used to maintain the system's pH. The concentration of HCO_3^- in solution is associated with the percentage of CO_2 in the gas stage. Alkalinity generally supplies sufficient buffering ability to survive reasonable shock loads of VFA. However, the optimum pH range in an anaerobic reactor should be 6.8 to 7.2, although the process can tolerate a pH range of 6.5 to 8.0 (Manyi-Loh *et al.,* 2013; Cioabla *et al.,* 2012; Labatut & Gooch, 2012: Deublein & Steinhauser 2011; Romano & Zhang, 2011; Trzcinski & Stuckey, 2009; Monson *et al.,* 2007). Liu *et al.* (2008) report that the pH range is widespread in the plant-scale operations and the optimal pH fluctuates with substrate quality and the biodegradability of the contaminants in the system.

2.3.7.3 Organic loading rate (OLR) and hydraulic retention time effect (HRT)

HRT is defined as the average residence time of waste suspension within the reactor. As a result, the high biogas production is obtained at higher rates, which is calculated according to Equation: 2-9:

$$HRT = V/Q \qquad \text{Equation: 2-9}$$

Where, HRT = hydraulic retention time (h)
V = fluid volume in the reactor (m^3)
Q = wastewater withdrawal rate (m^3/h)

OLR is one of the significant parameters in the operation and design of the biological system of wastewater treatments (Matovic, 2013; Monson *et al.,* 2007). OLR is the quantity of organic matter (COD) that is loaded in one volumetric reactor per unit time, and it is calculated by using Equations: 2-10 or 2-11:

$$OLR = (COD_{feed} \times Q)/V \qquad \text{Equation: 2-10}$$

The ratio between HRT and the reactor volume is equivalent to the flowrate, hence Equation: 2-9 becomes Equation: 2-10:

$$OLR = \ COD_{feed}/HRT \qquad \text{Equation: 2-11}$$

Where, OLR = organic loading rate (kg COD/m^{3}d)

$$COD_{feed} = \ OLR \times HRT$$

Organic matter removal efficiency measures the performance of the reactor and is determined by Equation: 2-12:

$$\eta = \left[\frac{COD_{in} - COD_{out}}{COD_{in}}\right] \times 100\% \qquad \text{Equation: 2-12}$$

The OLR value is generally coupled with HRT, as a result, the shorter the HRT, the higher the OLR. However, the value of the OLR varies with the corresponding HRT as the organic matter concentrations changes. There is a potential risk that rapidly increasing the OLR would result in hydrolysis and acidogenic bacteria producing intermediate products quickly (Matovic, 2013; Monson *et al.,* 2007). As the replication time of the methanogenic bacteria lowers, the bacteria will not be able to consume fatty acids at the same rate, and which results in a drop in pH and causes the system to be unsuccessful (Grosser *et al.,* 2013; Manyi-Loh *et al.,* 2013). An additional process parameter is retention time, which consists of HRT and solid retention time (SRT), which refers to the time that any part of portion of a solid, feed, water feed and microbial biomass remains in a digestion reactor.

2.3.7.4 Effect of nutrients

The microorganisms in the AD process requires micronutrients and trace elements such as calcium, cobalt, copper, iron, magnesium, manganese, nitrogen, nickel, phosphorous, sulphur, potassium and zinc for optimal growth. The existence of heavy minerals in the wastewater is a crucial parameter, as this affects the reactor’s stability and granulation process.

However, it is essential that these elements are present in extremely low range concentrations (Deublein & Steinhauser 2011; Monson *et al.,* 2007; Rajeshwari *et al.,* 2000). The absence of these nutrients has an effect on AD and in such cases the wastewater should be supplemented prior to treatment. According to Russel (2006) and Rajeshwari *et al.* (2000), the vital optimal C:N:P ratio for enriched yield of CH_4 should be 100:5:1 or 100:2.5:0.5 for ideal bacterial growth. The minimum concentration of micro and macronutrients can be calculated from the wastewater COD biodegradable concentration (Rajeshwari *et al.,* 2000). Additionally, carbon and nitrogen are the two nutrients that require attention for AD loading rates (Deublein & Steinhauser 2011; Monson *et al.,* 2007).

2.3.7.5 Upflow velocity (V_{up}) effect

The other important parameter during AD is the upflow velocity, V_{up}. As V_{up} increases, the turbulence in the reactor also increases, and hence the contact between the wastewater and the granular sludge will improve (Cervantes, 2009). The relationship between the height of the reactor (H) and V_{up} is described by Equations: 2-13; 2-14 and 2-15:

$$V_{up} = Q/A \qquad \text{Equation: 2-13}$$

Or

$$V_{up} = V/(HRT \times A) \qquad \text{Equation: 2-14}$$

Or

$$V_{up} = H/HRT \qquad \text{Equation: 2-15}$$

Where,

A = cross sectional area (m^2)
H= height (m)

In general, V_{up} ranges from 0.5 to 2 m/h for UASB, and up to 10 m/h for the EGSB (Cervantes, 2009).

2.4 Anaerobic digestion (AD) for the beverage industry's wastewater

Treatment of wastewater from the beverage industry typically comprises physical pre-treatment for the removal of suspended matter, followed by biological treatment, either anaerobic or aerobic (Hsine *et al.,* 2010; Matošić *et al.,* 2009; Guven, 2001). AD has been utilized worldwide in the treatment of wastewater from the food and beverage industry, including the alcohol industry, brewery industry, coffee industry, dairy industry, fish processors, fruit and vegetable industries, softdrink industry and winery industry (Poh & Chong , 2009; de Lemos Chernicharo, 2007). The brewery industry has been at the origin of utilizing anaerobic technology (Fillaudeau *et al.,* 2006). Wastewater that is generated by beverage industries is mainly composed of readily biodegradable organic matter, thus AD is a recognized technology for the successful treatment and removal of organic matter removal (Torres *et al.,* 2011; Alvarado-Lassman *et al.,* 2008; Connaughton *et al.,* 2006; Fillaudeau *et al.,* 2006).

Tables 2.5 and Table 2.6 indicate several potential treatment methods that have been used for wastewater from alcoholic and non-alcoholic beverage industries, respectively. According to Kanai *et al.* (2010); Wijekoon *et al.* (2011); Satyawali & Balakrishnan (2009); Zhang *et al.* (2006); Coetzee *et al.* (2004); Brito *et al.* (2007); Ganesh & Rajinikanth (2010); Melamane *et al.* (2007); Wolmarans & De Villiers (2002); Yu *et al.* (2006); Parawira *et al.* (2005); Torres, *et al.* (2011); Alvarado-Lassman *et al.* (2008); Zupančič *et al.* (2007) and Shao *et al.* (2008), in the beverage industry, anaerobic wastewater treatment processes are generally adopted as a pre-treatment method.

2.4.1 Application of anaerobic digestion (AD) for alcoholic beverage industry

As shown in Table 2.5, AD has been presented as being appropriate for treating distillery wastewater from low strength 700 mg/L (Zhang *et al.,* 2006) to high strength 190 000 mg/L (Acharya *et al.,* 2008); winery wastewater from 800 mg/L (Artiga *et al.,* 2005) to 27 200 mg/L (Eusébio *et al.,* 2004) and brewery wastewater of 500 mg/L (Dai *et al.,* 2010) to 62 300 mg/L (Zupančič *et al.,* 2007); since the C:N:P ratio is unstable for aerobic treatments, which requires nitrogen and phosphorus addition. AD technology is acceptable for the bioremediation of distillery, winery and brewery wastewater due to the low cost of operation, and since the technology is considered to be environmentally friendly (Valderrama *et al.,* 2012; Torres *et al.,* 2011; Wijekoon *et al.,* 2011; Kanai *et al.,* 2010; Ling *et al.,* 2009; Melamane, 2007; Moletta, 2005; Driessen & Vereijken, 2003; Wolmarans & de Villiers, 2002; Cornelissen *et al.,* 2001).

2.4.1.1 Anaerobic digestion (AD) of distillery wastewater

A number of AD technologies have been applied and investigated at full scale, pilot and laboratory-scale to reduce the amount of pollution, which is present in distillery wastewater, as shown in Table 2-5. Distillery wastewater is usually treated via either anaerobic or aerobic biological methods, but in general, the integration of both methods has been applied (Mohana *et al.,* 2009). Distillery wastewater has a typical BOD:COD ratio of 0.5 to 0.6, indicating the water's suitability for biological treatment (Mohana *et al.,* 2009).

Table 2.5: Treatment methods for alcoholic beverages industry wastewater

Type of Wastewater	Application	Treatment Method & Type of Reactor	Configuration	Characteristics of Membrane	T^{1} (˚C)	HRT^{2} (d)	OLR^{3} (kg COD/m^{3}d)	COD$_{Feed}$ (mg/L)	TE^{4} (% COD)	References
Distillery: Synthetic Molasses	Laboratory	TAnMBR^	Sidestream	MF Tubular Ceramic (0.1 µm)	55	0.4 - 2	5 - 12	7 500 - 10 200	78 - 81	Wijekoon *et al.,* 2011.
Distillery: Synthetic Molasses	Laboratory	HT-MBR^	Submerged	6 x Flat Sheet Polymer (0.1; 0.8;2 µm)	21	0.8	-	2 250	80 - 85	Bilad *et al.,* 2011
Distillery Tequila Vinasse	Laboratory	CSTR-TD^	-	-	35 - 36	10	1	20 000 - 50 000	90 - 95	Méndez- Acosta, 2010.
Distillery: *Sochu*	Full	KSAMBR^	Submerged	Kubota	-	-	-	57 000 - 182 400	75 - 92	Kanai *et al.,* 2010.
Distillery	Laboratory	AMBR-PAC^	Submerged	Nylon Mesh Filter (30 µm)	21 - 26	7	3 - 5.71	-	53	Satyawali & Balakrishnan, 2008; Satyawali & Balakrishnan, 2009.
Distillery	Pilot	NF- RO^	-	-	28 - 30	-	-	4 000	91.3 - 96.8	Murthy & Chaudhari, 2009.
Distillery-Synthetic	Laboratory	AnSBBR	-	-	20 - 30	1	8.8	54 000	69.7	Mohan *et al.,* 2009.
Distillery	Laboratory	UAFFR^	-	-	37	8	23.25	110 000 - 190 000	64	Acharya *et al.,* 2008.
Distillery: Synthetic Grain Sorghum	Laboratory	AMBR^	Submerged	Flat Stainless Steel (0.2 µm)	30 - 45	0.4 - 1.3	0.6 - 2.8	700 - 1 500	94.7	Zhang *et al.,* 2006.
Distillery	Pilot	NF - RO^	-	-	27 - 29	-	-	2 900 - 100 000	99.9	Nataraj *et al.,* 2006.
Distillery	Laboratory	UASB-AF^	-	-	35	10 - 19	2.5 - 5.1	13 600	54 - 93	Blonskaja et *al.,* 2003.
Distillery	Laboratory	MCAB^	Sidestream	Tubular Hydrophobic Polypropylene (0.2 mm) ; Zirconia Skinned 0.14 mm. (0.2 µm)	55	13	3 - 3.5	38 400	>90	Kang *et al.,* 2002
Distillery: Whisky	Laboratory	GRABBR^	-	-	37	2 - 10	4.75	9 500	80 - 92.2	Akunna & Clark, 2000.
Winery	Pilot	AMBR^	-	Kubota	-	-	-	2 300 - 13 500	97	Valderrama *et al.,* 2012.
Winery	Laboratory	AMBB^	-	-	20	-	4 - 8	8 900	98.6	Oliveira & Duarte, 2011.
Winery: Synthetic	Laboratory	AnFBR^	-	-	35	-	22 - 42	18 000 - 21 000	81	Ganesh & Rajinikanth, 2010.
Winery: Distillery	Laboratory	AnMBR^	Submerged	UF Ceramic (0.2 µm)	-	0.5	-	4 300 - 6 100	99.5	Wijekoon *et al.,* 2011.
Distillery: Winery Grain	Laboratory	UASB^	-	-	37	0.04 - 3.4	5 - 48.3	16 500 - 22 520	80 - 97.3	Gao *et al.,* 2007.
Winery	Pilot	AnSBR	-	-	15 - 25	3.7 - 7.4	-	5 000	90	Brito *et al.,* 2007.

^: See Appendix A; 1.T: Temperature; 2. HRT: Hydraulic retention time; 3. OLR: Organic Loading Rate; 4. TE: Treatment Efficiency.

Table 2.5 (*cont.)*: Treatment methods for alcoholic beverages industry wastewater

Type of Wastewater	Application	Treatment Method & Type of Reactor	Configuration	Characteristics of Membrane	T^1 (°C)	HRT^2 (d)	OLR^3 (kg COD/m^3d)	COD_{Feed} (mg/L)	TE^4 (% COD)	References
Winery	Pilot	AMBR^	Submerged	Hollow Fibre - UF Zenon ZW-10	-	1.8	0.5 - 2.2	1 000 - 4 000	97	Artiga *et al.,* 2007.
Winery: Rice	Laboratory	UAF^	-	-	19 -27	0.3	37.7	8 340 - 25 760	82	Yu *et al.,* 2006)
Winery	Laboratory	AJLR^	-	-	39 - 42.2	0.8 - 1	20	3 100 - 27 200	80 - 90	Eusébio et *al.,* 2004.
Winery	Pilot	AnRBC^	Submerged	Stainless Steel	26 - 30	1	6.3	3 240	23	Coetzee *et al.,* 2004.
Winery	Laboratory	UASB^	-	-	35	2.2	5.1 - 10.1	-	90	Keyser *et al.,* 2003.
Winery: Distillery	Full	UASB^	-	-	34 - 36	-	2 -18	30 000	90	Wolmarans & de Villiers, 2002.
Winery	Laboratory	UASB^	-	-	35	2	6.1 - 18	3 000 - 5 000	>90	Laubscher *et al.,* 2001.
Winery	Laboratory	AMBB^	-	-	30	0.8	8.8	800 - 11 000	>90	Petruccioli *et al.,* 2000.
Winery	Laboratory	AJLR^	-	-	25 - 30	2.1 - 4.4	0.4 - 5.9	800 - 12 800	>90	Petruccioli *et al.,* 2002.
Brewery	Laboratory	AnMBR^	External	Ceramic (0.2 μm) & Polymeric (30nm)	30	-	12	21 000	97	Torres, et al., 2011.
Brewery	Laboratory	AO-MBR^	Submerged	Hollow Fibre Polyvinylidene Florde	-	0.8 - 1	0.2 - 0.4	500 - 1 600	96.1	Dai *et al.,* 2010.
Brewery: Synthetic	Laboratory	DSR-AnMBR^	Submerged	-	-	-	5 - 12.5	2 900 - 5 200	95.15	Ling *et al.,* 2009.
Brewery	Pilot	AnSBR^	-	-	33	1	1.5 - 5	22 500 - 32 500	90	Shao *et al.,* 2008.
Brewery	Laboratory	AnIFBR^	-	-	35	4	70	2 100	>90	Alvarado-Lassman *et al.,* 2008,
Brewery	Laboratory	AnSBR-UASB^	-	-	55	13.5 - 26	3.2 - 8.6	56 600 - 62 300	79.6 - 88.9	Zupančič *et al.,* 2007.
Brewery	Laboratory	ASBR^	-	-	25 -2 7	-	3.5	-	88.7 - 91.6	Wang *et al.,* 2007.
Brewery	Laboratory	UASB-CA^	-	-	37 - 39	0.1	23.1	2 000 - 6 000	92 - 96	Rao *et al.,* 2007.
Brewery	Laboratory	AO- UASB-SBR	-	-	-	-	15 - 30	13 200	70 - 80	Brito et al., 2007.
Brewery	Laboratory	GRABBR^	-	-	35	0.8 - 2.3	2.2 - 13.4	5 000	93 - 96	Baloch *et al.,* 2007.
Brewery	Laboratory	EGSB-AF^		-	37	0.8 - 2	1.6 - 4.5	2 900 - 3 400	85.5 - 92.6	Connaughton *et al.,* 2006.
Brewery: Opaque Beer	Full	UASB^	-	-	37 - 39	1	6	12 540	57	Parawira *et al.,* 2005.
Brewery	Laboratory	PBUAR & MBR^	submerged	Polyethylene MF		0.3	4.5	1 200	92 - 96	Huei, 2005.
Brewery	Laboratory	UASB^	-	-	-	0.04	28	1 300	93	Ahn & Speece, 2004.
Brewery	Laboratory	AnMBR^	Submerged	-	-	-	-	2 300	95	Cornelissen *et al.,* 2001.

^: See Appendix A; 1.T: Temperature; 2. HRT: Hydraulic retention time; 3. OLR: Organic Loading Rate; 4. TE: Treatment Efficiency.

Treatment methods include biological flocculation (Zhang *et al.,* 2009) and anaerobic fermentation (Wilkie *et al.,* 2000) by using anaerobic upflow fixed film glass column reactors (UAFFR) (Acharya *et al.,* 2008); granular bed anaerobic baffled reactor (GRABBR) (Akunna & Clark, 2000); anaerobic filters with upflow anaerobic sludge bed (UASB-AF) reactors (Blonskaja *et al.,* 2003); anaerobic digestion in a continuous stirred zone reactor with a typical digester (CSTR-TD) (Méndez- Acosta, 2010); and anaerobic sequencing batch biofilm reactor (AnSBBR) (Mohan *et al.,* 2009). An anaerobic sequencing batch reactor (AnSBR) was used for the treatment of cassava stillage with a high COD of 40 000 to 70 000 mg/L. The reactor was maintained at 55°C by a circulating hot water bath at HRT of 5 d. The COD removal efficiency was 90.8%. The COD:N:P ratio of 200:5:1 was maintained with minimal nutrient supplementation (Luo *et al.,* 2009).

AD of distillery industry wastewater, having a very high COD of 110 000 to 190 000 mg/L and BOD of 50 000 to 60 000 mg/L, was studied in a continuously UAFFR at 37˚C and an optimum operational pH, which ranged from 6.0 to 8.0 was reported by Acharya *et al.* (2008) under HRT 8 d and OLR of 23.3 kg COD/m^{3}d, leading to a 64% COD reduction. Akunna and Clark (2000) investigated the performance of a GRABBR, which was used in the treatment of a whisky distillery wastewater comprising 9 500 mg/L of COD at various values of HRT (10, 7, 4 and 2 days) corresponding to OLR of (1, 1.3, 2.4 and 4.8 kg COD/m^{3}d). The pH values remained reasonably constant at approximately 7 with up to 80% COD removal. Mohan *et al.* (2008) studied the hydrogen (H_2) production in an anaerobic sequencing batch biofilm reactor (AnSBBR) by using distillery wastewater as substrate at two operating pH values of 3 and 6 for 24h at an ambient temperature of around 28˚C. The performance of the reactor was found to be reliant on the operating pH and favoured H_2 of 26, 6.98, 7 and 1.6 mmol/d, respectively. Nevertheless, COD removal efficiency was 69.7%; and was discovered to be successful at a neutral pH of 7.

AD of distillery industry wastewater was studied in mesophilic temperatures of 36 to 37˚C in a two-stage system comprising of an anaerobic filter (AF) and an UASB reactor. HRT ranged from 10 to 19 d at OLR of 2.5 to 5.1 kg COD/m^{3}d in the first stage and HRT of to 20 to 39 d, corresponding to OLR of 0.6 to 2.5 kg COD/m^{3}d in the second stage. The COD removal treatment efficiency that was achieved was 54% in the first stage and 93% in the second stage, respectively. The acidogenic reactor provided reasonable conversion of COD to volatile fatty acids (VFAs), averaging 20.5% and a self-regulating pH in the methanogenic reactor was in the range of 7.2 to 7.8 (Blonskaja *et al.,* 2003). Conversely; Gao *et al.* (2007) investigated the performance of an UASB reactor treating a distiller's grains wastewater with an acidic pH 3.3 to 4.3 at 37°C. Removal efficiencies of COD achieved was 80 to 97.3% at a HRT of 2 to 11 h, and OLR of 5 to 48.3 kg COD/m^{3}d.

A model was developed by Turkdogan-Aydınol and Yetilmezsoy (2010) to predict biogas and methane production rates in a mesophilic UASB reactor by treating molasses wastewater, which was characterized by a moderate acidic pH of 4 to 5, high COD from 65 000 to 130 000 mg/L and the reactor showed a significant performance with an average COD removal efficiency of 93%, and an average volumetric COD removal rate of 6.9 kg COD/m^{3}d. A study of biomethanation of distillery industry wastewater with COD of 71 544 mg/L was also carried out by Banerjee and Biswas (2004) in a semi-batch digester at different OLRs of (1.54, 2.12, 2.74, 3.28 and 4.5 kg COD/m^{3}d) at different digestion temperatures in the range of 35 to 55 $^\circ$C for HRT of 14 d and under a controlled pH in the range of 6.8 to 7.2.

2.4.1.2 Anaerobic digestion of winery wastewater

A number of technologies are applied for winery wastewater treatment by using AD at lab scale, pilot-scale and full-scale (Ganesh & Rajinikanth, 2010; Brito *et al*., 2007; Gao *et al.,* 2007; Yu *et al.,* 2006; Moleta, 2005; Coetzee *et al.,* 2004; Keyser *et al.,* 2003; Wolmarans & de Villiers, 2002; Laubscher *et al.,* 2001;). As shown in Table 2.5, different anaerobic treatment methods that were utilized includes an UASB (Gao *et al.,* 2007; Keyser *et al.,* 2003; Wolmarans & de Villiers, 2002; Laubscher *et al.,* 2001); an anaerobic fixed-bed reactor (AnFBR)(Ganesh & Rajinikanth, 2010); an upflow anaerobic filter (UAF) (Yu *et al.,* 2006); an anaerobic sequencing batch reactor (AnSBR) (Brito *et al*., 2007); and a rotating biological contactor (RBC) (Coetzee *et al.,* 2004). COD removal efficiencies of above 90% were achieved, which allowed the treated winery wastewater to be discharged into the municipal sewer.

Molasses-based wastewater was added with nutrient sources to get a C:N:P ratio of 100:5:1, and synthetic wastewater was used for separation of hydrolytic and methanogenic groups, respectively (Wejekoon *et al.,* 2011). The AD process was carried out in a CSTR type digester, which was utilized by Méndez- Acosta *et al.* (2010) to treat a tequila vinasses industry wastewater. The highly polluted wastewater with a temperature of around 90°C; pH of 4 and COD, which ranged from 20 000 to 50 000 mg/L was treated with a digester operated under mesophilic conditions, where the reactor temperature was regulated at 35 and 36°C. The COD removal ranged between 90 to 95% at a maintained OLR of 1 kg COD/m^{3}d.

Ganesh *et al*. (2010) investigated three upflow anaerobic fixed-bed reactors (AnFBR) for treatment of winery industry wastewater with COD of 18 000 to 21000 mg/L. The reactors were maintained at a mesophilic temperature of 35°C by using a hot water jacket. The feed wastewater with a liquid V_{up} between 0.8 and 0.9 m/h with a maximum OLR of 0.04 kg COD/m^{3}d and up to 80.5% removal efficiency was attained. A rotating biological contactor (RBC) was evaluated for the treatment of winery wastewater, with COD from 2 910 to 3 828 mg/L and an increased pH from 5.6 to 6.1 at an average HRT of 1 h. The system was able to achieve between 23 and 39% COD removal (Coetzee *et al.,* 2004). Three UASBs were evaluated for the treatment of winery wastewater by Keyser *et al.* (2003); the V_{up} in the reactors was set at 2 m/h and operated at 35˚C. The first reactor reached a 90% COD removal within 17 d at HRT of 24 h; the second reactor was seeded with brewery granules and achieved 85% COD removal rate with a HRT of 50 d, and the third was seeded with sludge, while there were problems encountered with conventional sludge seeding. Conversely, Laubscher *et al.* (2001) investigated two UASB reactors that were maintained at around 35°C and at a pH of 7 for treating synthetic winery wastewater with COD in the range of 2 000 to 5 000 mg/L. The OLR was 18 kg COD/m^3d and COD removal efficiency was above 90%.

2.4.1.3 Anaerobic digestion for brewery wastewater

Table 2.5 represents treatment methods for brewery wastewater. High-rate anaerobic, biological reactors such as: anaerobic filter and expanded granular sludge bed (EGSB-AF) (Connaughton *et al.,* 2006); UASB (Parawira *et al.,* 2005; Ahn & Speece, 2004); addition of carbon dioxide (CO_2) absorber (CA) into an UASB (UASB-CA) (Rao *et al.,* 2007); UASB sequencing batch reactor (UASB-SBR) (Brito *et al.,* 2007); GRABBR (Baloch *et al.,* 2007); anaerobic inverse fluidized bed reactors (AnIFBR) (Alvarado-Lassman *et al.,* 2008); and anaerobic sequencing batch reactors (AnSBR) (Shao *et al.,* 2008) were reported to be adequate for COD reduction of about 90% in the treatment of brewery wastewater.

An UASB reactor was utilized for treating brewery wastewater with COD of up to 1 290 mg/L at an OLR up to 28 kg COD/m^3d with a corresponding HRT of 1 h. The fluid V_{up} was maintained at 0.6 to 2.5 m/h at a temperature around 35˚C and the COD removal efficiency achieved was up to 93% (Ahn & Speece, 2004). Conversely, Parawira *et al.* (2005) evaluated an UASB reactor during anaerobic digestion to treat opaque beer brewery wastewater with HRT of approximately 24 h. The brewery wastewater had a COD of 8 240 to 20 000 mg/L and temperature was maintained at 37˚C, while the average COD reduction was 57% at OLR of 6 kg COD/m^3d. Studies were also carried out by Rao *et al.* (2007) with a carbon dioxide absorber (CA) to evaluate the usage of carbon dioxide (CO_2) in the UASB reactor to treat brewery wastewater with COD of 2 000 to 6 000 mg/L at an optimum OLR of 23.1 kg COD/m^3d and HRT of 2 h. The reactor pH was controlled in the range of 7 to 5 with COD reduction of 96%. The brewery wastewater was treated by an integrated system of UASB as a pre-treatment, and SBR as post-treatment, while both reactors were operated 35˚C. The pH in the UASB ranged from 6.5 to 7.9 and COD of 400 to 2 000 mg/L with V_{up} of

40 to 50 m/h .The average COD removal efficiencies of 70 to 80% was achieved in the UASB process (Brito *et al.,* 2007).

Brewery wastewater with COD of 22 500 to 32 500 mg/L was treated by utilizing an anaerobic sequencing batch reactor (ASBR) in which an OLR was controlled between 1.5 and 5.0 kg COD/m^{3}d, and HRT of 1 d. The COD removal efficiency was more than 90% (Shao *et al.,* 2008). The AnIFBR technology was investigated for the removal of organic matter in the brewery wastewater with COD of 2 083 mg/L and removal efficiencies greater than 90%. The reactor showed an excellent COD removal with OLR values of up to 0.07 kg COD/m^{3}d, and the reactor temperature was kept at 35˚C in a controlled pH of 7 (Alvarado-Lassman *et al.,* 2008).

Baloch *et al.* (2007) studied the performance characteristics of a GRABBR treating brewery wastewater with a high COD of 115 000 to 125 000 mg/L. The reactor achieved COD removal of 93% to 96% with high methane production when operated at OLRs of 3.4, 5 and 13.4 kg COD/m^{3}d, with corresponding HRTs of 36, 22 and 19.2 h, respectively. Two identical expanded granular sludge bed-anaerobic filter (EGSB-AF) bioreactors were fed with brewery wastewater COD discharged at a range between 1 000 to 6 000 mg/L. The average pH was 7.3 and the treatment experiments were separated into four different operational periods, which were characterized either by the change in the HRT or $V_{up.}$ The fluid V_{up} was 2.5, 3 and 5 m/h at a HRT of 18, 24 and 48 h. The reactors were maintained at 37˚C and around 15˚C, respectively. The OLR was up to 4.5 kg COD/m^{3}d (Connaughton *et al.,* 2006). The sequence batch reactor (SBR) was utilized for treatment of brewery wastewater at HRT of 6 h and temperature around 25˚C. The aeration flowrate to reactor was 500 L/h and a stable removal efficiencies of 88.7% COD was achieved (Wang *et al.,* 2007). Treatment of brewery wastewater with COD between 20 000 and 22 000 mg/L in a thermophilic anaerobic sequencing batch reactor (ASBR) was studied under different OLR from 3.2 to 8.6 kg COD/m^{3}d. The ASBR COD degradation removal efficiency was from 79.6 to 88.9% (Zupančič *et al.,* 2007).

2.4.2 Application of anaerobic digestion (AD) for the non-alcoholic beverage industry

Table 2.6 presents treatment methods for non-alcoholic beverages wastewater. AD treatment technologies have been reported for laboratory scale application in the treatment of non-alcoholic beverage industry wastewater (El-Kamah *et al.,* 2010; Guven, 2001; Kalyuzhnyi, *et al.,* 1997).

Table 2.6: Treatment methods for non-alcoholic beverages industry wastewater

Type of Wastewater	Application	Treatment Method & Type of Reactor	Configuration	Characteristics of Membrane	T[1] (°C)	HTR[2] (days)	OLR[3] (kg COD/m^{3}d)	COD_{Feed} (mg/L)	TE[4] (% COD)	References
Fruit Juice	**Laboratory**	**AH-SBR^**	**-**	**-**	**26**	**0.5**	**5.3 - 11.8**	**-**	**67.4**	**Tawfik & El-Kamah, 2012.**
Fruit Juice	**Laboratory**	**UASR-AS^**	**-**	**-**	**25**	**1**	**8.7**	**2 300 - 10 900**	**97.5**	**El-Kamah *et al.*, 2010.**
Fruit Juice-Sourcherry & Apple	**Laboratory**	**UASB-ASBR^**	**-**	**-**	**35 - 37**	**2.3 - 5.7**	**5**	**1 000 - 8 000**	**95**	**Ozbas *et al.*, 2006.**
Fruit Juice	**Laboratory**	**AMBR-UV-NF^**	**Submerged**	**Tubular (0.04 µm)**	**-**	**-**	**-**	**5 000 - 40 000**	**>95**	**Chmiel *et al.*, 2003.**
Fruit Juice	**Pilot**	**AMBR-UV-NF^**	**Submerged**	**Hollow Fibre MF polymer (0.4 µm)**	**20 - 35**	**0.42 - 1.4**	**4 -13**	**2 500 - 6500**	**95 - 97**	**Noronha *et al.*, 2002.**
Fruit Juice	**Laboratory**	**AMBR-UV-NF^**	**Submerged**	**Tubular (0.04 µm)**	**13 - 34**	**-**	**-**	**1 800 - 6 600**	**>95**	**Blöcher *et al.*, 2002.**
Fruit Juice	**Laboratory**	**AH-SBR^**	**-**	**-**	**40 - 66**	**0.41 - 0.42**	**3.5**	**-**	**67.4**	**Tawfik & El-Kamah, 2012.**
Softdrink: Bottle Washing	**Pilot**	**CF- RO- IE^**	**-**	**-**	**40 - 50**	**-**	**-**	**-**	**>99**	**Haroon *et al.*, 2013.**
Softdrink: Bottling Washing	**Pilot**	**AMBR^**	**Submerged**	**Zenon: ZW-10 (0.4 µm)**	**-**	**0.21 - 0.33**	**-**	**288 - 2 990**	**94**	**Matošić *et al.*, 2009.**
Softdrink	**Laboratory**	**AnCSBR^**	**-**	**-**	**35 - 37**		**0.004 - 0.09**	**72 900 - 145 000**	**74 - 98**	**Guven, 2001.**
Softdrink: Bottling Washing	**Full**	**CF-NF^**	**-**	**-**	**-**	**-**	**-**	**33**	**70**	**Miyaki *et al.*, 2000.**
Softdrink	**Laboratory**	**UASB-HR^**	**-**	**-**	**35**	**<1**	**13 - 16.5**	**1 100 - 30 700**	**80 - 82**	**Kalyuzhnyi, *et al.*, 1997.**

^: See Appendix A; 1.T: Temperature; 2. HRT: Hydraulic retention time; 3. OLR: Organic Loading Rate; 4. TE: Treatment Efficiency.

The selection of technology depends on the wastewater characteristics, the required specification of the final product, and the simplicity of manufacturing and costs (Guimarães *et al.,* 2012; Hsine *et al.,* 2010; Guven, 2001). Treatment of wastewater from fruit juice and softdrink industries usually comprises some sort of physical pre-treatment method for the removal of suspended matter, followed by biological treatment methods (Ozbas *et al.,* 2006; Blöcher *et al.,* 2003; Chiemel *et al.,* 2003; Noronha *et al.,* 2002; Tawfik & El-Kamah, 2012).

2.4.2.1 Anaerobic digestion for fruit juice industry wastewater

The AD methods reported for fruit juice wastewater include: coagulation and flocculation (Amuda & Amoo, 2007; Amuda *et al.,* 2006); an UASB reactor and aerobic sequence batch reactor (UASB-ASBR) (Ozbas *et al.,* 2006); polyurethane trickling filter and anaerobic hybrid reactor (PTF-AH) (Tawfik & El-Kamah, 2012); and an activated sludge with upflow anaerobic sponge reactor (UASR-AS) (El-Kamah *et al.,* 2010). El-Kamah *et al.* (2010) studied the treatment of fruit juice industry wastewater with a maximum COD of 10 913 mg/L by utilizing a three batch activated sludge (AS) system and operated at HRT of 48 h; a two-stage upflow anaerobic sponge reactors (UASRs) were operated at HRT of 13 h, corresponding to an OLR of 8.7 kg COD/m^{3}d. The third treatment scheme consisted of a two-stage UASR followed by an AS system, which was operated at three different HRTs, namely 10, 12, and 14 h. The combined system attained an overall removal efficiency of 97.5% for COD producing wastewater suitable for reuse for agricultural purposes.

An integrated system, which comprised of AH–PTF was used to treat tomato processing industry wastewater with fluctuating COD of approximately 795 to 1 000 mg/L and an OLR of 1 to 4.5 kg COD/m^3.d. The results attained for the combined system was very successful for the treatment of tomato industry wastewater at an HRT of not more than 10 h, while the COD removal efficiency was 96% (Tawfik & El-Kamah, 2012). An UASB reactor was used as a pre-treatment of tomato processing industry wastewater at a feed flowrate of 20 L/d and COD of 5 000 to 7 000 mg/L at an V_{up} of 0.2 m/h. The reactor OLR was in the range of 2.9 to 7.7 kg COD/m^3 d at HRT of 0.75 d. The average COD removal efficiency of 85 to 95.6% was achieved (Gohil & Nakhla, 2006).

2.4.2.2 Anaerobic digestion for softdrink industry wastewater (SDIW)

The total soluble organic matter present in SDIW is high at about 62% (Matošić *et al.,* 2009), and hence cannot be removed by physical or mechanical means. SDIW are in concordance with COD: total organic carbon (TOC) ratio of about 3.8, signifying the presence of an oxidation phase for carbon, thus indicating that wastewater is suitable for biological treatment. Therefore, chemical and biological oxidation becomes the key for SDIW treatment (Hsine *et al.,* 2010).

Table 2.6 shows some of the studies that have been reported on the treatment of SDIW on a lab, pilot and full-scale. Guven (2001) treated highly biodegradable SDIW with COD of 72 900 to 145 000 mg/L under anaerobic conditions by using two AnCSBRs. For an OLR ranging from 0.004 to 0.09 kg COD/m^{3}d the reactors were able to achieve up to 98% removal efficiency. The SDIW was studied by utilizing anaerobic treatment of UASB and a hybrid reactor (HR) (Kalyuzhnyi *et al.,* 1997), with a fluctuating COD between 1 100 to 30 700 mg/L and a varying pH between 4.3 and 13.0. Both reactors were maintained at 35°C and AH and UASB attained OLR of 13 and 16.5 kg COD/m^{3}d, respectively, with the treatment efficiency of about 80% at HRT of 1 d. From Tables 2.5 and 2.6 it is clear that extensive research has been published on the beverage industry in general, but limited studies have been reported for the softdrink industry.

2.5 Anaerobic digestion and subsequent biogas production

As explained in section 2.3.6; a complex microbiological process lies behind the efficient production of biogas. In biogas production large organic molecules such as fats, proteins and sugars are sequentially broken down into CH_4, CO_2 and a gas mixture called biogas (Schnurer & Jarvis, 2010), as previously presented in Figure 2-2. AD gained broader recognition, as more information became available, governing conditions that permit more consistent production of biogas, which can be used as an energy source (Strong *et al.,* 2008). Biogas typically refers to a gas, which is produced during anaerobic digestion of biomass or wastes. It is classified as a renewable energy source such as solar and wind energy. As previously indicated in Equations 2-2 to 2-7; the biogas composition is determined by the operating conditions and waste material that are used during anaerobic digestion. A guarantee of optimal generation of biogas depends on the C:N ratio of the input (Ošlaj & Muršec, 2010). In addition to CH_4 and CO_2, biogas contains several trace elements such as halogenated organic compounds, sulphur compounds, siloxanes, water (H_2O), ammonia (NH_3), oxygen (O_2), carbon monoxide (CO) and nitrogen (N_2). All of these compounds are harmful to human health, the environment, as well as equipment (Monteleone *et al.,* 2011). However, the most corrosive, harmful and toxic compound is hydrogen sulphide (H_2S) (Monteleone *et al.,* 2011; Schomaker *et al.,* 2000). In addition, biogas recovery from AD guarantees renewable energy supply and decreases greenhouse gas emissions (Wijekoon *et al.,* 2011).

Table 2.7 shows the typical composition of biogas. According to Wilkie *et al.* (2000), AD converts more than half of wastewater COD into biogas. Industrial biogas is normally produced at mesophilic (35 - 37°C) and thermophilic (50 - 60°C) conditions from 1) landfills; 2) sewage treatment plants; 3) industrial processing plants; and 4) during agricultural organic digestion (Ryckebosch *et al.,* 2011; Rasi *et al.,* 2007).

Table 2.7: Typical composition of biogas (Monteleone *et al.,* 2011; Ryckebosch *et al.,* 2011; Rasi *et al.,* 2007; Von Sperling, 2007; Krich *et al.,* 2005; Wellinger & Lindberg, 2005; Schomaker *et al.,* 2000)

Components	Percentage (%)
Methane (CH_4)	40 - 70
Carbon dioxide (CO_2)	15 - 60
Water (H_2O) $_{moisture}$	5 - 10
Hydrogen sulphide (H_2S)	0.005 - 2
Siloxanes	0 - 0.02
Halogenated hydrocarbons	VOC < 0.6
Ammonia (NH_3)	< 1
Oxygen (O_2)	0 - 1
Carbon monoxide (CO)	< 0.6
Nitrogen (N_2)	0 - 2

2.5.1 Biogas implementation and utilization in South Africa

Currently, biogas production technologies are mainly located in rural areas and consist of a fixed dome plant that uses bio-digesters to produce biogas from waste material, which is supplied to heating appliances in rural and peri-urban areas. These technologies assist in addressing challenges of access to energy in SA. Societies utilize biogas technology to produce energy, which is used for cooking, lighting, warmth and even electricity generation. Biogas also subsidizes towards a cleaner environment as it reduces the necessity for wood fires, hence there is a reduction in greenhouse gas emissions, deforestation, and prevents the inhalation of smoke from wood fires, which can have a negative impact on human health (Gas Act, 2001 (Act No. 48 of 2001)). Biogas has the big advantage that the energy it generates is largely CH_4 and can be used to meet specific demand. However, the raw plant materials employed as the energy source must be sustainably grown and used efficiently in order for this to hold true. Although there are barriers that are associated with biogas utilization, some beverage industries have implemented this technology. According to Agama Biogas (2010) and Boyd (2012), South African Breweries (SAB) utilize an on-site digester to treat process wastewater and organic waste to produce biogas of 9 200 L/d.

Biogas technology is utilized worldwide, yet its application in SA is insignificant (Boyd, 2012). Since 2011 biogas production operations in rural communities in KwaZulu-Natal, Limpopo, as well as Pretoria were registered and approved by the National Energy Regulator of South Africa (NERSA). According to the national legislation, the owners of biogas processes that are not connected to the national gas pipeline are required to register with NERSA, but do not have to be licensed. There are some barriers, which are associated with biogas application in the agricultural, domestic and industrial sector, as presented in Table 2.8.

Table 2.8: Industrial barriers associated with biogas application and installation (Boyd, 2012)

Implementation barriers	Incentive options
Lack of knowledge and awareness	Operationalize and build on global knowledge
Municipal by-law limits	Adapt municipal by-laws to encourage industrial participation
No incentives: no charges for CO_2 tax and low tipping charges	Implement environmental policies which increase tipping cost and CO_2 tax
Motivation for biogas digesters are for waste management not energy generation	
High capital cost and lack of feed-in tariff.	

2.5.2 Anaerobic technology used for biogas production in the beverage industry

Treatment methods of wastewater from beverage industries are presented in Table 2.9. Several anaerobic reactors were used and reported for the production of biogas in the treatment of both alcoholic and non-alcoholic beverage industries. These reactors include: anaerobic hybrid (AH) reactors (Tawfik & El-Kamah, 2012); continuous stirred zone reactor (CSTR) digesters (Méndez-Acosta *et al.,* (2010); anaerobic granular bed baffled reactors (GRABBR) (Baloch *et al.*, 2007; Akunna & Clark, 2000); semi continuous batch reactor (SBR) (Banerjee & Biswas, 2004); anaerobic sequencing batch reactors (ASBR) (Shao *et al.,* 2008); UASB (Turkdogan-Aydinol & Yetilmezsoy, 2010; Gao *et al*., 2007; Rao *et al.,* 2007; Gohil & Nakhla, 2006; Moletta, 2005; Blonskaja *et al.,* 2003; Kalyuzhnyi *et al.*, 1997); upflow anaerobic fixed film bioreactors (UAFFR) (Acharya *et al.,* 2008); and expanded granular sludge bed-anaerobic filter (EGSB-AF) (Connaughton *et al.,* 2006).

