Cape Peninsula
University of Technology

# MEMBRANE BIOREACTOR APPLICATION WITHIN THE SOUTH AFRICAN TEXTILE INDUSTRY: PILOT TO FULL-SCALE

by

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# DECLARATION

I, Debbie de Jager, declare that the contents of this thesis represent my own unaided work, and that the 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.

**Signed** **Date**

# ABSTRACT

To date, limited information has been published on textile wastewater treatment, for re-use, in South Africa (SA), with treatment processes focusing on conventional wastewater treatment methods. A large contributor to the contamination of water within textile industries is from dyehouse processes. A major concern in textile wastewater treatment is the release of azo dyes and their metabolites, some of which are carcinogenic and mutanogenic, into the environment since they are xenobiotic and aerobically recalcitrant to biodegradation. A necessity therefore exists to find an effective treatment method capable of removing both the strong colour and the toxic organic compounds from textile wastewater. Membrane bioreactors (MBRs) are favoured when treating high-strength wastewater, since the membrane area is determined by the hydraulic throughput and not the biological load; no sludge is wasted and all bacteria are retained within the reactor, including specific bacteria capable of degrading the toxic, non-biodegradable constituents present in textile wastewater. MBR systems, using various configurations have been utilised extensively in the rest of the world to treat textile wastewater at both lab and pilot-scale.

This DTech project formed part of a collaborative Water Research Commission (WRC) funded project K5/1900 - Pilot application of a dual-stage membrane bioreactor (dsMBR) for industrial wastewater treatment. The main purpose of this study was the on-site evaluation of a pilot-scale dsMBR incorporating two ultrafiltration (UF) sidestream membrane modules for the treatment, recovery and re-use of textile wastewater.

The objectives of this project were to determine the treatment efficiency of the system; to evaluate the degree of colour removal from the textile wastewater; to improve residual colour removal within the system using treatment processes, such as NF and RO, as well as to propose a design and cost for a full-scale plant.

A textile industry located in Bellville, Western Cape, was chosen as the industrial partner for the on-site evaluation of a semi-automated pilot wastewater treatment MBR plant using two 5.1 m$^2$ Norit X-flow Airlift$^{TM}$ membrane modules. Since the wastewater treatment system was located on the premises, real continuously changing industrial wastewater was being treated. The industrial textile wastewater was treated in a series of tanks: 1) an anaerobic tank, which cleaved the azo bonds of the reactive dyes; 2) an anoxic tank containing reduced amounts of dissolved oxygen, in which denitrification occurred; and 3) an aerobic tank, in which i) nitrification, as well as ii) mineralisation of the aromatic amines occurred. The UF-membrane modules would account for the removal of any organic material.

The wastewater stream was characterised by a chemical oxygen demand (COD) range of between 45 to 2,820 mg/L and an average biological oxygen demand (BOD) of 192.5 mg/L. The dsMBR achieved an average COD reduction of 75% with a maximum of 97% over the 220 day test period. The COD concentration obtained after dsMBR treatment averaged at 191 mg/L, which was well within the City of Cape Town industrial wastewater discharge standard. The average reduction in turbidity and TSS was 94% and 19.6%, respectively, during the UF-MBR stage of the system. Subsequent treatment of the UF permeate with nanofiltration (NF) for 4 days, alternated with reverse osmosis (RO) for 14 days removed both the residual colour and salt present in the UF permeate. A consistent reduction in the colour of the incoming wastewater was evident. The colour in the wastewater was reduced from an average of 659 ADMI units to ~12 ADMI units in the NF permeate, a lower American dye manufacturing index (ADMI) (i.e. method of colour representation) compared to the potable water (~17 ADMI units) utilised by the industrial partner in their dyeing processes. The colour was reduced from an average of 659 to ~20 ADMI units in the RO permeate, a lower ADMI and therefore colour when compared to the potable water. An average conductivity rejection of 91% was achieved with conductivity being reduced from an average of 7,700 to 693 µS/cm and the TDS reduced from an average of 5,700 to 473 mg/L, which facilitated an average TDS rejection of 92%. Based on the composition of the UF permeate fed to the RO membrane a maximum removal of 98.7% was achieved for both conductivity and total dissolved solids (TDS).