Akunna and Clark (2000) utilized a GRABBR for the treatment of whisky distillery wastewater with an OLR 1 to 4.8 kg.COD/m^3 d. Biogas production increased with increasing loading rates from 10 to 22 L/d and the CH_4 content from 60 to 70%. GRABBR was utilized by Baloch *et al.* (2007) for the treatment of brewery wastewater to produce biogas. It was reported that as the OLR increased from 2.2 to 13.4 kg.COD/m^{3}d, the biogas production rate also increased from 16 to 62 L/d with a high CH_4 generation, which varied from 62% to 75%. Biogas containing CO_2 in the range of 30% to 40% and a methane yield in the range of 0.3 m^3CH_4/kg COD was consumed. An upflow anaerobic fixed film bioreactor (UAFFR) was utilized for the treatment of distillery wastewater, containing COD up to 230 300 mg/L leading to a biogas production of 7.2 L/d (Acharya *et al.,* 2008).

Table 2.9: Biogas production and technology used for alcoholic and non-alcoholic industries

Wastewater Type	Reactor Type	pH	Organic loading rate (OLR) (kg.COD/m^{3}d)	Biogas (L/d)	Biogas composition (CH_4 %)	References
Distillery	**UAFFR^**	**8 - 8.5**	**23.25**	**7.2**	**-**	**Acharya *et al.*, 2008.**
Molasses	**AnMBR^**	**7.2**	**5 -12**	**5.5**	**55 - 65**	**Wijekoon et al., 2011.**
Distillery: Shochu	**KSAMBR^**	**-**	**-**	**1-10**	**60**	**Kanai *et al.*, 2010.**
Distillery grain	**UASB^**	**7**	**33.3**	**1.6 - 11.51**	**57 - 60**	**Gao *et al.*, 2007.**
Distillery	**SBR^**	**6.8 - 7.2**	**2.74**	**6.15 - 7.381**	**73.23**	**Banerjee & Biswas, 2004.**
Distillery	**UASB+AF^**	**7.2 - 7.8**	**0.6 - 5.1**	**1 - 6**	**55 - 75**	**Blonskaja *et al.*, 2003.**
Distillery	**GRABBR^**		**0.99 - 4.75**	**10 - 22**	**60 - 70**	**Akunna & Clark, 2000.**
Distillery: molasses vinasse	**UASB^**	**-**	**1.9 - 16.56**	**36 - 490**	**50 - 70**	**Turkdogan-Aydinol & Yetilmezsoy, 2010.**
Disillery: molasses vinasse	**UASB^**	**6.5 - 8**	**5-15**	**400 - 600**	**60 - 70**	**Moletta, 2005.**
Brewery	**ASBR^**	**6 -7**	**5.0**	**2.40**	**68**	**Shao *et al.*, 2008.**
Brewery	**GRABBR^**	**7.1 - 7.2**	**2.16 - 13.38**	**16 - 62**	**62 - 75**	**Baloch *et al.* 2007.**
Brewery	**EGSB-AF^**	**~ 7.1 - 7.2**	**4.47**	**4.1**	**~ 57.8 - 73.8**	**Connaughton *et al.*, 2006.**
Brewery	**UASB^**	**7 - 7.5**	**23.1**	**4.62 - 5.2**	**-**	**Rao et al., 2007.**
Brewery	**ASBR^**	**7.4**	**3.23 - 8.57**	**2.4**	**63 - 66**	**Zupančič *et al.*, 2007.**
Brewery	**UASB^**	**7.3**	**6.3**	**23**	**-**	**Keyser *at al.*, 2003.**
Tomato Juice	**AH^**	**-**	**2 – 4**	**0.48**		**Tawfik & El-Kamah, 2012.**
Tomato Juice	**UASB^**		**-**	**0.43**	**78 - 82**	**Gohil & Nakhla, 2006.**
Fruit Juice	**UASB^**		**-**	**0.8 - 11**		**Ozbas *et al.*, 2006.**
Softdrink	**UASB + HR^**	**6 - 7**	**0.013 - 0.165**	**-**	**60 - 65**	**Kalyuzhnyi *et al.*, 1997.**

^ Refer to appendix A; 1: OLR: Organic loading rate; 2: CH_4: methane

An anaerobic sequencing batch reactor (ASBR) was utilized to treat brewery wastewater, which effectively produced biogas of 2.4 L/d at a controlled OLR between 1.5 and 5 kg COD/m^{3}d. The average CH_4 composition in the biogas was 68% (Shao et al., 2008). An ASBR was investigated for the treatment of brewery wastewater under different OLR, ranging from 3.2 to 8.6 kg.COD/m^{3}d, and resulting in a CH_4 yield of 248 L/kg COD, which was consumed. Conversely, Banerjee and Biswas (2004) designed a semi continuous batch reactor (SBR) to investigate the biomethanation of distillery wastewater, while a total CH_4 production of 73.2% occurred at an OLR of 2.7 kg.COD/m^{3}d. Anaerobic treatment of tequila vinasses in a CSTR digester was investigated by Méndez-Acosta *et al. (*2010), where the biogas production rate was 1 to 14 L/d at an OLR of 0.01 kg.COD/m^{3}d, leading to a CH_4 production of greater than 65%.

The performance of an UASB reactor, which treats distiller's grain wastewater, was investigated by Gao *et al*. (2007) with an OLR of 33.3 kg.COD/m^{3}d. The biogas mainly consisted of 57 to 60% CH_4 and 38 to 41% CO_2. The biogas stillage treatment plants for *shochu* generated 60% CH_4; 40% CO_2 and other trace elements, including H_2S. Anaerobic digestion of distillery wastewater was in a mesophilic two-stage system, consisting of an anaerobic filter (AF) and an UASB reactor with an OLR of 0.6 and 5.1 kg.COD/m^{3}d. Biogas production of up to 6 L/d containing 55% to 75% of CH_4 was recorded by Blonskaja *et al.* (2003). Rao *et al.* (2007) studied the effect of a carbon dioxide absorber (CA) to evaluate the usage of CO_2 in the biogas UASB reactor using brewery wastewater. The biogas produced was between 4.6 to 5.2 L/d at an optimal OLR of 23.1 kg.COD/m^{3}d. An UASB reactor was used for the treatment of diluted beet molasses vinasse wastewater. The OLR ranged from 2 to 16.6 kg.COD/m^{3}d and the daily biogas production rates ranged between 36 to 490 L/d. The biogas produced contained CH_4 and CO_2 between 50% and 70%, and of 16% and 44%, respectively (Turkdogan-Aydinol & Yetilmezsoy, 2010). The treatment of winery wastewater was investigated by Moletta (2005) using an UASB with an OLR of between 5 and 15 kg.COD/m^{3}d. The biogas production was between 400 and 600 L/kg COD removed with a 60 to 70% CH_4 content. An expanded granular sludge bed-anaerobic filter (EGSB-AF) bioreactor was used for the treatment of brewery wastewater. A maximum OLR of 4.5 kg COD/m^{3}d was achieved at an applied liquid V_{up} of 2.5 m/h. In addition, the CH_4 content of the biogas was ~57.8 to ~73.8 % (Connaughton *et al.,* 2006).

An UASB reactor was applied for the treatment of tomato processing wastewater with a biogas yield of 0.43 m^3/kg COD and CH_4 content of 78 to 82% (Gohil & Nakhla, 2006). Ozbas *et al.* (2006) treated fruit juice wastewater by utilizing an UASB. The biogas that was produced was 0.8 to 11 L/d, which was produced with an average CO_2 ratio of 20%. An AH reactor was utilized for the treatment of tomato juice wastewater. The biogas production rate was 2.6 to 4 L/d, which was equivalent to 0.48 m^3/kg COD that was consumed (Tawfik & El-Kamah, 2012). The production of hydrogen (H_2) from softdrink wastewater was evaluated by Peixoto *et al.* (2011) for using two upflow anaerobic packed bed reactors. The results show that softdrink wastewater is a good source for H_2 generation. Data from both reactors indicated that the reactor without nutrients provided a higher H_2 yield of 3.5 H_2/mol of sucrose when compared to the reactor with nutrients containing 3.3 H_2/mol of sucrose. Better H_2 and biogas production rates were observed in the reactor, which produced 0.4 to 1 L/d biogas and contained 15.8% H_2, compared to a second reactor, which produced 0.2 to 1 L/d biogas with 2.6% H_2. The anaerobic treatment of softdrink wastewater, which utilized an UASB reactor and a hybrid sludge bed reactor was studied by Kalyuzhnyi *et al.* (1997). Both reactors produced a satisfactorily stable CH_4 content, ranging from 60 to 65% and CO_2 content, ranging from 35% to 40%.

2.5.3 Biogas utilization in beverage industry

Biogas utilization covers a diverse group of markets, including electricity, heat and vehicle fuels (AEBIOM, 2009). The biogas, which is recovered from anaerobic treatment guarantees a renewable energy source supply and decreases greenhouse gas emissions, thus contributing to sustainable waste and environmental impact management systems for a beverage producing facility (Brito *et al.,* 2007; Parawira *et al.,* 2005). The produced CH_4 can be readily utilized as a fuel for the ethanol production process.

According to Driessen and Vereijken (2003), anaerobic wastewater treatment can subsidize a brewery operation by saving up to 8% of a brewery's energy requirements, which is approximately 170 MJ/hL. Muster-Slawitsch *et al.* (2011) also reported that the energy that was generated from the biogas produced during AD of brewery wastewater exceeds the thermal process energy demand of 37 MJ/hL manufacture of beer. In breweries, direct application of biogas in a boiler is regularly the favoured solution. However, due to the high investment costs for a combined heat and power unit, extensive biogas generation system is required (Ince *et al.,* 2000).

The application of anaerobic membrane bioreactors (AnMBR) for the treatment of brewery wastewater was studied by Torres *et al.* (2011), and biogas was injected inside the membranes to stimulate a plug-flow regime. Kubota's submerged anaerobic membrane biological reactor (KSAMBR) has been successfully utilized in a number of full-scale food and beverage industries. The biogas was collected in the factory that treats *shochu* distillery wastewater and was utilized either for power generation or in the boiler, thus recovering energy for the industry (Kanai *et al.,* 2010). A winery industry in Wellington, SA, utilizes an UASB reactor for wastewater pre-treatment and the biogas produced after purification is used on-site (Wolmarans & de Villiers, 2002). The biogas produced from a submerged AnMBR system for pulp and paper wastewater treatment was used for scouring the membrane surfaces to reduce membrane fouling (Liao *et al.,* 2006).

2.6 Anoxic and aerobic wastewater treatment

According to Futselaar *et al.* (2013), the removal of TN and TP is usually required to meet international discharge standards. However, an anaerobic system is not capable of accomplishing these requirements when used as the sole and primary wastewater treatment technology. Therefore, either intermittent aerobic or anoxic processes within the bioreactor can be employed for TN and TP removal (Dai *et al.,* 2010; Cicek, 2003). The removal of both the nitrate formed through nitrification and phosphate present demands configuring the process to produce zones which are depleted (anoxic or anaerobic) and/or enriched (aerobic) with dissolved oxygen (DO) (Grady *et al.,* 2011; Judd, 2011; Mutombo, 2004). Grady *et al.* (2011) and Mutombo (2004) recommend that aerobic treatment should be used for COD concentrations of lower than 1 500 mg/L and anaerobic treatment for higher COD values of up to 50 000 mg/L. The supply of oxygen in the aerobic process is accomplished by using mechanical equipment, principally mechanical aerators. To attain a DO level of 2 mg/L, mixing and the maintenance of the biomass is essential (Mutombo, 2004).

2.6.1 Denitrification

Nitrogen removal or denitrification is the biological reduction of nitrate (NO_3-N) to nitrogen gas (N_2) under anoxic conditions by heterotrophic bacteria, which utilizes organic matter as food supply and NO_3-N as a supply of oxygen (O_2) (Fiss & Stein, 2002). Equation: 2-16 represents the reaction as follows:

$$6NO_3 + 5CH_3O \rightarrow 3N_2 + 5CO_2 + 7H_2 + 6OH^- + New\ Cells \qquad \text{Equation: 2-16}$$

Denitrification takes place only under anoxic conditions in the absence of DO, but NO_3-N is accessible. Thus, denitrification will normally initiate at DO less than 0.5 mg/L. Anoxic wastewater treatment methods are usually utilized for the reduction of nitrogen, and the process of the biological removal of nitrogen is called denitrification. Organisms in the anoxic treatment utilize the nitrate as an electron acceptor and discharge nitrogen in the form of nitrogen oxides or nitrogen gas (Wang *et al.,* 2009). Equation: 2-17 defines the denitrification reaction:

$$6NO_3^- + 5CH_3OH \rightarrow 3N_2 + 5CO_2 + 7H_2O + 6OH \qquad \text{Equation: 2-17}$$

Due to the anaerobic nature of anoxic wastewater treatment, the microorganism bed must remain submerged (Wang *et al.,* 2009). A minimal DO should be introduced into the system recycle stream or wastewater, and DO should be between 0.2 to 0.5 mg/L to attain higher denitrification rates (Wang *et al.,* 2009). WWTP has to deal with tough wastewater discharge standards, and nitrogen and phosphorus are one of the problems.

2.6.2 Nitrification

The biological transformation of ammonium to nitrate is known as nitrification. The microorganisms, known as "nitrifies," are strict "aerobes;" which means that, they must have DO in order to carry out aerobic treatment of wastewater. The main species responsible for nitrification are nitrobacteria and nitrosomonas (Fiss & Stein, 2002). Nitrification takes place only under aerobic conditions at DO levels of 1.0 mg/L or more. The optimum pH is between 7.5 and 8.5. However, most treatment plants are able to effectively nitrify at a pH of 6.5 to 7.0. However, at a pH below 6 the nitrification process stops (Al-Hashimia *et al.,* 2013). The nitrification process is described by Equations: 2-18 and 2.19 (Fiss & Stein, 2002):

$$2NH_4^+ + 3O_2 \rightarrow 2NO_2^- + 2H_2O + 4H^+ + new\ cells \qquad \text{Equation: 2-18}$$

$$2NO_2^- + O_2 \rightarrow 2NO_3^- + new\ cells \qquad \text{Equation: 2-19}$$

2.6.3 Phosphorus removal

Phosphorus removal can be achieved by chemical methods or biological methods. Factors that affect the phosphate removal in the reactor are pH, temperature and sludge age. A number of bench-scale studies have been reported on the effects of parameters, which affect nitrogen and phosphorus removal such as carbon sources, hydraulic retention time (HRT), recycling time, recycling rate, and SRT, which have influence on nitrogen and phosphorus removal, directly or indirectly (Song *et al.,* 2009). Presently, nutrient removal has attracted huge awareness in wastewater treatment for recycle (Tatiana *et al.,* 2011). Wastewater recycling can equally decrease the demand of freshwater and reduce the discharge of wastewater into the environment (Qadir *et al,* 2010).

2.6.4 The modified Ludzack-Ettinger (MLE) treatment method

There are two types of stages for the anoxic zone, namely the single stage zone and the multistage zone. The single stage zones are classified according to their flow regime and the staging of anoxic and aerobic arrangement. They rely on zones or compartments for denitrification to occur. For denitrification to occur, it is essential for nitrate and carbon supply to be present. The organic carbon can be supplied as BOD of the wastewater (Wang *et al.,* 2009).

According to Wang *et al.* (2009), a single stage denitrification process was reported by Wuhrmann for the treatment of domestic wastewater. Conversely, Ludzack and Ettinger (1962) was also developed a similar system. The system used was designed by Ludzack and Ettinger positioned the anoxic zone ahead of the aerobic zone. Conversely, Barnard improved the Ludzack and Ettinger method, as shown in Figure 2.3; on condition that the internal mixed liquor suspended solids (MLSS) are recycled from the aerobic stage to return nitrified MLSS to the anoxic zone at a synchronized rate, thereby, increasing the system's efficiency. The recycle should be up to five times the feed flow rate (Sheldon *et al.,* 2012; Hatch Mott MacDonald, 2010).

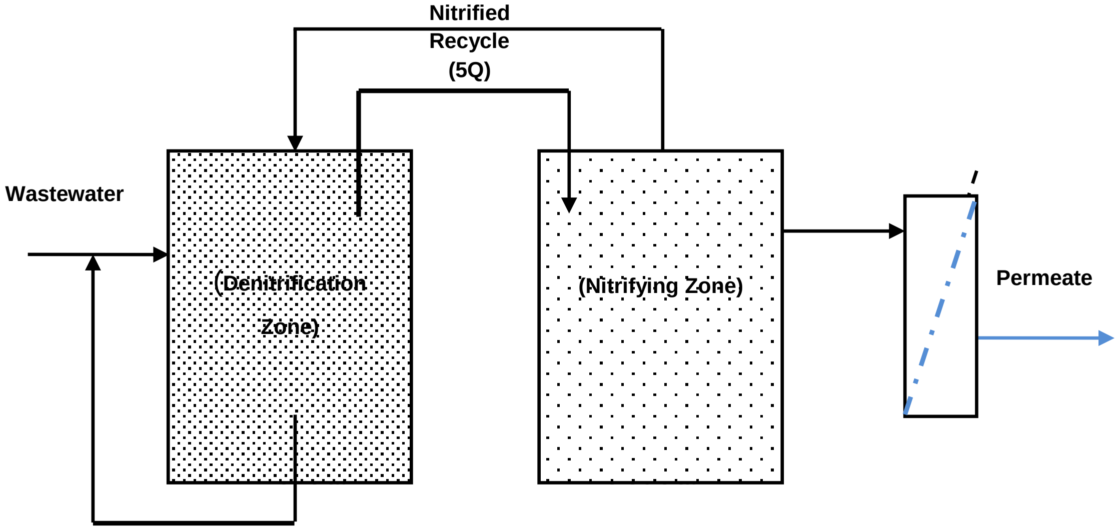


Figure 2.3: Modified Ludzack - Ettinger (MLE) process (Spellman & Drinan, 2012; Wang *et al.,* 2009)

2.6.5 UCT and modified UCT processes

The UCT process was proposed by Rabinowitz and Marais in 1980, where the recycle stream is taken from the anoxic instead of the aerobic zone and the introduction of nitrate in the anaerobic zone is avoided as presented in Figure 2.4 (Wang *et al.,* 2013; Monclús *et al.,* 2010). The modified UCT (MUCT) process was designed to make sure that the introduction of nitrate in the anaerobic zone is impractical. The anoxic zone is divided into two portions, introducing the return sludge in the upstream and using the downstream for denitrification of the nitrate recycled with recirculation as shown Figure 2.5. Under these circumstances, denitrification will be thorough in the anoxic zone and no nitrate will be returned to the anaerobic zone. The disadvantage of this MUCT is that the anoxic zone is under-loaded with nitrate (Wang *et al.,* 2013; Vaiopoulou & Aivasidis, 2008).

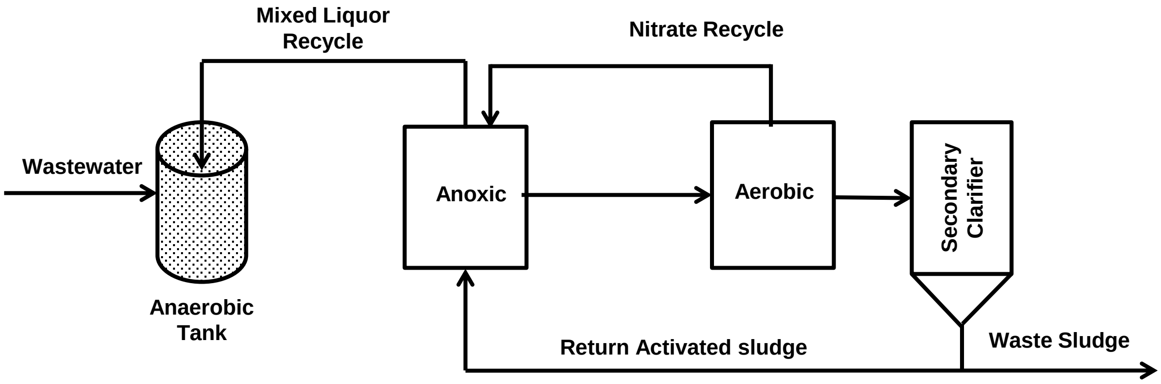


Figure 2.4: Schematic diagram for UCT process modified from (Monclús *et al.,* 2010)

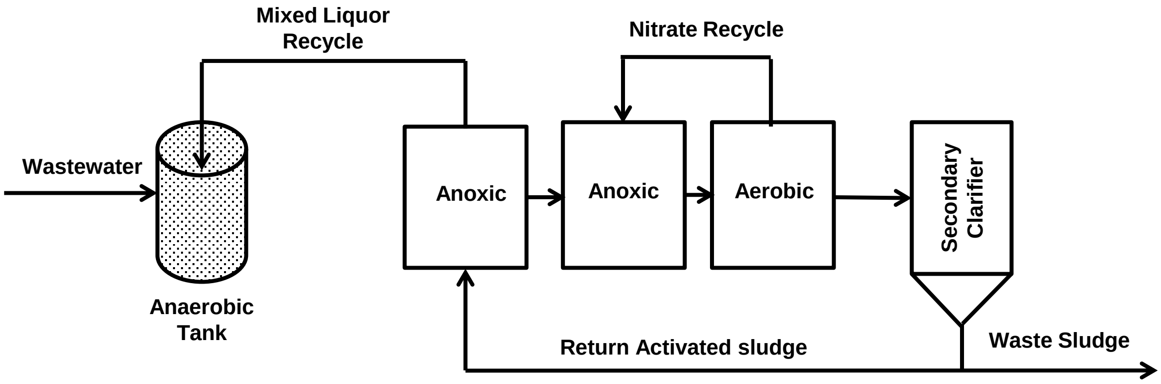


Figure 2.5: Schematic diagram for modified UCT process modified from (Vaiopoulou & Aivasidis, 2008).

2.6.6 Comparison of anaerobic and aerobic wastewater treatment method

Table 2.11 indicates a general comparison between anaerobic and conventional aerobic biological treatment systems. When discharging into surface water bodies, anaerobic pre-treatment, combined with subsequent aerobic post-treatment for organic or nutrient removal, is considered to be an optimal solution (Wijekoon *et al.,* 2011; Brito *et al.,* 2007; Connaughton *et al.,* 2006; Parawira *et al.,* 2005).

Table 2.10: Anaerobic treatment as compared to aerobic treatment (Torres *et al.,* 2011; Simate *et al.,* 2011; Wijekoon *et al.,* 2011; Ganesh *et al.,* 2010; Strong *et al.,* 2008; Artiga *et al.,*2007; Melamane, 2007; Huei, 2005; Parawira *et al.,* 2005; Driessen, Vereijken, T. 2003)

Operational parameters	Anaerobic systems	Aerobic Systems
Energy consumption	Low	High
Energy production	Yes	No
Biosolids production	Low	High
COD removal	70% - 85%	90% - 98%
Nutrients (N/P) removal	Low	High
Space requirement	Low	High
Discontinuous operation	Easy	Difficult

2.7 Membrane bioreactors (MBRs) and configurations

MBRs are defined as systems, which integrate biological degradation of waste products with membrane filtration (Lin *et al.,* 2012; Singhania *et al.,* 2012; Judd, 2011; Torres *et al.,* 2011; Cicek, 2003). MBR processes are designed to eradicate nitrogen and phosphorus concurrently (Ahn *et al.,* 2003). In the late 1960s Dorr-Oliver commercially established the first MBRs. MBR treatment methods have been put into operation in more than 200 countries and SA is one of the national key drivers when it comes to the utilization of membrane technology. The way that the membrane reactor is mounted including geometry is crucial to determine the overall process performance (Judd, 2011).

MBR systems were basically implemented based on two configurations: an immersed configuration (iMBR) and a sidestream (sMBR) configuration, as shown in Figure 2.6. Usually, the sidestream configuration provides more direct hydrodynamic control of fouling, and offers the advantages of easier membrane replacement, and high fluxes, but with high energy consumption and costs that are associated with frequent cleaning of the membranes. Several distinct advantages of the submerged configuration are their much lower energy consumption and fewer rigorous cleaning procedures, as well as milder operational conditions due to the lower tangential velocities (Lin *et al.,* 2012; Singhania *et al.,* 2012; Judd, 2011; Torres *et al.,* 2011; Cicek, 2003).

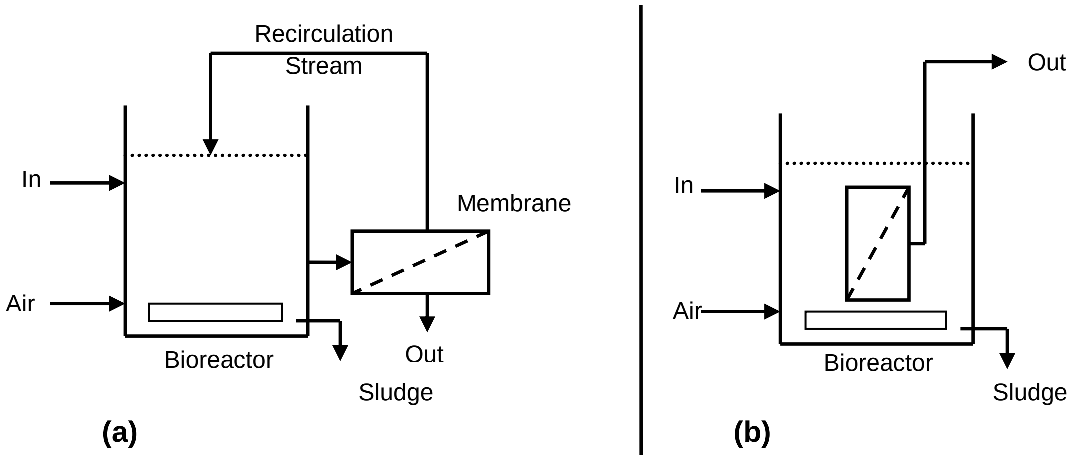


Figure 2.6: Configurations of a membrane bioreactor: (a) sidestream and (b) immersed Modified from (Judd, 2011)

2.7.1 MBR for beverage industry wastewater

MBRs are capable of treating high strength wastewater; hence efforts were made to evaluate their effectiveness within the treatment of alcoholic beverage industry wastewater. However, capital investment costs and high pressure requirements have resulted in the lack of full-scale implementation of many such treatment methods (Liao *et al.,* 2006; Dai *et al.,* 2010; Cicek, 2003). As shown in Table 2-6, several studies have reported that they utilized sidestream/external membranes in the treatment of alcoholic beverage industry wastewater (Bilad *et al.,* 2011; Torres, *et al.,* 2011; Kang *et al.,* 2002). Sidestream/external membrane configurations are considered to be more appropriate for high organic strength, high temperatures, and difficult to filter, waste streams (Mutamim *et al.,* 2012; Singhania *et al.,* 2012; Judd, 2011; Cicek, 2003). The application of the Kubota's submerged anaerobic membrane biological reactor (KSAMBR) process has been effectively developed in a number of full-scale alcoholic industries (Valderrama *et al.,* 2012; Kanai *et al.,* 2010).

2.7.1.1 Membrane bioreactor (MBR) for alcoholic beverages' wastewater treatment

The application of MBRs for the treatment of distillery wastewater has been studied at lab-scale, pilot-scale and was also implemented at full-scale (Valderrama *et al.,* 2012; Bilad *et al.,* 2011; Wijekoon *et al.,* 2011; Kanai *et al.,* 2010; Satyawali & Balakrishnan, 2009; Satyawali & Balakrishnan, 2008; Artiga *et al.,* 2007; Melamane *et al.,* 2007; Zhang *et al.,* 2006, Kang *et al.,* 2002). The effect of the OLR on VFA was examined in a two-stage thermophilic anaerobic MBR (TAnMBR), which was used to treat high strength molasses-based synthetic wastewater operated in a sidestream configuration. The COD removal efficiency was between 78% and 81% (Wijekoon *et al.,* 2011). COD removal of up to 53% was obtained in the study of the effects of powdered activated carbon addition on the operation of an MBR (AMBR-PAC), treating sugarcane molasses based distillery wastewater (Satyawali & Balakrishnan, 2009; Satyawali & Balakrishnan, 2008).

The performance of a calefactive aerobic membrane bioreactor (AMBR) equipped with a submerged stainless steel membrane, treating grain-sorghum based simulated distillery, was investigated by Zhang *et al.* (2006); obtaining COD and TN removal efficiencies of 94.7% and 84.4%, respectively. Bilad *et al.* (2011) also studied the influence of membrane pore size during the treatment of molasses based distillery wastewater. Bilad *et al.* (2010) evaluated the performance of an aerobic membrane bioreactor (AMBR), treating diluted synthetic molasses wastewater containing COD of 2 250 mg/L. The aeration system was positioned underneath the membrane modules to provide a distributed air flowrate of 0.6 m^3/h. The system was kept at a psychrophilic temperature of 21°C. The reactor operated at HRT of 18 to 20 h and a mixed liquor suspended solid (MLSS) of 800 to 1 200 mg/L. The COD removal efficiencies that were obtained were between 80% and 85%. Satyawali and Balakrishnana (2009) investigated the effect of powdered activated carbon (PAC) addition on the operation of a MBR treating sugarcane molasses based distillery wastewater with COD, ranging from 30 to 40 000 mg/L. The reactor was equipped with a submerged 30µm nylon mesh filter with 0.05 m^2 filtration area. OLR varied from 4.2 to 6.9 kg COD/m^3d and at an enhanced critical flux of around 23%. The transmembrane pressure (TMP) stayed constant at 0 to 0.1 kPa. Similar studies were reported by Satyawali and Balakrishnana (2008) who used a similar MBR at OLR ranging from 3 to 5.7 kg COD/m^3d and COD removal and biomass growth in the reactor, which was up to 41%.

In Japan, KSAMBRs are being utilized by 15 full-scale plants for the treatment of *shochu* spirit wastewater. The recovered energy from the KSAMBR process was on average 12 GJ/d, which was used for steam production. The COD removal efficiencies were between 75% and 92% (Kanai *et al.,* 2010). Additionally, Kang *et al.* (2002) compared the filtration characteristics of organic and inorganic membranes by using a membrane coupled anaerobic bioreactor (MCAB) for the treatment of distillery wastewater. Due to the seasonal nature of winery wastewater and the irregular organic matter, the application of a MBR was a suitable treatment method for winery wastewater as they efficiently combine the BOD, SS, TN and TP removal and microbial sanitization (Valderrama *et al.,* 2012; Artiga *et al.,* 2007). According to Ganesh and Rajinikanth (2010) and Moleta (2005), a number of technologies are used for winery wastewater treatment. Different aerobic treatment methods have been utilized, including an aerobic jet-loop activated sludge reactor (AJLR) (Eusébio et *al.,* 2004; Petruccioli *et al.,* 2002); and an air micro-bubble bioreactor (AMBB) (Oliveira & Duarte, 2011; Petruccioli *et al.,* 2000). COD removal efficiencies of above 90% were achieved, allowing the treated winery wastewater to be discharged into the municipal sewer. Table 2.5 shows winery wastewater treatment methods, which use MBR. Artiga *et al.* (2007) utilized an MBR to treat a synthetic winery wastewater under aerobic conditions and recorded COD removal of above 97%. The MBR winery permeate was appropriate for agricultural, urban, and recreational reuse (Valderrama *et al.,* 2012; Artiga *et al.,* 2007; Melamane *et al.,* 2007), and meeting the quality stipulation defined by worldwide guidelines and regulations for water reuse and reclamation (Valderrama *et al.,* 2012). Winery wastewater was treated by evaluating an ABBR with COD, which ranged from 800 to 11 00 mg/L at the maximum OLR of 8.8 kg COD/m^{3}d, and HRT was approximately 0.8 d. The bioreactor efficiency was above 90% (Petruccioli *et al.,* 2000). An aerobic jet-loop activated sludge reactor (AJLR) was utilized for the treatment of winery wastewater with COD that ranged between 800 and 12 800 mg/L and OLR from 0.4 to 5.9 kg COD/m^{3}d with a varied HRT from 2.1 to 4.4 d.

A comparative study was carried out by Valderrama *et al.* (2012) for the winery wastewater in order to evaluate MBR pilot plant and compared it to a full-scale conventional activated sludge system in order to reach the quality determined international guidelines and regulations for wastewater reuse and reclamation. The winery wastewater has a fluctuating COD from 100 mg/L to more than 8 000 mg/L. The flat sheet Kubota membrane had a surface area of 20 m^2 permeation of 9 min and relaxation of 1 min, with a permeation flux of 12.5 L/m^{2}h, and membrane fouling, which was prevented by trough air scouring. A comparative evaluation of a conventional activated sludge (CAS) system with a MBR was studied by Valderrama *et al.* (2012). The MBR successfully reduced the COD concentration with a removal efficiency of close to 97%. In addition, the MBR performance showed a fairly stable operation in terms of transmembrane pressure (TMP) and permeability regardless of the high inconsistency of the treated wastewater. Artiga *et al.* (2007) utilized a MBR to treat a synthetic winery wastewater under aerobic conditions and recorded COD removal of above 97%. A fungal pre-treatment step followed by a submerged MBR and a digester in series was investigated by Melamane *et al.* (2007). High rate anaerobic digestion of the winery distillery wastewater was achieved, as high polyphenols and COD removals of 99.5 and 87% were recorded, respectively. The MBR winery permeate was appropriate for agricultural, urban, and recreational reuse (Valderrama *et al.,* 2012; Artiga *et al.,* 2007; Melamane *et al.,* 2007), and met the quality stipulation defined by worldwide guidelines and regulations for water reuse and reclamation (Valderrama *et al.,* 2012).

A submerged ZW-10 hollow fibre ultrafiltration (UF) with a surface area of 0.9 m^2 and AMBR was fed with simulated white wine wastewaters of COD, ranging between 1 000 and 4 000 mg/L and OLR 0.5 to 2.2 kg COD/m^{3}d. Dissolved oxygen (DO) concentration was maintained in the range of 3 to 4 mg/L. The UF membrane module was operated in cycles of 15 min of permeation and 45 s of backwashing with permeate, and TMP was always kept below 50 kPa. The system was able to achieve COD removal efficiency higher than 97% (Artiga *et al.,* 2005).

The treatment of brewery wastewater, which used MBRs' has been studied mostly at lab-scale, as shown in Table 2.5. The application of a sidestream AnMBR was studied by Torres *et al.* (2011) for the treatment of brewery wastewater, which contained high amounts of organic SS. COD removal levels were above 97%. A submerged AnMBR was studied by Cornelissen *et al.* (2001) for the treatment of brewery wastewater with a constant COD removal of 95%, while Huei (2005) evaluated the performance of a packed-bed upflow anaerobic reactor (PBUAR), coupled with a MBR. The COD reduction during the MBR stage ranged between 92% and 96 %. COD removal efficiency of 95.15% was obtained in a submerged double-shaft rotary DSR-AnMBR, which was used for the treatment of synthetic brewery wastewater (Ling *et al.,* 2009).

A multi-segment biochemical treatment (AO/MBR) process, which included an anoxic and aerobic stage, was used in the integration of an UASB-MBR to treat beer brewing wastewater (Dai *et al.,* 2010). The average COD and TN removals were 96.1% and 92%, respectively. Using the MBR technology to treat brewery wastewater accomplished high removal rates for COD (96.1%) and NH_4-N (92%). The permeate quality parameters that were assessed were better than those that were set to treat wastewater for the purpose of reuse (Dai *et al.,* 2010). The SBR post-treatment was utilized for the treatment of brewery wastewater with COD ranging from 400 to 2 000 mg/L, characterized by the use of aerobic zone, followed by an anoxic zone. There was an oxygen limitation when DO was 3.7 mg/L with HRT of 1.9 in the aerobic-anoxic sequence, characterized by a pre-denitrification step and thereafter the aerated phase in the anoxic–aerobic–anoxic sequence and HRT of 1.2 d with DO of 2.8 mg/L in the aerated period. The maximum observed specific nitrogen removal rate was 0.04 kg/kg and NO_3^-N in the permeate was lower than 8 mg/L. The results attained when acetate was used to increase the mass C:N ratio to 1.3 during the anoxic phase, leading to a complete nitrate removal, confirmed the soluble carbon source. Conversely, denitrification was inhibited throughout the aerated period when the DO was increased to 7 mg/L. The nitrogen removal efficiency was 50%, resulting in a wastewater NO_3^-N above 15 mg/L. An upflow anaerobic sludge bed and membrane bioreactor (UASB+MBR) was investigated by using various operations to treat brewery wastewater of COD around 500 to 1 000 mg/L and pH of 7.7. The DO of the aerobic zone was controlled at a range of 2 to 4 mg/L. The system treatment efficiency was up to 96% (Dai *et al.,* 2010).

The brewery wastewater of COD_s, which ranged between 2 000 and 6 000 mg/L was treated by an integrated system of packed bed upflow anaerobic reactor (PBUAR) and a three flat shaped microfiltration (MF) membrane of 0.4 µm pore size and a surface area of 0.1 m^2 were submerged in a bioreactor, which operated at HRT of 2.12 and 18 h. The average OLR for PBUAR was approximately 4.5 kg COD/m^3d and for MBR was 1 kg COD/m^3d with the air flow rate at 0.02 m^3/h. The COD removal for PBUAR was 75% and the MBR was between 92% and 96% (Huei, 2005).

2.7.1.2 Membrane bioreactor for non-alcoholic beverages' wastewater treatment

MBRs have also been evaluated for the treatment of fruit juice and softdrink industry wastewater along with further membrane filtration (Blöcher *et al.,* 2003; Chiemel *et al.,* 2003; Noronha *et al.,* 2002) in order to facilitate water reuse (Haroon *et al.,* 2013; Alam *et al.,*2007; Miyaki *et al.,* 2000;Tay & Jeyaseelan, 1995), and to meet the required discharged standards (Drogui *et al.,* 2008; Amuda & Amoo, 2007; Oktay *et al.,* 2007; Amuda *et al.,* 2006; Kuyuzhnyi, *et al.,* 1997). Table 2.4 indicates the treatment methods for non-alcoholic beverage wastewater. Blöcher *et al.* (2003) implemented a system that comprised of MBR and a combined NF/UV disinfection phase. During the MBR stage the COD removal efficiencies were above 95%; with similar results obtained by Chiemel *et al.* (2003). These integrated processes treated water to meet the legal bacteriological standards for drinking water (Blöcher *et al.,* 2003; Chiemel *et al.,* 2003; Noronha *et al.,* 2002).

The AMBR was used to treat a fluctuating softdrink industry wastewater with COD between 200 and 3 000 mg/L. The MBR accomplished constant organic compound removal from the wastewater with removal efficiencies of 94% for both COD and TOC, and 98% for BOD. Hence, these properties of MBR systems and the variations of flow and COD concentrations of beverage industry wastewaters, the treatment of these wastewaters in a MBR is motivating (Matošić *et al.,* 2009). As a result, MBR post-treatment method could be utilized for the treatment of softdrink industry wastewater. Aerobic conditions were also studied by using iMBR under at pilot-scale for the treatment of softdrink wastewater from a bottling plant for further filtration and water reuse (Matošić *et al.,* 2009).

2.7.2 Types of membranes and membranes filtration methods

Membranes are classified according to the type of filtration and separation methods, which will determine the membrane pore size. Membrane filtration occurs when force is applied to water or wastewater through the membrane (Judd, 2011; Huei, 2005). According to Judd (2011); Simate *et al.* (2011); and Huei (2005), there are four main pressure driven membrane separation and filtration methods from which water and wastewater permeate:

1) Microfiltration (MF): Separation and filtration of SS from wastewater is achieved by sieving through macropores (>50 nm);
2) Ultrafiltration (UF): Separation and filtration occur by sieving through mesopores (2-50 nm);
3) Nanofiltration (NF): Separation and filtration are attained through a combination of solubility-diffusion, sieving and charge rejection through micropores (<2 nm); and

4) Reverse Osmosis (RO): Separation and filtration are achieved by the features of different diffusion rates of wastewater, solutes and solubility through the membrane.

2.7.3 Membrane materials and filtration methods in beverage industry

In general, there are two main different types of membrane materials that are utilized to form membranes, namely ceramic and polymeric (Judd, 11). A number of configuration and membrane types have been used in MBR applications for the treatment of beverage industry wastewater treatment, which include tubular, rotary disk, hollow fibre, organic (polyethylene, polyethersulfone, polysulfone and polyolefin), metallic, stainless steel, inorganic (for example, zirconia, ceramic) MF and UF membranes (Valderrama *et al.,* 2012; Bilad *et al.,* 2011; Torres, *et al.,* 2011; Wijekoon *et al.,* 2011; Kanai *et al.,* 2010; Satyawali & Balakrishnan, 2009; Matošić *et al.,* 2009; Artiga *et al.,* 2007; Melamane, 2007; Coetzee *et al.,* 2004; Chmiel *et al.,* 2003; Noronha *et al.,* 2002; Blöcher *et al.,* 2002; Miyaki *et al.,* 2000). Most membrane modules used in AnMBRs were implemented by using MF or UF membranes, with either hollow fibre, flat sheet (plate or frame) or tubular configurations due to their high packing density and cost efficiency. Hollow fibre membrane modules are popularly used in iMBRs. In recent years, wastewater pre-treatment in recent years have shifted towards the use of low-pressure membrane processes such as UF and MF (Agana *et al.,* 2013; Judd, 2011; Kim *et al.,* 2008).

The configuration of MBR is based on either cylindrical or planar geometry. There are six main membrane configurations in practise: 1) flat sheet (FS); 2) hollow fibre (HF); 3) (multi) tubular (MT); 4) capillary tube (CT); 5) pleated filter cartridge (FC); and 6) spiral-wound (SW) (Judd, 2011; Henze *et al.,* 2008). However, for MBR only, HF, FS and MT are appropriate since they are easy to clean, and support a high amount of turbulence. HF modules operate with the flow passing from outside to inside (Judd, 2011). Optimal membrane efficiency should be arranged in the following style: 1) a design that allows modularisation that allows easy cleaning; 2) a design that permits easy cleaning; 3) membrane area should be higher to module volume ratio; 3) a high degree of turbulence on the feed side should promote mass transfer; 4) volume of product water produced should require low energy requirements per unit; and 5) membrane low costs (Judd, 2011; Henze *et al.,* 2008).

The removal of TSS present in beverage production wastewater is a significant pre-treatment step throughout water recovery. This guarantees that the effect of fouling on a primary treatment method such as high-pressure membrane filtration (NF and RO) is reduced. Processes that are normally used for the pre-treatment of beverage production wastewater comprises of coagulation/flocculation (Amuda & Amoo, 2007; Amuda *et al.,* 2006), and low-pressure membrane filtration (UF and MF) (Agana *et al.,* 2013; Chmiel *et al.,* 2003). The decision on whether to utilize MF or UF as pre-treatment for a specific wastewater usually depends on the nature and particle size range of contaminants that are present. Usually, UF rejects large dissolved molecules and colloidal particles in the 0.01 to 0.1 µm size range, and MF rejects suspended solids in the 0.1 to 1 µm size range (Agana *et al.,* 2013). Although both types of membranes can normally be used for the pre-treatment of wastewater, an UF membrane is usually favoured in industrial surroundings (Vedavyasan, 2007). UF membrane can successfully eliminate TSS and emulsified oils present in industrial wastewater (Zhang *et al.,* 2008; Karakulski & Morawski, 2000). UF filtrate water quality has been shown to be higher than MF (Teng *et al.,* 2003).

Ceramic membranes are the choice in the pre-treatment of highly polluted industrial wastewater due to their thermal resistance and chemical stability (Lee & Cho, 2004). They function well within a pH range of 1 to 14 and can be operated at temperatures as high as 500˚C (Van Gestel *et al.,* 2002). One of the typical qualities of ceramic membranes is their capability to tolerate high residual chlorine concentrations under alkaline pH (Agana *et al.,* 2013). Other advantages of ceramic membranes include a longer lifetime, lower cleaning frequency and higher average flux (Agana *et al.,* 2013). Although ceramic membranes may offer a number of advantages in comparison to polymeric membranes, their use in industrial wastewater treatment has been restricted due to their high initial cost and their intrinsic brittleness. In addition, sudden temperature changes and pressure surges can cause thermal and physical shocks, respectively, which may fracture the membranes (Agana *et al.,* 2013).

A comparative study was performed by Kang *et al. (*2002) with regard to the filtration characteristics of organic and inorganic membranes in a membrane-coupled anaerobic bioreactor (MCAB). The reactor was maintained at a thermophilic temperature of 55˚C. The two tubular membranes that were chosen for comparison were: a zirconia skinned inorganic membrane with a pore size of 0.14 µm and a hydrophobic polypropylene membrane with a pore size of 0.2 µm; and treating distillery industry wastewater COD of 38 400 mg/L. With acidic pH of 2.0 backflushing, the flux was approximately doubled for the organic membrane. Three types of membranes were tested by Torres *et al.* (2011), namely ceramic membrane of 0.2 µm and two polymeric membranes of 30 nm at different internal diameters of 5 and 8 mm for the treatment of brewery wastewater, which presented an average COD of 21 000 mg/L. Reactor and membrane modules were operated at a temperature of 30˚C and pH of 6.9. Membrane modules were operated with cycles consisting of 5 min of filtration, followed by 30 s of backflush.

2.8 Advantages and disadvantages of membrane bioreactors (MBRs)

The advantages offered by MBR methods over conventional anaerobic systems and aerobic MBR are broadly acknowledged (Lin *et al.,* 2012; Mutamim *et al.,* 2012; Singhania *et al.,* 2012; Judd, 2011; Liao *et al.,* 2006). MBR systems have proven relatively efficient in eliminating organic and inorganic contaminants, as well as biological microorganisms from wastewater. Advantages of the MBR include high quality permeate free of bacteria, good management of biological activity and pathogens, higher organic loading rates and smaller plant sizes (Lin *et al.,* 2012; Mutamim *et al.,* 2012; Singhania *et al.,* 2012; Judd, 2011; Liao *et al.,* 2006; Cicek, 2003). A disadvantage related to MBRs are primarily high capital costs due to fouling problems, which can result in the regular cleaning of membranes (Mutamim *et al.,* 2012; Singhania *et al.,* 2012; Cicek, 2003).

2.9 Membrane fouling and the effect of air scouring

Membrane fouling is initiated by the deposition of colloidal, particulate, or soluble material on the membrane surface or inside the pores. Hydrodynamic forces can be applied to remove deposit on membrane surfaces. However, these forces do not only affect the deposition layer, but might have negative effects on soluble microbial products and a reduction in flocculant dosage. Due to the high costs associated with fouling, a substantial number of studies have focused on the cause and control of fouling in MBRs over the past decade (Böhm *et al.,* 2012). The significance of the multi-phase fluid dynamics for controlling fouling has been generally addressed with air scouring, which is also applied commercially to create positive hydrodynamic flow on the surrounding area of the membrane surface. These flow fields are induced by the complex interactions between flux, cross flows, as well as turbulent eddies, which are created by the increasing the number of bubbles (Böhm *et al.,* 2012; Logsdon, 2011).

Membrane fouling is the main obstacle that holds back quicker commercialization of MBRs. Membrane fouling results in a reduction of permeate flux or an increase of TMP, depending on the operation mode. The permeate flux is defined by the product of water flow, which is divided by the surface area of the membrane (Böhm *et al.,* 2012; Logsdon, 2011; Le-Clech *et al.,* 2006). Permeate flux is calculated according to Equation: 2-20:

$$J = Q/A \qquad \text{Equation: 2-20}$$

Where, J = filtrate flux (L/h.m^2)
Q = flowrate (L/h)
A = Membrane surface area (m^2)

TMP is transmembrane, which is the pressure difference from the average feed concentration to the permeate transversely to the membrane wall. Operating parameters that have been reported to affect membrane performance are TMP, cross flow velocity, pH, as well as temperature (Ramaswamy *et al.,* 2013). Equation: 2-21 represents transmembrane pressure:

$$TMP = P_{f} - P_{p} \qquad \text{Equation: 2-21}$$

Where, TMP = Transmembrane Pressure (kPa)
P_f = feed pressure (kPa)
P_p = permeate pressure (kPa)

2.10 Beverage industry wastewater recycling or reuse

A tertiary wastewater treatment gives attention to the disinfections of wastewater streams. This is generally performed through oxidation methods such as ozonation, chlorination and UV. Low-pressure membrane filtration is more recognized and has advantages over conventional treatment methods. These advantages include: 1) compact modular construction; 2) continuous operation under steady-state conditions; 3) an efficient separation process even without a change of phase; and 4) reliable permeate water quality in spite of differences in the quality of feed water (Agana *et al.,* 2013; Chen, *et al.,* 2006). Membrane treatment methods have also been considered for their disinfectant characteristic, while MF and RO membranes have been used to impart the disinfection of industrial wastewater. One of the objectives and being part of the water-stressed world is to reduce the amount of freshwater, which is utilized by increasing the volume of recycled water. The benefits of recycling industrial wastewater is the reduction in freshwater intake and wastewater disposal cost, as well as saving in chemical costs (Milne *et al.,* 2009; Tamime, 2009).

Although recycling is possible in the food and beverage industry, it is important to consider all regulations, standards and by-laws, including health and safety (Milne *et al.,* 2009; Tamime, 2009). There are direct perceptions of industries that, regardless of the level of treatment methods applied and the quantity of microbial infectivity in the recycled streams, direct contact should be avoided merely because of this perception. Additionally, consumers are reliant on confidence and trust (Tamime, 2009; Casani *et al.,* 2005). Typical uses of recycled water include in-house water replacements for cooling tower make-up and boiler feed, CIP water rinse, floor cleaning, toilet flushing and irrigation. However, CIP, cooling tower make-up and boiler feed are the most intensive water processes. Although mixed wastewater streams may be recycled, most studies have been reported on the treatment and recycling of specific streams. NF permeate can produce treated wastewater that is suitable for boiler feed and RO permeate for cooling towers. Beverage industries usually recycle their wastewater from bottle washing streams (Milne *et al.,* 2009; Tamime, 2009; Mavrov *et al.,* 2001).

Treatment methods such as UF, RO, UV and ion exchange (IE) have been previously investigated for beverage industry wastewater to facilitate reuse (Haroon *et al.,* 2013; Murthy & Chaudhari, 2009; Nataraj *et al.,* 2006; Chmiel *et al.,* 2003; Noronha *et al.,* 2002; Blöcher *et al.,* 2002). Table 2-5 and Table 2-6 indicate that UF, MF, IE and RO units were utilized in areas where discharge requirements were stringent, or direct reuse or recycling of the wastewater was required for both alcoholic and non-alcoholic beverage industries (Haroon *et al.,* 2013; Acharya *et al.,* 2008; Nataraj *et al.,* 2006; Miyaki e*t al.,* 2000; Gao *et al.,* 2007; Artiga *et al.,* 2007; Chmiel *et al.,* 2003; Noronha *et al.,* 2002). These methods have been used for the treatment of distillery wastewater to remove the colour and contaminants, with COD removal efficiencies of up to 99.9%. The treated wastewater was suitable to be reused or recycled for both industrial and domestic purposes (Agarwal *et al.,* 2010; Murthy & Chaudhari, 2009; Acharya *et al.,* 2008; Nataraj *et al.,* 2006; Mohana *et al.,* 2007). The membrane based treatment method for beverage industry wastewater recycling are UF and RO. In both cases the removal efficiencies were higher than 94%. However, salt reduction was higher in RO systems than was predictable (Murthy & Chaudhari, 2009; Tamime, 2009; Acharya *et al.,* 2008; Nataraj *et al.,* 2006; Mavrov & Bélières, 2000).

An integrated membrane filtration process comprised of an NF and ultraviolet (UV) disinfection stage, which was developed to produce water of drinking quality out of polluted fruit juice industry wastewater with an average COD of 4 030 mg/L. A membrane flux of 12-15 L/m^{2}h was achieved at a TMP of 60 to 100 mbar. The pH and temperature were maintained at an average of 8.2 and 26˚C, respectively. The nutrients were adjusted in the bioreactor with C:N and C:P at a ratio of 20:1 and 100:1, respectively, and the bioreactor COD removal rates were above 95% (Blöcher *et al.,* 2002). Similar studies were also performed by Chmiel *et al.* (2003) for fruit juice industry wastewater with COD of 1 800 to 6 600 mg/L for reuse. The NF membrane flux was at 14 L/m^{2}h and the reactor pH was 7.8 to 9. The COD removal rates achieved was also above 95%. A water recycling method was developed and implemented by Miyaki *et al.* (2000) for reuse in a beverage industry that produces both carbonated and non-carbonated softdrinks, while a floating media filtration followed by NF, was used. The COD removal from NF was above 70%. A combination of cartridge filter (CF), RO and IE pilot-scale system was used by Haroon *et al.* (2013) for the treatment of wastewater from a softdrink facility. The integrated system was capable of removing more than 99% of the contaminants.

CHAPTER THREE
MATERIALS AND METHODS

3.1 Background

The softdrink industry wastewater (SDIW) that was used was obtained from a continuous softdrink processing industry (located in the Western Cape, SA), which produces carbonated softdrinks and carbonated flavoured water. The SDIW was a combined stream that was generated from different operations and comprises a blend of water from bottle-washing, colorants, flavoring agents, lubricants from conveyor belts, syrup, cleaning of floors, machinery, pipes and work zones. Water is an essential raw material, which is used in softdrink production, with a consumption of 2.5 to 3.5 L of freshwater per litre of softdrink production (Hsine *et al.,* 2010). The wastewater from the industrial partner was directed into an equalization tank located on-site before being discharged to the municipal sewer. The wastewater that was used in this study was collected from the equalization tank. A laboratory-scale plant comprising of an anaerobic EGSB pre-treatment and MBR post-treatment was utilized for the treatment of this SDIW. The purpose of this study was to perform a technology evaluation on the effectiveness and efficiency of the combined EGSB and MBR technology for the treatment of SDIW, and to access the possibility of wastewater recycling or reuse.

3.2 Softdrink industry potable water

Potable water samples before and after pre-treatment on-site were collected and analysed in order to compare the composition of this water to the drinking water quality standards and guidelines, as specified in SANS 241:2011. Eight 2L potable water samples were collected from the tap every second week and four 2 L samples were collected from the pre-treatment plant when operational. These samples were also taken to compare the treated water from this study. Sample analysis methods will be covered in Section 3.5.1.

3.3 Softdrink industry wastewater (SDIW)

Eight 2 L bottle-washing wastewater samples were collected on-site every second week for analysis. Eight batches of SDIW (500 L each) collected from the equalization tank on-site. Initially it was planned to collect samples every third week, however, to stop the biological activity in the holding tank the SDIW was collected every second week and used as the feed for the EGSB pre-treatment system in this study. After pumping this wastewater into a 1000 L holding tank on campus and circulating for 30 minutes by using a submersible pump a sample was taken and analyzed. Sample analysis will be covered in Section 3.5.1.

3.4 Experimental setup

A four-stage process consisting of a biological (anaerobic, anoxic and aerobic) treatment stages followed by a membrane separation unit, as shown in Figures 3.1, 3.2, 3.3 and 3.4 was utilized for the treatment of the SDIW. The SDIW was fed from the holding tank and passed through a (30 L) settling tank fitted with a 1 mm fine mesh filter, followed by a filter (Effast 0.5 mm cartridge filter) in order to remove any fibrous and/or particulate matter, under a vacuum of between -10 and -100 kPa before entering the anaerobic EGSB pre-treatment phase (Li *et al.,* 2008).

3.4.1 Anaerobic expanded granular sludge bed (EGSB) pre-treatment

The anaerobic EGSB reactor, made of polyvinylchloride (PVC) (ID of 104 mm, 2070 mm high and a 24 L working volume) was incorporated to effectively reduce the organic load of the SDIW prior to aerobic treatment. Refer to the piping and instrumentation diagram (P&ID) in Figure 3.3 for a detailed design of the anaerobic EGSB system. The EGSB design, which was also used in the study by Sheldon *et al.* (2012), was based on the design by Zhang *et al.* (2008).

The discharge stream at the top of the EGSB reactor was divided into two, namely: (i) recirculation stream; and (ii) EGSB product stream. The recirculation stream from the top of the EGSB reactor was mixed with the filtered wastewater feed in a mixing tank to ensure a consistent feed into the bottom of the EGSB reactor. The fluid upflow velocity (V_{up}) was maintained by supplying a wastewater recirculation stream at the bottom of the EGSB to improve mixing. The pH of the EGSB reactor was measured by means of a pH probe, which was located in the stirred mixing tank. The mixing tank is an essential feature for pH control and homogeneous environmental conditions. A dosing pump was installed to regulate the pH, as shown in Figure 3.3. The flowrates from the EGSB feed and EGSB product were measured three times daily. A gas-liquid-solids separator was utilized at the top of the column to allow for separation of the solids and biogas from the liquid phase, as shown in Figure 3.4.

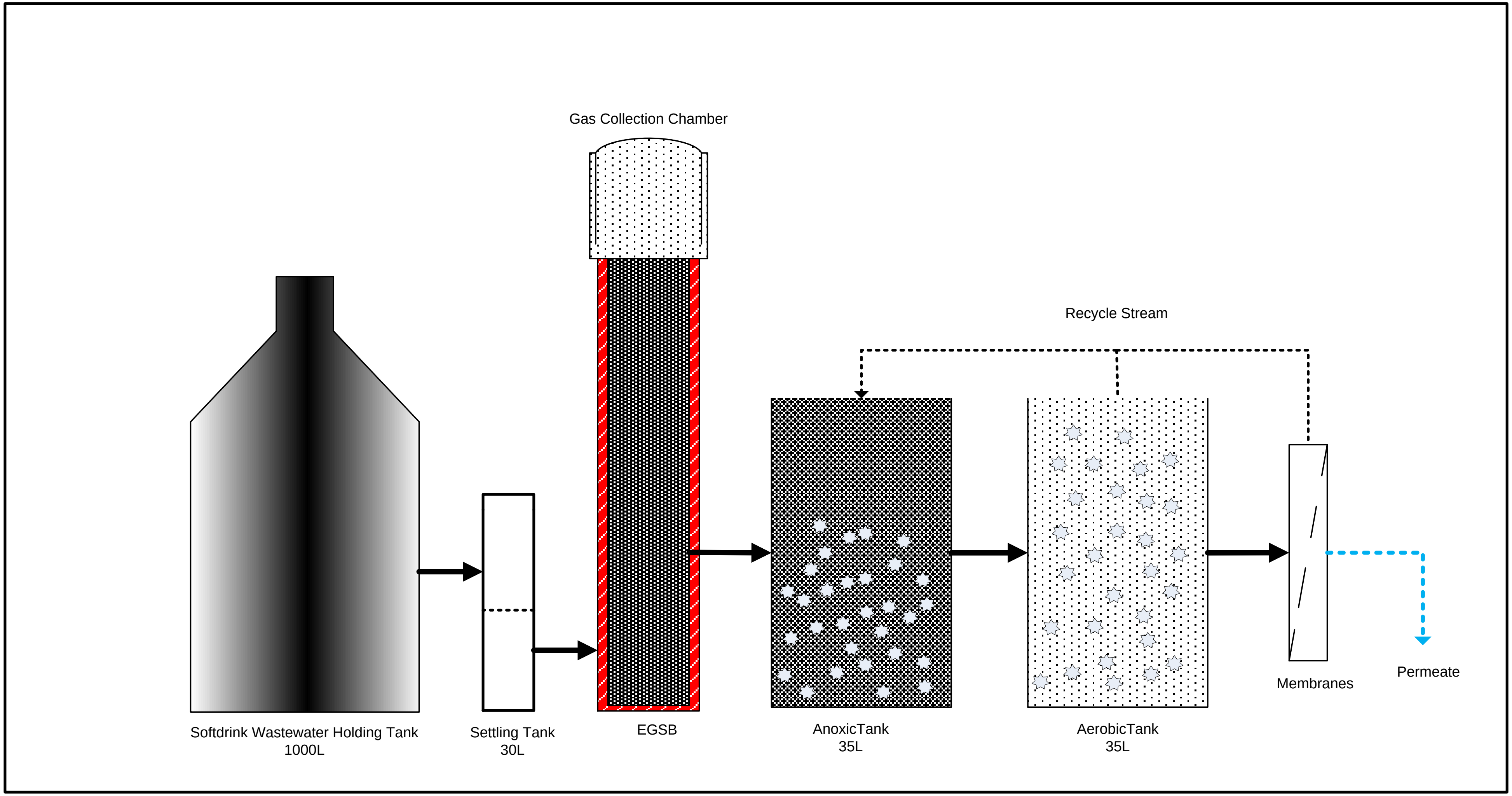


Figure 3.1: Block flow diagram showing the laboratory integrated EGSB and UF-MBR system used in the treatment of softdrink industry wastewater

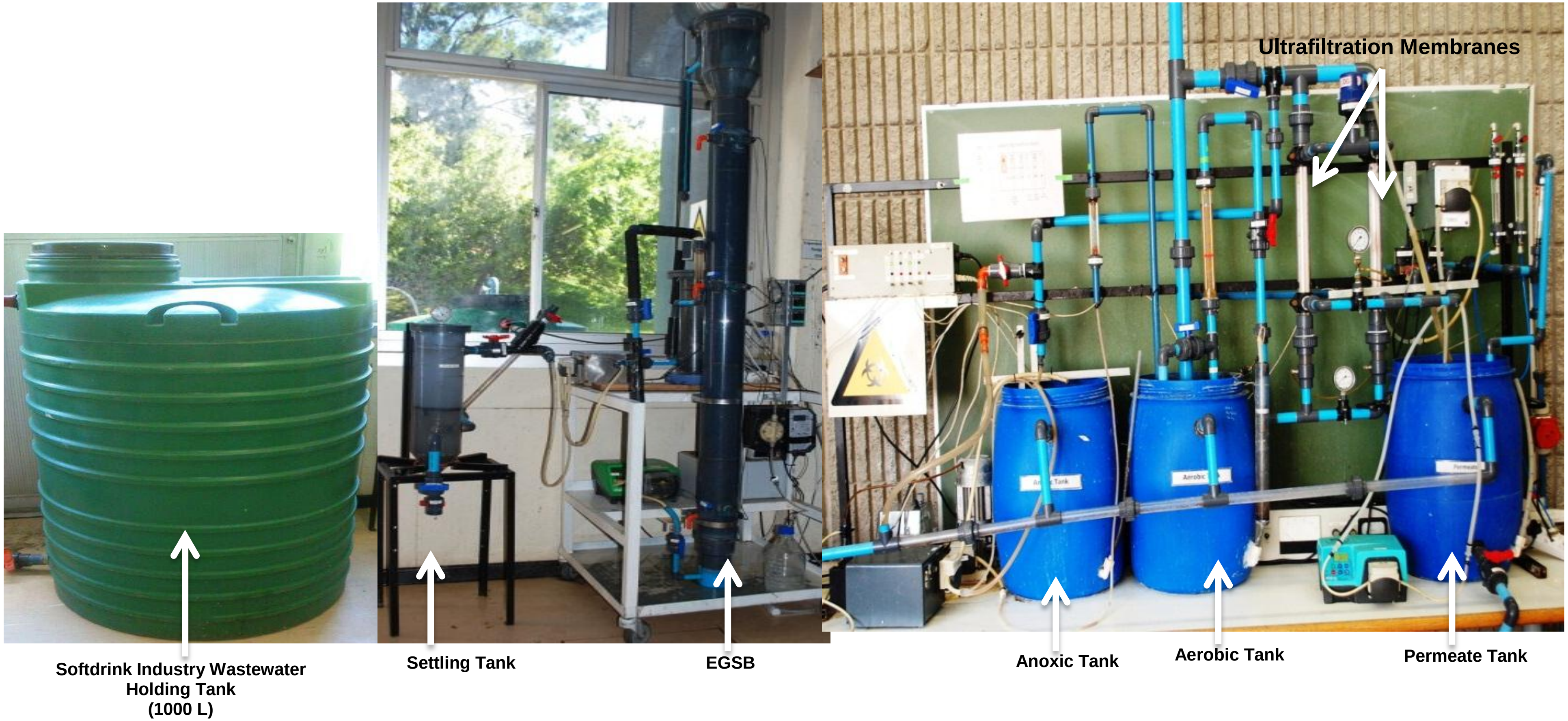
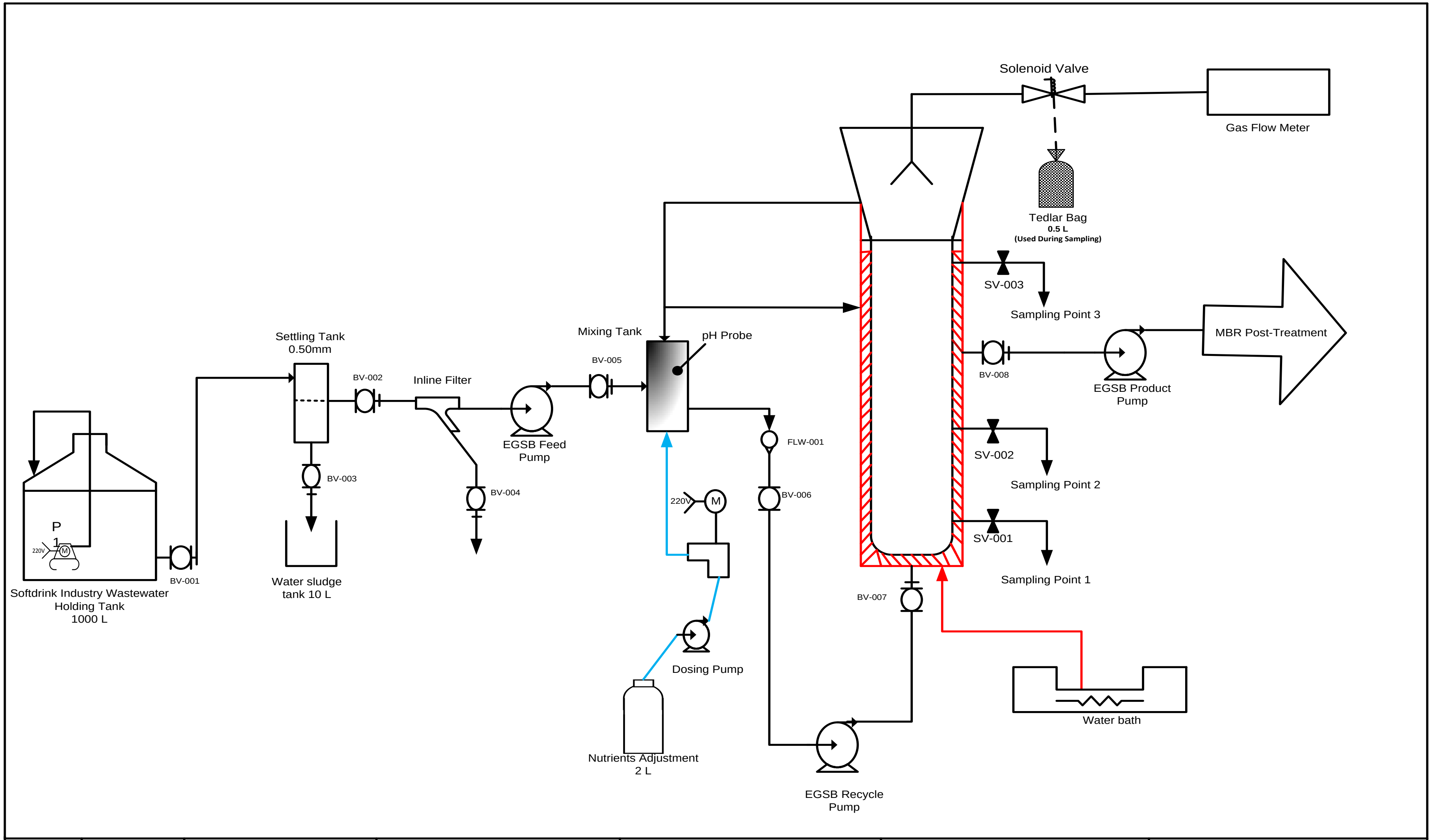


Figure 3.2: Laboratory integrated EGSB and UF-MBR system used in the treatment of softdrink industry wastewater



Revision No.	Date	Description
A	29-06-2011	
B	15-12-2014	Modified

Client: Softdrink Industry
Drawing No.:DWG/CL/022-2011
Scale: NOT TO SCALE

Project: Softdrink Industry Wastewater
Author: I. Erdogan
Checked By: Prof Sheldon
Status: Draft

Drawing Title: P&ID of EGSB Pre-Treatment

Cape Peninsula
University of Technology

Figure 3.3: P&ID of anaerobic Expanded Granular Sludge Bed Reactor (EGSB) pre-treatment [Drawn using Microsoft Visio 2010]

3.4.1.1 EGSB inoculation

The EGSB was inoculated with granular anaerobic inoculum (9 L) with total suspended solids (TSS) of 1 182 mg/L and volatile suspended solids (VSS) of 24.5%. The granular anaerobic inoculum was collected from a full-scale UASB reactor treating brewery wastewater (SABMiller plc, Newlands, South Africa). Activated sludge (AS) (3 L) with a TSS of 1 064 mg/L and VSS of 1.5 % was obtained from the Athlone Wastewater Treatment Works (City of Cape Town, South Africa). This combined inoculum was used in order to reduce the start-up time and acclimatisation period of the EGSB. The EGSB was allowed to stabilise by putting it on a recycle operational mode for 24 h. Prior to inoculation, the SDIW was characterized in terms of its organic matter biodegradability. The SDIW from the industrial partner had a COD of 5 000 mg/L and a BOD of 1 150 mg/L. Due to the nature of the main ingredients used by the softdrink industry during production, sugars are the major pollutant present in the SDIW and can, therefore, readily biodegraded by micro-organisms. This wastewater had a BOD:COD ratio of 0.23, which was a prediction of good biodegradability (Hsine *et al.,* 2010; Chmiel *et al.,* 2003; Guven, 2001; Kalyuzhnyi *et al.,* 1997).

3.4.1.2 EGSB operation and operating conditions

The reactor was equipped with a hot water jacket to maintain a mesophilic temperature of between 35 to 37˚C, which is optimal for AD to occur (Matovic, 2013; Manyi-Loh *et al.,* 2013; Sheldon *et al.,* 2012; Trzcinski & Stuckey, 2009; Monson *et al.,* 2007). During the acclimatization phase, phosphoric acid (H_3PO_4) was used for dosing in order to decrease the feed pH. The EGSB was dosed with 0.5M H_3PO_4, which assisted in attaining the optimum C:N:P ratio of 100:1.8:0.4 (Peixoto *et al.,* 2011). An average COD_t value of 4 197 mg/L requires the ammonium and organically bound nitrogen, as well as the phosphorous concentrations in the wastewater to be 75.6 and 16.8 mg/L, respectively. However, the actual C:N:P ratio was 100:0.09:0.02. Refer to Appendix B1 for the preparation of a 0.5M H_3PO_4 dosing solution. The SDIW had a high pH value of ~ 10.1, which was due to the use of sodium hydroxide (NaOH) solution that was used to clean the bottles. However, it was discovered that the SDIW, without added nutrients can enhance hydrogen generation (Peixoto *et al.,* 2011). Gas chromatograph (GC) showed that the major constituents of the organic matter in the EGSB reactor were acids, alcohols and esters.

In this study the EGSB was overdosed with H_3PO_4 which resulted in a stress period. A stress period is defined as the stability of an anaerobic treatment system, which occurs due to changes in environmental conditions such as pH, temperature and heavy metals (Mudhoo & Kumar, 2013).The EGSB failed during this stress period and as a result, the dosing with H_3PO_4 was stopped after 75 days. A second start-up was initiated.

The C:N:P ratio of the SDIW was appropriate for bioconversion to methane (CH_4), and it was not essential to supplement the EGSB reactor with nutrients during the second start-up, which was in agreement with observation made by Luo *et al.* (2009) and Yu *et al*. (2006) in the treatment of cassava sillage and winery wastewater. The pH of the raw wastewater was adjusted to between 6.5 and 7.2 by using 1M sodium hydrogen carbonate ($NaHCO_3$) for effective AD (Wijekoon *et al.,* 2011; Turkdogan-Aydinol & Yetilmezsoy, 2010; Gohil & Nakhla, 2006). Following acclimatization, the operation of the EGSB was started immediately with the actual SDIW as the carbon source containing feed.

The EGSB operating conditions were measured daily and are shown in Tables 3.1 and 3.2, which were a modification to operational parameters used for the paper mill wastewater treatment (Sheldon *et al.,* 2012) and brewery wastewater treatment (Connaughton *et al.,* 2006). The EGSB was operated for 135 days and the treatment experiments were divided into three different operational modes. Each mode was characterised by an adjustment in the hydraulic retention time (HRT). With each HRT, the V_{up} was changed stepwise, up or down, to determine the performance of the system. According to Sheldon *et al.* (2012), during the treatment of paper mill wastewater, the EGSB had an V_{up} of 1.1 m/h; hence, this was used during the start-up and acclimatization phase. However, on day 22 it was noticed that the reactor bed had risen to the top of the column; and as a result, the V_{up} was decreased to 0.59 m/h to prevent system failure.

3.4.2 Biogas production

Biogas was produced during the anaerobic pre-treatment phase, as explained in Section 2.5.2 in the literature review. A custom made on-line biogas measuring unit, which was obtained from the University of Cape Town (UCT) was installed at the top of the EGSB to measure the flowrate of the biogas that was produced, as shown in Figure 3.4. Biogas production was monitored and measured daily by using a water displacement technique. The volume of water that was displaced from the biogas flow meter was equivalent to the volume of gas that was produced. As soon as the EGSB reached a steady state for each specific operating condition, biogas samples were collected in duplicate by using a 0.5 L Tedlar bag (Méndez-Acosta *et al.,* 2010) as shown in Figure 3.4, which was attached to the biogas exit stream. Biogas samples were sent to J. Muller laboratories (SA) for analysis of the chemical composition. An extraction fan was used to discharge the biogas from the laboratory to the atmosphere.

Table 3.1: EGSB pre-treatment planned operation conditions from literature (Sheldon *et al.*, 2012; Connaughton *et al.*, 2006).

Reactor Operational Conditions	Start-up		Mode-1a	Mode-1b	Mode-2a	Mode-2b	Mode3a	Mode3b
HRT (d)	2.5	2	1	1	0.8	0.8	0.5	0.5
HRT (h)	58.7	48	24	24	18	18	12	12
Feed Rate (L/d)	10	12.2	24.5	24.5	32.6	32.6	48.9	48.9
Feed Rate (L/h)	0.5	0.5	1.0	1.0	1.4	1.4	2.0	2.0
Product Rate (L/d)	10	12.2	24.5	24.5	32.6	32.6	48.9	48.9
Product Rate L/h	0.4	0.5	1.0	1.0	1.4	1.4	2.0	2.0
V_{up} m/h)	1.1	1.1	1.1	0.85	0.85	1.1	1.1	0.85

Table 3.2: EGSB pre-treatment actual operation conditions measured

Reactor Operational Conditions	Acclimatisation		Start-up 1		Stress period		Start-up 2	Mode-1a	Mode-1b	Mode-2a	Mode-2b	Mode3a	Mode3b
Days (d)	0 - 21	22 - 41	42 - 48	49 - 62	63 - 69	70 – 74	77 - 90	91 - 97	98 - 104	105 - 111	112 - 118	119 -125	126 - 135
HRT (d)	2.4 - 2.6	2.4 - 2.6	2 - 2.03	1.9 - 2.0	1 - 1.1	11.0	2.0 - 2.03	1.0	1.0	0.7 - 0.8	0.8	0.5	0.5
HRT (h)	57 - 62.1	58.4 - 61.3	48.2 - 48.6	46 - 48.4	24.1 - 25.1	24.4 – 25	47.6 - 48.8	23.7 - 24.6	24 - 24.1	17.7 - 18	18 - 18.3	12 - 12.5	12.0 - 12.3
Feed Rate (L/d)	9. 5 - 9.8	9.58 - 10.3	12.1 - 12.2	12.1 - 12.8	23.4- 24.4	23.5 - 24.1	12.0 - 12.3	23.8 - 24.9	24.4 - 25.4	32.6 - 34.8	32.2 - 34.6	48.0 – 49.0	48.3 – 49.0
Feed Rate (L/h)	0.4 - 0.4	0.4 - 0.4	0.5 - 0.5	0.5 - 0.5	1.0	1.0	0.5	1.0	1.0-1.1	1.4 - 1.5	1.3 - 1.4	2.0	2.0
Product Rate (L/d)	9.5	9.5	12.3 - 12.4	12.3 - 12.8	23.4 - 24.5	23.6 - 24.4	12.1 - 12.2	24.2 - 24.8	24.1- 24.9	32.3 - 35	33.2 - 33.9	48.0 - 48.8	48.0
Product Rate (L/h)	0.4	0.4	0.51 - 0.52	0.5 - 0.5	1.0	1.0	0.5	1.0	1.0	1.3 - 1.5	1.4	2.0	2.0
V_{up} (m/h)	1.1	0.59	0.85	1.1	1.1	0.85	1.1	1.1	0.85	0.85	1.1	1.1	0.85
OLR (kg COD/m^3d)	0.7 - 1.2	0.7 - 0.9	0.9 - 1	0.6 - 1.9	4.6 - 5.4	4.4 - 4.7	4.4 - 5.4	2.8 - 3.4	2.3 - 2.7	7.6 - 7.9	6.2 - 7.2	10.9 - 13.2	8.2 - 9.9

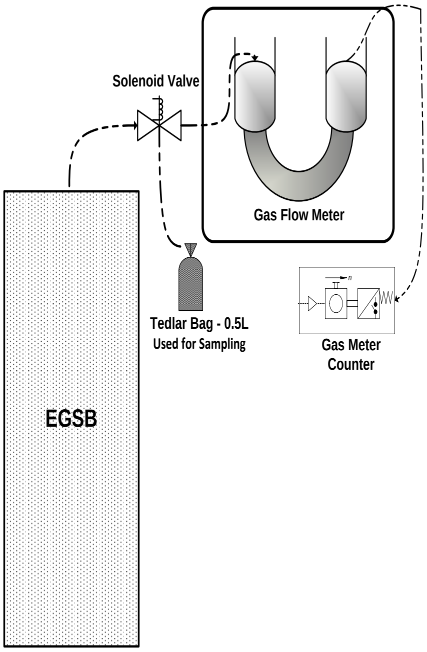


Figure 3.4: Schematic representation of biogas measuring unit [Drawn using Microsoft Visio 2010]

3.4.3 UF-Membrane bioreactor (MBR) post-treatment for softdrink industry wastewater

A modified Ludzack-Ettinger (MLE) process configuration (Spellman, 2013) was adopted for the biological as post-treatment phase, as shown in Figure 3.5. Denitrification and nitrification were achieved with an anoxic zone (35 L), followed by an aerobic zone (35 L), respectively. Both zones were inoculated with 10 L of sewage sludge, which were obtained from Athlone Wastewater Treatment Works (City of Cape Town, South Africa), with a TSS concentration of 1 064 mg/L and VSS of 15%. The anoxic and aerobic zones both contained 30 plastic bioballs of about 3 cm in diameter, which were used as biofilm carriers on which the biomass was immobilised (Ng *et al.,* 2011). The biomass in the anoxic zone was kept in suspension by using a HP-6606 submersible pump. The flowrates (Q) from the anoxic and aerobic zones, as well as from the ultrafiltration (UF) permeate, were measured thrice daily. Aeration was accomplished by means of air diffusers, which were located at the bottom of the aerobic zone. Aeration with specified dissolved oxygen (DO) concentrations in the aerobic zone was monitored based on the operating conditions of 2, 2.8 and 3.7 mg/L DO (Sheldon *et al.,* 2012; Brito *et al.,* 2007).

The product from the aerobic zone was fed through two airlift UF-membrane modules 0.058 m^2 per module, with inner diameter of 0.065 m and active length of 0.47 m, each consisting of six ceramic aluminium oxide (Al_2O_3) tubular membranes with pores of 0.2 and 0.4 µm, respectively. Permeate was extracted on the shell side of the membrane modules, while the retentate was recycled back to the anoxic and aerobic zones. The feedrate to the UF-membranes was dependant on the operating conditions of the EGSB product flowrate. The average feed flowrate to the UF membranes was approximately 128.2 L/d for both modules, of which the retentate had to be recycled back to the anoxic and aerobic zones to provide ideal denitrification (Spellman, 2013). Correspondingly, membrane fouling was preventatively controlled through continuous air scouring, which was gradually increased from 0.5 to 2.5 L/min per module (Sheldon *et al.,* 2012; Zhang *et al.,* 2006) on the lumen side of the membranes. Further fouling prevention included a backflush system to the membrane modules by using reverse permeate flow, controlled via a cyclic timer. Following 5 min of the forward cycle, the backflush cycle would start for 7 s at a set flowrate, pumping UF permeate back into the membranes.

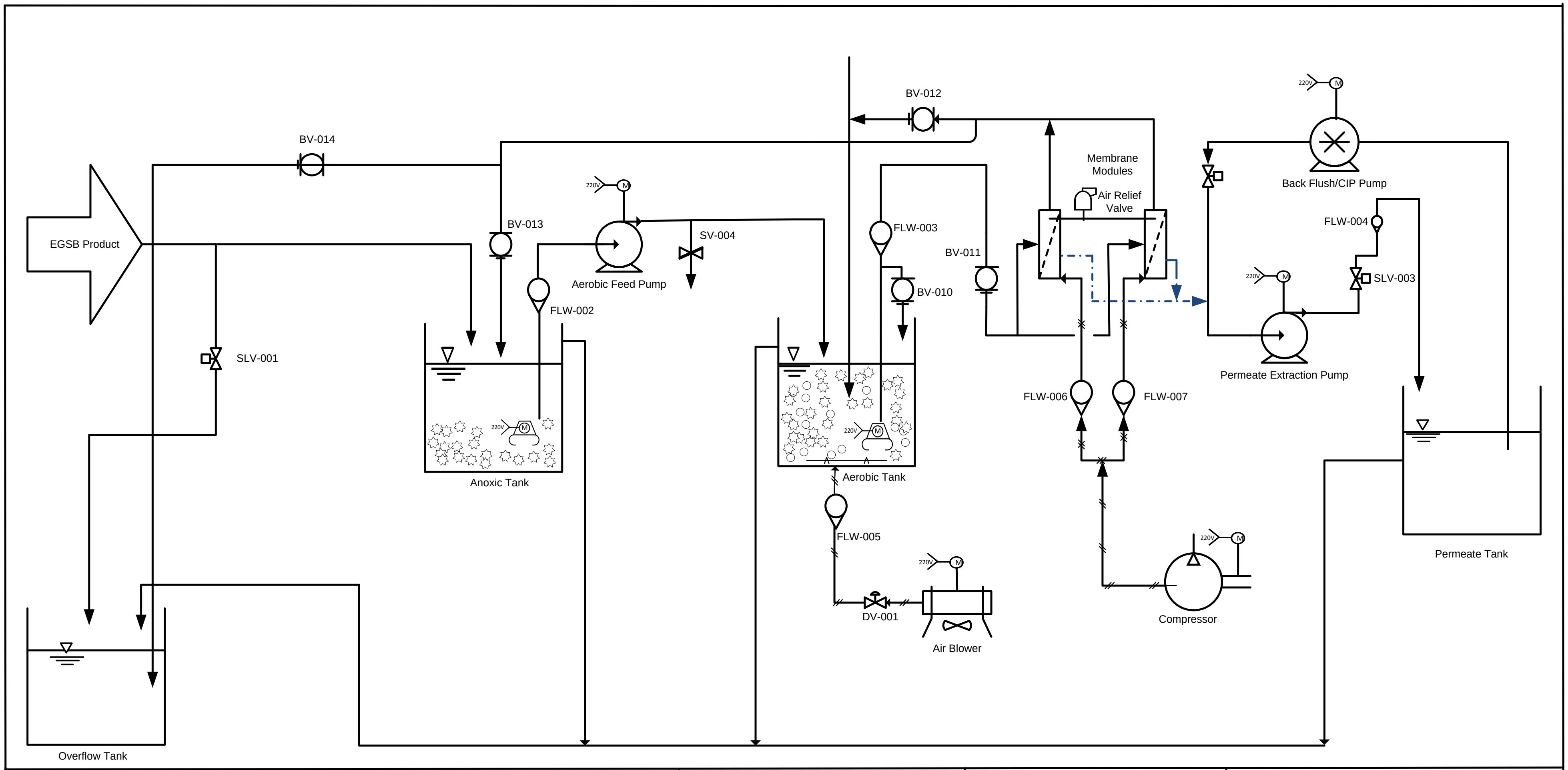
3.4.3.1 UF-MBR operating conditions

The MBR phase was started 36 days after the EGSB phase to allow the EGSB to first stabilize. The MBR was put on recycle for 24 h during this period with an average mixed liquor suspended solid concentration (MLSS) in both anoxic and aerobic zones of 530 and 455 mg/L, respectively. The UF-MBR was operated for 95 days starting with 10 L/d EGSB product as the carbon source with a COD_t concentration of approximately 1 650 mg/L. The system was allowed to acclimatize for 3 days. The MBR was operated for 13 days during Mode-1a of operation without bioballs. The target operating conditions were adjusted from Sheldon *et al.* (2012) and Brito *et al.* (2007), which were used for paper mill and brewery wastewater treatment, respectively, and are presented in Table 3.2. The DO was measured daily in both the anoxic and the aerobic zones by using the YSI Professional Plus (Pro Plus-No.: 605596) handheld multiparameter analyser. The flowrates and HRTs for the different operating modes are indicated in Table 3.3 and the flux through the membranes was calculated using equation 2-20, as shown in Appendix E.