The proposed full-scale plant would incorporate a UF-MBR system, followed by NF, RO, flocculation and a filter press. Therefore, the two waste products produced during operation of the proposed full-scale plant, would be the solid filter cakes and the liquid filtrate from the filter press. Implementing the proposed full-scale plant it would cost the industrial partner an operating cost of ZAR 113.85 and ZAR 3,415.49 to treat 97.1 m$^3$ and 2,913 m$^3$ of textile wastewater, respectively, per day and per month. This results in an annual saving of ZAR 845,848 on potable water expenses.

This research, would provide SA textile industries, with an option to: 1) reduce their water consumption, thereby utilising less of a valuable decreasing commodity; 2) meet the SA government discharge standards and reduce their discharge costs; 3) reduce their carbon footprint (i.e. reduce their impact on the environment) by re-using their treated wastewater and therefore using less water from the municipality; and 4) decrease their annual expenditure on water, since the treated wastewater would be available for re-use.

# ACKNOWLEDGEMENTS

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# DEDICATION

For my mom, who supports and believes in me unconditionally and gave me the wings to follow my dreams

# PREFACE

This thesis was written according to the guidelines provided by CPUT for a traditional thesis.

- Chapter 1 includes a brief introduction and background into textile wastewater. It provides the rationale and motivation for doing this project and provides information with regards to the proposed pilot plant to be used during the study.
- Chapter 2 is a literature review relating to membrane bioreactors and textile wastewater.
- Chapter 3 is a materials and methods chapter which includes: 1) a detailed layout of both the UF-dsMBR and NF/RO pilot-scale systems complete with tank sizes and stream flow rates; 2) a detailed experimental time line; 3) lists of the different analyses performed; as well as 4) figures representing all the operational data recorded.
- Chapter 4 is a results chapter and gives an overview of how the pilot-scale MBR plant was operated daily for ~8-months (i.e. 250 days) and the results obtained with regard to the treatment efficiency over 220 days.
- Chapter 5 is a results chapter which elaborates on the residual colour and salt removal from the ultrafiltration (UF) permeate for 5 sample sets taking the hydraulic retention time (HRT) into account for a fixed feed flow rate.
- Chapter 6 describes how the pilot plant can be scaled up to full-scale size and includes the design and cost of the proposed full-scale plant.
- Chapter 7 summarises all the results obtained along with an overall discussion.
- Chapter 8 summarises all the conclusions drawn, as a whole, from the results obtained during the study and includes recommendations for future studies.

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# LIST OF SYMBOLS

| | |
|---|---|
| $A$ | Weight of the glass fibre filter and sample residue, as well a the aluminium dish (mg) |
| $B$ | Weight of the glass fibre filter and the aluminium dish (mg) |
| $b$ | Cell path length (cm) |
| $C$ | Volume of sample filtered (ml) |
| $C_1$ | Initial concentration (mg/L) |
| $C_2$ | Final concentration (mg/L) |
| $C_L$ | Designed dissolved oxygen concentration (kg $O_2$/L) |
| € | Euro |
| $F$ | Calibration factor |
| $Con.N$ | Oxygen required for nitrification (kg $O_2$/kg $N_2$) |
| $C_S$ | Saturation concentration of water (kg/L) |
| $C_{s,T}$ | Oxygen saturation concentration corrected for temperature and altitude (kg/L) |
| $N_{Effective}$ | 60% of the average ammonium (mg/L) |
| % | Percentage (dimensionless) |
| $Q$ | Volumetric Flow Rate (L/h) |
| $R$ | Universal gas constant (J.K$^{-1}$.mol) |
| $R_1$ | Aromatic constituents in dye molecules (dimensionless) |
| $R_2$ | Aromatic constituents in dye molecules (dimensionless) |
| $ | Dollar (monetary unit) |
| $T$ | Inlet temperature of aerobic tank (°C) |
| $T_T$ | True turbidity (NTU) |
| $T_D$ | Turbidity of diluted sample (NTU) |
| $T_{ref}$ | Ambient temperature (°C) |

| | |
|---|---|
| $V_1$ | Volume to add (ml or L) |
| $V_2$ | Final volume (ml or L) |
| $V_D$ | Volume of dilution water (ml) |
| $V_S$ | Volume of sample before dilution (ml) |
| $V_X$ | Munsell value for X |
| $V_Y$ | Munsell value for Y |
| $V_Z$ | Munsell value for Z |
| $x$ | Unknown (g or ml) |