The overall MBR HRT was gradually decreased from 14.4 to 3.3 h, as shown in Table 3.3. The MBR was operated in three sequential modes (Modes-1 to -3) with the feed flowrate, ranging from 12.2 to 48.9 L/d and corresponding UF membrane flux gradually increasing from 1.4 to 5.6 L/m^2h for each mode, as shown in Table 3.3.

3.4.3.2 Cleaning-in-Place (CIP) of ultrafiltration (UF) membranes

The UF membranes were chemically cleaned-in-place (CIP) by using 400 mg/L sodium hypochlorite (NaClO) solution to remove any bio-fouling and a 1% citric acid solution to remove any chemical fouling and scaling, according to the Norit X-Flow manufacturer specifications, every two weeks. CIP was performed on the UF-membrane modules initially once every two weeks. However, when increased fouling of the membranes was noticed when the backwash pressure reached 60 to 70 kPa, CIP was performed weekly.



Revision No.	Date	Description	Client:	Softdrink Industry	Project:	Softdrink Industry Wastewater
A	29-06-2011		Drawing No.:	DWG/CLI/022-2011_022	Author:	I. Erdogan
B	20-12-2014	Modified	Scale:	NOT TO SCALE	Checked By:	Prof Sheldon
			Status:	DRAFT	Drawing Title:	MBR P&ID Post-Treatment

Figure 3.5: P & ID of the Modified Ludzack-Ettinger (MLE)-MBR post-treatment phase [Drawn using Microsoft Visio 2010]

During CIP the anoxic and aerobic zones were maintained in the recycle mode, while the UF membranes were soaked with 400 mg/L NaOCl for 24 h, followed by a 1% citric acid for 4 h to remove any inorganic matter, including scaling from the membranes. Refer to Appendix B2 for the preparation of the CIP solutions.

Table 3.3: MBR actual operating conditions

Operating parameters	Start-up	Mode-1a	Mode-1b	Mode-2a	Mode-2b	Mode-3a	Mode-3b
Days (d)	1- 3	4 -17	20 - 29	37 - 48	51 – 63	64 - 73	79 - 95
Overall MBR HRT(d)	0.6	0.5	0.3	0.5	0.3	0.2	0.1
Overall MBR HRT (h)	14.4	11.9	6.1	11.9	6.2	4.6	3.3
HRT Aerobic tank (35L) (d)	0.02	0.02	0.02	0.02	0.02	0.02	0.02
HRT Aerobic tank (35L) (h)	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Feed to anoxic (L/d)	10	12.2	24.8	12.2	24.1	33.7	48.8
Feed to anoxic (L/h)	0.4	0.5	1.0	0.5	1.0	1.4	2.0
Recycle from membrane modules to anoxic tank (L/d)	50.0	61.0	123.8	61.0	120.7	168.6	243.8
Recycle from membrane modules to anoxic tank (L/h)	2.1	2.5	5.2	2.5	5.0	7.0	10.2
HRT Anoxic tank (35L) (d)	0.6	0.5	0.2	0.5	0.2	0.2	0.1
HRT Anoxic tank (35L) (h)	14.0	11.5	5.7	11.5	5.8	4.2	2.9
Feed to aerobic (L/d)	60.0	73.2	148.6	73.2	144.8	202.3	292.6
Feed to aerobic (L/h)	2.5	3.1	6.2	3.1	6.0	8.4	12.2
Recycle from membrane modules to aerobic tank (L/d)	198	1 966	1 892	1 968	1 898	1 841	1 752
Recycle from membrane modules to aerobic tank (L/h)	82.5	82.0	78.8	82.0	79.1	76.7	73.0
Feed to membrane modules (L/d)	2 040	2 040	2 041	2 042.	2 043	2 044	2 045
Feed to membrane modules (L/h)	85.0	85.0	85.0	85.1	85.1	85.2	85.2
Total recycle from membrane modules (L/d)	2 030	2 027	2 016	2 029	2 018	2 010	1 996
Total recycle from membrane modules (L/h)	84.6	84.5	84.0	84.6	84.1	83.7	83.2
Permeate flowrate (L/d)	10.0	12.2	24.8	12.2	24.1	33.7	48.8
Permeate flowrate (L/h)	0.4	0.5	1.0	0.5	1.0	1.4	2.0
Flux (J) (L/m^2h)	3.4	4.3	8.6	4.3	8.6	12.1	17.2

3.5 Analytical methods

Four samples were collected from four different points at the softdrink industrial partner, namely potable water (2L), pre-treated water (2L), bottle-washing water (2L), and 500 L of wastewater from the equalizing tank every second week, and were analysed. HRT samples were also taken for each biological treatment stage during the entire study and subsequently labelled HRT1, HRT2, HRT3 and HRT4. For sugar analysis, the samples were filtered by using a 45 µm filter unit, centrifuged at 13 000 rpm for 10 min to remove the supernatant and stored at -80˚C before analysis with a high performance liquid chromatography (HPLC).

3.5.1 Softdrink industry water and wastewater

The softdrink industry potable, pre-treated water and wastewater samples that were collected were analysed for: total chemical oxygen demand (COD_t); soluble chemical oxygen demand (COD_s); colour; electrical conductivity (EC); ammonium concentration (NH_4^+); nitrate concentration (NO_3^-); pH; orthophosphate (PO_4^{3-}); sugars, namely: fructose and sucrose; temperature; total dissolved solids (TDS); total suspended solids (TSS); and turbidity. Refer to Appendix C for COD, PO_4-P, TSS and turbidity measurement methods. Selected samples were also sent to independent South African National Accreditation System (SANAS) accredited laboratories (Bemlab, South Africa and Scientific Services, City of Cape Town) for full chemical water analysis. Biological oxygen demand (BOD_5); fats oil and grease (FOG) samples were sent to an independent accredited laboratory (Scientific Services, CCT) and were analysed using EPA (1983 method 405.1), and for sugar analysis samples were sent to the Biocatalysis and Technical Biology (BTB) Research laboratory (CPUT) and analysed using HPLC method.

3.5.2 Softdrink industry wastewater (SDIW) in the holding tank

SDIW samples were collected weekly from the holding tank of the lab-scale treatment plant, and analysed, as indicated in Table 3.4 The following parameters were analysed: COD_t; COD_s; colour; EC; NH_4^+; NO_3^-; pH; PO_4^{3-}; sugars, namely: fructose and sucrose; temperature; TDS; TSS and turbidity.

Table 3.4: Softdrink industry wastewater (SDIW) analysis in the holding tank

Parameters	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
COD_t (mg/L)	√		√		√		
COD_s (mg/L)	√		√		√		
Colour (mg Pt/L)	√		√		√		
EC (µS/cm)	√	√	√	√	√	√	√
NH_4-H (mg/L)	√	√	√	√	√	√	√
NO_3-N (mg/L)	√	√	√	√	√	√	√
pH	√	√	√	√	√	√	√
PO_4^{3-} (mg/L)	√		√		√		
Sugars (mg/L)	√		√		√		
TDS (mg/L)	√	√	√	√	√	√	√
Temperature (°C)	√	√	√	√	√	√	√
TSS (mg/L)	√		√		√		
Turbidity (NTU)	√		√		√		

3.5.3 Sample analysis during EGSB pre-treatment phase

Samples from the EGSB feed and product were collected and analysed, as indicated in Table 3.5. The following parameters were analysed: COD_t; COD_s; colour; EC; NH_4^+; NO_3^-; pH; PO_4^{3-}; sugars namely; fructose and sucrose; temperature; TDS; TSS and turbidity.

Table 3.5: EGSB feed and product analysis

Parameters	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
COD$_t$ (mg/L)	√		√		√		
COD$_s$ (mg/L)	√		√		√		
Colour (mg Pt/L)	√		√		√		
EC (µS/cm)	√	√	√	√	√	√	√
NH_4^+ (mg/L)	√	√	√	√	√	√	√
NO_3^- (mg/L)	√	√	√	√	√	√	√
pH	√	√	√	√	√	√	√
PO_4^{3-} (mg/L)	√		√		√		
Sugars (mg/L)	√		√		√		
TDS (mg/L)	√	√	√	√	√	√	√
Temperature (^oC)	√	√	√	√	√	√	√
TSS (mg/L)	√		√		√		
Turbidity (NTU)	√		√		√		

3.5.2 EGSB biogas analysis

Biogas samples were collected in duplicate per specific operating condition as soon as the system stabilised by using 0.5 L Tedlar bags. The composition of the biogas was analysed by using gas chromatography at J. Muller laboratories (SA).

3.5.3 MBR post-treatment phase sample analysis

Water samples that were collected from the anoxic and aerobic zones, as well as UF permeate from the MBR, were analysed, as indicated in Table 3.6: NH_4^+; COD$_t$; COD$_s$; colour; EC; NO_3^-; pH; PO_4^{3-}; sugars, namely: fructose and sucrose; temperature; TDS; TSS and turbidity.

Table 3.6: MBR post-treatment analysis

Parameters	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
COD$_t$ (mg/L)	√		√		√		
COD$_s$ (mg/L)	√		√		√		
Colour (mg Pt/L)	√		√		√		
DO (mg/L)	√	√	√	√	√	√	√
EC (µS/cm)	√	√	√	√	√	√	√
NH_4^+ (mg/L)	√	√	√	√	√	√	√
NO_3^- (mg/L)	√	√	√	√	√	√	√
pH	√	√	√	√	√	√	√
PO_4^{3-} (mg/L)	√		√		√		
Sugars (mg/L)	√		√		√		
TDS (mg/L)	√	√	√	√	√	√	√
Temperature (^oC)	√	√	√	√	√	√	√
TSS (mg/L)	√		√		√		
Turbidity (NTU)	√		√		√		

The internal laboratory analysis was performed on all samples by means of the following methods, as presented in Table 3.7. All methods that were used are standard established analytical methods with certificates, and are explained in Appendix C.

Table 3.7: In-house analysis for EGSB pre-treatment and MBR post-treatment

Parameters	Method	References
COD$_t$ (mg/L)	Merck Spectroquant test kits No. 1.14555.0001	**Appendix C1**
COD$_s$ (mg/L)	Merck Spectroquant test kits No. 1.09773.0001	**Appendix C1**
Colour (mg Pt/L)	NOVA60 Spectroquant	
DO (mg/L)	YSI Professional Plus (Pro Plus-No.: 605596)	**-**
EC (µS/cm)	PCSTestr 35	**-**
NH_4^+ (mg/L)	YSI Professional Plus (Pro Plus-No.: 605596)	**-**
NO_3^- (mg/L)	YSI Professional Plus (Pro Plus-No.: 605596)	**-**
pH	PCSTestr 35	**-**
PO_4^{3-} (mg/L)	Merck Spectroquant test kit No. 1.14543.0001	**Appendix C4**
Sugars (mg/L)	HPLC	**HPLC**
TDS (mg/L)	PCSTestr 35	**-**
Temperature (^oC)	PCSTestr 35	**-**
TSS (mg/L)	ESS Method 340.2	**Appendix C2**
Turbidity	Turbidimeter TN-100	**Appendix C3**

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Softdrink industry potable water (municipal water)

The softdrink industry uses potable water supplied by the municipality for various purposes such as 1) the actual preparation of the product; 2) processing and equipment, including bottle-washing, boilers and refrigeration; and 3) general tasks such as cleaning of walls, surfaces, floors, trucks, gardening and sanitizing processing vessels. The purity and quality of the potable water is of primary concern since it is used to produce products that will be consumed.

Softdrink industry comprises about 87% to 92% of potable water, which makes up the volume of a carbonated softdrink and 100% of bottled water (Shachman, 2004). Potable water intake by the softdrink industry is 2.5 to 3.5 m^3 H_2O/m^3 of softdrink that is produced (Almukhtar, 2012; CSIR, 2010). This potable water often contains trace amounts of different ions that can alter the taste of the final product. The potable water supplied by the municipality, may vary in chemical and physical characteristics due to environmental and seasonal changes, contamination in pipelines, and other problems encountered at municipal treatment plants. Potable water supplied for human consumption in most cases meets stringent health standards and quality as set by South African National Standard (SANS 241:2011).

The potable water is usually pre-treated further on-site before it is used in order to ensure uniformity of the finished product. The potable water of the industrial partner that was used in this study was pre-treated separately on-site before being used in the production process to meet product quality specifications. Figure 4.1 presents a flow diagram of a typical softdrink manufacturing process.

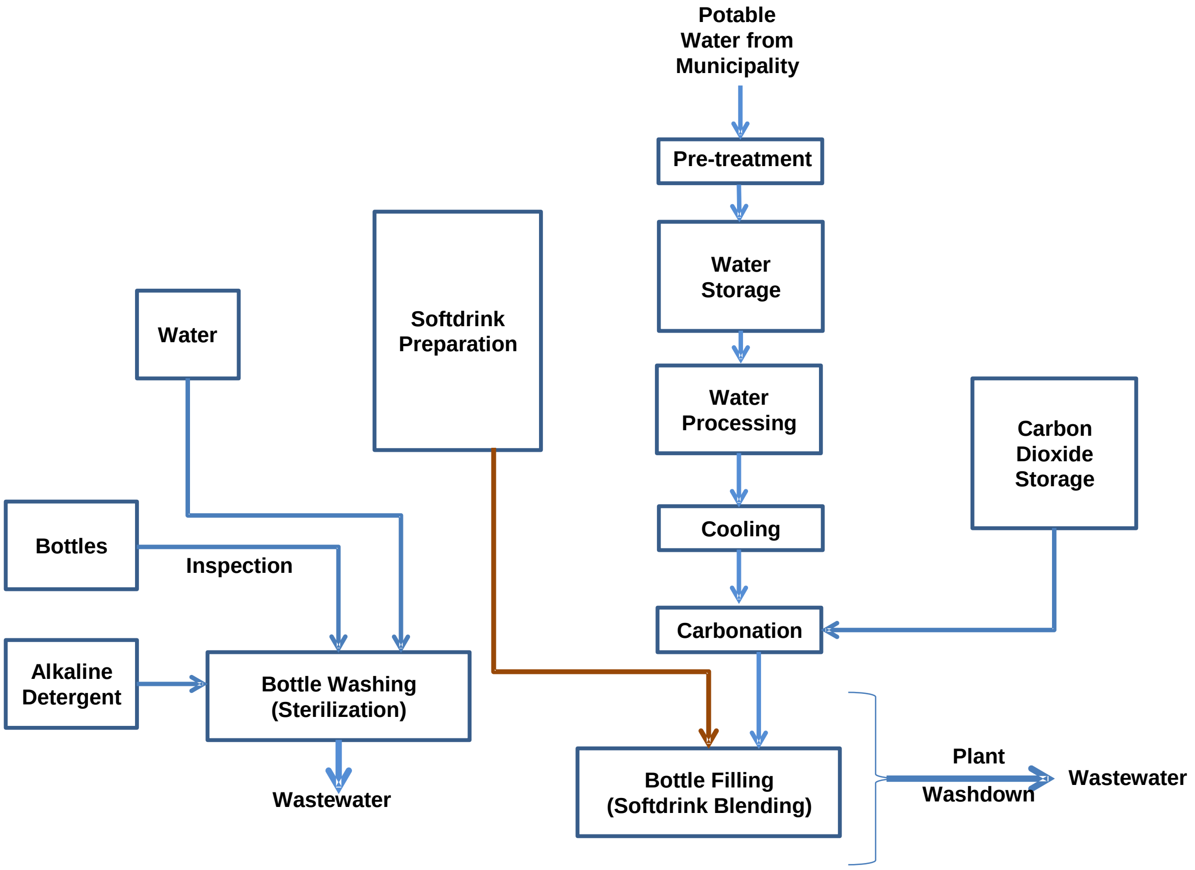


Figure 4.1: Schematic diagram of softdrink manufacturing processes (modified from Woodard, 2001). [Drawn using PowerPoint 2010]

4.1.1 Potable water characteristics for industrial partner

The composition of the industrial partner's potable water and pre-treated water is presented in Table 4.1, along with the SANS 241:2011 and (European Council drinking water standards 80/778/EEC:1980). The physical characteristics, which affect the potable water quality, are pH, conductivity, colour, and turbidity. The potable water pH ranged from 7.7 to 10.5 with an average of 9. The total dissolved solids (TDS) values varied from 52.8 to 80.7 mg/L, with an average value of 62.3 mg/L. The average hardness, which includes copper, manganese, iron, calcium and magnesium, was 0.02, 0.01, 0.04, 13.4 and 0.9 mg/L; respectively, and is an additional concern for the softdrink industry (Haroon *et al.,* 2013). The alkali earth ions, which are sodium (Na^+) and potassium (K^+) values ranged from 3.9 to 13.1 mg/L and from 0 to 1.1 mg/L, respectively. The organic matter present in the form of COD varied from 59.2 to 157 mg/L, with an average of 105.6 mg/L, which was too high when compared to COD of 2 mg/L stipulated by the Council Directive 80/778/EEC:1980 in relation to the quality of water intended for softdrink production. Turbidity ranged from 0.2 to 1.1 NTU, while TSS fluctuated between 8 and 17 mg/L, and sugars in the form of fructose and sucrose were less than 1.7 mg/L and 6.3 mg/L, repetitively. Both physio-chemical properties and inorganic properties were within the suitable limit stipulated by SANS 241:2011.

Table 4.1: Composition of potable water, reverse osmosis water for industrial partner, SANS 241 and European Council 80/778/EEC

Parameters	Potable Water			Pre-treated Water			SANS 241^^	European Council 80/778/EEC^^
	Minimum	Maximum	Average	Minimum	Maximum	Average		
Physio-chemical properties								
pH	**7.7**	**10.5**	**9.0**	**7.3**	**7.5**	**7.4**	**≥5 ≤ 9.7**	**≥6.5 ≤ 8.5**
Temperature (°C)	**13.8**	**25.0**	**20.9**	**19.0**	**23.3**	**21.2**	**-**	**-**
Alkalinity (mg/L)	**11.0**	**30.1**	**22.8**	**4.0**	**5.0**	**4.5**	**-**	**-**
Conductivity (µS/cm)	**51.3**	**92.9**	**79.0**	**1.0**	**2.8**	**1.6**	**≤ 170**	**500**
TDS (mg/L)	**52.8**	**80.7**	**62.3**	**2.3**	**7.0**	**4.8**	**≤1 200**	**500**
COD_t (mg/L)	**59.2**	**157**	**105.6**	**2.0**	**2.7**	**2.4**	**-**	**2**
COD_s (mg/L)	**46.5**	**145**	**92.7**	**1.5**	**2.5**	**2**	**-**	**-**
BOD_5 (mg/L)	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
Turbidity (NTU)	**0.2**	**1.1**	**0.8**	**0.01**	**0.2**	**0.1**	**≤ 1**	**1**
TSS (mg/L)	**8.0**	**17.0**	**11.8**	**3.0**	**9.0**	**5.3**	**-**	**-**
FOG (mg/L)	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
Colour (mg Pt/L)	**<1**	**<1**	**<1**	**0.0**	**0.0**	**0.0**	**≤ 15**	**20**
Fructose (mg/L)	**-**	**-**	**-**	**-**	**-**	**-**	**-**	**-**
Sucrose(mg/L)	**<1.7**	**<1.7**	**<1.7**	**0.1**	**0.1**	**0.1**	**-**	**-**
Inorganic components								
Sodium	**3.9**	**13.1**	**7.1**	**3.7**	**5.3**	**4.5**	**≤ 200**	**150**
Boron (mg/L)	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**-**	**-**
CO_3 (mg/L)	**0.0**	**1.5**	**0.8**	**-**	**-**	**-**	**-**	**-**
HCO_3 (mg/L)	**1.5**	**41.9**	**15.7**	**-**	**-**	**-**	**-**	**-**
Calcium (mg/L)	**7.7**	**17.3**	**13.4**	**0.4**	**13.5**	**7.0**	**-**	**-**
Chloride (mg/L)	**11.1**	**14.5**	**12.4**	**3.4**	**6.8**	**5.1**	**≤ 300**	**20**
Fluoride (mg/L)	**0.0**	**0.4**	**0.1**	**0.0**	**0.3**	**0.1**	**≤ 1.5**	**1.5**
Ammonium (mg/L)	**0.0**	**0.8**	**0.4**	**0.0**	**0.6**	**0.4**	**≤ 1.5**	**-**
Nitrate (mg/L)	**0.0**	**0.7**	**0.3**	**0.0**	**0.5**	**0.3**	**≤ 11**	**1.5**
Potassium (mg/L)	**0.0**	**1.1**	**0.6**	**0.2**	**0.6**	**0.4**	**-**	**-**
Magnesium (mg/L)	**0.5**	**1.4**	**0.9**	**0.0**	**0.8**	**0.4**	**-**	**-**
Sulphates (mg/L)	**3.2**	**7.3**	**5.5**	**0.0**	**0.01**	**0.01**	**≤ 250**	**10**
Zinc (mg/L)	**0.0**	**0.03**	**0.01**	**0.0**	**0.001**	**0.01**	**≤ 5**	**5**
Copper (mg/L)	**0.0**	**0.02**	**0.01**	**-**	**-**	**-**	**≤ 2**	**3**
Iron (mg/L)	**0.0**	**0.1**	**0.04**	**0.0**	**0.0**	**0.0**	**≤ 0.3**	**0.2**
Manganese (mg/L)	**0.0**	**0.01**	**0.01**	**0.0**	**0.0**	**0.0**	**≤0.1**	**0.03**
Phosphate (mg/L)	**0.0**	**0.8**	**0.3**	**0.2**	**0.7**	**0.41**	**-**	**3**
Orthophosphate (mg/L)	**0.4**	**1.0**	**0.7**	**-**	**-**	**-**	**-**	**-**

^^: SANS 241-1:2011 South African National Standard (SANS 241) (Department of Water Affairs, 2011)
^^: Council Directive 80/778/EEC relating to the quality of water intended for human consumption (European Union 1980)

In Europe the minimum prerequisites of all potable water used in softdrink production must meet the requirements in terms of drinking water standards European Council 80/778/EEC (1980). However, according to Steen and Ashurst (2008), all softdrink manufacturers must have their individual potable water standards.

4.1.2 Pre-treated water characteristics of the industrial partner

Softdrink producers have used pre-treatment plant for several years in addition to other treatment steps to bring potable water to within ingredient quality (Almukhtar, 2012). Pre-treated water treatment contributes a significant amount to the softdrink production. The purifying plant is effective for the removal of water impurities, including salt forming ions, microorganisms and any other pollutants (Ioannou *et al.,* 2013; Zhou *et al.,* 2012; Judd, 2011). It is observed from Table 4.1 that the physio-chemical parameters and inorganic compounds present in the potable water decreased after pre-treatment. Pre-treatment is used for the high rejection of the average organic matter [alkalinity from 22.8 to 4.5 mg/L; COD_t from 105.6 to 23.5 mg/L; sucrose from 6.3 to 1.9 mg/L; EC from 79 to 1.6 µS/cm; TSS from 11.8 to 5.3 mg/L; TDS from 62.3 to 4.8 mg/L and ions (calcium of 13.5 mg/L; chloride of 6.8 mg/L; magnesium of 0.4 mg/L)]. However, the pre-treatment plant could be difficult to maintain and operate, and as a result COD_t was up to 2.7 mg/L. This could have been due to fouling.

4.1.3 Bottle-washing wastewater

One of the most significant processes in softdrink production is bottle-washing. The quality of the final product is subjected mainly to how thoroughly the bottles are washed instantly before filling. The bottle-washing process begins with the passing of the returnable bottles through a washer and rinser in sequence. Then, the washed bottles are inspected carefully before they automatically go through sequential stages of filling, blending, capping, labelling, packaging and wrapping (Almukhtar, 2012; Hsine *et al.,* 2010; Miyaki *et al.,* 2000). According to Eckenfelder and Cleary (2013), Camperos *et al.* (2004) and Ramirez *et al.* (2004) water from bottle-washing accounts for 50% of the total softdrink industry wastewater and the consumption of water in the bottle-washing units reach 16 m^3/d for cleaning processes (Almukhtar, 2012). Therefore, this warrants a comprehensive investigation. A summary of chemical analysis of the bottle-washing process wastewater, compared to the CCT industrial wastewater by-laws is presented in Table 4.2. The pH ranged from neutral to slightly alkaline at between 7 and 9.9 with an average of 8.5. The contaminating flow was between 99 to 1 065 mg/L COD_t with an average of 453.8 mg/L, while BOD values of less than 1 mg/L were recorded. The main contamination is in dissolved form COD_s up to 997.5°mg/L. TDS; turbidity; TSS values varied up to 73.8 mg/L, 1.2 NTU; and 11 mg/L, respectively.

Table 4.2: Physico-chemical properties of softdrink industry wastewaters and City of Cape Town by-laws, 2006

Parameters	**Bottle-washing wastewater**			**Softdrink industry wastewater**			**EGSB product**			**City of Cape Town By-Laws^^**
	Minimum	**Maximum**	**Average**	**Minimum**	**Maximum**	**Average**	**Minimum**	**Maximum**	**Average**	
Physiochemical properties										
pH	**7.0**	**9.9**	**9.0**	**6.1**	**11.8**	**9.0**	**6.6**	**8.9**	**7.3**	**5.5 -12.0**
Temperature (^{o}C)	**13.7**	**24.2**	**20.0**	**17.3**	**25.2**	**25.1**	**18.8**	**28.0**	**22.3**	**0 - 40**
Alkalinity (mg/L)	**20.1**	**35.1**	**28.0**	**160.6**	**589.9**	**348.1**	**-**	**-**	**-**	**-**
EC (µS/cm)	**72.2**	**86.7**	**80.0**	**300.6**	**1 483**	**660.1**	**524.0**	**7 311**	**4 057**	**≤ 500**
TDS (mg/L)	**52.0**	**73.8**	**59.0**	**226**	**1 050**	**461.9**	**356.0**	**4 674**	**2 725**	**1 000**
COD_t (mg/L)	**99.0**	**1 065**	**453**	**2 242**	**11 717**	**5 533**	**240.0**	**4 373**	**2 245**	**≤ 5 000 mg/L**
COD_s (mg/L)	**84.5**	**997.5**	**404**	**2 217**	**11 345**	**5 220**	**187.0**	**4 163**	**2 051**	**-**
BOD_5 (mg/L)	**<1**	**<1**	**<1**	**400**	**1 150**	**640.0**	**-**	**-**	**-**	**-**
Turbidity (NTU)	**0.6**	**1.2**	**0.9**	**12.0**	**65.3**	**26.4**	**12.1**	**137.0**	**50.7**	**-**
TSS (mg/L)	**8.0**	**11.0**	**9.5**	**28.0**	**66.0**	**39.0**	**20.0**	**274.0**	**99.0**	**4 000**
FOG (mg/L)	**-**	**-**	**-**	**16.0**	**40.0**	**28.0**	**-**	**-**	**-**	**400**
Colour (mg Pt/L)	**0.8**	**12.0**	**4.7**	**21.0**	**42.0**	**32.4**	**-**	**-**	**-**	**-**
Fructose (mg/L)	**3.9**	**72.3**	**37.0**	**10.3**	**1 071**	**518.0**	**3.3**	**461.6**	**189.0**	**1 500**
Sucrose (mg/L)	**133.3**	**696.6**	**421**	**1 346**	**6 901**	**3 983**	**46.6**	**1 505**	**631.0**	**1 500-**
Inorganic parameters										
Sodium	**3.3**	**7.9**	**6.0**	**83.1**	**183.0**	**115.9**	**87.4**	**1 355**	**537.6**	**1 000**
Boron (mg/L)	**0.0**	**0.01**	**0.01**	**0.0**	**0.01**	**0.01**	**0.0**	**0.0**	**0.0**	**5**
CO_3 (mg/L)	**-**	**-**	**-**	**30.1**	**241**	**100**	**-**	**-**	**-**	**-**
HCO_3 (mg/L)	**35.1**	**43.5**	**39.0**	**137.8**	**421.1**	**228.1**	**367.5**	**2 787**	**1 623**	**-**
Calcium (mg/L)	**13.7**	**17.7**	**16.0**	**13**	**22.7**	**19.9**	**13.2**	**21.0**	**16.4**	**-**
Chloride (mg/L)	**13.6**	**14.0**	**14.0**	**19.8**	**44.2**	**28.7**	**22.1**	**44.2**	**32.3**	**1 500**
Fluoride (mg/L)	**0.0**	**0.0**	**0.2**	**0.5**	**1.4**	**1.1**	**0.0**	**0.5**	**0.2**	**-**
Ammonium (mg/L)	**0.1**	**0.42**	**0.3**	**0.5**	**1.8**	**0.8**	**0.1**	**38.7**	**4.2**	**-**
Nitrate (mg/L)	**0.1**	**1.1**	**0.6**	**0.1**	**2.2**	**0.7**	**0.01**	**9.4**	**2.6**	**-**
Potassium (mg/L)	**0.3**	**1.0**	**0.7**	**1.3**	**5.1**	**2.9**	**1.4**	**6.9**	**3.3**	**-**
Magnesium (mg/L)	**1.3**	**1.4**	**1.4**	**0.6**	**2.4**	**1.5**	**0.7**	**3.3**	**1.9**	**-**
Sulphates (mg/L)	**5.0**	**6.0**	**5.7**	**6.4**	**20.8**	**13.3**	**3.4**	**17.0**	**10.1**	**1 500**
Zinc (mg/L)	**0.0**	**0.0**	**0.0**	**0.02**	**0.1**	**0.0**	**0.0**	**0.1**	**0.0**	**30**
Copper (mg/L)	**0.0**	**0.02**	**0.01**	**0.02**	**0.01**	**0.03**	**0.0**	**0.0**	**0.0**	**20**
Iron (mg/L)	**0.0**	**0.04**	**0.02**	**0.3**	**0.7**	**0.5**	**0.1**	**1.6**	**1.0**	**50**
Manganese (mg/L)	**0.0**	**0.0**	**0.0**	**0.01**	**0.05**	**0.02**	**0.0**	**0.5**	**0.1**	**-**
Phosphate (mg/L)	**0.02**	**2.46**	**1.0**	**1.2**	**2.6**	**1.7**	**0.3**	**23.1**	**8.2**	**25**
Orthophosphate (mg/L)	**1.4**	**1.35**	**1.4**	**5.6**	**6.2**	**5.9**	**4.9**	**18.8**	**7.4**	**-**

^^: City of Cape Town: Wastewater and industrial wastewater by-law (South Africa (Western Cape), 2006

The sugar, namely fructose and sucrose concentrations, ranged from 3.9 to 72.3 mg/L with an average of 37.3 mg/L and 133.3 to 696.6 mg/L with an average of 421.4 mg/L, respectively. Both the physio-chemical properties and inorganic properties of the bottle-washing wastewater were within the suitable limit stipulated by the CCT's industrial by-laws. However, it did not meet all the parameters stipulated by SANS 241:2011 for reuse or recycling. It was reported by Hsine *et al. (*2010) that a bottling-washing plant generates wastewater that is good for biological treatment. However, it varies significantly in quantity and quality, depending on the production plans and products that are mixed. The quality of wastewater is in turn often influenced by a reduced water intake, bottle-washing methods and the unpredictable nature of the process.

4.1.4 Softdrink industry wastewater (SDIW) on-site

Results from the eight batches of SDIW from the on-site equalization tank are presented in Table 4.2. SDIW consists of wasted softdrinks, syrups, sugars, and water from the backwashing; washing of bottling machines, equipment floors and pipe work during flavour changing; and operational performances, washing of bottles, which contains caustics, detergents and lubricants that are used in the machinery and conveyor belts. Therefore, the significant associated wastewater pollutants will include TSS, TDS, BOD_5, COD and nitrate. There are large variances in both the quality and quantity of SDIW.

The quantity and quality of the SDIW is proportional to potable water intake, but is greatly influenced by the nature of the process and product. The data recorded in Table 4.2 clearly shows wastewater with a high organic content and fluctuating pH, but it was mainly alkaline. The pH values fluctuated from 6.1 to 11.8. The high pH of 11.8 was due to sodium hydroxide (NaOH), which is indicative of the usage at the bottle-washing utilities. The alkalinity value ranged from 160 to 589 mg/L. This could be attributed to the wastewater containing components of detergents that were used in the bottle-washing plant, which made pH control more difficult.

The temperature for SDIW fluctuated between 17.3 and 19.4˚C during the winter season, and increased during the summer season to between 20.7 and 25.2˚C. An increase in temperature, together with an increase in the sugar concentrations in the wastewater equalization tank, intensified the biological activities in the wastewater and thus accelerated an excessive growth of micro-organisms. The SDIW had high BOD levels, which ranged from 400 to 1 150 mg/L due to the dissolved sugars, namely fructose from 10.3 to 1 071 mg/L and sucrose from 1 346 to 6 900 mg/L. However, the fructose concentration for batches 6, 7 and 8 were lower than 36.3 mg/L due to the industrial partner discontinuing the use of fructose for sweetening flavoured water.

There was a wide range of contaminants. The SDIW was classified as a medium to high-strength wastewater (Chmiel *et al.,* 2003); with COD_t varying between 2 242 and 11 717 mg/L with an average of 5 638 mg/L, which is above the target specific contamination load of untreated SDIW COD of 3 500 mg/L (CSIR, 2010). The COD_t was above the CCT's industrial wastewater discharge standards by-law (South Africa, 2006). The preparation of syrups and sugars caused the most contamination, as it produced wastewater that was rich in sugars, with most contaminants being the dissolved type including those which contribute to COD_s. The COD_s varied between 2 217 and 11 345 mg/L, with an average of 5 337 mg/L. It was observed that the COD_t consisted mostly of COD_s.

The SDIW was comprised of TDS, EC with maximum values of 1 050 mg/L and 1 483 µS/cm; respectively, and did not meet the CCT's industrial wastewater discharge standards by-law (South Africa, 2006). The samples were generally and characterized by TSS values, which ranged from 28 to 66 mg/L. FOG ranged from 16 to 40 mg/L, which is indicative of the FOG generated during CIP processes in the production plant and of the lubricants used in the conveyor belts. However, the sugars, TSS and FOG were all within the CCT's industrial wastewater discharge standards by-law (South Africa, 2006). Turbidity and colour were recorded to a maximum of 65.3 NTU and 42 mg Pt/L, respectively. This could be an indication of product spillage or bacterial population in the SDIW. The SDIW was not a significant source of boron, iron, fluoride, ammonium, nitrate, potassium, magnesium, zinc, copper, iron, manganese and phosphates that may have degraded the equalization tank environment, as they were all below 5 mg/L, a higher limit concentration value with regard to the quality parameters require for discharging the wastewater into fresh water resources (DWA, 2010).

About 62% of the total organic matter present in SDIW is soluble and as a result cannot be removed by mechanical or physical means (Hsine *et al.,* 2010; Matošić *et al.,* 2009). Therefore, biological and chemical oxidation becomes a feasible option to treat this type of wastewater (Hsine *et al.,* 2010; Chmiel *et al.,* 2003).

4.2 Performance of the EGSB reactor

The performance of the EGSB, with respect to COD_t and COD_s treatment efficiencies (TE), was evaluated under a varying range of HRT_s and V_{up}, as previously presented in Tables 3.1 and 3.2. Performance results are presented in Figures 4.2 and 4.3, indicating the variation of both COD_t and COD_s TE during the entire study. In Figures 4.2 and 4.3 the vertical lines with numbers indicate when a new batch of SDIW was collected and loaded into the holding tank. Figure 4.4 presents the HRT used and TE over time while the vertical lines indicate V_{up} in m/h.

4.2.1 EGSB initial start-up

The acclimatization phase can be considered as the first 21 days of operation with the initial of the SDIW being loaded into the holding tank with COD_t, fructose and sucrose concentration values of 3 267, 528.7 and 2 901 mg/L, respectively. During acclimatization the EGSB feed COD_t and COD_s fluctuated between 1 691 to 3 277 mg/L and 1 515 to 2 255 mg/L, respectively, with fructose concentration of 580 mg/L while sucrose of 913 mg/L. COD_t and COD_s TE of 66% and 67% were obtained, respectively, at an average OLR of 1.2 kg COD/m^{3}d.

The holding tank developed a pungent odour and serious operating difficulties were experienced with the occurrence of the foam as shown in Figure 4.5. Foam formation could be associated with various issues such as low to high pH, toxic material, low dissolved oxygen, anaerobic conditions, fermentation and change in ambient temperature. The SDIW COD_t and pH decreased from 3 267 to 2 233 mg/L and 6.1 to 5.4, respectively. This was an indication that the holding tank was becoming anaerobic. Special attention was paid to prevent anaerobic conditions of the holding tank by collecting fresh SDIW more frequently. According to Bellmer and Hasan (2012), the dissolved waste's sugar content provides the potential for fermentation to occur. Thus, preliminary testing was conducted to determine how easily softdrinks could be fermented. Using a gas chromatography (GC) unit, unexpected acids, esters and ethanol-type fermentation was discovered at the top of the EGSB. According to Isla *et al.* (2013), SDIW can be regarded as a medium for alcoholic fermentation since this wastewater was comprised of a high sugar content of approximately 1 346 to 6 901 mg/L. The average EGSB feed concentration values for COD_t was 1 890 mg/L and for COD_s was 1 777 mg/L. The COD_t TE increased up to 90.4% at an OLR of 0.7 kg COD/m^{3}d.

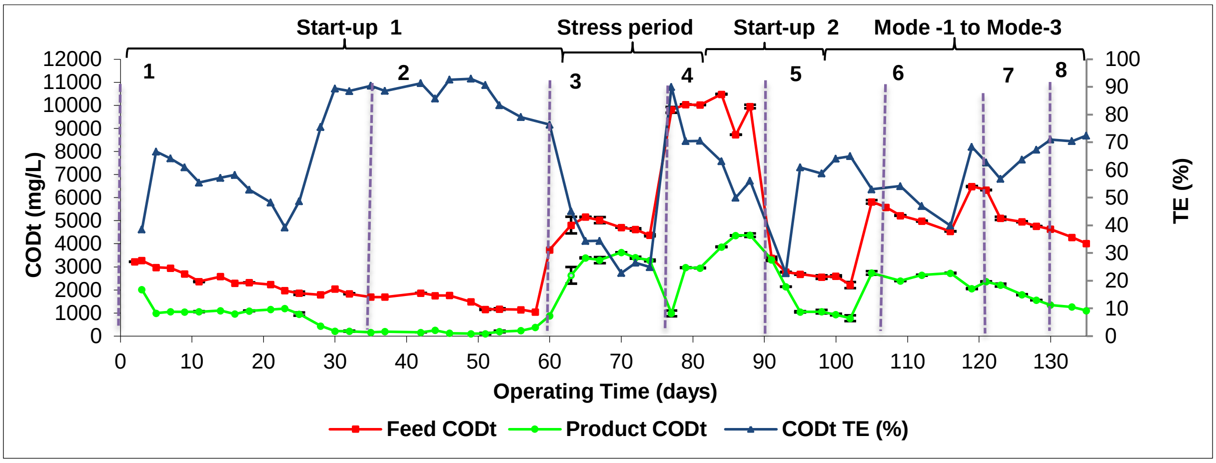


Figure 4.2: EGSB pre-treatment efficiency and COD_t of softdrink industry wastewater

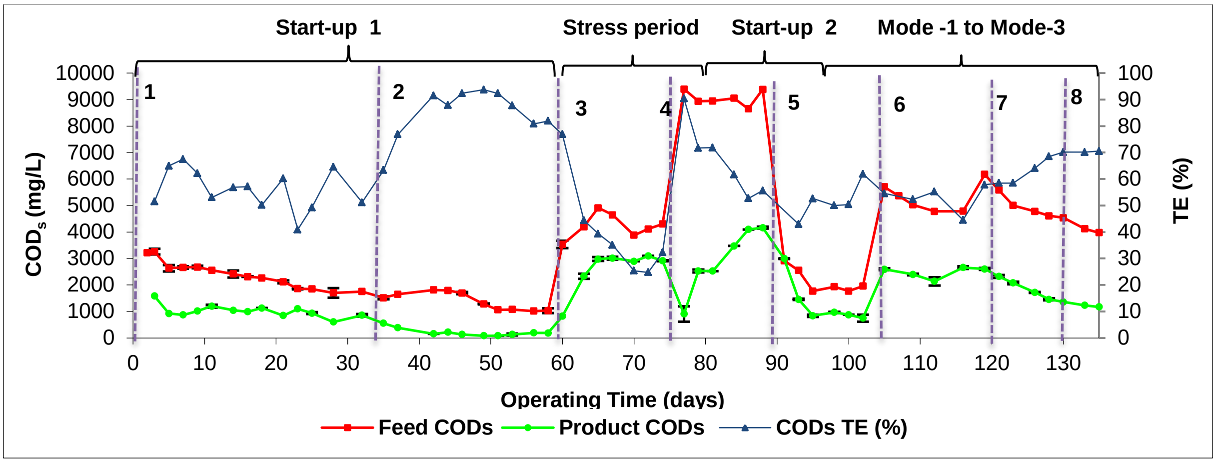


Figure 4.3: EGSB pre-treatment efficiency and COD_s of softdrink industry wastewater

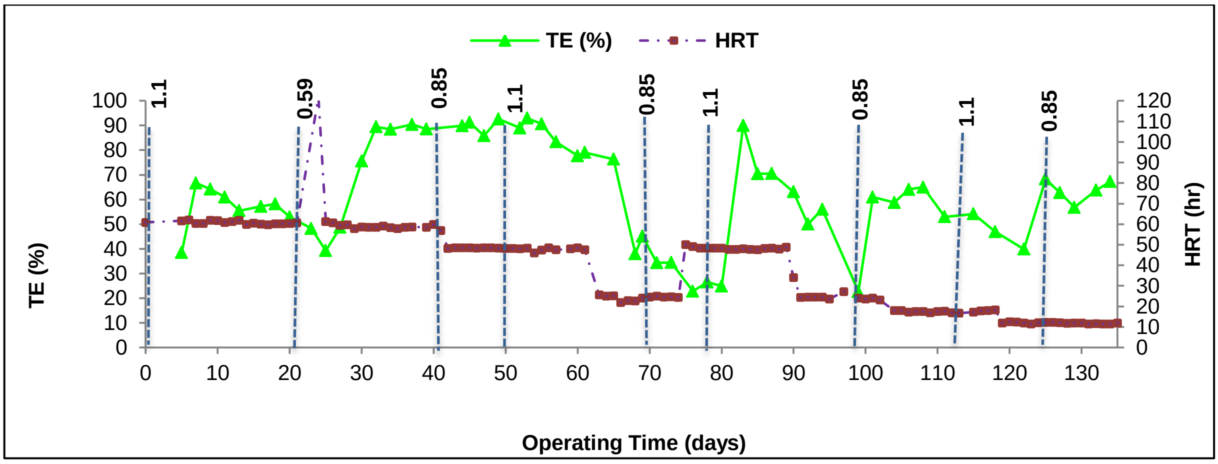


Figure 4.4: EGSB HRT, V_{up} and COD_t treatment efficiency

Figure 4.5: Foam formation in the holding tank

On day 35, batch two of the SDIW was loaded into the holding tank with a COD_t of 2 242 mg/L, fructose of 342.21 mg/L, sucrose of 2 067 mg/L and BOD_5 of 400 mg/L, and the EGSB COD_t TE of 89% was observed. For an average feed, i.e. COD_t of 1 797 mg/L and COD_s of 1 765 mg/L, fructose of 203.2 mg/L, sucrose of 1 346 mg/L and a maximum OLR of approximately 0.95 kg COD/m^3d, the COD_t and COD_s TE was increased to 93 and 92%, respectively.

Based on the composition of the wastewater that was fed into the EGSB, the average TSS and turbidity decreased from 110 to 36 mg/L and 41 to 18 NTU, as shown in Figures 4.6 and 4.7. Other parameters such as EC and TDS increased in the EGSB product during the initial start-up period by 55% (486 to 1 444 µS/cm) and 53% (353 to 988 mg/L), respectively as shown in Figures 4.8 and 4.9. The increase in EC and TDS in the product could be due to fluctuations in the ion content such as H^+, hydroxide (OH^-), and the presence of nutrients such as nitrate and phosphates (Levlin, 2007). This was attributed to dosing the system with phosphoric acid (H_3PO_4) to achieve the desired C:N:P ratio. Similarly, the average ammonium, nitrate and orthophosphate of the EGSB product increased from 0.5 to 8.2 mg/L; 0.8 to 2.8 mg/L; and 2.1 to 6.6 mg/L, respectively.

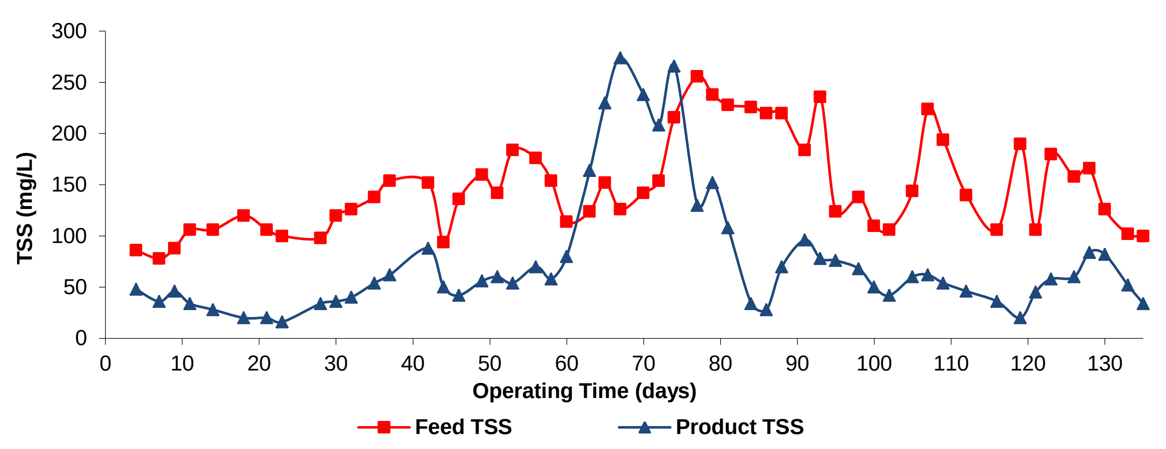


Figure 4.6: TSS during EGSB pre-treatment of softdrink industry wastewater

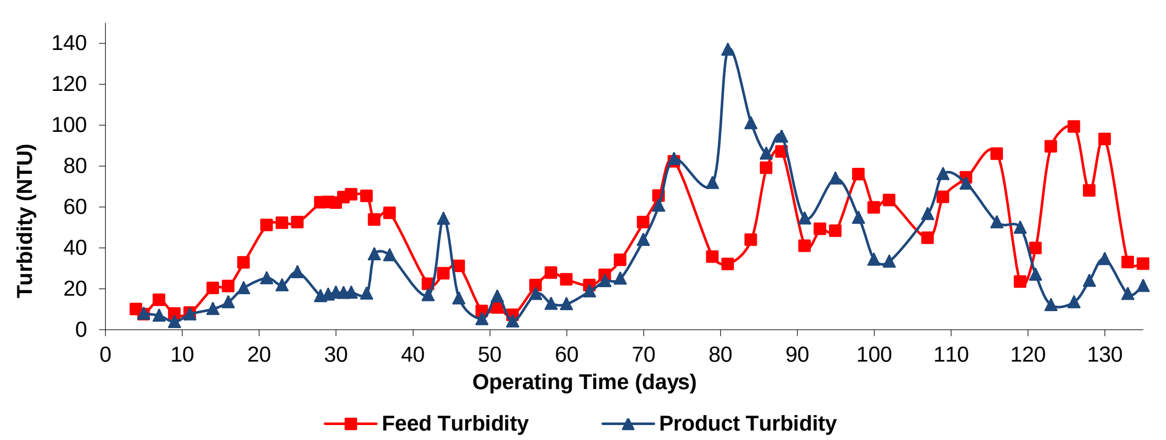


Figure 4.7: Turbidity during EGSB pre-treatment of softdrink industry wastewater

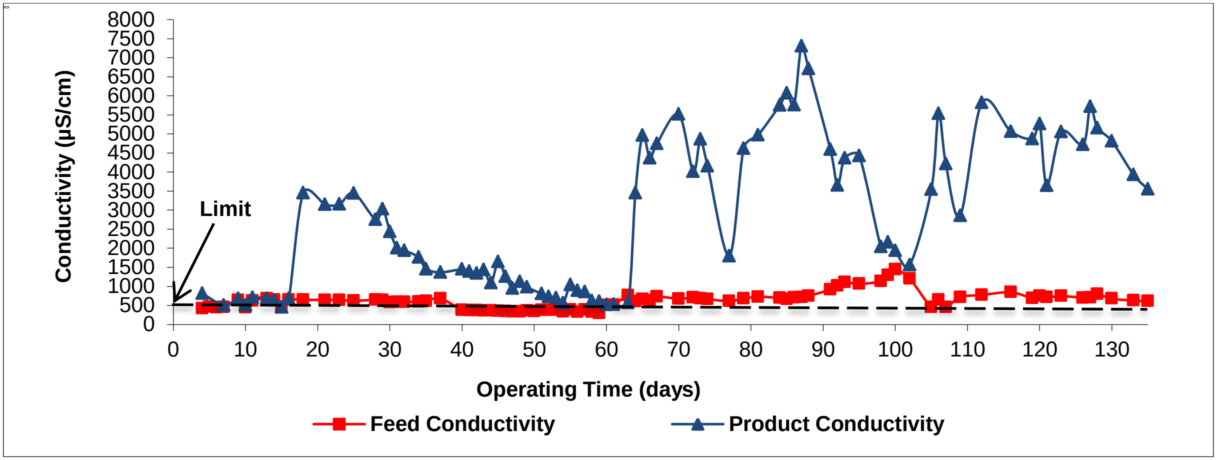


Figure 4.8: EC during EGSB pre-treatment of softdrink industry wastewater

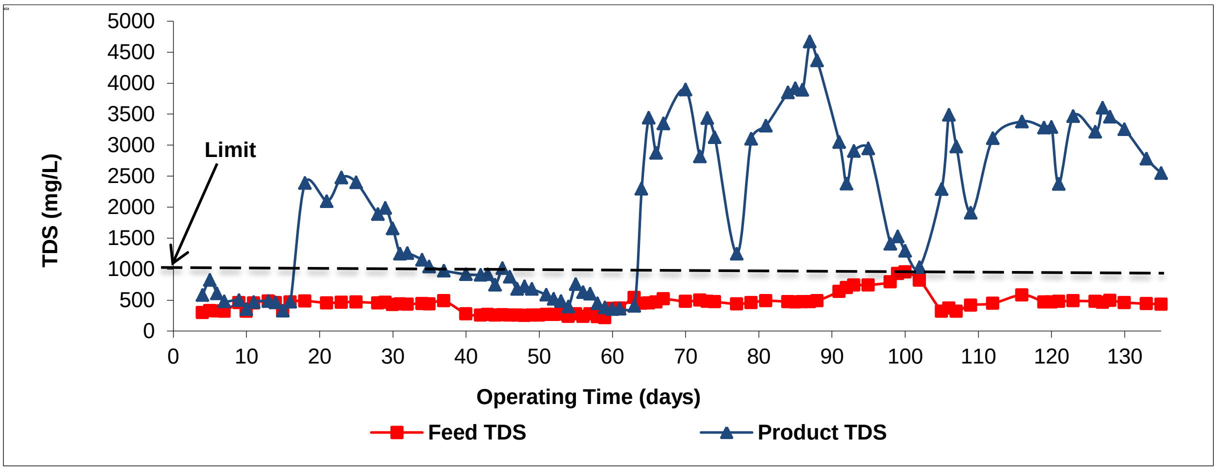


Figure 4.9: TDS during EGSB pre-treatment of softdrink industry wastewater

During the initial-start-up it was noted that the ammonium concentration in the EGSB product fluctuated from 2.9 to 39.5 mg/L, as shown in Figure 4.10. This wide range could be due to changes in the characteristics of the SDIW, environmental conditions, acclimatisation periods and inoculum quality (Rajagopal *et al.,* 2013; Chen *et al.,* 2008). According to González-Fernández and García-Encina (2009) ammonium is the end-product of the AD of proteins, nucleic acids and urea. Unlike the importance of ammonium for bacterial growth at lower concentration, high concentration of ammonium may cause a severe disruption in the AD, that is, cause decrease in microbial activities (Rajagopal *et al.,* 2013; Zhang *et al.,* 2011).

An increase in nitrate$^-$ from 0.5 to 4.5 mg/L was also recorded, as shown in Figure 4.11. This could be an indication of an ANAMMOX process, which utilizes autotrophic bacteria that converts ammonium directly into nitrogen gas under anaerobic conditions (Ahn, 2006; Van Loosdrecht & Salem, 2005). An advantage of the ANAMMOX process is that because the bacteria are autotrophic, there is no need for an external carbon source for denitrification (Phillips *et al.,* 2006). This also agrees with the pilot study conducted by Gut *et al.* (2006) on a WWTP, which used a fixed film moving bed bioreactor (MBBR).

The average orthophosphate concentration increased in the EGSB product from 1.1 to 5.1 mg/L, when compared to the EGSB feed, according to Phillips *et al.* (2006) and during AD, approximately 60% of the stored total phosphorus in the feed sludge is released as orthophosphate, which may triple the phosphorus load that enters with the raw wastewater (Figure 4.12).

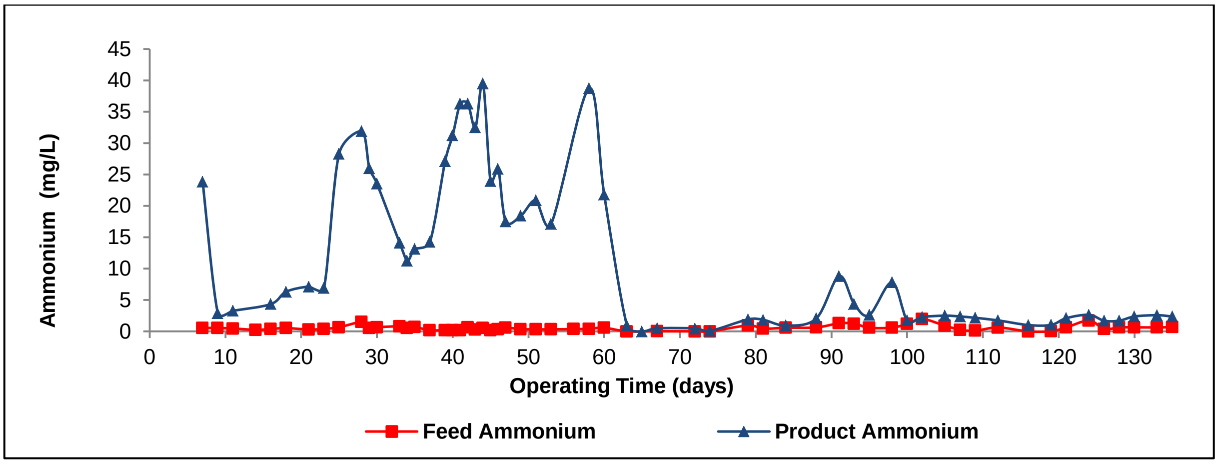


Figure 4.10: Ammonium concentration during EGSB pre-treatment for softdrink industry wastewater

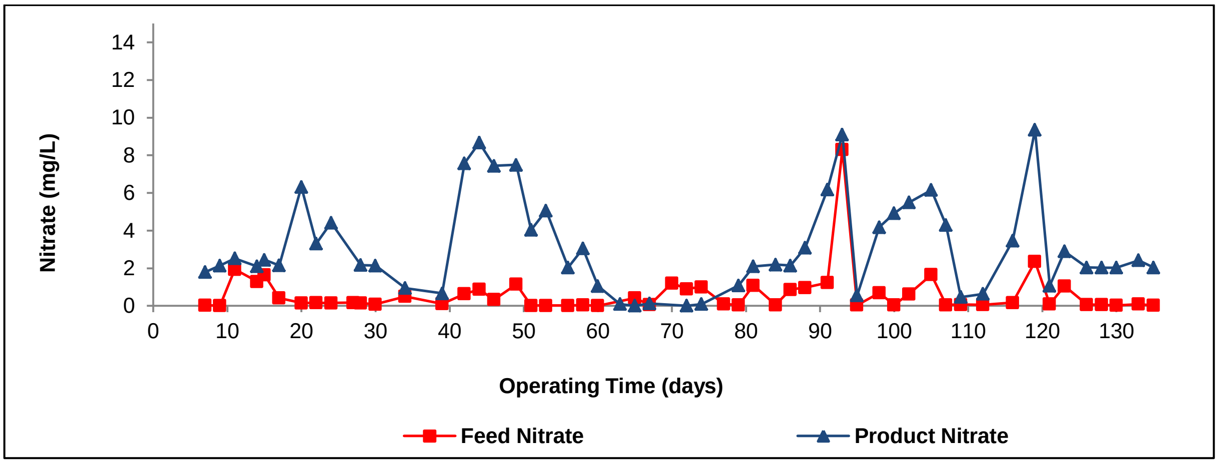


Figure 4.11: Nitrate concentration during EGSB pre-treatment for softdrink industry wastewater

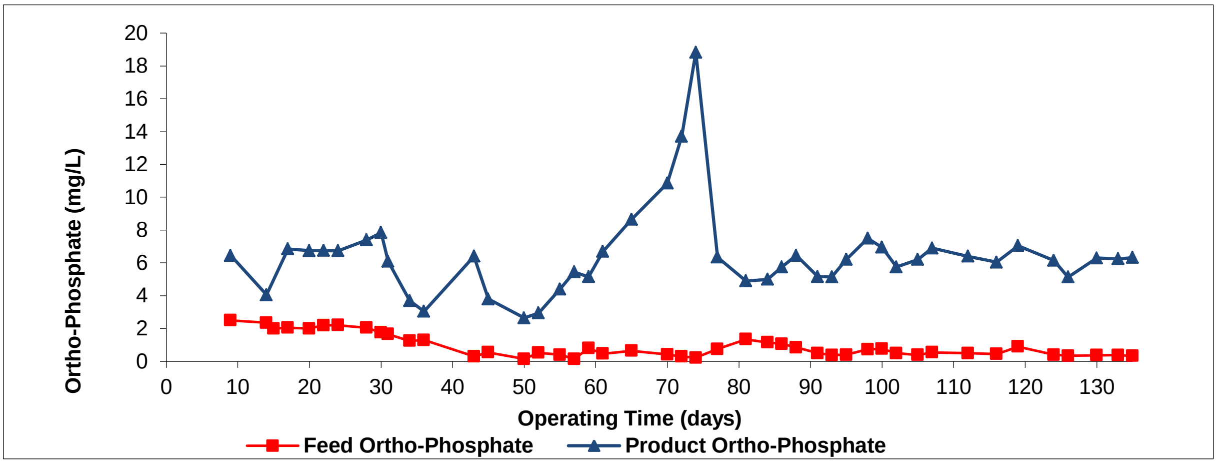


Figure 4.12: Orthophosphate concentration during EGSB pre-treatment for softdrink industry wastewater

4.2.2 EGSB stress period

On day 59 d, a third batch of SDIW was loaded to the holding tank with an average COD_t of 5 495 mg/L, fructose of 63.6 mg/L and sucrose of 788 mg/L, and a low BOD_5 of 76 mg/L. A slight decrease in COD_t and COD_s TE to 76% and 77%, respectively was observed at an OLR of 1.9 kg COD/m^3d. After day 61 the EGSB was overdosed with H_3PO_4 resulting in a drop in COD_t and COD_s TE down to 45 and 44%, respectively, at a V_{up} of 1.1 and HRT of 24h. The ammonium, nitrate and orthophosphate increased from 0.5 to 20.2 mg/L, 0.02 to 2.1 mg/L and 0.7 to 8.7 mg/L, respectively.

When the V_{up} was decreased to 0.85 m/h with an average COD_t of 4 564 mg/L and COD_s of 4 097 mg/L and an OLR of 4.6 kg COD/m^3d a further decrease was observed in TE down to 24.7% and 27.5% for COD_t and COD_s, respectively. Methanogens and acidogens are extremely sensitive to pH change. During this period, the pH in the EGSB decreased to 4.5, resulting in severe toxicity. This correlated with the undissolved volatile fatty acids (VFA), which were produced by the acidogenic microorganisms. Preferably the concentration of VFA should be less than 200 mg/L, since VFA is being consumed by the methanogenic bacteria. A pH or toxic load variation results in an increase in the VFA concentration (Gray, 2008; Paulsen, 2006). This resulted in system failure and the EGSB required re-inoculation. It was also noted that during this period the ammonium varied from 0.1 to 38.7 mg/L as shown in Figure 4.9, while it is reported that ammonium is one of the main causes of reactor failure because of its inhibition of bacterial activity (Rajagopal *et al.,* 2013; Chen *et al.,* 2008).

4.2.3 EGSB re-inoculation and second start-up

The EGSB system was re-inoculated on day 76 with both anaerobic sludge and aerobic granules with a TSS of 3 032 mg/L and TSS of 1 705 mg/L, respectively. The system was put on recycle for two days to improve the granular sludge microbial interactions. A fourth batch of the SDIW was loaded into the holding tank with a COD_t concentration of 11 717 mg/L, fructose of 34.7 mg/L, sucrose of 6 900 mg/L and BOD_5 of 500 mg/L, with a BOD:COD ratio of 0.043.. The COD_t TE increased to 90% initially but subsequently decreased from 90% to 50%. The low TE could have been due to a high OLR between 4.4 and 5.4 kg COD/m^3d, which were higher than those suggested by Sheldon *et al.* (2012) during the start-up period, namely 4.1 kg COD/m^3d, for paper mill wastewater treatment using the EGSB. The EC and TDS increased from 692.1 to 5 375 µS/cm and from 471 to 3 546 mg/L. Turbidity also increased from 56 to 98 NTU and the maximum TSS removal that was achieved was only 87%. The average ammonium, nitrate and phosphate concentrations increased from 0.7 to 1.7 mg/L; 0.5 to 2.1 mg/L; and 1.0 to 5.7 mg/L, respectively.

4.2.4 EGSB treatment efficiency

SDIW (batch 5) with a COD_t of 2 662 mg/L, fructose of 54.3 mg/L, sucrose of 794.7 mg/L and a low BOD_5 of 30 mg/L, was introduced into the holding tank on day 90 prior to Mode-1a of operation, with BOD:COD ratio of 0.023. The average COD_t and COD_s was 2 946 and 2 410 mg/L, respectively, with an OLR 3.4 kg COD/m^3d. During days 91 to 94, the EGSB began to achieve a certain level of stability, although the TE that were observed were low. However, the process stabilised after day 95 with COD_t and COD_s TE increasing up to 61% and 53%, respectively. The average EC of 1 926 µS/cm and TDS of 1 315 mg/L remained high in the EGSB product when compared to the EGSB feed. The system stabilized after day 98 and the TE of COD_t and COD_s stabilised at 65 and 62%, respectively, at an OLR of 2.7 kg COD/m^3d. The average ammonium, nitrate and phosphate concentrations increased in the product to 3.4, 4.9 and 6.7 mg/L, respectively.

SDIW (batch 6) was introduced into the holding tank on day 104 with COD_t values of 6 962 mg/L, a low fructose of concentration 10.3 mg/L when compared to batches 1 to 5, sucrose of 1346 mg/L and BOD_5 of 650 mg/L. The HRT was decreased from 24 to 18 h. The COD_t and COD_s removal efficiencies that were obtained at an OLR of 7.9 kg COD/m^3d were 54% and 55%, respectively. The average EC and TDS increased in the product to 4 038 µS/cm and 2 666 mg/L, respectively. An increase in the average ammonium (2.4 mg/L) and nitrate (3.6 mg/L) concentrations were observed with a slight decrease in the phosphate concentration to 6.6 mg/L when compared to Mode-1b of operation.

On day 112 the V_{up} was increased to 1.1 m/h with a corresponding HRT of approximately 18 h. The COD_t and COD_s concentrations of the EGSB feed were 4 540 and 4 982 mg/L with an OLR of 7.19 kg COD/m^3d. The COD_t removal decreased to 47%, while the COD_s removal rate increased to 55%. A decrease in COD_t and increase in COD_s could be due to the microbial hydrolysis of insoluble organic matter such as carbohydrates, which are broken-down to a soluble end-product that becomes accessible for other microorganisms. Acidogenic microorganisms then convert the amino acids and sugars into organic acids, CO_2, H_2 and N_2. Finally, the methanogens microorganisms then convert these products to CH_4 and CO_2 (Matovic, 2013; Manyi-Loh *et al.,* 2013; Tabatabaei *et al.,* 2010; Song *et al.,* 2005).

SDIW with COD_t concentration values of 7 760 mg/L, fructose of 7.5 mg/L and sucrose of 4 514 mg/L, and BOD_5 of 500 mg/L were loaded on day 118 with an OLR of 13.2 kg COD/m^3d. The COD_t and COD_s TE that were attained were 56.8% and 68.3%, respectively. On day 130 the V_{up} was decreased to 0.85 m/h and the SDIW (batch 7) with COD_t, fructose, sucrose, BOD_5 values of 4 692 mg/L, 5.30 mg/L, 6 301 mg/L and 150 mg/L, respectively, were loaded into the holding tank. On day 135 the average COD_t concentration in the EGSB feed was 4 786 mg/L and the average was COD_s of 4 641 mg/L at OLR of 9.9 kg COD/m^3d. The COD_t and COD_s TE that were attained were 71% and 70.1%, respectively.

4.2.5 EGSB treatment summary

Table 4.3 represents operating conditions in relation to COD at various operating conditions. The EGSB operation remained fairly consistent during 45 days of stable operation, as presented in Figures 4.2 and 4.3. Throughout mode 1 to 3 of operations, the average amount of COD_t that was removed was 62.3% with a maximum of 93%; while the average COD_s that were removed amounted to 61% with a maximum of 94%. The average TSS and turbidity removal was 8.2% and 22.4%, respectively.

The ammonium and nitrate concentrations increased from 0.57 to 2.04 mg/L, 0.1 to 0.7 mg/L, respectively, during AD. This could be due to an amino acid such as alanine being oxidatively decarboxylated and the hydrogen produced during the reaction that was used to reductively transform an additional amino acid such as glycine, to acetate and ammonia, as indicated in Equations 4.1 and 4.2 (Gallert, & Winter, 2005).

$$CH_3 - CHNH_2 - COOH + 2H_2O \rightarrow CH_3 - COOH + CO_2 + NH_3 + 2H_2$$ Equation 4.1

$$\Delta G = +7.5 kmol^{-1}$$

$$2CH_2NH_2 - COOH + 2H_2 \rightarrow 2CH_3COOH + 2NH_3$$ Equation 4.2

$$\Delta G = -38.9\ kmol^{-1}$$

Table 4.3: EGSB treatment efficiency for different operating conditions

Operating Stages	Operating Conditions				COD_t			COD_s		
	HRT		V_{up}	OLR	Feed	Product	TE	Feed	Product	TE
	Days	h	m/h	kg COD/m^3d	mg/L	mg/L	%	mg/L	mg/L	%
Start-up 1	2	48.4	0.59	0.9	1 167	194.50	83.3	1 072	131	87.8
Stress period	1.1	25.1	1.1	5	4 622	3 397	26.5	4 115	3 095	24.8
Start-up 2	2	47.8	1.1	5	9 950	4 372	56.1	9 380	4 162	55.6
Mode-1a	1	23.7	1.1	2.8	2 212	775	65	1 955	745.	61.9
Mode-1b	1	23.6	0.85	2.7	5 582	2 547	54.4	5 365	2 360	56.0
Mode-2a	0.7	16.9	0.85	7.6	4 540	2 730	39.9	4 782	2 655	44.5
Mode-2b	0.8	18	1.1	6.2	5 103	2 205	56.8	5 004	2 078	58.5
Mode-3a	0.5	11.4	1.1	10.9	4 637	1 345	71	4 536	1 355	70.1
Mode-3b	0.5	11.8	0.85	9.9	4 007	1 104	72.4	3 975	1 173	70.5

The nitrate increased from 0.5 to 2.6 mg/L in the EGSB product which agrees with Chen *et al.'s* (2008) in the review of AD that showed significant variation in the toxicity/inhibition levels and the main reason for these dissimilarities is the complexity of the AD process, where mechanisms such as antagonism, synergism, acclimation, and complexing could significantly affect the phenomenon of inhibition. Reduced levels of inhibition occurred with alcohol derivatives and oxides (Amaral *et al.,* 1998). Anaerobic ammonium oxidation (ANAMMOX) is attained by a highly specialized group of microorganisms, which belongs to the planctomycete group and, which oxidises ammonium to N_2 gas by using ammonium as an electron donor and nitrate simultaneously as an electron acceptor while producing alkalinity (Yang *et al.,* 2010), as presented in Equation 4.3:

$$NH_3 + 1.32NO_2 + H^+ \rightarrow 1.02N_{2+}0.26NO_3 + 2H_2O \qquad \text{Equation 4.3}$$

According to Fiss and Stein (2002), an increase in the phosphate concentration from 0.48 to 5.48 mg/L during AD could be due to certain microbes named phosphate accumulating organisms (PAOs) which generate phosphorus. PAOs utilize VFAs as a food source. If a sugar solution or other alternative carbohydrates are added to the anaerobic phase as the carbon source to enhance biological processes, the sugars are quickly fermented by heterotrophic bacteria, which are called acid-forming bacteria to produce VFAs. The carbohydrates themselves cannot be utilized by the PAOs. However, since the sugars are quickly fermented to VFAs in a suspended growth process under anaerobic conditions, the addition of sugar instead of acetic acid does not have an observable impact on the efficiency of the enhanced biological process.

4.3 EGSB biogas production and composition

The biogas that was produced during the anaerobic treatment was primarily composed of methane (CH_4), carbon dioxide (CO_2), nitrogen (N_2), oxygen (O_2) and traces elements of hydrogen sulphide (H_2S) and hydrogen (H_2). The biogas production was monitored and analysed at different operating conditions, as shown in Table 4.4. The biogas productions in relation to COD TE and OLR throughout the study are shown in Figures 4.13 and 4.14.

4.3.1 Biogas composition during EGSB initial start-up

During the initial start-up the OLR was maintained at ~ 0.9 kg COD/m^{3}d and the biogas flowrate that was obtained was low at 2.5 L/d. The CH_4, CO_2, H_2S, N_2 and O_2 content in the biogas were 32.3%, 18%, 0.7%; 45.4% and 9.6 %, respectively, with H_2, which was less than 1%.

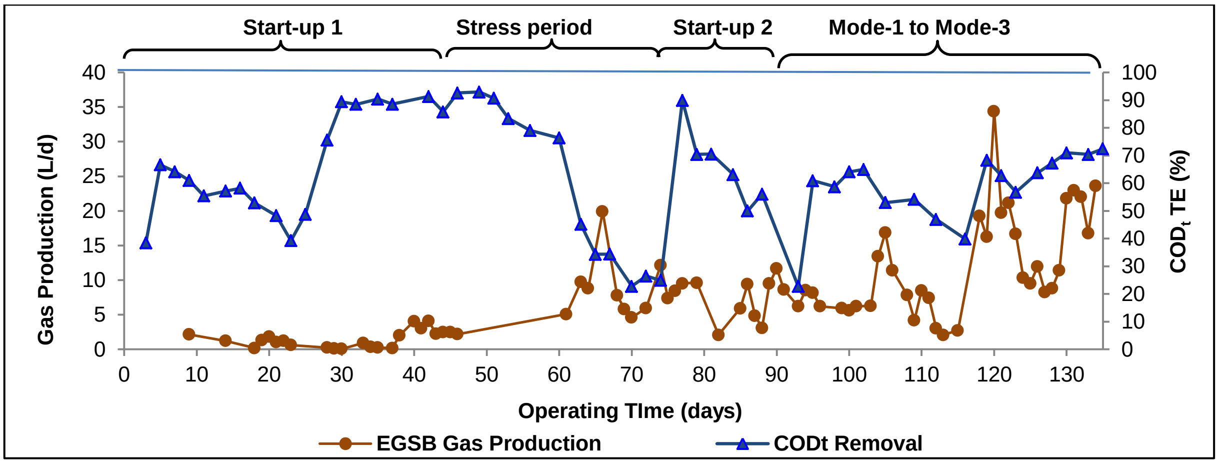


Figure 4.13: Biogas production in relation to COD_t TE during EGSB pre-treatment

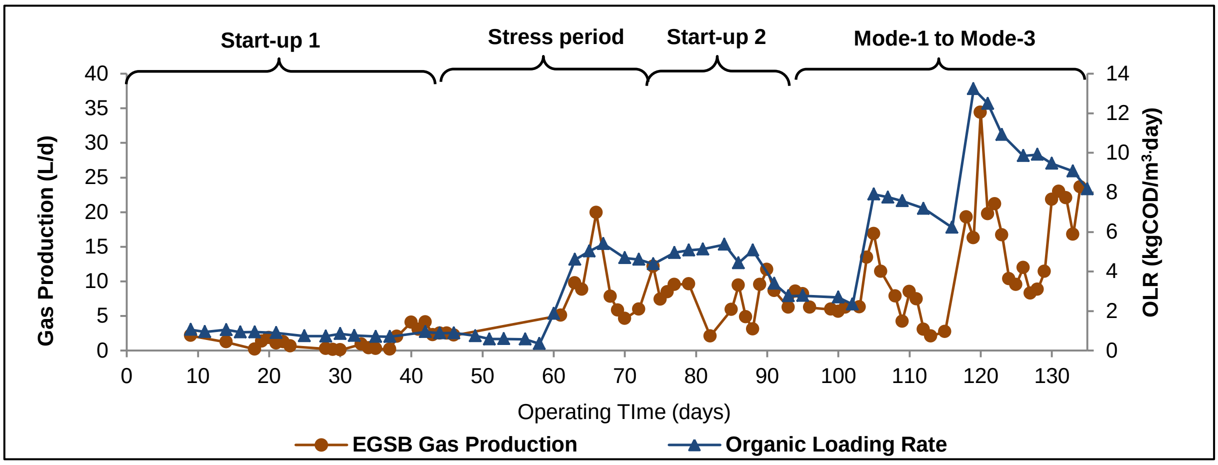


Figure 4.14: Biogas production in relation to OLR during EGSB pre-treatment

4.3.2 Biogas composition during the EGSB stress period

During the stress period the OLR was maintained at 5kg COD/m^{3}d with a COD_t concentration in the feed of 4 622 m/L and COD_s of 4 115 mg/L. A reduction in COD_t, TE and biogas CH_4 production was observed in the reactor down to 26.5% and 10.4%, respectively. This coincided with a gradual decrease in the reactor pH from 7.6 to 4.6. However, the biogas flowrate measured was 8.8 L/d and the biogas contained CH_4, CO_2, H_2S, N_2 and O_2 content of 9.4 to 10.4%, 23.2 to 25.1%, 0.8 to 0.2%; 50 and 15 to 16.2%, respectively, with traces of H_2, which was less than 1%. The low CH_4 concentration was an indication of changes in the EGSB operating conditions, in this case, the introduction of an acid, which resulted in a system imbalance. It has been reported that the degree of CH_4 production decreases when the pH value drops below 6.3 (Nelson, 2010; El-Mashad *et al.,* 2004). A slight increase in ammonium and a decrease in nitrate concentration values from 0.02 to 0.5 mg/L and 0.9 to

0.01 mg/L, respectively, were observed. According to Yang *et al.* (2010), the lower the ammonium and the nitrate concentration in the reactor product, the lower the CH_4 production.

4.3.3 Biogas composition during EGSB second start-up

As determined by gas chromatograph (GC), the major constituents of the organic matter were acids, alcohols and esters in the EGSB reactor. The C:N:P ratio of the SDIW was 100:0.09:0.02, which was appropriate for bioconversion to CH_4. It was not necessary to supplement nutrients to the EGSB reactor during the second start-up period, which concurs with Luo *et al.* (2009) and Yu *et al*. (2006) for the treatment of cassava sillage and winery wastewater.

It was observed that there was a slight difference in biogas composition during the initial start-up and the second start-up. The biogas content of the EGSB during the second start-up was 26.2 to 30%, 17 to 18.1%, 0.15 to 0.2%, 41 to 41.3% and 11.7 to 14.1% for CH_4, CO_2, H_2S, N_2 and $O_{2,}$ respectively, with traces H_2, which was less than 1%. However, the feed concentration values of COD_t and COD_s were high at 9 950 mg/L and 9 380 mg/L, respectively. The OLR was maintained at 5.1 kg COD/m^{3}d, higher than the initial start-up of 0.9 kg COD/m^{3}d, and a higher biogas flowrate of 9.5 L/d was obtained, compared to 2.5 L/d. The COD_t TE was 56.1%, which was much lower than the initial start-up of 87.8%. Conversely, ammonium and nitrate increased in the EGSB product from 0.64 to 2.1 mg/L and from 1 to 3.1 mg/L, respectively; hence ammonium was partly oxidized to nitrite through the ANAMMOX reaction (Lu *et al.,* 2013).

During Mode-1a the EGSB demonstrated a recovery in process performance with the TE COD_t of 65%. The biogas flowrate was 8.6 L/d at an OLR of 2.8 kg COD/m^{3}d with feed concentrations of COD_t of 2 212 mg/L and COD_s of 1 955 mg/L. The biogas content for CH_4, CO_2, N_2 and O_2 was 47%, 9.6%, 35.6%; and 7.9%, respectively, with traces of H_2S and H_2 of less than 1%. Ammonium and nitrate concentration ranging from 1.99 to 2.31 mg/L and 0.6 to 5.5 mg/L, respectively. On day 112 at an OLR of approximately 2.7 kg COD/m^{3}d the biogas flowrate varied between 5.7 and 6.3 L/d. The HRT was maintained at 23.6 h with feed COD_t concentration of 5 582 mg/L. However, the COD_t TE decreased to 54.4%. The biogas composition increased in CH_4 to between 60.4% and 70%, with a slight increase in CO_2 from 10.4% to 11%. Ammonium and nitrate concentration values ranged from 0.27 to 2.4 mg/L and from 0.05 to 4.3 mg/L, respectively.

Table 4.4: Biogas composition at different operation conditions during EGSB anaerobic treatment for softdrink industry wastewater (SDIW)

	Operating Conditions			COD_t			Average Biogas Flowrate	Biogas Composition					
Operating Stages	HRT	V_{up}	OLR	Feed	Product	TE		CH_4	CO_2	H_2S	H_2	N_2	O_2
	h	m/h	kg COD/m^{3}d	mg/L	mg/L	%	L/d	%	%	%	%	%	%
Start-up 1	48.4	0.59	0.9	1 167	194.50	83.3	2.5	30 - 32.3	16.2 - 18	0.5 - 0.71	<0.1	39.3 - 45.4	7.6 - 9.6
Stress period	25.1	1.1	5	4 622	3 397	26.5	8.8	9.4 - 10.4	23.2 - 25.1	0.8 - 0.2	<0.1	50	15 - 16.2
Start-up 2	47.8	1.1	5.1	9 950	4 372	56.1	9.5	26.2 - 30	17 - 18.1	0.2 - 0.15	<0.1	41 - 41.3	11.7 - 14.1
Mode-1a	23.7	1.1	2.8	2 212	775	65	8.6	47.0	9.60	<0.1	<0.1	35.6	7.9
Mode-1b	23.6	0.85	2.7	5 582	2 547	54.4	6.2	60.4 - 70	10.4 - 11	<0.1	<0.1	14.8 - 20.9	4.1 - 5.3
Mode-2a	16.9	0.85	7.6	4 540	2 730	39.9	7.9	44 - 44.7	15.2 - 16	<0.1	<0.1	30.2 - 31.6	8.5 - 9.7
Mode-2b	18	1.1	6.2	5 103	2 205	56.8	2.7	30.3 - 31.6	12.7 - 13.5	<0.1	<0.1	40.6 - 41.3	14.9 -15.1
Mode-3a	11.4	1.1	10.9	4 637	1 345	71	16.7	36.9 - 37.7	13.1 - 16.2	<0.1	<0.1	34.2 - 35.6	11.5 - 12.7
Mode-3b	11.8	0.85	9.9	4 007	1 104	72.4	23	52.4 - 52.6	10.6 - 11.2	<0.1	<0.1	27 - 27.1	9.3 - 9.6

During Mode-2a of operation the OLR was increased to approximately 7.9 kg COD/m^{3}d and the HRT was decreased to approximately 18 h. The varying feed COD$_t$ concentration of 4 540 to 4 982 mg/L at a V$_{up}$ of 0.85 m/h. However, the reactor displayed a decrease in performance with a TE of COD$_t$ of between 38.9% and 47%. The biogas flowrate varied from 7.9 to 11.4 L/d and the CH_4 and CO_2 concentrations were approximately 44 to 44.7% and 15.2 to 16%, respectively, while N_2 and O_2 concentration values varied from 30.2 to 31.6% and from 8.5 to 9.7%, respectively. There was a slight increase in ammonium and nitrate concentration values from 0.6 to 1.8 mg/L and 0.06 to 0.6 mg/L, respectively. At an OLR of 6.2 kg COD/m^{3}d, and the feed concentration of COD$_t$ 5 103 mg/L, an Increasing TE that was obtained was 56.8% and 58.5%, respectively. The biogas flowrate was low at 2.7 L/d with a decrease in CH_4 and CO_2 from 30.3 to 31.6%, and from 12.7 to 13.5%, respectively. N_2 and O_2 concentration values in the biogas increased from 40.6 to 41.3% and 14.9 to 15.1%, respectively. The ammonium and nitrate increased from 0.64 to 2.12 mg/L and from 0.09 to 1.07 mg/L, respectively, in the EGSB product.