*Greek letters*

| | |
|---|---|
| $\alpha$ | Transfer coefficient for oxygen from potable water (dimensionless) |
| $\beta$ | Wastewater factor that inhibits oxygen transfer (dimensionless) |
| $\theta$ | Arrhenius constant used to correct the effects of temperature (°$C^{-1}$) |
| $\rho_{Air}$ | Density of air ($kg/m^3$) |
| $\tau$ | Temperature correction factor for saturation concentration of water (dimensionless) |
| µm | Micrometres |
| µS/cm | Micro Siemens per centimetre |
| $\omega$ | Pressure correction factor for saturation concentration of water (dimensionless) |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| AAR | Actual Air Required |
| ABR | Anaerobic Baffled Reactor |
| ADMI | American Dye Manufacture Index (ADMI units) |
| ADUF | Anaerobic Digestor Ultrafiltration |
| AEC-MBR | Airlift External Circulation Membrane Bioreactor |
| AGI | Agitator |
| AMBR | Aerobic Membrane Bioreactor |
| AOMBR | Anaerobic-oxic Membrane Bioreactor |
| AOR | Actual Oxygen Required |
| AOX | Adsorbable Organic Halide |
| BFD | Block Flow Diagram |
| BF-MBR | Biofilm Membrane Bioreactor |
| BLW | Blower |
| BNR | Biological Nutrient Removal |
| BOD | Biological Oxygen Demand (mg/L) |
| BP | Backward Pump |
| BSMBR | Biomass Separation Membrane Bioreactor |
| C:N:P | Carbon:Nitrate:Phosphate |
| CAPEX | Capital Expenditure |
| CAS | Conventional Activated Sludge |
| CCT | City of Cape Town |
| CFV | Crossflow Velocity (m/s) |
| cHz | Cyclic Hertz |
| CIP | Cleaning-in-place |
| $Cl^-$ | Chloride |
| cm | Centimetre |
| $CO_2$ | Carbon dioxide |
| COD | Chemical Oxygen Demand (mg/L) |
| CPMP | Centrifugal Pump |
| CPUT | Cape Peninsula University of Technology |
| CT | Capillary Tube |
| CTICC | Cape Town International Convention Centre |
| d | Day |
| Da | Daltons |
| DE | Adams Nickerson colour difference |

| | |
|---|---|
| DEWATS | Decentralised Wastewater Treatment Systems |
| dMBR | Diffusive Membrane Bioreactor |
| DO | Dissolved Oxygen (mg/L) |
| DOC | Dissolved Organic Carbon (mg/L) |
| dsMBR | Dual-Stage Membrane Bioreactor |
| DWA | Department of Water Affairs |
| DWAF | Department of Water Affairs and Forestry |
| DWEA | Department of Water and Environmental Affairs |
| EBPR | Enhanced Biological Phosphate Removal |
| EFC | Eutectic Freeze Crystallisation |
| eMBR | Extractive Membrane Bioreactor |
| EPS | Extracellular polymeric substances |
| Eq. | Equation |
| ERD | Energy Recovery Device |
| err | Error |
| ESS | Environmental Sciences Section |
| EWRP | eMalahleni Water Reclamation Plant |
| F/M | Food/Micro-organism |
| FC | Pleated Filter Cartridge |
| $Fe^{2+}$ | Ferrous Iron |
| FIC | Flow Indicator Controller |
| FLW | Flow meter |
| FMB-CFMF | Floating Media Biofilter-Crossflow Microfiltration System |
| FO | Forward Osmosis |
| $FO_x$ | Fenton Oxidation |
| FP | Forward Pump |
| FRP | Fibreglass Reinforced Plastic |
| FS | Flat Sheet |
| FU | Flow Unit |
| g | Grams |
| g/L | Grams per litre |
| g/mol | Grams per mol |
| GAC | Granular Activated Carbon |
| h | Hour |
| $H_2O$ | Water |
| $H_2O_2$ | Hydrogen Peroxide |
| $H_2SO_4$ | Sulphuric acid |
| $H_3PO_4$ | Phosphoric acid |

| | |
|---|---|
| HCl | Hydrochloric acid |
| HF | Hollow Fibre |
| HMI | Human Machine Interface |
| HRT | Hydraulic Retention Time |
| HWWTW | Hammarsdale Wastewater Treatment Works |
| Hz | Hazen |
| $Hz_{pump}$ | Hertz |
| iCOD | Insoluble Chemical Oxygen Demand |
| IEMBR | Ion Exchange Membrane Bioreactor |
| iMBR | Immersed or Submerged Membrane Bioreactor |
| kg | Kilogram |
| $kg/m^3$ | Kilogram per cubic metre |
| kL | Kilolitres |
| kPa | Kilopascals |
| kW | Kilowatt |
| L | Litres |
| L/d | Litres per day |
| *L. casei* | *Lactobacillus casei* |
| LS | Level Switch |
| Ltd. | Limited |
| M | Molar |
| m | Metres |
| $m^2$ | Metres squared |
| $m^3$ | Cubic metre |
| MAB | Membrane Aeration Bioreactor |
| MBR | Membrane Bioreactor |
| MCC | Motor Control Centre |
| MF | Microfiltration |
| mg | Milligram |
| mg/L | Milligram per litre |
| mg Pt/L | Milligram Platimun per litre |
| MGR | Membrane Gradostat Bioreactor |
| min | Minutes |
| ML | Megalitres |
| ml | Millilitres |
| ml/g | Millilitre per gram |
| ML/d | Megalitres per day |
| MLSS | Mixed Liquor Suspended Solids (mg/L) |

| | |
|---|---|
| mm | Millimetre |
| mmol/L | Millimol per litre |
| Mo | Motor |
| MT | Multi-tubular |
| $M_w$ | Molecular weight |
| MWCO | Molecular Weight Cut Off |
| N/A | Not Applicable |
| $Na^+$ | Sodium |
| NaCl | Sodium Chloride (i.e. fine salt) |
| NaOH | Sodium Hydroxide |
| NaOCl | Sodium Hypochlorite |
| $Na_2SO_4$ | Sodium Sulphate (i.e. Glaubers salt) |
| NF | Nanofiltration |
| $NH_3$ | Ammonia |
| $NH_3$-N | Ammonium nitrogen |
| $NH_4^+/NH_4$ | Ammonium (mg/L) |
| ni | Not Indicated |
| nm | Nanometre |
| No. | Number |
| $NO_3$ | Nitrate (mg/L) |
| NRF | National Research Foundation |
| NTU | Nephlometric Turbidity Units |
| OLR | Organic Loading Rate |
| OPEX | Operational Expenditure |
| OTR | Oxygen Transfer Rate |
| P | Pump |
| P&G | Preliminary and General |
| PAC | Powdered Activated Carbon |
| PAN | Polyacrylonitrile |
| PD | Positive Displacement |
| PDIA | Pressure Differential Indicator Alarm |
| PES | Polyethersulfone |
| PFD | Process Flow Diagram |
| PG | Pressure Gauge |
| PI | Pressure Indicator |
| PIC | Pressure Indicator Controller |
| P&ID | Piping and Instrumentation Diagram |
| PLC | Programmable Logic Control |

| | |
|---|---|
| $PO_4$ | Phosphate (mg/L) |
| $PO_4$-P | Orthophosphate (mg/L) |
| PP | Polypropylene |
| ppm | Parts Per Million |
| PPMP | Peristaltic Pump |
| PSO | Polysulfone |
| Pty | Proprietary |
| PVC | Polyvinyl Chloride |
| PVDF | Polyvinylidene Fluoride |
| QIS | Quantity Indicator Switch |
| RO | Reverse Osmosis |
| s | Seconds |
| SA | South Africa |
| SAB | Submerged Aerated Biofilter |
| SANAS | South African National Accreditation System |
| SAMBR | Submerged Anaerobic Membrane Bioreactor |
| SANS | South African National Standard |
| SBR | Sequencing Batch Bioreactor |
| sCOD | Soluble Chemical Oxygen Demand |
| SCR | Screen |
| SMABR | Submerged Membrane Adsorption Bioreactor |
| sMBR | Sidestream Membrane Bioreactor |
| $SO_3$ | Sulphide |
| SOR | Standard Oxygen Required |
| SPMP | Submersible Pump |
| SRT | Sludge Retention Time |
| SS | Suspended Solids (mg/L) |
| SVI | Sludge Volume Index (ml/g) |
| SW | Spiral Wound |
| tCOD | Total Chemical Oxygen Demand (mg/L) |
| TDS | Total Dissolved Solids (ppm) |
| TFC | Thin Film Composite |
| TISA | Temperature Indicator Shutdown Alarm |
| TMP | Transmembrane Pressure |
| TNK | Tank |
| TOC | Total Organic Carbon (mg/L) |
| TS | Temperature Switch |
| TSS | Total Suspended Solids (mg/L) |