During Mode-3a with a high OLR of 10.9 kg COD/m^{3}d and the V$_{up}$ was maintained at 1.1 m/h, the EGSB feed COD$_t$ concentration was 4 637 mg/L and the performance of the EGSB was good with TE of 71 and 70.1%, respectively. The biogas flowrate was increased to 16.7 L/d with a slight increase in CH_4 and CO_2 content from 36.9 to 37.7% and 13.1 to 16.2%, respectively, and a slight decrease in N_2 and O_2 content from 34.2 to 35.6%, 11.5 to 12.7%, respectively. Ammonium and nitrate concentrations increased from 0.66 to 2.39 mg/L and 0.03 to 2.03 mg/L, respectively. Subsequently, the V$_{up}$ was decreased during Mode-3b to 0.85 m/h; the HRT was maintained at approximately 11.8 h, and the OLR decreased slightly to 9.9 kg COD/m^{3}d. The feed concentration was COD$_t$ of 4 007 mg/L and the TE was approximately 72.4%. The biogas flowrate was almost double when compared to Mode-2a at 23 L/d, with an increase in CH_4 content to 52.6%, and a slight decrease in CO_2, N_2 and O_2 content of 10.6 to 11.2%, 27.1% and 9.3 to 9.6%, respectively.

4.3.4 Biogas summary

Biogas production was low at the beginning of the study due to the low OLR. The performance of the EGSB was satisfactory during Mode-3a and Mode-3b of operation. When, the V$_{up}$ was decreased to 0.85 m/h and CH_4 production increased to 52.6%. The EGSB system showed a continuous increase in biogas production rate with increasing OLR, as shown in Figure 4.14, which could be attributed to the continuous growth of methanogenic microbial activity, allowing the EGSB to operate at even higher loading rates (Wijekoon *et al.,* 2011).

CH_4, CO_2, H_2S, N_2 and O_2, concentrations of up to 70%, 11%, 14.8%, and 4.1%, respectively. This period, combined with the applied process changes, was further characterised by varied EGSB feed COD_t concentration, which ranged from 2 212 to 5 582 mg/L. This resulted in unpredictable process performance as displayed by the EGSB COD_t TE, varying from 44.5 to 70.5%, as shown in Figures 4.13 and 4.14. Specifically, COD_t removal during Mode-3 was greater than Mode-1 and Mode-2. However, the treatment efficiency during Mode-2 showed more variations from 39.9 to 47% and 56.8% with an increasing V_{up} from 0.85 to 1.1 m/h at a HRT of between 11.4 and 11.8 h.

Despite the difference in start-up times, an overall similarity in process performance was observed throughout the study, which emphases, in this case the comparability of results that were generated from the EGSB anaerobic treatment for SDIW. Towards the end of the study the analogous performance of the reactor was particularly evident with respect to values for COD removal, biogas volumes and CH_4 content, as indicated in Figure 4.13. The process stability in terms of wastewater treatment and CH_4 production was apparent in the reactor at this time. This difference was more pronounced during Mode-3, when the reactor was exposed to the highest OLR of 10.9 kg COD/m^{3}d. This can be an indication of two factors, namely increased microbial activity and increased organic matter (Wijekoon *et al.,* 2011). At a V_{up} of 0.85 m/h the EGSB produced more biogas than at V_{up} of 1.1 m/h. The average CH_4, CO_2, N_2 and O_2 content in the biogas during the study (Mode-1 to Mode-3) was 46.2%, 12.4%, 31.2% and 9.7%, respectively. However, the highest CH_4 production content was obtained at a V_{up} of 0.85 m/h, HRT of 24 h and OLR of 2.7 kg COD/m^{3}d.

4.4 Performance of the Modified Ludzack-Ettinger (MLE) membrane bioreactor post-treatment

Application of the membrane bioreactor (MBR) was investigated as a post-treatment phase for SDIW. Refer to Table 3.3 for operating conditions in Section 3.4.3.1.

4.4.1 MBR start-up

The EGSB product with a COD_t concentration of 1 092 mg/L was fed to the anoxic zone at 10 L/d for 5 days to allow the system to acclimatize to the SDIW. The EGSB product showed significant variations in composition, as shown in Table 4.5.

4.4.2 MBRTreatment efficiency

Figure 4.15 represents the COD_t TE for the MBR system. The composition of the wastewater in both the anoxic and aerobic zones at different operating modes is presented in Appendices D1 and D2. The average MLSS was 356.3 mg/L in the anoxic zone and 399.8

mg/L in the aerobic zone, which was low for a typical MBR sludge retention time (SRT) of 14 days. However, this may be due to several electrical and mechanical problems, which interrupted the MBR system during acclimatisation which resulted in sludge washout. The MLSS should be higher to enhance the degradation process in order to get the best performance in treating high strength wastewater (Mutamim *et al.,* 2012). The average OLR was approximately 0.14 kg COD/m^{3}d for the anoxic zone and 0.9 kg COD/m^{3}d for the aerobic zone, with the average inlet COD$_t$ to the MBR of 158.4 mg/L. The average COD$_t$ TE was approximately 64.7%. A COD$_t$ of 104.4 mg/L for the treated SDIW in the UF permeate was within the CCT wastewater and industrial discharge standards, as shown in Table 4.5. The EC, TDS, TSS and turbidity concentrations were 1 012 µS/cm, 729 mg/L, 98.7 mg/L and 13.6 NTU (Figures: 4.16, 4.17, 4.18 and 4.19).

On day 17 it was noted that the MLSS in both the anoxic and aerobic zones further decreased to 70 mg/L and 180 mg/L, respectively. As a result, on day 18 the anoxic and aerobic zones were re-inoculated with AS with TSS of 3 032 mg/L and VSS of 3.5%. Bioballs were added to each zone and the system was put on recycle for 2 days so that acclimatization could occur. On day 20 the MLSS for the anoxic zone increased to 712.1 mg/L and 3 488 mg/L for the aerobic zone. The OLR was 1.9 and 3.7 kg COD/m^{3}d for both the anoxic and aerobic zones, respectively, with an inlet COD$_t$ of 94.4 mg/L. The COD$_t$ TE that was achieved was 79.7%, which is higher when compared to Mode-1a. The UF permeate results indicate a slight decrease in turbidity levels 3.4 NTU and an increase in TSS to 99.6 mg/L, EC to 1 530 µS/cm and TDS to 1 077 mg/L.

Table 4.5: EGSB, MBR and overall treatment efficiencies

Parameters	EGSB Feed	EGSB Product	TE%	MBR Permeate	MBR TE %	Overall TE%	CCT^	SANS 241^^
Physio-chemical properties								
pH	5.2	7.3	-	9.2	-	-	5.5 -12.0	≥5 ≤ 9.7
Temperature (^{o}C)	22.8	22.3	-	22.0	-	-	0 - 40	-
Alkalinity (mg/L)	136.8	1 724	-	4 016	-	-	-	-
Conductivity (µS/cm)	560.1	4 057	-	4 129	-	-	≤ 500	≤ 170
Total Dissolved Solids (mg/L)	361.9	2 725	-	2 753	-	-	1 000	≤1 200
COD_t (mg/L)	5 433	2 245	58.7	253.5	88.7	95.3	≤ 5 000	1 000 – 2 400
COD_s (mg/L)	5 140	2 051	60.1	185.5	91.0	96.4	-	-
Fructose (mg/L)	201.3	9.0	95.5	0.0	100	100	1 500	-
Sucrose (mg/L)	2 597	631.0	75.7	25.0	96.0	99.0	1 500	-
Turbidity (NTU)	22.4	50.7	-	79.1	-	-	-	≤ 1
TSS (mg/L)	33.4	99.0	-	211.0	-	-	4 000	-
Colour (mg Pt/L)	19.0	18.8	0.9	34.3	-	-	-	≤ 15
Inorganic parameters								
Sodium	122.2	537.6	-	756.0	-	-	1 000	≤ 200
Boron (mg/L)	0.0	0.0	-	0.0	-	-	5	-
CO_3 (mg/L)	-	-	-	-	-	-	-	-
HCO_3 (mg/L)	76.5	1 623	-	1 856	-	-	-	-
Calcium (mg/L)	19.0	16.4	13.3	16.0	2.4	15.8	-	-
Chloride (mg/L)	77.9	32.3	58.6	44.1	-	43.4	1 500	≤ 300
Fluoride (mg/L)	0.5	0.2	51.9	1.0	-	-	-	≤ 1.5
Ammonium (mg/L)	1.2	4.2	-	0.5	88.1	59.5	-	≤ 1.5
Nitrate (mg/L)	0.3	2.6	-	2.7	-	-	-	≤ 11
Potassium (mg/L)	1.4	3.3	-	8.1	-	-	-	-
Magnesium (mg/L)	28.0	1.9	93.3	2.8	-	90.1	-	-
Sulphates (mg/L)	15.1	10.1	33.4	8.6	14.9	43.4	1 500	≤ 250
Zinc (mg/L)	23.9	0.0	99.9	0.3	-	98.8	30	≤ 5
Copper (mg/L)	0.0	0.0	55.2	0.6	-	-	20	≤ 2
Iron (mg/L)	1.9	1.0	46.5	0.1	90.0	92.2	50	≤ 0.3
Manganese (mg/L)	0.2	0.1	29.1	0.0	100	92.9	-	≤0.1
Phosphate (mg/L)	1.1	7.4	-	27.0	-	-	25	-
Orthophosphate (mg/L)	0.9	6.4	-	7.0	-	-	-	-

^: City of Cape Town: Wastewater and industrial wastewater by-law (South Africa (Western Cape), 2006
^^: SANS 241-1:2011 South African National Standard (SANS 241) (Department of Water Affairs, 2011)

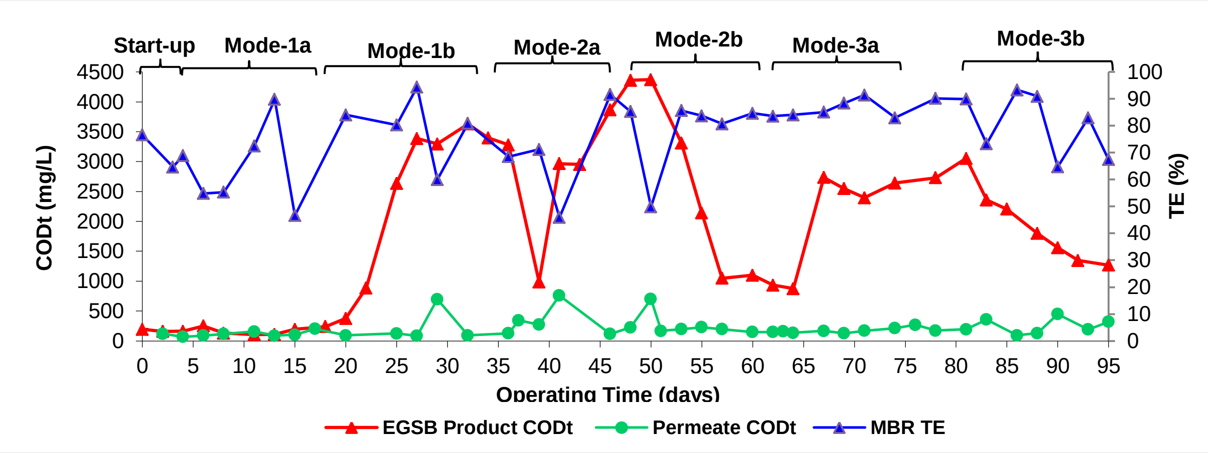


Figure 4.15: MBR treatment efficiency

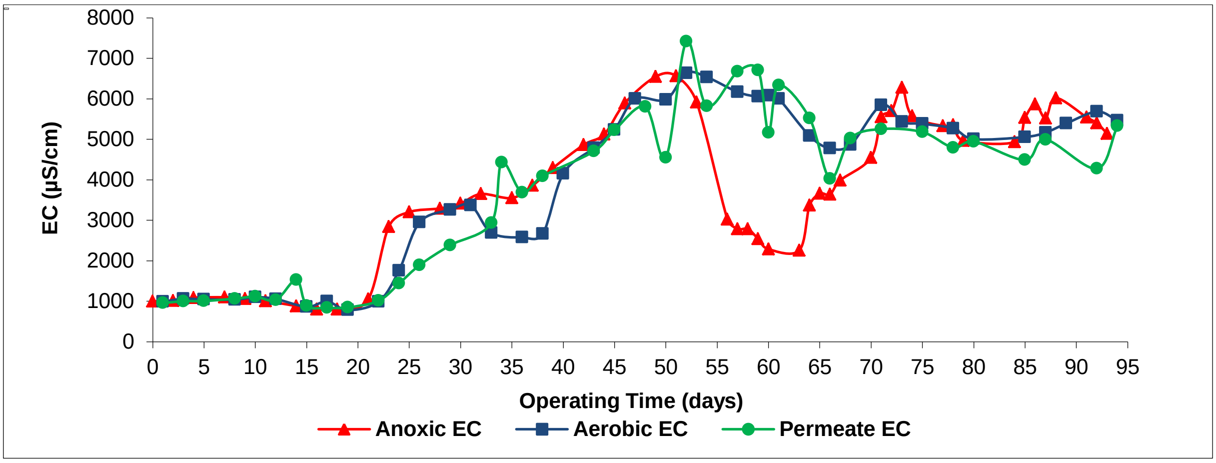


Figure 4.16: EC concentrations for anoxic, aerobic and permeate during MBR treatment

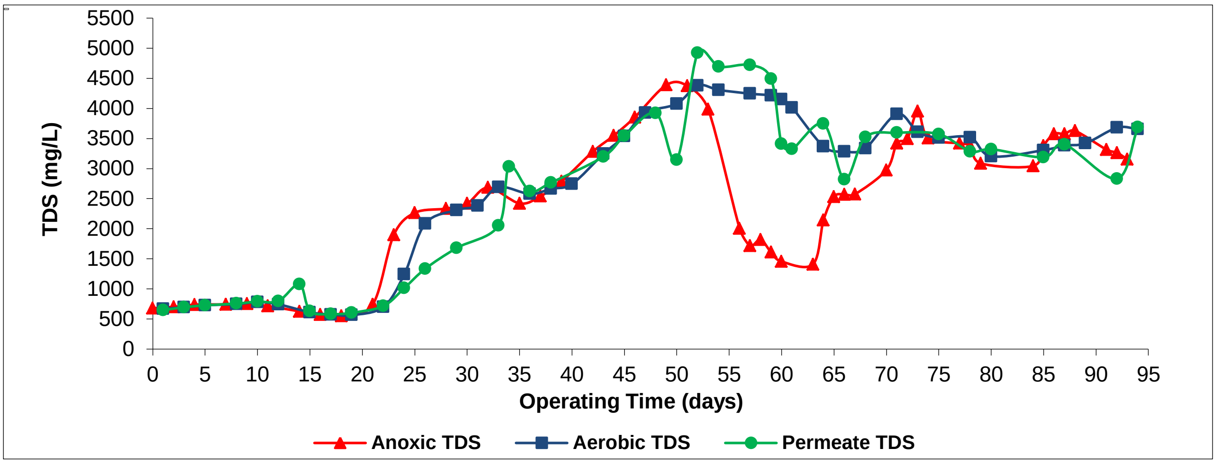


Figure 4.17: TDS concentrations for anoxic, aerobic and permeate during MBR treatment

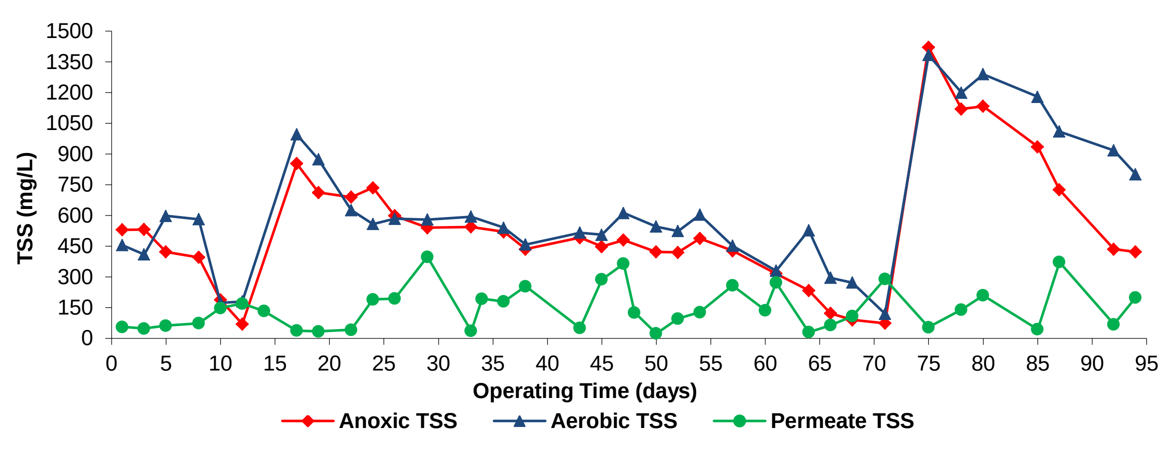


Figure 4.18: TSS concentration for anoxic, aerobic and permeate during MBR Treatment

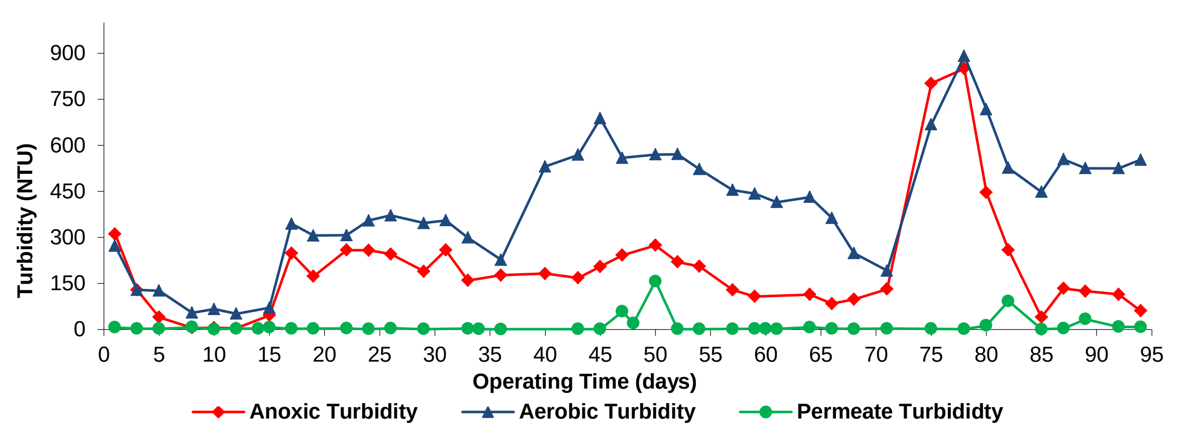


Figure 4.19: Turbidity concentration for anoxic, aerobic and permeate during MBR Treatment

While the EGSB system was being re-inoculated due to system failure resulting from the stress period (Section 4.4.3), the MBR system was put on recycle and the UF membranes were cleaned in place. The MBR was restarted on day 37 and the flowrate was decreased to an average of 12.2 L/d during Mode-2a, the anoxic zone HRT was 11.5 h and aerobic zone HRT was 0.41 h with corresponding OLR of 1.1 and 1.5 kg COD/m^{3}d, respectively. The average MLSS in both the anoxic and aerobic zones was 474 and 505 mg/L, respectively, which decreased rapidly due to overflowing of the tanks with subsequent biomass loss. The EGSB product to the anoxic zone had an average COD$_t$ of 3 251 mg/L, and the average removal efficiency that was obtained was up to 73.5%. It can be noted that there was a slight decrease in the COD$_t$ when compared to Mode-1b. The average UF permeate EC and TDS increased to 4 428 µS/cm and 3 032 mg/L, respectively. The average BOD, TSS, COD$_t$ were high in the UF permeate, 10; 193; 344.3 mg/L, respectively, and turbidity of 36.6 NTU; which is usually considered high for recycling or re-use. High turbidity as a result of membrane failure (refer to Section 4.4.4).

The average OLR was 2.8 and 1.1 kg COD/m^{3}d with a HRT of 5.8 h and 0.41 h for the anoxic and aerobic zones, respectively. The average COD$_t$ that was fed to the MBR system was 1 707 mg/L, with an increasing average TE of 83.9% when compared to Mode-2a. The average MLSS in the anoxic and aerobic zones were 439.5 and 531.5 mg/L, respectively. Turbidity in the permeate decreased to 21.9 NTU and TSS to 124.5 mg/L. However, the average EC and TDS concentrations increased to 5 882 µS/cm and 3 983 mg/L, respectively, in the UF permeate, which was too high when compared to the CCT's industrial wastewater quality discharge by-laws (2006).

The feed flowrate to the MBR was increased to an average of 33.7 L/d for Mode-3a from day 64 to 73 with an OLR of 2.3 and 3.1 kg COD/m^{3}d for both anoxic and aerobic zones, respectively. The average MLSS decreased to 139.3 mg/L and 257.7 mg/L for the anoxic and aerobic zones, respectively, with HRT decreasing to 4.2 h in the anoxic zone and to 0.41 h for the aerobic zone. A decrease was noted as the system failed to meet the required HRT, and this increased membrane fouling and clogging. Despite the problems experienced with membranes fouling, the MBR was able to achieve an average COD$_t$ TE of 86.4% at an average COD$_t$ MBR feed concentrations of 2 239 mg/L. The average EC and TDS decreased slightly in the UF permeate to 5 162 µS/cm; 3 408 mg/L, respectively; turbidity increased to 22.7 NTU and TSS to 137 mg/L when compared to Mode-2a.

On day 76, sewage sludge was added to the MBR and both tanks were re-inoculated as the MLSS of 74.0 mg/L and 120.0 mg/L for the anoxic zone and the aerobic zones were too low for SRT of 22 days. The system was put on recycle for 2 days so that acclimatization could occur, and for the UF membranes to be cleaned. Mode-3b operated from day 79 to 97 when the system was operated at an increased feed flowrate of 48.8 L/d and HRT of 2.9 h for the anoxic zone, and at 0.41 h for the aerobic zone. Throughout this operating mode, the average feed COD$_t$ concentration to the anoxic tank was 1 935 mg/L with an OLR of 3.1 kg COD/m^{3}d for the anoxic zone and 2.4 kg COD/m^{3}d for the aerobic zone. However, COD$_t$ TE decreased to 78.6% compared to Mode-3a. Lower COD$_t$ removal may be correlated to less organic matter concentration in both the anoxic and aerobic zones. The average TSS, EC and TDS of the UF permeate remained high at 155.4 mg/L, 4 937 µS/cm and 3 357 mg/L, respectively.

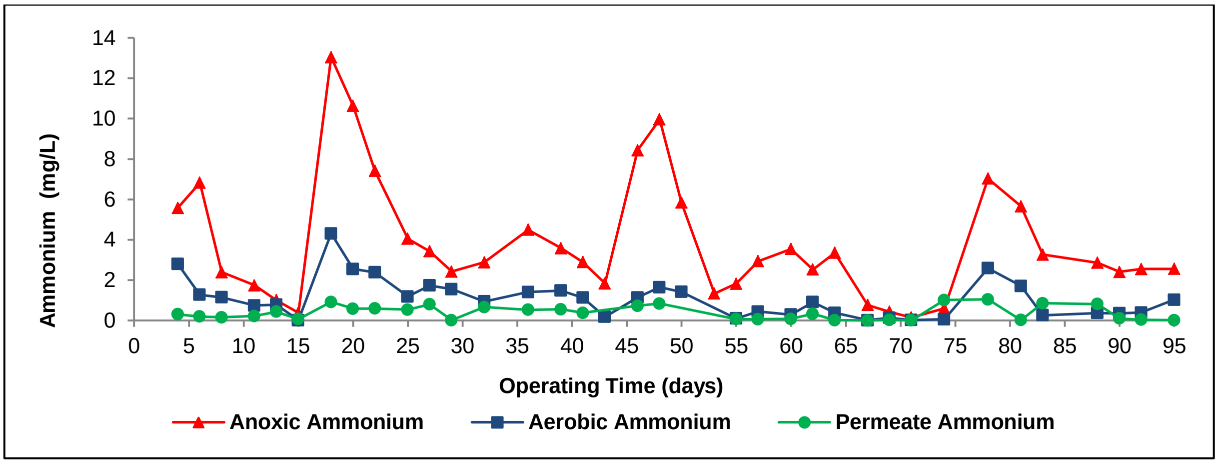


Figure 4.20: Ammonium concentration during MBR treatment

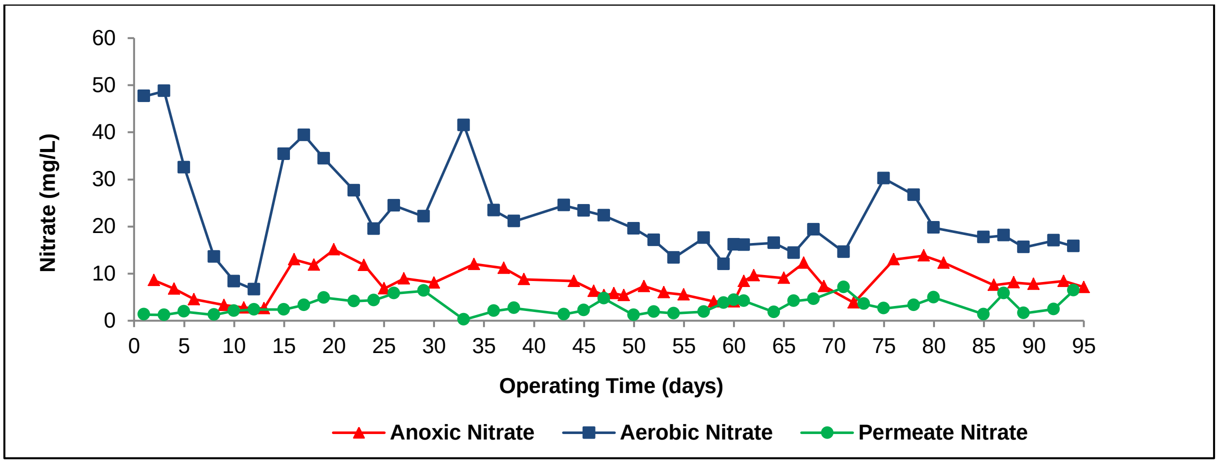


Figure 4.21: Nitrate concentration during MBR treatment

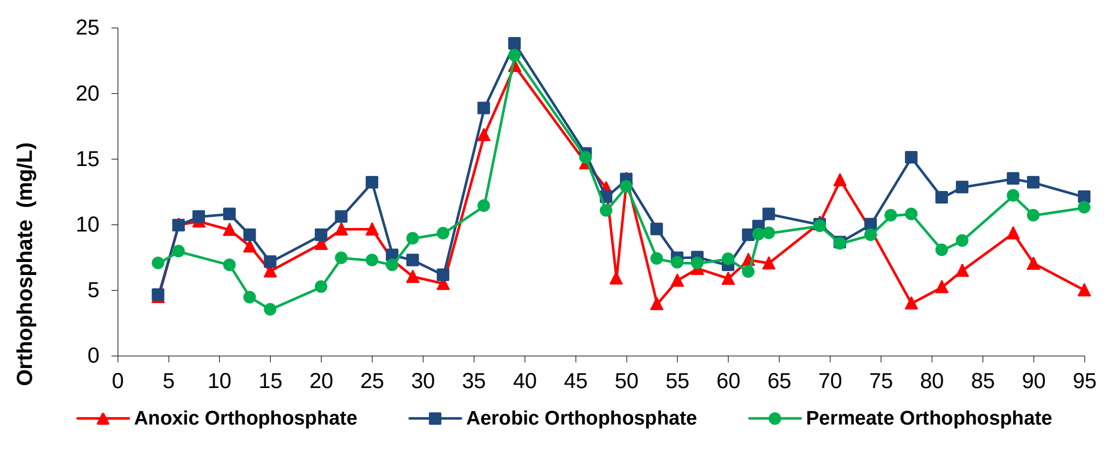


Figure 4.22: Orthophosphate concentration during MBR treatment

4.4.3 Relationship between dissolved oxygen (DO) and nutrient removal during MBR post-treatment

During this study, the aeration was adjusted to obtain suitable dissolved oxygen (DO) in the anoxic and aerobic zones. As previously discussed in Chapter 2, Section 2.6 of the literature review, wastewater can be comprised of nitrogen in the form of organic nitrogen, ammonium (NH_4^+), nitrite (NO_2-N) and nitrate (NO_3-N). Biological treatment of wastewater for the removal of nitrogen occurs in three stages: 1) ammonification (breakdown of organic N to NH_3-N); 2) nitrification (oxidation of NH_3-N to NO_3-N); and 3) denitrification (conversion of NO_3-N to N_2) (Wang *et al.,* 2009). The pH value is considered as an indication of alkalinity in the mixed liquor. Most microorganisms, mainly the denitrifying and nitrifying microorganisms, are sensitive to alkalinity (Meng *et al.,* 2013).

The ammonium, nitrate and orthophosphate concentrations for anoxic, aerobic and UF permeate are presented in Figures 4.20, 4.21 and 4.22. During Mode-1a, the average DO was 1.2 mg/L in the anoxic zone. However, a higher DO is detrimental for anoxic conditions to proceed, since DO must be less than 0.5 mg/L for denitrification to occur (Wang *et al.,* 2009). DO in the aerobic zone of 7.0 mg/L was suitable for nitrification to take place at a corresponding aeration rate of 14.7 L/min. The anoxic and aerobic zones average temperature and pH were 22.5˚C and 7.9; and 21.2˚C and 8.5, respectively. The NH_4^+ present in the anoxic zone was converted into nitrate in the aerobic zone. However, the average concentration of ammonium, nitrate and orthophosphate in the UF permeate was 0.2, 1.7, and 7.2 mg/L, respectively. Therefore, the alternation to nitrification without any external carbon source addition to the anoxic and aerobic zones resulted in ammonium, nitrate and orthophosphate removal in the MBR of 85.4%, 53.5% and 14.2%, respectively. Despite this, the average ammonium and nitrate concentration of the permeate was lower than the limit 1.5 and 11 mg/L, which is stipulated in SANS 241:2011. The aeration supplied to the aerobic zone was 11.78 L/min with an average DO of 5.7 mg/L during Mode-1b and DO of 2.1 mg/L in the anoxic zone, which corresponded to Mode-1a of operation. The anoxic and aerobic zones average temperature and pH were 22.1˚C and 8.3; and 21.5˚C and 8.8, respectively, during Mode-1b. The average ammonium, nitrate and orthophosphate removal increased to 89.9%; 55.3% and 38.6%, respectively, with the average concentration of ammonium, nitrate and orthophosphate in the UF permeates being 0.6; 3.6, and 6.6 mg/L, respectively. Decreased average ammonium, nitrate and orthophosphate concentrations in the UF permeate were observed when compared to Mode-1a.

The average DO for the anoxic and the aerobic zones were decreased to 0.6 and 4.6 mg/L, respectively, as compared to Mode-1b of operation in spite of an increase in the aeration rate of the aerobic zone to 16.4 L/min during Mode-2a. The anoxic and aerobic zones average temperature and pH increased to 23.4˚C and 9.1; and 22.5˚C and 9.3, respectively. The average concentrations of ammonium and nitrate in the UF permeate were 0.6 and 2.1 mg/L, respectively, and the percentage removal increased to 90.9% and 75.1%, respectively. A high average orthophosphate concentration value of 16.4 mg/L was recorded in the UF permeate when compared to Mode-1b. A decrease in orthophosphate removal could be due to the loss in orthophosphate biomass; hence, orthophosphate concentration levels did not meet the SANS 241:2011 drinking water standards and CCT's, industrial wastewater quality by-laws (2006).

The average DO was 1.3 mg/L for the anoxic zone and 6.2 mg/L for the aerobic zone, with a corresponding aeration of 17.1 L/min during Mode-2b. The average ammonium and nitrate concentrations in the UF permeate were 0.14 and 2.1 mg/L, respectively, while the ammonium removal increased to 94.7%. Conversely, nitrate removal decreased to 50%. A decrease in the percentage removal of nitrate could be due to the problems experienced with the nitrification cycle or with a process called simultaneous nitrification and denitrification (SND) which suggested that SND occurred during MBR because of the coexistence of the anoxic and aerobic zones (Zang *et al.,* 2013). SND starts with an incomplete nitrification of NH_4^+ to nitrite and afterwards carries on with a direct nitrite reduction to N_2 gas (Jenicek *et al.,* 2004; Lai *et al.,* 2004). SND nitrification and denitrification occur at the same time in the same zone under identical operating conditions (Breisha & Winter, 2010). According to Dai *et al.* (2010) and Gao *et al.* (2001), when the DO concentration is higher than 4 mg/L, bacterial decomposition of carbohydrates increases, and the hydrophobic constituents would be adsorbed on the membrane pore, which will be difficult to wipe off. Higher DO lead to a gradual decrease in orthophosphate removal, which is triggered by nitrate influence. Also, nitrate concentration levels gradually increased as increased oxygen hampers the degree of SND in the MBR system. It was noted that an increasing DO increased the permeate concentrations of orthophosphate to 7.3 mg/L. Overall, the degree of orthophosphate removal is influenced by the nitrate concentration in the MBR (Nopens *et al.,* 2007).

During Mode-3a the ammonium moved that was up to 97.4% and nitrate removal increased to 53.2% when the DO was about 2.3 mg/L for the anoxic zone, and 6.5 mg/L for the aerobic zone with a corresponding aeration of 11.4 L/min; which indicated that nitrification was a key process equivalent to Mode-3. The average ammonium and nitrate concentration of feed water was 0.2 and 4.4 mg/L, respectively. However, the orthophosphate concentration increased to 9.2 mg/L. The temperature and pH recorded in the anoxic zone was 25.6˚C and 9.1; and 23.5˚C and 10 in the aerobic zone. It is reported that an increase in pH above 8.6 triggers a substantial increase in nitrite accumulation and a decrease in the nitrate removal rate (Lee *et al.,* 2003). Higher DO concentrations were maintained during Mode-3b in order to promote nitrification, DO of approximately 2.5 mg/L in the anoxic and DO of 6.1 mg/L in the aerobic zone with a corresponding aeration rate of 13.7 L/min. The temperature and pH recorded in the anoxic zone was 27.7°C and 8.6; and 24.9°C and 9.8 in the aerobic zone. The average ammonium and nitrate concentration values were 0.4 mg/L and 3.6 mg/L, respectively, with a decreasing ammonium removal to 89.7% and increasing nitrate to 60.8%, whereas the orthophosphate concentration increased to 10.7 mg/L when compared to Mode-3a.

4.4.4 Membrane perfomance, air scouring and CIP

Over the entire period of this study, the membrane performance was monitored by the development of backwash pressure as a function of membrane permeates flux and air scouring intensities. It was noticed that physical backwashing, to some degree, improved membrane permeability to its original values, but the reversible fouling and leakages was found to gradually accumulate after repeated physical backwashing due to the accumulation of the sludge that remained on the UF membranes.

CIP was performed twice during this operating Mode-3a since the backflush pressure went above 70 kPa and sludge build-up was noticed on the UF membranes as shown in Figure 4.23. It is noticeable that backflush pressure increased due to the fouling of the UF membrane. Backflush pressure reached 80 kPa on day 67, which was the quickest fouled UF membrane. As a result, the air scouring rate was increased and maintained to 2.5 L/min and the UF membrane flux to 3.9 L/m^{2}h for both Mode-3a and 3b. In this phase biological, colloidal, particle, and organic materials quickly accumulated onto the membranes, and formed a sludge build-up, which led to a quick or rise in the backflush pressure. This could be caused by two types of fouling: 1) irreversible fouling cause by solute and small colloids that block membrane pores, and CIP cleaning may be required (Hosseinzadeh *et al.,* 2013); and 2) reversible fouling caused by deposition of large constituent and flocculants on the surface of the membrane that can be achieved with effective physical cleaning (Stoller *et al.,* 2013).

The average TSS of the UF permeate was high at 152.9 mg/L. This was due to visible cracks on the membranes as the epoxy became lose, as shown in Figure 4.24. During this period CIP was conducted after manually removing the sludge that accumulated on the shell side of the UF membrane modules. This tendency of sludge collecting on the shell side of membrane modules indicated that membrane fouling was increased during operation of the MBR. CIP was performed thrice during this period, since the backflush pressure went above 70 kPa and sludge build-up and leakages were noticed on the UF membranes

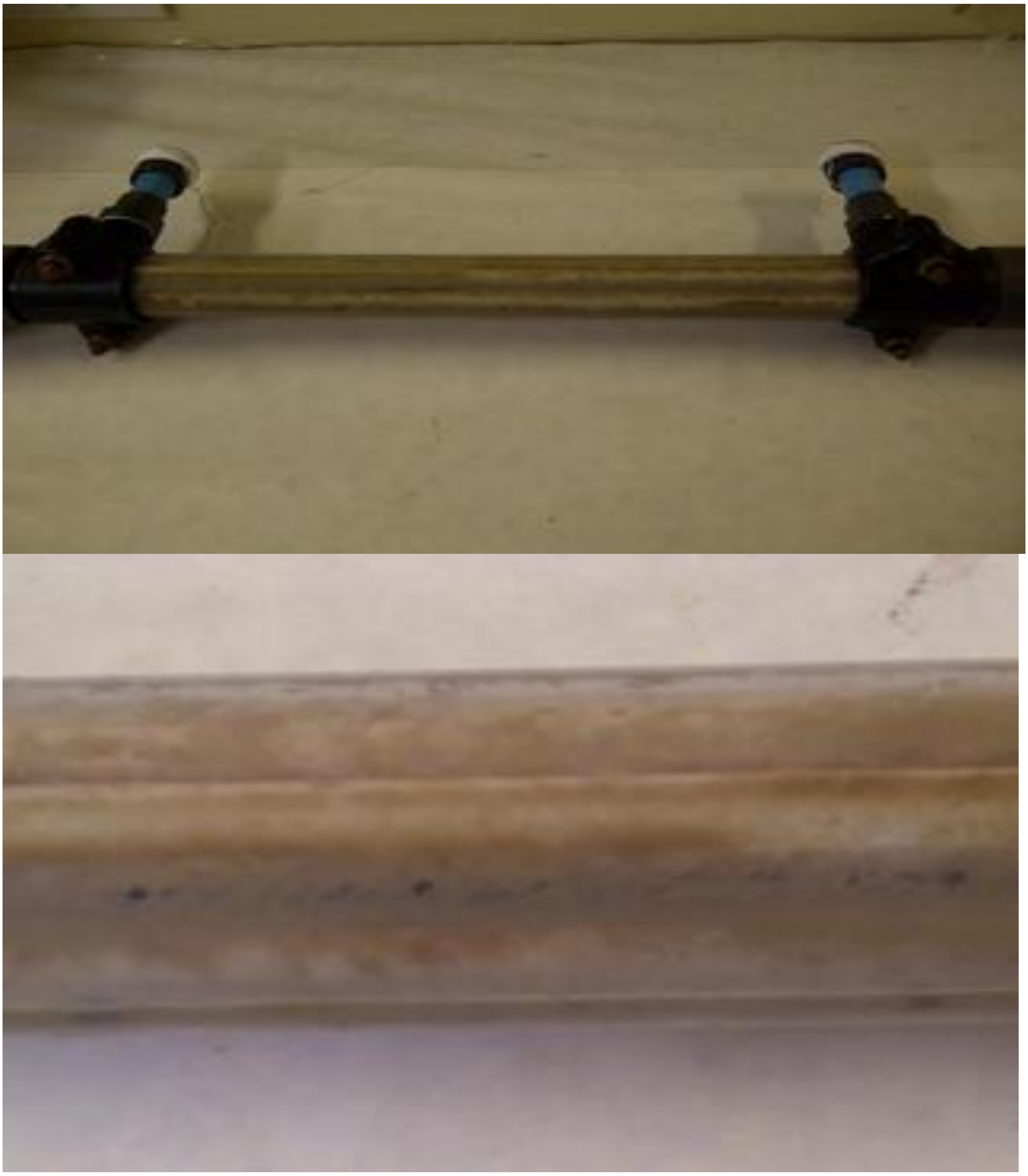


Figure 4.23 (a) and (b: Sludge permeation through the UF membranes

During Mode-1 of operation the MBR system operated at a low flux of 1.4 L/m^{2}h to limit severe and rapid membrane fouling. It was noted that MBR fouling decreased with an increasing air souring rate to 2 L/min and during Mode-2 of operation flux also increased from 1.8 to 2.8 L/m^{2}h. Furthermore increasing air scouring rates had little or no influence on fouling reduction during Mode-3, since the membranes were CIPed frequently. During Mode-3, membrane fouling was unavoidable in MBR permeate flux decline, as CIP helped to reduce the amount of TMP, but did not restore the full capacity of the membrane. Initially the cracks on the UF membranes were not visible. Towards the end of the study the cracks were noticed, which indicated that the UF membranes were near the end of its life as a result of the experiments were discontinued. In general, the recovery of membrane permeability was strongly governed by operating conditions, membrane materials and sludge characteristics.

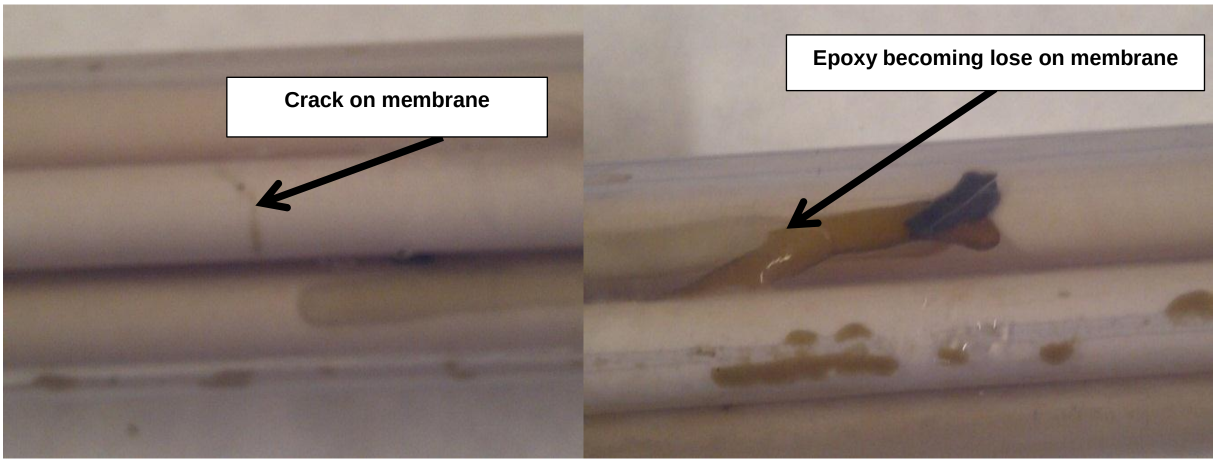


Figure 4.24: Cracks on the membranes and epoxy becoming lose

4.4.5 MBR post-treatment summary

Despite the problems experienced with low MLSS and membrane cracks, the MBR was able to achieve an average COD_t removal of 86.4%. The study indicated that the air scouring of 2.5 L/min and UF flux of 3.85 L/m^2h had influence on the membrane fouling. The UF permeate showed high amounts of the EC and TDS. This could be the result of low biomass concentration in the bioreactor, resulting in efficient biodegradation of the contaminants present in the wastewater.

Mode-1b provided satisfactory nutrient removal at an aeration rate of 11.8 L/min in the aerobic zone. DO was 5.7 mg/L and DO of 2.1 mg/L in the anoxic zone, although it was higher than the required DO in the anoxic zone of 0.2 to 0.5 mg/L, as reported by Wang *et al.* (2009). The average ammonium, nitrate and orthophosphate removal increased to 89.9%; 55.3% and 38.6%, respectively. However, increasing DO during Mode-2b and 3b was only efficient for ammonium removal, while it had a negative influence on nitrate removal because of the simultaneous nitrification and denitrification (SND). The orthophosphate concentrations that entered the MBR system varied unpredictably. The MBR did not decrease the orthophosphate concentration to within the SANS 241:2011 drinking water standards. This result was rather unexpected, as the orthophosphate content is considered to be significant in the biological treatment of wastewater. However, the ammonium and nitrate level decreased to below that of the potable, which was used by the softdrink industrial partner, as shown in Table 4.6, Section 4.1.

4.5 Overall EGSB and MBR treatment efficiency

The EGSB pre-treatment reactor was operational for 135 d during which the average COD_t removal efficiency was up to 58.7%, with a maximum of 92.9%. The EGSB pre-treatment reactor successfully degraded organic material from SDIW and provided stable results for 94 days. The MBR reactor was highly efficient and the average removal of COD_t with a maximum of 89%. Figure 4:25 represents the overall TE for the integrated EGSB and MBR. Membrane modules played a significant role in the permeate quality in terms of COD, to make up for the variability of the biological treatment. The overall treatment efficiency achieved was above 90%. Table 4.6 presents the comparison between EGSB TE, MBR TE, and overall EGSB-MBR TE along with the CCT (2006) and SANS: 241 2011.

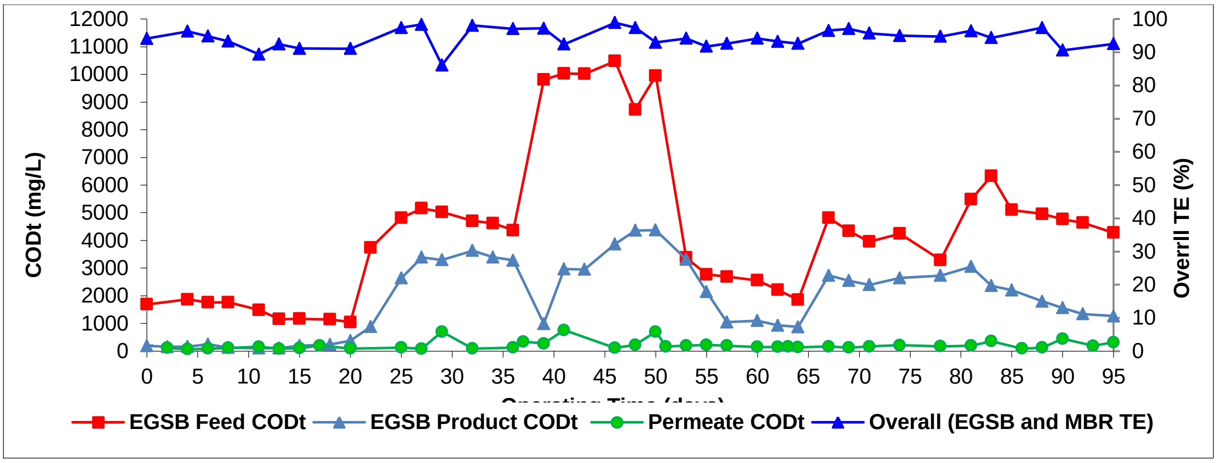


Figure 4. 25: Overall treatment efficiency for integrated during EGSB and MBR

The MBR post-treatment provided a satisfactory anoxic and aerobic environment, which was favourable to the removal of ammonium and nitrate. The average UF permeate COD_t and COD_s was below 250.5 and 218.8 mg/L, and ammonium and nitrate concentration was about 0.9 and 4.1 mg/L. The integrated processes EGSB-MBR SDIW treatment COD_t and COD_s removal rates reached up to 94.1% and 81.6%, indicating that the biological process was operated fairly stable, and was promising for future applications. The biological process produced suitable removal of organic components and it was capable of achieving a high removal of COD. Despite the satisfactory MBR performance, the orthophosphate concentration values and colour in the UF permeate were high during the entire study, and was not within the SANS 241:2011 drinking water standards.

Figure 4.26 represents colour difference. A consistent increase in the colour of the incoming SDIW was evident, and unsatisfactory colour reduction was recorded in the UF permeate up to 34.3 mg Pt/L. Due to the presence of colour, as well as high concentrations of EC and TDS present after MBR treatment, the UF permeate will have to be further treated.

Figure 4.26: Colour comparison of the wastewater fed from the holding tank to the permeate tank

4.6 Comparison of MBR permeate to potable water

A summary of the average MBR permeate, potable water and pre-treated water quality that was measured during the entire study, is provided in Table 4.6. The MBR permeate was still within SANS 241:2011, although it was noted that the ammonium and nitrate levels did not decrease below that of the potable and pre-treated water that was used by the softdrink industrial partner. The water of pre-treated plant had a COD and TSS of 2.4 and 5.3 mg/L and EC less than 1.6 µS/cm, respectively, as shown in Table 4.6. The MBR permeate can be treated by methods such as NF, RO, UV and ion exchange (IE), which have been previously investigated for beverage industry wastewater to facilitate re-use (Haroon *et al.,* 2013; Murthy & Chaudhari, 2009; Nataraj *et al.,* 2006; Chmiel *et al.,* 2003; Noronha *et al.,* 2002;

Blöcher *et al.,* 2002). These methods have been used for the treatment of distillery wastewater to remove the colour and contaminants, with COD TE of up to 99.9%. The treated wastewater was suitable to be reused or recycled for both industrial and domestic purposes (Agarwal *et al.,* 2010; Murthy & Chaudhari, 2009; Acharya *et al.,* 2008; Nataraj *et al.,* 2006; Mohana *et al.,* 2007).

According to Côté and Mourato (2004), combining two membrane filtration processes, namely UF and RO, allows industries to produce recycled water. The benefits of an MBR, as a pre-treatment for the RO processes, are the improved wastewater and almost complete rejection of TSS. The MBR and RO treatment processes can assist to save potable water intake, and might influence sustainable water management.

Table 4.6: Composition of average MBR permeate and potable water, pre-treated water for industrial partner

Parameters	**Potable Water**	**MBR Permeate**	**Pre-treated Water**
Physio-chemical properties			
pH	**8.99**	**9.2**	**7.4**
Temperature (^{o}C)	**20.9**	**22.0**	**21.2**
Alkalinity (mg/L)	**22.8**	**4 016**	**4.5**
Conductivity (µS/cm)	**79**	**4 129**	**1.6**
TDS (mg/L)	**62.3**	**2 753**	**4.8**
COD_t (mg/L)	**105.6**	**253.5**	**2.4**
COD_s (mg/L)	**92.7**	**185.5**	**2**
BOD_5 (mg/L)	**-**	**0.0**	**-**
Turbidity (NTU)	**0.78**	**25.0**	**0.1**
TSS (mg/L)	**11.75**	**79.1**	**5.3**
FOG (mg/L)	**-**	**211.0**	**-**
Colour (mg Pt/L)	**<1**	**34.3**	**0**
Fructose (mg/L)	**<1.7**	**9.2**	**-**
Sucrose(mg/L)	**5.5**	**22.0**	**1**
Inorganic parameters			
Sodium	**7.1**	**756.0**	**4.5**
Boron (mg/L)	**0.0**	**0.0**	**0**
CO_3 (mg/L)	**0.8**	**-**	**-**
HCO_3 (mg/L)	**15.7**	**1 856**	**-**
Calcium (mg/L)	**13.4**	**16.0**	**7.0**
Chloride (mg/L)	**12.4**	**44.1**	**5.1**
Fluoride (mg/L)	**0.1**	**1.0**	**0.14**
Ammonium (mg/L)	**0.4**	**0.5**	**0.4**
Nitrate (mg/L)	**0.3**	**2.7**	**0.3**
Potassium (mg/L)	**0.6**	**8.1**	**0.4**
Magnesium (mg/L)	**0.9**	**2.8**	**0.4**
Sulphates (mg/L)	**5.5**	**8.6**	**0.01**
Zinc (mg/L)	**0.01**	**0.3**	**0.01**
Copper (mg/L)	**0.01**	**0.6**	**-**
Iron (mg/L)	**0.04**	**0.1**	**0.0**
Manganese (mg/L)	**0.01**	**0.0**	**0.0**
Phosphate (mg/L)	**0.3**	**27.0**	**0.41**
Orthophosphate (mg/L)	**0.7**	**7.0**	**-**

CHAPTER FIVE
CONCLUSIONS AND RECOMEMNDATIONS

5.1 Conclusions

Objective 1: Evaluate the efficiencies of the anaerobic, anoxic, aerobic and membrane treatment stages under different operating conditions and evaluate the extent of contaminant removal in the individual stages, as well as the overall process.

For the EGSB technology, the capability to apply OLR of 10.9 kg COD/m^{3}d and the COD$_t$ removal was between 62.3% and 92.9% during 45 days of normal operation. The optimum up flow velocity was 0.85 m/h, respectively. The reactor performed better at HRT of approximately 11.8 h, and the TSS and turbidity reduction of 8.2% and 22.4%, respectively. However, during this study all macronutrients (NH_4^+, NO_3^- and PO_4^{3-}) were below the discharged standards, which are stipulated by the CCT's industrial wastewater quality by-laws (2006). The MBR showed that it could successfully operate with the feed flowrate up to 33.7 L/d at an OLR of 2.3 and 3.1 kg COD/m^{3}d for both anoxic and aerobic zones, respectively, with HRT of 4.2 h in the anoxic zone and 0.41 h for the aerobic zone. Despite the problems experienced with the membranes, the MBR was able to achieve the average COD$_t$ removal was up to 86.4%.

Objective 2: Evaluate the gas production and composition under different hydraulic retention times (HRT); upflow velocities and organic loading rates (OLR) in the anaerobic stage.

Anaerobic digestion of SDIW in the EGSB showed high COD$_t$ removal and methane production under different operational conditions. Besides COD$_t$ reduction during EGSB pre-treatment it showed that it has the capability to produce energy. The biogas gas production reached as high as 16.7 L/d. This research has shown that the anaerobic acetogenesis of SDIW is an achievable alternative to biogas production. When the reactor was exposed to a highest OLR of 10.9 kg COD/m^{3}d and a V$_{up}$ of 0.85 m/h the EGSB produced more biogas. The CH_4 and N_2 content in the biogas reached up to 70% and 41.3%, respectively.

Objective 3: Evaluate the performance of an integrated anaerobic EGSB pre-treatment combined with an MBR post-treatment system to treat SDIW.

The combination of the system EGSB-MBR represents a promising option for the treatment of softdrink industry wastewater. The EGSB-MBR for SDIW treatment COD$_t$ and COD$_s$ removal rates reached up to 94.1% and 81.6%, respectively. The nutrient removal at an aeration rate of 11.8 L/min in the aerobic zone and DO of 5.7 mg/L and DO of 2.1 mg/L in the anoxic zone was with ammonium, nitrate and orthophosphate removal increasing to 89.9%; 55.3% and 38.6%, respectively. The MBR did not decrease the orthophosphate concentration to within the SANS 241:2011 drinking water standards.

5.2 Recommendations

- Further research should be conducted to collect additional data on the use of organic acids, which are produced from the anaerobic digestion of SDIW treatment.
- The challenges related to the EGSB operation at higher OLR and lower HRT should be addressed, which include clogging and poor EGSB product and biogas quality.
- The study also revealed that CH_4 was available as a source of energy, and biogas generated from the SDIW during anaerobic digestion can be effectively used in production plant boilers.
- Additional research should be conducted to gain more understanding of the stable performance of the EGSB against various process inhibitors such as ammonia.
- Fouling could be limited by optimising the operating conditions such as: air scouring rates, low flux operation, chemical and physical cleaning and membrane relaxation after backwash.
- Subsequent treatment of the UF permeate with NF or RO for use in agriculture, washing of trucks and cars, bottles, floors and walls and flushing of toilets. It can also be used for firefighting purposes, cooling of power and industrial equipment.
- In addition, financial and technical feasibility studies of the economic and environmental aspects of the industrial application EGSB and MBR technology as an alternative should be conducted.

All of the above views were suggested for the possible expansion of EGSB and MBR technology in the future, both on a pilot-scale and full-scale.

REFERENCES

Acharya, B. K., Mohana, S. & Madamwar, D. 2008. Anaerobic treatment of distillery spent wash-A study on upflow anaerobic fixed film bioreactor. *Bioresource Technology*, 99(11):4621-4626.

Adams, G. M. 2004. *Identifying and Controlling Odour in the Municipal Wastewater Environment: Phase I: Literature Search and Review*. London. IWA Publishing.

Agama Biogas, 2010. Sustainable Cities: Biogas Energy from Waste: *A Feasibility Study and Guidelines for Implementing Biogas from Waste Projects in Municipalities.* Cape Town.

Agana, B. A., Reeve, D. & Orbell, J. D. 2013. Performance optimization of a 5nm TiO_2 ceramic membrane with respect to beverage production wastewater. *Desalination,* 311:162-172.

Agarwal, R., Lata, S., Gupta, M. & Singh, P. 2010. Removal of melanoidin present in distillery effluent as a major colorant: A review. *Journal of Environmental Biology,* 31(4):521-528.

Ahn, K.- H., Song, K.- G., Cho, E., Cho, J., Yun, H., Lee, S. & Kim J. 2003. Enhanced biological phosphorus and nitrogen removal using a sequencing anoxic/anaerobic membrane bioreactor (SAM) process. *Desalination,* 157(1-3):345-352.

Ahn, Y. H. & Speece, R. E. 2004. Settleability assessment protocol for anaerobic granular sludge and its application. *Water SA*, 29(4):419-426.

Ahn, Y. H. 2006. Sustainable nitrogen elimination biotechnologies: A review. *Process Biochemistry,* 41(8):1709-1721.

Akpor, O. B. & Muchie, B. 2013. Environmental and public health implications of wastewater quality. *African Journal of Biotechnology,* 10(13):2379-2387.

Akpor, O. B. 2011. Wastewater effluent discharge: Effects and treatment processes. In 3rd International Conference on Chemical, Biological and Environmental Engineering, IACSIT Press, Singapore.

Akunna, J.C. & Clark, M. 2000. Performance of a granular-bed anaerobic baffled reactor (GRABBR) treating whisky distillery wastewater. *Bioresource Technology,* 74:257-261.

Alam, A. S. M. M., Hossain, K. M., Hossain, B., Ahmed, A. & Hoque, M. J. 2007. A Study on Industrial Waste Effluents and Their Management at Selected Food and Beverage Industries of Bangladesh. *Journal of Applied Sciences and Environmental Management,* 11(4):5-9.

Al-Hashimia, M. A. I., Abbas, T. R. & Jasema, Y. I. 2013. Performance of sequencing anoxic/anaerobic membrane bioreactor (SAM) system in hospital wastewater treatment and reuse. *European Scientific Journal,* 9(15):169-180.

Allen, G. J. & Albala, K. 2007. *The business of food: Encyclopaedia of the food and drink industries*. ABC-CLIO.

Almukhtar, R. S. 2012. Water Recycling/Reuse in Factories Case Study Soft Drink Factory. *Engineering and Technology Journal,* 30(1):1-14.

Alvarado-Lassman, A., Rustrian, E., Garcia-Alvarado, M. A., Rodriguez-Jimenez, G. C. & Houbron, E. 2008. Brewery wastewater treatment using anaerobic inverse fluidized bed reactors. *Bioresource Technology*, 99(8):3009-3015.

Amaral, J. A., Ekins, A., Richards, S. R. & Knowles, R. 1998. Effect of selected monoterpenes on methane oxidation, denitrification, and aerobic metabolism by bacteria in pure culture. *Applied and Environmental Microbiology,* 64(2):520-525.

Amuda, O. S., Amoo, I. A. & Ajayi, O. O. 2006. Performance optimization of coagulant/flocculant in the treatment of wastewater from a beverage industry. *Journal of Hazardous Materials,* 129(1): 69-72.

Amuda, O.S. & Amoo, I.A. 2007. Coagulation/flocculation process and sludge conditioning in beverage industrial wastewater treatment. *Journal of Hazardous Materials,* 141:778-783.

Artiga, P., Carballa, M., Garrido, J. M. & Mendez, R. 2007. Treatment of winery wastewaters in a membrane submerged bioreactor. *Water Science and Technology,* 56(2):63-69.

Baloch, M. I., Akunna, J. C. & Collier, P. J. 2007. The performance of a phase separated granular bed bioreactor treating brewery wastewater. *Bioresource Technology*, 98(9):1849.

Banerjee, S. & Biswas, G. K. 2004. Studies on biomethanation of distillery wastes and its mathematical analysis. *Chemical Engineering Journal,* 102(2):193-201.

Bellmer, D. & Hasan A. 2012. Soft Drink Waste for Ethanol Production-Fermentation of numerous different soft drink products has been conducted at Oklahoma State University. *Resource-Engineering and Technology for a Sustainable World*, 19:2-10.

Bilad, M. R., Declerck, P., Piasecka, A., Vanysacker, L., Yan, X. & Vankelecom, I. F. 2011. Treatment of molasses wastewater in a membrane bioreactor: Influence of membrane pore size. *Separation and Purification Technology,* 78(2):105-112.

Bitton, G. 2011. *Wastewater microbiology*. New Jersey: John Wiley & Sons.

Blöcher, C., Noronha, M., Fünfrocken, L., Dorda, J., Mavrov, V., Janke, H. D. & Chmiel, H. 2002. Recycling of spent process water in the food industry by an integrated process of biological treatment and membrane separation. *Desalination*, 144(1):143-150.

Blonskaja, V., Menert, A. & Vilu, R. 2003. Use of two-stage anaerobic treatment for distillery waste. *Advances in Environmental Research*, 7(3):671-678.

Böhm, L., Drews, A., Prieske, H., Bérubé, P. R. & Kraume, M. 2012. The importance of fluid dynamics for MBR fouling mitigation. *Bioresource Technology,* 122:50-61.

Bolzonella, D., Fatone, F., Pavan, P. & Cecchi, F. 2010. Application of a membrane bioreactor for winery wastewater treatment. *Water Science and Technology*, 62(12):2754-2759.

Boyd, A. 2012. Informing international UNFCCC technology mechanisms from the ground up: Using biogas technology in South Africa as a case study to evaluate the usefulness of potential elements of an international technology agreement in the UNFCCC negotiations process. *Energy Policy*, 51:301-311.

Breisha, G. Z. & Winter, J. 2010. Bio-removal of nitrogen from wastewaters: *A review. Journal of American Science,* 6(12):508-528.

Brito, A. G., Peixoto, J., Oliveira, J. M., Oliveira, J. A., Costa, C., Nogueira, R. & Rodrigues, A. 2007. Brewery and winery wastewater treatment: some focal points of design and operation. *In Utilization of by-products and treatment of waste in the food industry,* 3:109-131.

Bustamante, M. A., Paredes, C., Moral, R., Moreno-Caselles, J., Pérez-Espinosa, A. & Pérez-Murcia, M. D. 2005. Uses of winery and distillery effluents in agriculture: characterisation of nutrient and hazardous components. *Water Science and Technology*, 51(1):145-151.

Camperos, E. R., Nacheva, P. M. & Diaz Tapia, E. 2004. Treatment techniques for the recycling of bottle washing water in the soft drinks industry. *Water Science and Technology,* 50(2): 107-112.

Casani, S., Rouhany, M. & Knøchel, S. 2005. A discussion paper on challenges and limitations to water reuse and hygiene in the food industry. *Water Research,* 39(6):1134-1146.

Cervantes, F. J. 2009. *Environmental technologies to treat nitrogen pollution*. London: IWA Publishing.

Chae, K.J., Jang, A., Yim, S.K. & Kim, I. S. 2008: The effects of digestion temperature and temperature shock on the biogas yields from the mesophilic anaerobic digestion of swine manure. *Bioresource Technology*, 99:1-6.

Chen, J. P., Mou, H., Wang, L. K. & Matsuura, T. 2006. *Membrane filtration. In Advanced Physicochemical Treatment Processes,* 4th Ed. Toronto: Humana Press.

Chen, T. T., Zheng, P. & Shen, L. D. 2013. Growth and metabolism characteristics of anaerobic ammonium-oxidizing bacteria aggregates. *Applied Microbiology and Biotechnology,* 97(12):5575-5583.

Chen, Y., Cheng, J. J. & Creamer, K. S. 2008. Inhibition of anaerobic digestion process: a review. *Bioresource Technology,* 99(10):4044-4064.

Cheremisinoff, N. P. 2001. *Handbook of water and wastewater treatment technologies.* USA. Butterworth-Heinemann.

Chmiel, H., Kaschek, M., Blocher, C., Noronha, M. & Mavrov, V. 2003. Concepts for the treatment of spent process water in the food and beverage industries. *Desalination*, 152(1):307-314.

Choorit, W. & Wisarnwan, P. 2007: Effect of temperature on the anaerobic digestion of palm oil mill effluent. *Electron Journal of Biotechnology,* 10(3):376.

Cicek, N. 2003. A review of membrane bioreactors and their potential application in the treatment of agricultural wastewater. *Canadian Biosystems Engineering,* 45:6-37.

Cioabla, A. E., Ionel, I., Dumitrel, G. A. & Popescu, F. 2012. Comparative study on factors affecting anaerobic digestion of agricultural vegetal residues. *Biotechnology for Biofuels*, 5(1):1-9.

Coetzee, G., Malandra, L., Wolfaardt, G. M. & Viljoen-Bloom, M. 2004. Dynamics of a microbial biofilm in a rotating biological contactor for the treatment of winery effluent. *Water SA,* 30(3):407-412.

Connaughton, S., Collins, G. & O'Flaherty, V. 2006. Psychrophilic and mesophilic anaerobic digestion of brewery effluent: A comparative study. *Water Research,* 40(13): 2503-2510.

Cornelissen, E. R., van Buggenhout, S., van Ermen, S., De Smedt, M., Van Impe, J. & Koning, J. 2001. Anaerobic treatment of brewery wastewater with an internal membrane bioreactor. *Mededelingen (Rijksuniversiteit te Gent. Fakulteit van de Landbouwkundige en Toegepaste Biologische Wetenschappen),* 66(3):135-138.

Côté, P., Masini, M. & Mourato, D. 2004. Comparison of membrane options for water reuse and reclamation. *Desalination,* 167:1-11.

CSIR, A. 2010. A CSIR Perspective on Water in South Africa. *Council for Scientific and Industrial Research*.

Dai, H., Yang, X., Dong, T., Ke, Y. & Wang, T. 2010. Engineering application of MBR process to the treatment of beer brewing wastewater. *Modern Applied Science*, *4*(9):103-109.

De Lemos Chernicharo, C. A. 2007. *Anaerobic Reactors*. New York: IWA Publishing.

Deublein, D. & Steinhauser, A. 2011. Biogas from waste and renewable resources: *An introduction*. Winheim: John Wiley & Sons.

Dinamarca, C. 2014. Anaerobic expanded granular sludge bed (EGSB) reactor for the removal of sulphide by autotrophic denitrification. *Journal homepage: www. International Journal of Energy and Environment. International Journal of Energy and Environment Foundation. Organization*, *5*(1):111-118.

Dinsdale, R. M., Hawkes, F. R. & Hawkes, D. L. 2000. Anaerobic digestion of short chain organic acids in an expanded granular sludge bed reactor. *Water Research,* 34(9):2433-2438.

Driessen, W. & Vereijken, T. 2003. Recent developments in biological treatment of brewery effluent. In Institute and Guide of Brewery Convention, Livingstone, Zambia.

Drogui, P., Asselin, M., Brar, S. K., Benmoussa, H. & Blais, J. F. 2008. Electrochemical removal of pollutants from agro-industry wastewaters. *Separation and Purification Technology,* 61(3):301-310.

Eckenfelder. W. W. & Cleary. J. G. 2013. *Activated Sludge Technologies for Treating Industrial Wastewaters.* Pennsylvaniaa: DEStech Publications.

El-Kamah, H., Tawfik, A., Mahmoud, M. & Abdel-Halim, H. 2010. Treatment of high strength wastewater from fruit juice industry using integrated anaerobic/aerobic system. *Desalination,* 253(1):158-163.

El-Mashad, H. M., Zeeman, G., van Loon, W. K., Bot, G. & Lettinga, G. 2004. Effect of temperature and temperature fluctuation on thermophilic anaerobic digestion of cattle manure. *Bioresource Technology,* 95(2):191-201.

España-Gamboa, E., Mijangos-Cortes, J., Barahona-Perez, L., Dominguez-Maldonado, J., Hernández-Zarate, G. & Alzate-Gaviria, L. 2011. Vinasses: characterization and treatments. *Waste Management and Research,* 29(12):1235-1250.

Eusébio, A., Petruccioli, M., Lageiro, M., Federici, F. & Duarte, J. C. 2004. Microbial characterisation of activated sludge in jet-loop bioreactors treating winery wastewaters. *Journal of Industrial Microbiology and Biotechnology*, 31(1):29-34.

Feroz, S., Rao, D. G., Senthilkumar, R. & Byrne, J. A. 2012. *Wastewater treatment: Advanced processes and technologies*. New York: CRC Press.

Fillaudeau, L., Blanpain-Avet, P. & Daufin, G. 2006. Water, wastewater and waste management in brewing industries. *Journal of Cleaner Production,* 14(5):463-471.

Fiss, E. & Stein, R. 2002. Achieving Biological Nutrient Removal in a POTW Using a Soft-Drink Waste as a Supplemental Carbon Source. Water Environment Federation Annual Meeting. North Carolina.

Forstmeier, M., Goers, B. & Wozny, G. 2005. Water network optimisation in the process industry-case study of a liquid detergent plant. *Journal of Cleaner Production*, 13(5):495-498.
Fritzmann, C., Löwenberg, J., Wintgens, T. & Melin, T. 2007. State-of-the-art of reverse osmosis desalination. *Desalination,* 216(1):1-76.

Futselaar, H., Rosink, R., Smith, G. & Koens, L. 2013. The anaerobic MBR for sustainable industrial wastewater management. *Desalination and Water Treatment,* 51(4-6):1070-1078.
Gallert, C. & Winter, J. 2005. Bacterial metabolism in wastewater treatment systems. Environmental Biotechnology. *Concepts and Applications,* 1(15):1-48.

Ganesh, R., Rajinikanth, R., Thanikal, J. V., Ramanujam, R. A. & Torrijos, M. 2010. Anaerobic treatment of winery wastewater in fixed bed reactors. *Bioprocess and Biosystems Engineering,* 33(5):619-628.

Gao chun-di, Wang shu-ying & Yin bo. 2001. Influence of DO on Organic Matter Degradation Rate and Sludge Settling Property. *China Water and Wastewater,* 17(5):12-15.

Gao, M., She, Z. & Jin, C. 2007. Performance evaluation of a mesophilic (37°C) upflow anaerobic sludge blanket reactor in treating distiller's grains wastewater. *Journal of Hazardous Materials,* 141(3):808-813.

Genovesi, A., Harmand, J. & Steyer, J. P. 2000. Integrated fault detection and isolation: application to a winery's wastewater treatment plant. *Applied Intelligence*, 13(1):59-76.

Gerardi, M. H. 2003. *The microbiology of anaerobic digesters*. New Jersey: John Wiley & Sons.

Ghosh, S. & Henry, M. P. 1981. Stabilization and gasification of soft-drink manufacturing waste by conventional and two-phase anaerobic digestion. (No. CONF-810577-1). Institute of Gas Technology, Chicago, IL.

Gleick, P. H. 2014. The World's Water Volume 8: *The Biennial Report on Freshwater Resources.* Washington: Island Press.

Gohil, A. & Nakhla, G. 2006. Treatment of tomato processing wastewater by an upflow anaerobic sludge blanket–anoxic–aerobic system. *Bioresource Technology*, 97(16):2141-2152.

González-Fernández, C. & García-Encina, P. A. 2009. Impact of substrate to inoculum ratio in anaerobic digestion of swine slurry. *Biomass and Bioenergy*, 33(8):1065-1069.

Gonzalez-Garcia, Y., Hernandez, R., Zhang, G., Escalante, F. M., Holmes, W. & French, W. T. 2013. Lipids accumulation in Rhodotorula glutinis and Cryptococcus curvatus growing on distillery wastewater as culture medium. *Environmental Progress and Sustainable Energy*, 32(1):69-74.

Grady Jr, C. L., Daigger, G. T., Love, N. G., Filipe, C. D. & Leslie Grady, C. P. 2011. *Biological wastewater treatment.* New York: IWA Publishing.

Gray, M. 2008. The activity of immobilized anaerobic bacteria in the treatment of industrial wastewater. (Unpublished Doctoral dissertation, Worcester Polytechnic Institute, New York).

Grosser, A., Worwag, M., Neczaj, E. & Kamizela, T. 2013. Co-digestion of Organic Fraction of Municipal Solid Waste with different organic wastes: A Review. *Environmental Engineering IV.* London: CRC Press.

Guimarães, L., Klabjan, D. & Almada-Lobo, B. 2012. Annual production budget in the beverage industry. *Engineering Applications of Artificial Intelligence*, 25(2):229-241.

Gut, L., Plaza, E., Trela, J., Hultman, B. & Bosander, J. 2006. Combined partial nitritation/Anammox system for treatment of digester supernatant. *Water Science and Technology,* 53(12):149-159.

Guven, E. 2001. Soft drink and cookie industry wastewater treatment by anaerobic contact sequencing batch reactors*.* Unpublished Doctoral dissertation, Faculty of the Graduate School, Marquette University.

Haroon, H., Waseem, A. & Mahmood, Q. 2013. Treatment and Reuse of Wastewater from Beverage Industry. *Journal of Chemical Society of Pakistan*, 35(1):5-11.

Hayes, T. 2004. The electrodialysis alternative for produced water management. GasTIPS, Summer, 15-20.

Henze, M. 2008. *Biological wastewater treatment: Principles, modelling and design.* London: IWA Publishing.

Hosseinzadeh, M., Bidhendi, G. N., Torabian, A. & Mehrdadi, N. 2013. Evaluation of membrane bioreactor for advanced treatment of industrial wastewater and reverse osmosis pre-treatment. *Journal of Environmental Health Science and Engineering,* 11(1):34-42.

Hsine, E., Benhammou, A. & Pons, M. N. 2010. Water resources management in soft drink industry-water use and wastewater generation. *Environmental Technology,* 26(12):1309-1316.

Huei, L.C. 2005. Biodegration of brewery effluent using packed-bed upflow anaerobic reactor (PBUAR) and membrane bioreactor (MBR). Faculty of Civil Engineering, University Teknology Malaysia.

Ince, B. K., Ince, O., Sallis, P. J. & Anderson, G. K. 2000. Inert COD production in a membrane anaerobic reactor treating brewery wastewater. *Water Research,* 34(16):3943-3948.

Isla, M. A., Comelli, R. N. & Seluy, L. G. 2013. Wastewater from the soft drinks industry as a source for bioethanol production. *Bioresource Technology,* 136:140-147.

ISO 14001. 2004. Environmental management systems: Requirements with guidance for use.

Jenicek P, Svehla P, Zabranska J, & Dohanyos M. 2004 Factors affecting nitrogen removal by nitritation/denitritation. *Water Science and Technology,* 49:73-79.

Judd, S. 2011. *The MBR Book: Principles and Applications of Membrane Bioreactors for Water and Wastewater Treatment.* Oxford: Butterworth-Heinemann.

Kalyuzhnyi, S. V., Saucedo, J. V. & Martinez, J. R. 1997. The anaerobic treatment of soft drink wastewater in UASB and hybrid reactors. *Applied Biochemistry and Biotechnology,* 66(3):291-301.

Kanai, M., Ferre, V., Wakahara, S., Yamamoto, T. & Moro, M. 2010. A novel combination of methane fermentation and MBR-Kubota Submerged Anaerobic Membrane *Bioreactor process. Desalination,* 250(3):964-967.

Kang, I. J., Yoon, S. H. & Lee, C. H. 2002. Comparison of the filtration characteristics of organic and inorganic membranes in a membrane-coupled anaerobic bioreactor. *Water Research*, 36(7):1803-1813.

Kangle, K. M., Kore, S. V., Kore, V. S. & Kulkarni, G. S. 2012. Recent trends in anaerobic codigestion: a review. *Universal Journal of Environmental Research and Technology,* 2(4):210-219.

Kaparaju, P., Buendia, I., Ellegaard, L. & Angelidakia, I. 2008. Effects of mixing on methane production during thermophilic anaerobic digestion of manure: Lab-scale and pilot-scale studies. *Bioresource Technology*, 99(11):4919-4928.

Karakulski, K. & Morawski, W. A. 2000. Purification of copper wire drawing emulsion by application of UF and RO. *Desalination,* 131(1):87-95.

Karia G.L. & Christian, R.A. 2006. *Wastewater Treatment: Concepts and Design Approach*. London: PHI Learning.

Keyser, M., Witthuhn, R. C., Ronquest, L. C. & Britz, T. J. 2003. Treatment of winery effluent with upflow anaerobic sludge blanket (UASB)-granular sludges enriched with *Enterobacter sakazakii. Biotechnology Letters,* 25(22):1893-1898.

Kim, J., DiGiano, F. A. & Reardon, R. D. 2008. Autopsy of high-pressure membranes to compare effectiveness of MF and UF pretreatment in water reclamation. *Water Research,* 42(3):697-706.

Krich K, Augenstein A, Batmale J, Benemann J, Rutledge B, & Salour D. 2005. Upgrading dairy biogas to biomethane and other fuels. In: Andrews K., Editor. Biomethane from Dairy Waste - A Sourcebook for the Production and Use of Renewable Natural Gas in California. California: Clear Concepts, 47-69.

Labatut, R. A. & Gooch, C. A. 2012. Monitoring of Anaerobic Digestion Process to Optimize Performance and Prevent System Failure. Enhanced Environmental and Economic Sustainability Conference. Holiday Inn, Liverpool, *New* York, 28-29 March 28-29.

Lai E, Senkpiel S, Solley D, & Keller J. 2004. Nitrogen removal of high strength wastewater via nitritation/denitritation using a sequencing batch reactor. *Water Science and Technology,* 50:27-33.

Laubscher, A. C. J., Wentzel, M. C., Le Roux, J. M. W. & Ekama, G. A. 2001. Treatment of grain distillation wastewaters in an upflow anaerobic sludge bed (UASB) system. *Water SA,* 27(4):433-444.
Le-Clech, P., Chen, V. & Fane, T. A. 2006. Fouling in membrane bioreactors used in wastewater treatment. *Journal of Membrane Science,* 284(1):17-53.

Lee, K. C. & Rittmann, B. E. 2003. Effects of pH and precipitation on autohydrogenotrophic denitrification using the hollow-fiber membrane-biofilm reactor. *Water Research,* 37(7):1551-1556.

Lee, S. & Cho, J. 2004. Comparison of ceramic and polymeric membranes for natural organic matter (NOM) removal. *Desalination,* 160(3):223-232.
Lettinga, G. 1995. Anaerobic digestion and wastewater treatment systems. *Antonie van Leeuwenhoek,* 67(1):3-28.

Levlin, E. 2007. Conductivity measurements for controlling municipal waste-water treatment. *In Proceedings of a Polish-Swedish-Ukrainian Seminar* (pp. 51-62).

Levy, G., Fine, P., & Bar-Tal, A. 2011. *Treated Wastewater in Agriculture: Use and impacts on the soil environments and crops*. United Kingdom: Blackwell.

Li, Y., Kang, J. H., Lau, S. L., Kayhanian, M., & Stenstrom, M. K. 2008. Optimization of settling zone design to remove particles and metals. *Journal of Environmental Engineering,* 134(11):885-894.

Liao, B. Q., Kraemer, J. T. & Bagley, D. M. 2006. Anaerobic membrane bioreactors: applications and research directions. *Critical Reviews in Environmental Science and Technology,* 36(6):489-530.

Libralato, G. 2013. The small-scale approach in wastewater treatment. *In Wastewater Reuse and Management* (pp. 195-224). Springer Netherlands.

Lim, S. J. & Kim, T. H. 2014. Applicability and trends of anaerobic granular sludge treatment processes. *Biomass and Bioenergy,* 60:189-202.

Lin, H., Gao, W., Meng, F., Liao, B. Q., Leung, K. T., Zhao, L. & Hong, H. 2012. Membrane bioreactors for industrial wastewater treatment: a critical review. *Critical Reviews in Environmental Science and Technology,* 42(7):677-740.

Ling, B., Chao, L., Kan, H. & Shenxiang, W. 2009. Characteristics of membrane fouling in a submerged anaerobic double-shaft rotary membrane bioreactor. *Industrial Water Treatment,* 8:001-009.

Liu, C. F., Yuan, X. Z., Zeng, G. M., Li, W. W. & Li, J. 2008. Prediction of methane yield at optimum pH for anaerobic digestion of organic fraction of municipal solid waste. *Bioresource Technology,* 99(4):882-888.

Ioannou, L. A., Michael, C., Vakondios, N., Drosou, K., Xekoukoulotakis, N. P., Diamadopoulos, E. & Fatta-Kassinos, D. 2013. Winery wastewater purification by reverse osmosis and oxidation of the concentrate by solar photo-Fenton. *Separation and Purification Technology,* 118: 659-669.

Logsdon, G. S. 2011. *Water filtration practices: Including slow sand filters and pre-coat filtration.* Washington: Water Works Association.

Lu, J. C., Gao, D. W. & Sun, X. Y. 2013. A novel municipal wastewater treating process for energy production and autotrophic nitrogen removal based on ANAMMOX*. Huan Jing ke Xue,* 34(4):1435-1441.
Luo, G., Xie, L. & Zhou, Q. 2009. Enhanced treatment efficiency of an anaerobic sequencing batch reactor (ASBR) for cassava stillage with high solids content. *Journal of Bioscience and Bioengineering,* 107(6):641-645.

Maczulak, A. E. 2010. *Waste treatment: reducing global waste*. United Kingdom: InfoBase Publishing.

Madsen, E. L. 2011. *Environmental microbiology: from genomes to biogeochemistry*. New York: John Wiley & Sons.

Manyele, S. V., Peay, M., & Halfani, M. R. 2008. Treatment of beverage-processing wastewater in a three-phase fluidised bed biological reactor. *International Journal of Food Science and Technology*, 43(6):1058-1065.

Manyi-Loh, C. E., Mamphweli, S. N., Meyer, E. L., Okoh, A. I., Makaka, G. & Simon, M. 2013. Microbial Anaerobic Digestion (Bio-Digesters) as an Approach to the Decontamination of Animal Wastes in Pollution Control and the Generation of Renewable Energy. *International Journal of Environmental Research and Public Health,* 10(9):4390-4417.

Mason, K.D., Esteves, S.R., Guwyn, A.J. & Dinsdale, R.M. 2007 Anaerobic digestion of biodegradable municipal solid wastes *A Review*. University of Glamorgan.

Massoud, M. A., Fayad, R., Kamleh, R. & El-Fadel, M. 2010. Environmental Management System (ISO 14001) Certification in Developing Countries: Challenges and implementation strategies 1. *Environmental Science and Technology,* 44(6):1884-1887

Matošić, M., Prstec, I., Jakopović, H. K. & Mijatović, I. 2009. Treatment of beverage production wastewater by membrane bioreactor. *Desalination,* 246(1):285-293.

Matovic, M. 2013. *Biomass Now - Sustainable Growth and Use.* Intech.

Mavrov, V. & Bélières, E. 2000. Reduction of water consumption and wastewater quantities in the food industry by water recycling using membrane processes. *Desalination,* 131(1):75-86.

Mavrov, V., Chmiel, H. & Bélières, E. 2001. Spent process water desalination and organic removal by membranes for water reuse in the food industry. *Desalination*, 138(1):65-74.

Melamane, X. L. 2007. Treatment of wine distillery wastewaters by high rate anaerobic digestion and submerged membrane systems. *Doctoral dissertation,* Rhodes University, SA.

Melamane, X. L., Strong, P. J. & Burgess, J. E. 2007. Treatment of wine distillery wastewater: A review with emphasis on anaerobic membrane reactors. *South African Journal for Enology and Viticulture*, 28(1):25.

Méndez-Acosta, H. O., Snell-Castro, R., Alcatraz-González, V., González-Álvaro, V. & Playa-Ortiz, C. 2010. Anaerobic treatment of Tequila vinasses in a CSTR-type digester. *Biodegradation,* 21(3):357-363.