| | |
|---|---|
| UASB | Upflow Anaerobic Sludge Bed |
| UCT | University of Cape Town |
| UF | Ultrafiltration |
| USPW | Umbilo Sewage Purification Works |
| UV | Ultraviolet |
| UV/$H_2O_2$ | Chemical Oxidation Agent |
| UV/$O_3$ | Chemical Oxidation Agent |
| UV-VIS | Ultraviolet Visible Spectroscopy |
| VAT | Value Added Tax |
| VLV | Valve |
| VSS | Volatile Suspended Solids (mg/L) |
| WAS | Waste Activated Sludge |
| WDCS | Waste Discharge Charge System |
| WRC | Water Research Commission |
| WRF | White-Rot Fungus |
| WWTW | Wastewater Treatment Works |
| ZAR | South African Rand |

# GLOSSARY

| **Terms** | **Explanation** |
|---|---|
| **Aerobic** | Conditions where biochemical reactions are oxygen dependent and oxygen acts as an electron acceptor. During aerobic treatment organic compounds, such as biochemical oxygen demand (BOD) and chemical oxygen demand (COD), are removed and ammonia is oxidised to nitrate (i.e. nitrification) (Judd, 2011). |
| **Anaerobic** | Conditions where in the complete absence of oxygen biochemical reactions occur (i.e. oxygen independent) (Judd, 2006). |
| **Anoxic** | Conditions where biochemical reactions take place and another compound, other than oxygen, acts as the electron acceptor; and nitrate is reduced to nitrogen gas (i.e. denitrification) (Judd, 2011). |
| **Azo dyes** | Are electron deficient xenobiotic, aerobically recalcitrant aromatic compounds, possessing one or more azo (-N=N-) groups, as well as sulfonic (-$SO_3$) electron withdrawing groups that make up the largest and most diverse group of synthetic dyes found in the effluents of dye utilising industries (You & Teng, 2009, Pandey *et al.,* 2007; Żyłła *et al*., 2006; Chang *et al.,* 2001). |
| **Azo reductases** | Enzymes possessed by bacterial consortia capable of disrupting azo bonds under anaerobic conditions (negative redox potential) (Żyłła *et al.,* 2006). |
| **Biochemical oxygen demand (BOD)** | The amount of oxygen required by micro-organisms growing in aerobic conditions for the biodegradation of compounds (Bassa & Chetty, 2002). |

| **Terms** | **Explanation** |
|---|---|
| **Biodegradation** | The breakdown of compounds to its chemical constituents via living organisms. |
| **Carcinogenic** | A substance with cancer causing potential. |
| **Chemical oxygen demand (COD)** | The amount of oxygen required for complete chemical oxidation of compounds, present in both water and wastewater, to $CO_2$, $H_2O$ and $NH_3$ (Judd, 2011; Bassa & Chetty, 2002). |
| **Cytotoxic** | A substance toxic to living cells. |
| **Membrane Bioreactor (MBR)** | Combination of anaerobic and aerobic biological processes with membrane separation (Brik *et* al., 2006; Badani *et al.,* 2005; Van der Roest *et al.,* 2002). |
| **Mineralisation** | Mineralisation occurs when large organic molecules, such as aromatic amines, are biodegraded to carbon dioxide ($CO_2$), water ($H_2O$) and inorganic nitrogen products, like ammonia ($NH_3$) (Judd, 2006; Kodam & Gawai, 2006; Sponza & Işik, 2005). |
| **Mutagenic** | A substance capable of causing a mutation. |
| **Recalcitrant** | Resistant or non-responsive to change. |

# OUTPUTS FROM THIS STUDY

- **Technical Reports**

Edwards, W., Sheldon, M.S., Zeelie, P.J., **De Jager, D.**, Dekker, L.G. & Bezuidenhout, C.C. 2013. Water reuse using a dual-stage membrane bioreactor for industrial effluent treatment. WRC Report No. TT 556/13. Water Research Commission, Pretoria.

- **Publications**

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2012. Membrane bioreactor application within the treatment of high-strength textile effluent. *Water Science and Technology,* 65(5):907-914.

- **Conference Presentations**

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2011. Application of a pilot-scale MBR system for the removal of colour from industrial textile effluent. WISA-MTC 2011, Umhlanga, Durban, South Africa, 11 – 14 September 2011.

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2012. Colour removal from textile effluent using a pilot-scale UF-dsMBR and subsequent NF/RO system. WISA Biennial Conference and Exhibition, CTICC, Cape Town, South Africa, 6 – 10 May 2012.