Meng, F., Zhou, Z., Li, L., Li, R., Jian, X. & Li, S. 2013. A novel nearly plug-flow membrane bioreactor for enhanced biological nutrient removal. *Ache Journal,* 59(1):46-54.

Milne, N.A., Gray, S., Tjandaraatmadja, G., Diaper, C., Tailor, R., Zang, G. & Reeve., D. 2009. Guidance for the use of recycled water by industry. Victoria University. CSIRO and Smart water fund.

Mittal, A. 2011. Biological Wastewater Treatment. *Water Today, August, Fulltide*, 32-44.
Miyaki, H., Adachi, S., Suda, K. & Kojima, Y. 2000. Water recycling by floating media filtration and nanofiltration at a soft drink factory. *Desalination,* 131(1):47-53.

Mohana, S. Acharya, B.K. Madamwar, D. 2009. Distillery spent wash: Treatment technologies and potential applications. *Journal of Hazard Material,* 163:12-25.

Moletta, R. 2005. Winery and distillery wastewater treatment by anaerobic digestion. *Water Science and Technology,* 51(1):137-144.

Monclús, H., Sipma, J., Ferrero, G., Comas, J. & Rodriguez-Roda, I. 2010. Optimization of biological nutrient removal in a pilot plant UCT-MBR treating municipal wastewater during start-up. *Desalination*, *250*(2):592-597.

Mosse, K. P. M., Patti, A. F., Christen, E. W. & Cavagnaro, T. R. 2011. Review: winery wastewater quality and treatment options in Australia. *Australian Journal of Grape and Wine Research,* 17(2):111-122.

Mudhoo, A. & Kumar, S. 2013. Effects of heavy metals as stress factors on anaerobic digestion processes and biogas production from biomass. *International Journal of Environmental Science and Technology,* 10(6):1383-1398.

Murthy, Z. V. P. & Chaudhari, L. B. 2009. Treatment of distillery spent wash by combined UF and RO processes. *Global NEST Journal*, 11(2):235-240.

Musee, N., Trerise, M. A. & Lorenzen, L. 2007. Post-treatment of distillery wastewater after UASB using aerobic techniques. *South African Journal of Enology and Viticulture,* 28:50-55.

Muster-Slawitsch, B., Weiss, W., Schnitzer, H. & Brunner, C. 2011. The green brewery concept of energy efficiency and the use of renewable energy sources in breweries. *Applied Thermal Engineering,* 31(13):2123-2134.

Mutombo, D. T. 2004. Internal circulation reactor: pushing the limits of anaerobic industrial effluents treatment technologies. In Proceedings of the 2004. *Water Institute of Southern Africa (WISA) Bennial conference,* Cape Town, South Africa. 608-616.

Nataraj, S. K., Hosamani, K. M. & Aminabhavi, T. M. 2006. Distillery wastewater treatment by the membrane-based nanofiltration and reverse osmosis processes. *Water Research,* 40(12): 2349-2356.

Nelson, R. 2010. Methane generation from anaerobic digesters: considering different substrates. Environmental Biotechnology,-Iowa State University, USA.

NERSA, 2001. Registers biogas production activities in rural areas in terms of the Gas Act, 2001 (Act No. 48 of 2001).

Ng, K. K., Lin, C. F., Panchangam, S. C., Andy Hong, P. K. & Yang, P. Y. 2011. Reduced membrane fouling in a novel bio-entrapped membrane reactor for treatment of food and beverage processing wastewater. *Water Research,* 45(14):4269-4278.

Nielsen, P. H., Daims, H. & Lemmer, H. 2009. *FISH handbook for biological wastewater treatment: identification and quantification of microorganisms in activated sludge and biofilms by FISH*. London: IWA Publishing.

Nopens, I., Sin, G., Jiang, T., d'Antonio, L., Stama, S., Zhao, J. & Vanrolleghem, P. A. 2007. Model-based optimisation of the biological performance of a sidestream MBR. *Water Science and Technology,* 56(6):135-146.

Noronha, M., Britz, T., Mavrov, V., Janke, H. D. & Chmiel, H. 2002. Treatment of spent process water from a fruit juice company for purposes of reuse: hybrid process concept and on-site test operation of a pilot plant. *Desalination*, 143(2):183-196.

Nyilimbabazi, N., Banadda, N., Nhapi, I. & Wali, U. G. 2011. Characterization of brewery wastewater for reuse in Kigali, Rwanda. *Open Environmental Engineering Journal,* 4:89-96.

Oktay, S., Iskender, G., Germirli Babuna, F., Kutluay, G. & Orhon, D. 2007. Improving the wastewater management for a beverage industry with in-plant control. *Desalination*, 211(1):138-143.

Ozbas, E. E., Tufekci, N., Yilmaz, G. & Ovez, S. 2006. Aerobic and anaerobic treatment of fruit juice industry effluents. *Journal of Scientific and Industrial Research,* 65(10):830-837.

Pandey, R. A., Malhotra, S., Zonehiwale, A., Pande, S., Pathe, P. P. & Kaul, S. N. 2003. Treatment of biologically treated distillery effluent: A case study. *International Journal of Environmental Studies,* 60(3):263-275.

Pant, D. & Adholeya, A. 2007.Biological approaches for treatment of distillery wastewater: *A Review, Bioresource Technology,* 98:2321-2334.

Parawira, W., Kudita, I., Nyandoroh, M. G. & Zvauya, R. 2005. A study of industrial anaerobic treatment of opaque beer brewery wastewater in a tropical climate using a full-scale UASB reactor seeded with activated sludge. *Process Biochemistry*, 40(2):593-599.

Paulsen, C. 2006. Determination of the methanogenic potential of an apple processing wastewater treatment system. Unpublished Doctoral dissertation, Stellenbosch: University of Stellenbosch, SA.

Peixoto, G., Saavedra, N. K., Varesche, M. B. A. & Zaiat, M. 2011. Hydrogen production from soft-drink wastewater in an upflow anaerobic packed-bed reactor. *International Journal of Hydrogen Energy,* 36(15):8953-8966.

Peng, J., Lu, L. & Yang, H. 2013. Review on life cycle assessment of energy payback and greenhouse gas emission of solar photovoltaic systems. *Renewable and Sustainable Energy Reviews,* 19:255-274.

Petruccioli, M., Cardoso Duarte, J., Eusebio, A. & Federici, F. 2002. Aerobic treatment of winery wastewater using a jet-loop activated sludge reactor. *Process Biochemistry*, 37(8):821-829.

Phillips, H. M., Kobylinski, E., Barnard, J. & Wallis-Lage, C. 2006. Nitrogen and Phosphorus-Rich Sidestreams: Managing the Nutrient Merry-Go-Round. *Proceedings of the Water Environment Federation,* 2006(7):5282-5304.

Poh, P. E. & Chong, M. F. 2009. Development of anaerobic digestion methods for palm oil mill effluent (POME) treatment. *Bioresource Technology,* 100(1):1-9.

Qadir, M., D., Wichelns, L., Raschid-Sally, P. G., McComick, P., Drechsel, A., Bahri & Minhas P. S. 2010. The challenges of wastewater irrigation in developing countries. *Agricultural Water Management,* 97(4):561-568.

Rajagopal, R., Massé, D. I. & Singh, G. 2013. A critical review on inhibition of anaerobic digestion process by excess ammonia. *Bioresource Technology,* 143:632-641.

Rajeshwari, K. V., Balakrishnan, M., Kansal, A., Lata, K. & Kishore, V. V. N. 2000. State-of the- art of anaerobic digestion technology for industrial wastewater treatment. *Renewable and Sustainable Energy Reviews,* 4:135-156.

Ramaswamy, S., Huang, H. J. & Ramarao, B. V. 2013. *Separation and purification technologies in biorefineries.* New York: John Wiley & Sons.

Ramírez Camperos, E., Mijaylova Nacheva, P. & Díaz Tapia, E. 2004. Treatment techniques for the recycling of bottle washing water in the soft drinks industry. *Water Science and Technology*, 50(2):107-12.

Rao, A. G., Reddy, T., Prakash, S. S. Vanajakshi, J., Joseph, J. & Sarma, P. N. 2007. pH regulation of alkaline wastewater with carbon dioxide: A case study of treatment of brewery wastewater in UASB reactor coupled with absorber. *Bioresource Technology,* 98(11):2131-2136.

Romano, R.T. & Zhang, R. 2011. Anaerobic digestion of onion residuals using a mesophilic anaerobic phased solids digester. *Biomass Bioenergy,* 35(10):4174-4179.

Rügner, H., Schwientek, M., Beckingham, B., Kuch, B. & Grathwohl, P. 2013. Turbidity as a proxy for total suspended solids (TSS) and particle facilitated pollutant transport in catchments. *Environmental Earth Sciences,* 69(2): 373-380.

Russell, D. L. 2006. *Practical wastewater treatment*. New Jersey: John Wiley & Sons.

Ryckebosch, E., Drouillon, M. & Vervaeren, H. 2011. Techniques for transformation of biogas to biomethane. *Biomass and Bioenergy*, 35(5):1633-1645.

Saha, N. K., Balakrishnan, M. & Batra, V. S. 2005. Improving industrial water use: case study for an Indian distillery. *Resources, Conservation and Recycling,* 43(2):163-174.

Sangave, P. C. & Pandit, A. B. 2006. Enhancement in biodegradability of distillery wastewater using enzymatic pre-treatment. *Journal of Environmental Management*, 78(1):77-85.

Satyawali, Y. & Balakrishnan, M. 2008. Treatment of distillery effluent in a membrane bioreactor (MBR) equipped with mesh filter. *Separation and Purification Technology,* 63(2):278-286.

Schomaker, A. H. H. M., Boerboom, A. A. M., Visser, A. & Pfeifer, A. E. 2000. Anaerobic digestion of agro-industrial wastes: information networks: technical summary on gas treatment. *AD-NETT Project FAIR-CT96-2083 (DG12-SSMI)*.

Shachman, M. 2004. *The soft drinks companion: A technical handbook for the beverage industry.* Florida: CRC Press.

Shachman. M. 2004. *The soft drinks companion: A technical handbook for the beverage industry*. Florida: Taylor and Francis.

Shao, X., Peng, D., Teng, Z. & Ju, X. 2008. Treatment of brewery wastewater using anaerobic sequencing batch reactor (ASBR). *Bioresource Technology,* 99(8):3182-3186.

Sheldon, M. S., Zeelie, P. J. & Edwards, W. 2012. Treatment of paper mill effluent using an anaerobic/aerobic hybrid side-stream Membrane Bioreactor. *Water Science and Technology,* 65(7):1265-1272.

Simate, G. S., Cluett, J., Iyuke, S. E., Musapatika, E. T., Ndlovu, S., Walubita, L. F. & Alvarez, A. E. 2011. The treatment of brewery wastewater for reuse: State of the art. *Desalination*, 273(2):235-247.

Singhania, R. R., Christophe, G., Perchet, G., Troquet, J. & Larroche, C. 2012. Immersed membrane bioreactors: An overview with special emphasis on anaerobic bioprocesses. *Bioresource Technology,* 122:171-180.

Song, H., Clarke, W. P. & Blackall, L. L. 2005. Concurrent microscopic observations and activity measurements of cellulose hydrolyzing and methanogenic populations during the batch anaerobic digestion of crystalline cellulose. *Biotechnology and Bioengineering,* 91(3):369-378.

Song, K. G., Cho J. & Ahn, K. H. 2009. Effects of internal recycling time mode and hydraulic retention time on biological nitrogen and phosphorus removal in a sequencing anoxic/anaerobic membrane bioreactor process. *Bioprocess and Biosystems Engineering,* 32(1):135-142.

Sonune, A. & Ghate, R. 2004. Developments in wastewater treatment methods. *Desalination,* 167, 55-63.

South Africa, Department of Public Works. 2012. Small wastewater treatment works, Design guidelines.

South Africa, Department of Water Affairs and Forestry. 2004. Guidelines for Water Conservation and Water Demand Management: Volume 1: CMA Planning Framework, Pretoria.

South Africa, The National Water Act. 1998. (Act No. 36 of 1998). *Government Gazette,* SA.

South Africa. 2003. Department of Water Affairs and Forestry. 2003. Water Quality Management Series: Towards a Strategy for a Waste Discharge Charge System. Sub-Series No. MS 11. Pretoria: Government Printer.

South Africa. 2006. City of Cape Town: Wastewater and industrial effluent by-law. Province of Western Cape Provincial Gazette, 6378:1562-1563, September 1.

South Africa. Department of Water Affairs and Forestry. 2005. SANS 241:2005: A drinking water quality framework for South Africa.

South Africa. Department of Water Affairs. 2011. SANS 241-1:2011: South African National Standard: Drinking Water. Part 1: Microbiological, physical, aesthetic and chemical determinants. Pretoria: SABS Standards Division.

Spellman, F. R. & Drinan, J. E. 2012. *The drinking water handbook.* New York: CRC Press.

Spellman, F. R. 2013. *Handbook of water and wastewater treatment plant operations.* New York: CRC Press.

Sreethawong, T. & Chavadej, S. 2008. Colour removal of distillery wastewater by ozonation in the absence and presence of immobilized iron oxide catalyst. *Journal of Hazardous Materials,* 155(3):486-493.

Stoller, M., De Caprariis, B., Cicci, A., Verdone, N., Bravi, M. & Chianese, A. 2013. About proper membrane process design affected by fouling by means of the analysis of measured threshold flux data. *Separation and Purification Technology,* 114: 83-89.

Strong, P. J. & Burgess, J. E. 2008. Treatment methods for wine-related and distillery wastewaters: a review. *Bioremediation Journal,* 12(2):70-87.

Sulaiman, A., Busu, Z., Tabatabaei, M., Yacob, S., Abd-Aziz, S., Hassan, M. A. & Shirai, Y. 2009. The effect of higher sludge recycling rate on anaerobic treatment of palm oil mill effluent in a semi-commercial closed digester for renewable energy. *American Journal of Biochemistry and Biotechnology*, *5*(1):1-6.

Surroop, D. & Mohee, R. 2011: Comparative assessment of anaerobic digestion of Municipal solid waste at mesophilic and thermophilic temperatures. *International Journal of Environmental Technology Management,* 14(1):238-251.

Tabatabaei, M., Rahim, R. A., Abdullah, N., Wright, A. D. G., Shirai, Y., Sakai, K. & Hassan, M. A. 2010. Importance of the methanogenic archaea populations in anaerobic wastewater treatments. *Process Biochemistry,* 45(8):1214-1225.

Tabatabaei, M., Sulaiman, A., Nikbakht, A. M., Yusof, N. & Najafpour, G. 2011. Influential parameters on biomethane generation in anaerobic wastewater treatment plants. Canada: Intech.

Tamime, A. Y. 2009. *Cleaning-in-place: dairy, food and beverage operations*. London: John Wiley & Sons.

Tawfik, A. & El-Kamah, H. 2012. Treatment of fruit-juice industry wastewater in a two-stage anaerobic hybrid (AH) reactor system followed by a sequencing batch reactor (SBR). *Environmental Technology,* 33(4):429-436.

Tay, J. H. & Jeyaseelan, S. 1995. Membrane filtration for reuse of wastewater from beverage industry. *Resources, Conservation and Recycling*, 15(1): 33-40.

Teerasong. S. Chan-Eam. S. Sereenonchai. K. Amornthammarong. N. Ratanawimarnwong. N. & Nacapricha. D. 2010. A reagent-free SIA module for monitoring of sugar, color and dissolved CO< sub> 2</sub> content in soft drinks. *Analytical Chemical,* 668(1):47-53.

Teng, C. K., Hawlader, M. N. A. & Malek, A. 2003. An experiment with different pretreatment methods. *Desalination,* 156(1):51-58.

Torres, A., Hemmelmann, A., Vergara, C. & Jeison, D. 2011. Application of two-phase slug-flow regime to control flux reduction on anaerobic membrane bioreactors treating wastewaters with high suspended solids concentration. *Separation and Purification Technology,* 79(1):20-25.

Trzcinski, A. P. & Stuckey, D. C. 2009. Continuous treatment of the organic fraction of municipal solid waste in an anaerobic two-stage membrane process with liquid recycle. *Water Research,* 43(9):2449-2462.

Turkdogan-Aydınol, F. I. & Yetilmezsoy, K. 2010. A fuzzy-logic-based model to predict biogas and methane production rates in a pilot-scale mesophilic UASB reactor treating molasses wastewater. *Journal of Hazardous Materials,* 182(1):460-471.

Vaiopoulou, E. & Aivasidis, A. 2008. A modified UCT method for biological nutrient removal: configuration and performance. *Chemosphere,* 72(7):1062-1068.

Valderrama, C., Ribera, G., Bahi, N., Rovira, M., Gimenez, T., Nomen, R. & Martinez-Llado, X. 2012. Winery wastewater treatment for water reuse purpose: Conventional activated sludge versus membrane bioreactor (MBR): A comparative case study. *Desalination*, 306:1-7.

Van Gestel, T., Vandecasteele, C., Buekenhoudt, A., Dotremont, C., Luyten, J., Leysen, R. & Maes, G. 2002. Salt retention in nanofiltration with multilayer ceramic TiO_2 membranes. *Journal of Membrane Science,* 209(2):379-389.

Van Haandel, A., Kato, M. T., Cavalcanti, P. F. & Florencio, L. 2006. Anaerobic reactor design concepts for the treatment of domestic wastewater. *Reviews in Environmental Science and Biotechnology,* 5(1):21-38.

Van Schoor, L. H. 2005. Guidelines for the management of wastewater and solid waste at existing wineries. *Winetech, South Africa*.

Vedavyasan, C. V. 2007. Pretreatment trends an overview. *Desalination,* 203(1):296-299.

Venkata Mohan, S., Mohanakrishna, G., Ramanaiah, S. V. & Sarma, P. N. 2008. Simultaneous biohydrogen production and wastewater treatment in biofilm configured anaerobic periodic discontinuous batch reactor using distillery wastewater. *International Journal of Hydrogen Energy,* 33(2):550-558.

Vlyssides, A. G., Barampouti, E. M. & Mai, S. 2005. Wastewater characteristics from Greek wineries and distilleries. *Water Science and Technology*, 51(1):53-60.

Von Sperling, M. 2007. *Basic principles of wastewater treatment.* London: IWA publishing.

Von Sperling, M., de Lemos Chernicharo, C. A., Andreoli, C. V. & Fernandes, F. 2005. Biological wastewater treatment in warm climate regions (Vol. 2). United Kingdom: IWA.

Wang, L. K., & Ivanov, V. 2010. *Environmental biotechnology*. New York: Springer.

Wang, L. K., Pereira, N. C. & Hung, Y. T. 2009. *Biological treatment processes.* New York: Springer.

Wang, S. G., Liu, X. W., Gong, W. X., Gao, B. Y., Zhang, D. H. & Yu, H. Q. 2007. Aerobic granulation with brewery wastewater in a sequencing batch reactor. *Bioresource Technology,* 98(11):2142.

Wang, X., Feng, Y. J. & Lee, H. 2008. Electricity production from beer brewery wastewater using single chamber microbial fuel cell. *Water Science and Technology*, 57(7):1117-1122.

Wang, Z., Liu, B., Liu, Y. D. & Wan, F. 2013. Evaluation of biological removal efficiency in a UCT process treating municipal wastewater during start-up stage. *Journal of Environmental Biology/Academy of Environmental Biology,* 34:459-464.

Ward, A.J., Hobbs, P.J., Holliman P.J. & Jones, D.L. 2008: Optimisation of the anaerobic digestion of agricultural resources. *Bioresource Technology,* 99:7928-7940.

Wen, Q., Wu, Y., Zhao, L. X., Sun, Q. & Kong, F. Y. 2010. Electricity generation and brewery wastewater treatment from sequential anode-cathode microbial fuel cell. *Journal of Zhejiang University Science B*, 11(2):87-93.

Wijekoon, K. C., Visvanathan, C. & Abeynayaka, A. 2011. Effect of organic loading rate on VFA production, organic matter removal and microbial activity of a two-stage thermophilic anaerobic membrane bioreactor. *Bioresource Technology,* 102(9):5353-5360.

Wilkie, A. C., Riedesel, K. J. & Owens, J. M. 2000. Stillage characterization and anaerobic treatment of ethanol stillage from conventional and cellulosic feedstock. *Biomass and Bioenergy,* 19(2):63-102.

Wolmarans, B. & De Villiers, G. H. 2004. Start-up of a UASB effluent treatment plant on distillery wastewater. *Water SA,* 28(1):63-68.

Yang, J., Zhang, L., Fukuzaki, Y., Hira, D. & Furukawa, K. 2010. High-rate nitrogen removal by the Anammox process with a sufficient inorganic carbon source. *Bioresource Technology,* 101(24):9471-9478.

Yang, W., Cicek, N. & Ilg, J. 2006. State-of-the-art of membrane bioreactors: Worldwide research and commercial applications in North America. *Journal of Membrane Science,* 270(1):201-211.

Yu, H. Q., Zhao, Q. B. & Tang, Y. 2006. Anaerobic treatment of winery wastewater using laboratory-scale multi-and single-fed filters at ambient temperatures. *Process Biochemistry,* 41(12):2477-2481.

Zhang, C., Wang, L., Yan, N., Zhang, Y. & Liu, R. 2013. Air-lift internal loop biofilm reactor for realized simultaneous nitrification and denitrification. *Bioprocess and Biosystems Engineering,* 36(5):597-602.

Zhang, S., Yang, F., Liu, Y., Zhang, X., Yamada, Y. & Furukawa, K. 2006. Performance of a metallic membrane bioreactor treating simulated distillery wastewater at temperatures of 30 to 45 ^{o}C. *Desalination,* 194(1):146-155.

Zhang, W., Xiong, R. & Wei, G. 2009. Biological flocculation treatment on distillery wastewater and recirculation of wastewater. *Journal of Hazardous Materials*, 172(2):1252-1257.

Zhang, Y., Yan, L., Chi, L., Long, X., Mei, Z. & Zhang, Z. 2008. Start-up and operation of anaerobic EGSB reactor treating palm oil mill effluent. *Journal of Environmental Sciences,* 20(6):658-663.

Zhang, Y., Zamudio Cañas, E. M., Zhu, Z., Linville, J. L., Chen, S. & He, Q. 2011. Robustness of archaeal populations in anaerobic co-digestion of dairy and poultry wastes. *Bioresource Technology,* 102(2):779-785.

Zhou, M., Tan, Q., Wang, Q., Jiao, Y., Oturan, N. & Oturan, M. A. 2012. Degradation of organics in reverse osmosis concentrate by electro-Fenton process. *Journal of Hazardous Materials,* 215, 287-293.

Zupančič, G. D., Stražiščar, M. & Roš, M. 2007. Treatment of brewery slurry in thermophilic anaerobic sequencing batch reactor. *Bioresource Technology,* 98(14):2714-2722.

INTERNET REFERENCES

AEBIOM, A. 2009. Biogas road map for Europe. *European Biomass Association.*

Council Directive_80/778/EEC of 15 July 1980 relating to the quality of water intended for human consumption - Repealed by Council Directive 98/83/European Council on the quality of water intended for human consumption. accessed [10 April 2014].

Fewtrell, L. & Bartram, J. 2001. Water Quality: Guidelines, Standards and Health: Assessment of Risk and Risk Management for Water-Related Infectious Disease. *International Water Association.*

http://www.worldcat.org/title/water-quality-guidelines-standards-and-health-assessment-of-risk-and-risk-management-for-water-related-infectious-diseases/oclc/496991738
viewed [21 March 2014].

Oliveira, M. & Duarte, E. 2011. Winery wastewater treatment: evaluation of the air micro-bubble bioreactor performance.
http://hdl.handle.net/10400.15/624 viewed [10 February 2014].

Softdrink 2014. In Encyclopædia Britannica. Inc. Encyclopaedia Britannica Online: http://www.britannica.com/EBchecked/topic/552397/soft-drink/66705/Carbon-dioxide-and-carbonation [Retrieved March 5. 2014].

South Africa (Western Cape). 2006. City of Cape Town: Wastewater and industrial effluent by-law. *Province of Western Cape Provincial Gazette,* 6378:1562-1563, September 1.
https://www.capetown.gov.za/en/PublicParticipation/Documents/Draft_Wastewater_By_law_2011_Eng.pdf**,** viewed [15 January 2014].

Wellinger A. & Lindberg A. 2005. Biogas Upgrading and Utilisation. IEA Bioenergy Task 24: Energy *from Biological Conversion of Organic Waste;* 2005
[accessed 20 January 2014].

APPENDICES

APPENDIX A: Treatment methods

Treatment method	Type of reactor
AD	Anaerobic digestion
AD-MBR	Anaerobic digestion & membrane bioreactor
AD-MCAB	Anaerobic digestion & membrane-coupled anaerobic bioreactor
AF	Anaerobic filter
AH-SBR	Anaerobic hybrid sequence batch reactor
AJLR	Aerobic jet-loop activated sludge reactor
AMBB	Air micro-bubble bioreactor
AMBR	Aerobic membrane bioreactor
AMBR-CSTR	Aerobic membrane bioreactor & continuous stirrer zone reactor
AMBR-PAC	Aerobic MBR and powdered activated carbon
AnCSBR	Anaerobic contact sequence batch reactor
AnMBR	Anaerobic membrane bioreactor
AnEMBR	Anaerobic external membrane bioreactor
AnFBR	Anaerobic fixed-bed reactor
AnIFBR	Anaerobic inverse fluidized bed reactors
AnRBC	Anaerobic rotating biological contactor
AnSBBR	Anaerobic sequencing batch biofilm reactor
AnSBR	Anaerobic sequencing batch reactor
AO&MBR	Anoxic/aerobic membrane bioreactor
CA-UASB	CO_2 Absorber & upflow anaerobic sludge bed
CF-RO	Cartridge filter & reverse osmosis
CF-RO-IE	Cartridge filter, reverse osmosis and ion exchange
CSTR	Continuous stirred zone reactor and type digester
DSR-AnMBR	Double-shaft rotary anaerobic membrane bioreactor
EGSB-AF	Expanded granular sludge bed & anaerobic filter
FBBR	Fluidised bed bioreactor
GRABBR	Granular bed anaerobic baffled bed reactor
HT-MBR	High-throughput and MBR
KSAMBR	Kubota’s submerged anaerobic membrane biological reactor

MBR-UV-NF	Membrane bioreactor and ultraviolet and nanofiltration
MCAB	Membrane-coupled anaerobic bioreactor
NF-RO	Nanofiltration and reverse osmosis
PBUAR	Packed-bed upflow anaerobic reactor
PTF-AH	Polyurethane trickling filter and anaerobic hybrid reactor
RBC	Rotating biological contactor
SBR	Aerobic sequence batch reactor
sMBR	Sidestream membrane bioreactor
TAnMBR	Thermophilic anaerobic membrane bioreactor
UAF	Upflow anaerobic filter
UAFFR	Anaerobic upflow fixed film reactor
UASB	Upflow anaerobic sludge bed
UASB-AF	Upflow anaerobic sludge bed and anaerobic filter
UASR-AS	Upflow anaerobic sponge reactor and activated sludge
UASB-ASBR	Upflow anaerobic sludge bed and aerobic sequence batch reactor
UASB-HR	Upflow anaerobic sludge bed and hybrid reactor
UASB-SBR	Upflow anaerobic sludge bed and sequence batch reactor

APPENDIX B: Preparation of dosing solution chemicals and cleaning-in-place (CIP) solution

Appendix B1: Dosing solution

Preparation of 2L 0.5 M phosphoric acid from 80% phosphoric acid (H_3PO_4)

1. Molecular weight of phosphoric acid (Mw (H_3PO_4)) = 97.99 g/mol
2. 0.5 M = 0.5 mol/L x Mw (H_3PO_4)

   = 0.5 x 97.95

   = 48.98 g/L

Concentration of undiluted H_3PO_4 (C_1) = 80%, therefore, C_1 = 800 g/L and V_1 = unknown volume

4. 2 L 0.5 M H_3PO_4 is required, therefore, C_2 = 48.98 g/L and V_2 = 2 L
5. $C_1V_1 = C_2V_2$
6. (800 g/L) x V_1 = (48.98 g/L) x (2 L)
7. V_1 = 0.122 L per 2 L water

Therefore, in order to prepare 2 L of 0.5 M H_3PO_4, 0.122 L (122 ml) of H_3PO_4 must be added to 2 L of distilled H_2O and mixed well.

Preparation of 2L 1M sodium hydrogen carbonate ($NaHCO_3$)

1. Molecular weight of sodium hydrogen carbonate (Mw ($NaHCO_3$)) = 84.007 g/mol
2. grams = (Mw ($NaHCO_3$)x (2 L)x 1M

   = 168.014 g

Therefore, in order to prepare a solution of 2L of 1M $NaHCO_3$, 168.014 g must be added to 2L of distilled H_2O and mixed well.

Appendix B2: CIP solution

Sodium Hypochlorite (NaOCl) 400ppm solution used to clean biological fouling off the membranes, and citric acid 1% solution prepared to clean chemical fouling off the membranes.

Preparation of 5 2Lof 400 ppm NaOCl from 12.5% NaOCl

1. Using the equation $C_1V_1 = C_2V_2$
2. Concentration of undiluted NaOCl (C_1) = 12.5%, which is equivalent C_1 = 125 g/L
3. 5 L 400 ppm NaOCl is required, equivalent C_2 = 0.4 g/L and V_2 = 2 L
4. $C_1V_1 = C_2V_2$ Equation B1
5. (125 g/L) x V_1 = (0.4 g/L) x (2 L)
6. V_1 = 0.0064 L per 2 L water

Therefore, 6.4 mL NaOCl is needed in 2L distilled H_2O

<u>Preparation of 2L 1% citric acid solution</u>

1% citric acid = 1 g/ 100 ml = 10 g /L

Therefore, 10g / 1L = x / 2L

x = (10 / 1) * 2

x = 20 g

Therefore, in order to prepare 2 L of 1% citric acid, 20 g (0.02 kg) of citric acid must be added to 2 L of distilled H_2O water and mixed well.

APPENDIX C: Procedure for determining analytical parameters

APPENDIX C1: Chemical oxygen demand (COD)

Determines the amount of organic pollutants found in surface water (for example, lakes and rivers) or wastewater, making COD a useful measure of water quality (Manyele *et al.,* 2008).

1.1.1) Method to determine total COD (COD_t):

a) All samples must be tested in duplicate.
b) All apparatus should first be checked to confirm that all is operational
c) The test cells should not be scratched and must be cleaned.
d) Switch on the Spectroquant thermoreactor TR 420 to the present setting of 148˚C for two hours and allow the thermoreactor to heat up to the desired temperature. This will take approximately 10 minutes.
e) Place approximately 100 ml of distilled water in a 250 ml beaker.

1.1.2) When using COD solutions A and B for the 500 to 10 000 mg/L range:

a) Use Merck COD Solution A, Cat. No. 1.14679.0495 and Merck COD Solution B, Cat. No. 14680.0495. Pipette 2.2 ml of COD solution A into the test cell.
b) Pipette 1.8 ml of COD solution B into the test cells into which COD solution A was pipetted. Pipette 1 ml of the sample into the test cells.
c) Tightly attach the screw caps to the test cells. Vigorously mix the contents of the test cells with a vortex mixer.
d) Heat the test cells in the Spectroquant thermoreactor TR 420 at 148˚C for 2 h. Carefully remove the test cells after 2 h and place in a test tube rack to cool.
e) Wait 10 minutes and mix the contents of the test cells with the vortex mixer again. Allow the test cells to cool down to room temperature for 30 minutes.
f) Clean the outside of the test cells and place in the Nova 60 Spectroquant.
g) Enter the code 024 for COD readings in the 500 to 10 000 mg/L range and the COD concentration of the sample in the test cell will be indicated on the display screen in mg/L.

1.1.3) When using COD solutions A and B for the 100 to 1500 mg/L range:

a) Use Merck COD Solution A, Cat. No. 1.14538.0065 and Merck COD Solution B, Cat. No. 1.14539.0495. This procedure is exactly the same as for COD solutions A and B for the 500 to 10000 mg/L range, with the exception of the following, which are outlined below.
b) Pipette 0.3 ml of COD solution A into the test cell. Pipette 2.3 ml of COD solution B into the test cell.

c) Pipette 3 ml of sample into the test cell.

1.1.4) ***Method to determine soluble COD (COD_s):***

a) A Büchner funnel is attached to a 500 ml suction flask.

b) The suction flask is either connected to a water pump or to a vacuum pump.

c) Glass microfibre filter discs, 5.5 cm in diameter with a 0.45 µm pore size, without organic binder (Whatman type GF/F (0.7 Fm)) is placed inside the Büchner funnel, and 50ml of the raw sample is filtered.

d) The COD of the filtered sample is then determined by using the same procedure as for total COD determination.

e) The insoluble COD (COD_i) is measured by the difference between the total COD (COD_t) and the soluble COD (COD_s), and is calculated by using Equation C1:

$$COD_t \text{ (mg / L)} - COD_s \text{ (mg / L)} = COD_i \text{ (mg / L)} \qquad \text{Equation. C1}$$

References

Merck COD Solution A, Cat. No. 1.14538.0065 and 1.14679.0495.

Merck COD Solution B, Cat. No. 1.14539.0495 and 1.14680.0495.

APPENDIX C2: Procedure to determine Total Suspended Solids (TSS)

A total suspended solid (TSS) gives the quantity of the turbidity of water or wastewater (Akpor & Muchie, 2013). TSS is defined as solids, which are retained by a glass fibre filter and dried to constant weight at 103 to 105°C.

a) Preparation of the glass fiber filter disk: Insert the filter disk onto the base and clamp on funnel.

- While the vacuum is applied, wash the disk with three successive 20 mL volumes of Milli-Q water.
- Remove all traces of water by continuing to apply the vacuum after water has passed through.
- Remove the funnel from the base and place the filter in the aluminium dish, and ignite in the muffle furnace at 550°C ± 50°C for 30 minutes.
- Rewash the filter with an additional three successive 20 mL volumes of Milli-Q water, and dry in an oven at 103-105°C for one hour.
- When needed, remove dish from the oven, desiccate, and weigh.

b) Select a sample volume (max. of 50 mL) that will yield no more than 50 mg of total suspended solids.

c) Place the filter on the base and clamp on the funnel and apply the vacuum.

d) Wet the filter with a small volume of Milli-Q water to seal the filter against the base.

e) Shake the sample vigorously and quantitatively transfer the sample to the filter by using a large orifice, volumetric pipet.

f) Remove all traces of water by continuing to apply the vacuum after the sample has passed through.

g) Rinse the pipet and funnel onto the filter with a small volume of Milli-Q water.

h) Remove all traces of water by continuing to apply the vacuum after water has passed through.

i) Carefully remove the filter from the base and dry for at least one hour at 103-105°C.

j) Cool in a desiccator and weigh.

Using equation C2 calculate Total Suspended Solids (TSS) as follows:

$$\text{Total Suspended Solids, mg/L} = (A-B) \times 1000/C \qquad \text{Equation C2}$$

Where: A = weight of filter and dish + residue in mg

B = weight of filter and dish in mg

C = volume of sample filtered in mL

References

Methods for the Chemical Analysis of Water and Waste. 1979. U.S. *Environmental Protection Agency,* EPA 600/4-79-020:160.2.

Standard Methods for the Examination of Water and Wastewater. 1985. 16th Edition, p. 96, Method 209C.

APPENDIX C3: Method to determine Turbidity

Turbidity is a measure of the degree to which the water or wastewater loses its transparency due to the presence of suspended solids (Rügner *et al.,* 2013). Turbidity is measured by an instrument, which is called a nephelometer and its units are called nephelometric turbidity units (NTU).

1.3.1) ***Calibration method:***

a) Place TN-100 turbidimeter on a flat level surface.
b) Insert the CAL 1 (800 NTU) calibration standard into the sampling well, aligning the mark on the vial with the mark on the meter.
c) Press the vial down until it snaps into the instrument.
d) Cover the vial with the light shield cap.
e) Press the ON/OFF key to switch on the meter. The meter will go into measuring mode after the start up sequence.
f) Press the CAL key to switch to calibration mode. The meter will prompt for the CAL 1 standard to be inserted.
g) Press the READ/ENTER key.
h) The annunciator will blink for 12 seconds and then prompt for the CAL 2 (200 NTU) calibration standard to be inserted.
i) Repeat steps a), b), d), g) and h) for CAL 2, CAL 3 (100 NTU) and CAL 4 (0.02 NTU) calibration standards.
j) After the CAL 4 (0.02 NTU) calibration standard is calibrated, the display will show STbY.
k) The nephelomete is now ready for measurement.

1.3.2) ***Turbidity measuring method:***

Preparation of vial sample:

a) Clean dry sample vial.
b) Hold the vial sample vial by the cap.
c) Wash the vial sample with roughly 10 ml of the sample, capping the vial with a black screw cap and inverting smoothly a number of times.
d) Dispose the used sample and repeat the washing method twice.
e) Fill the sample vial with approximately 10 ml of sample (up to the mark indicated on the sample vial).
f) Cap the vial with a black screw cap.
g) Wipe the vial sample with a soft cloth.
h) Make sure that the external of the vial is dry and clean.

*1.3.3) **Measurement method:***

a) Place the vial sample inside the sample well of the TN-100 turbidimeter and align the vial's index mark with the meter's index mark.
b) Push the vial sample until it is totally snapped in.
c) Cover the vial with the light protection cap.
d) Turn the meter on by pushing the ON/OFF key.
e) Following the power up sequence, the meter will go into measuring mode and the display will flash "Rd" roughly 10 times.
f) The measured reading will appear on the display.
g) If necessary, place a second sample vial into the sample well, push down until it is snapped into place and press the READ/ENTER key.
h) Wait for the measured reading to appear on the display screen.
i) Repeat steps a) to h) for all samples.

References

Standard Methods for the Examination of Water and Wastewater. 2012

APPENDIX C4: Method to determine orthophosphate:

Use a Merck Spectroquant Phosphate cell test for orthophosphate. Cat. No. 1.14543.0001.

a) Check the pH of the sample; it should be in the specified range of pH 0 – 10.
b) Add 5.0 ml of the filtered sample to a barcoded test cell with a P1000 pipette.
c) Place a cap on the barcoded test cell, and mix vigorously with a vortex mixer.
d) Add 5 drops of P-2K to the barcoded test cell.
e) Add 1 dose of P-3K to the barcoded test cell by using the blue dose-metering cap.
f) Place the cap on the barcoded test cell, and mix vigorously with the vortex mixer.
g) Wait for 5 minutes for the reaction to occur.
h) Place the test cells into the Nova 60 Spectroquant to measure for orthophosphate.

References

Merck Spectroquant Phosphate cell test for orthophosphate and total phosphorus, Cat. No. 1.14543.0001.

APPENDIX D: Composition of anoxic and aerobic zones in different operating conditions

Appendix D1: Characteristics of the anoxic zone for different modes of operation

Parameter	Mode -1a	Mode-1b	Mode-2a	Mode-2b	Mode-3a	Mode-3b
pH	7.9	8.3	9.1	8.8	9.1	8.6
COD_t (mg/L)	385.8	896.4	1 061	1 181	1 331	1 375
COD_s(mg/L)	109.8	755.3	822.5	939.6	1 262	981.3
DO (mg/L)	1.2	2.1	0.6	1.3	2.3	2.5
EC (µS/cm)	102 2	2 217	4 339	4 788	3 538	5 514
Fructose (mg/L)	2.9	60.5	63.4	97.7	33.9	3.9
Sucrose (mg/L)	46	680.2	2 392	1 271	958.4	771.4
TDS (mg/L)	708.3	1 567	2 916	3 163	2 299	3412
Temperature (°C)	22.5	22.1	23.4	23.8	25.6	27.7
TSS (mg/L)	356.3	667.9	474.0	447.6	167.2	885.1
Turbidity (NTU)	82.4	204	182.9	196.3	106.9	314.6
Ammonium (mg/L)	3.0	6.0	5.3	3.0	5.3	3.6
Nitrate (mg/L)	4.8	11.0	8.7	5.5	8.7	9.8
Phosphate	8.2	9.1	16.5	7.2	16.5	6.2

Appendix D2: Characteristics of the aerobic zone for different modes of operation

Parameter	Mode -1a	Mode-1b	Mode-2a	Mode-2b	Mode-3a	Mode-3b
pH	8.5	8.8	9.3	10.0	10.0	9.8
COD_t (mg/L)	496.4	893.3	974.2	1 119	1 381	1 508
COD_s(mg/L)	494.6	564.8	877.5	893.8	1 212	1 307
DO (mg/L)	7.0	5.7	4.6	6.2	6.5	6.1
EC (µS/cm)	1 327.	1 962.	3 884	6 229	5 442	5 318
Fructose (mg/L)	75.5	120.6	25.2	112.2	8.3	4.1
Sucrose (mg/L)	455.8	532.6	1 613	1 293	1 020	1 303
TDS (mg/L)	969.7	1 463	2 953	4 191	3 677	3 476
Temperature (°C)	21.2	21.5	22.5	23.0	23.5	24.9
TSS (mg/L)	515.6	596.0	505.0	547.6	309.2	801.6
Turbidity (NTU)	151.9	106.2	142.4	120.1	130.0	201.1
Ammonium (mg/L)	1.8	2.0	1.1	0.6	0.1	0.9
Nitrate (mg/L)	28.4	30.5	23.1	17.0	16.2	20.1
Phosphate	9.8	10.4	17.1	9.0	9.9	13.2

APPENDIX E: Calculations

Appendix E1: Membrane flux calculations

$$\mathrm{J} = \frac{\mathrm{Q}}{\mathrm{A}} = \frac{0.05\ \times 2}{0.4}$$

$$= \mathbf{3.45\ L/m^2h}$$

Appendix E2: Overall treatment efficiency calculations

$$TE\% = \frac{COD_{egsb-feed} - COD_{mbr-permeate}}{COD_{egsb-feed}}$$

$$= \frac{1870 - 69.5}{11870} \times 100$$

$$\boldsymbol{TE = 96.3\%}$$