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2012. Membrane bioreactors (MBRs) for the treatment of textile wastewater: The South African perspective. CPUT Postgraduate Research Conference, CPUT (Bellville campus), Cape Town, South Africa, 7 September 2012.

- **Conference Posters**

**De Jager, D.**, Schoeman, H.A., Sheldon, M.S. & Edwards, W. 2010. Membrane bioreactor application within the treatment of high-strength textile effluent. CPUT Research Day, Cape Town, South Africa, 3 December 2010.

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2011. Membrane bioreactor application within the treatment of high-strength textile effluent. 2nd Regional Conference of Southern African Young Water Professionals (YWP), CSIR, Pretoria, South Africa, 3 – 5 July 2011.

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2012. Application of an on-site UF-dsMBR system for the treatment of high-strength textile effluent. WISA Biennial Conference and Exhibition CTICC, Cape Town, South Africa, 6 – 10 May 2012.

- **International Conference Poster**

**De Jager, D.**, Sheldon, M.S. & Edwards, W. 2012. A case study on the treatability of industrial textile wastewater in South Africa using a combined MBR-NF/RO system for re-use. Singapore International Water Week: Water Convention, Sand Expo and Convention Centre, Marina Bay Sands, Singapore, 1 – 5 July 2012.

- **Awards**

Best student oral presentation (1st prize) at the International Water Institute of Southern Africa Membrane Technology Conference (WISA-MTC 2011); Umhlanga, Durban, South Africa, 11 – 14 September 2011.

Best oral presentation in the Faculty of Engineering at the CPUT Postgraduate Research Conference, CPUT (Bellville campus), Cape Town, South Africa, 7 September 2012.

# CHAPTER 1

# INTRODUCTION

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

This DTech project formed part of a collaborative Water Research Commission (WRC) funded project K5/1900 - Pilot application of a dual-stage membrane bioreactor (dsMBR) for industrial wastewater treatment. This project investigated the application of a dsMBR for the on-site treatment and recovery of industrial trade wastewater. The purpose of this evaluation was to determine if the use of membrane systems in combination with biological processes is a plausible method for textile wastewater treatment, recovery and re-use. This project aimed to provide a mobile, adaptable solution to specific industrial needs. A further challenge experienced by the wastewater treatment sector is the severe shortage of skilled labour. This project, therefore, also aimed to facilitate a skills development initiative exposing historically disadvantaged students through the Cape Peninsula University of Technology (CPUT) internship programme, as well as postgraduate students to on-site training in MBR technology. A textile industry located in the Western Cape was chosen as the industrial partner for the on-site evaluation of the pilot plant.

Dyehouse processes are a large contributor to the contamination of water within the textile industries. Dyehouse wastewater is complex and consists of concentrated waste process water, which contains a wide and varied range of dyes and other products (Badani *et al*., 2005; Fersi *et al*., 2005; Chakraborty *et al*., 2003; Barclay & Buckley, 2002; Robinson *et al.,* 2001). Azo dyes are aromatic compounds that make up the largest and most diverse group of synthetic dyes found in the wastewaters of dye utilising industries, such as textile, paper printing, food and cosmetic industries. A major concern in wastewater treatment is the release of azo dyes and their metabolites into the environment, as some may be mutagens and carcinogens (Pandey *et al.,* 2007; Żyłła *et al*., 2006; Chang *et al.,* 2001). Conventional aerobic wastewater treatment cannot efficiently decolourise wastewaters contaminated with azo dyes since these dyes are considered to be xenobiotic in nature and aerobically recalcitrant to biodegradation (Pandey *et al.,* 2007; Żyłła *et al*., 2006; Chang *et al.,* 2001). Azo dyes commonly utilised in the textile industry include reactive, acid and direct dyes (You & Teng, 2009). Anaerobic systems have previously been utilised to cleave azo bonds in the treatment of textile wastewater (Dos Santos *et al.,* 2007; O'Neill *et al.,* 1999a). Many bacterial consortia possess enzymes (i.e. azo-reductases) capable of disrupting the azo bonds under anaerobic conditions (Żyłła *et al.,* 2006). The product of this anaerobic degradation is colourless aromatic amines, which are carcinogenic. These aromatic amines can then be readily degraded via aerobic digestion (Van der Zee & Villaverde, 2005).

A fast emerging area of MBR applications is in the treatment of industrial trade wastewater. MBR systems in which a three stage process is used (i.e. anaerobic/aerobic and membrane filtration) show great promise in the treatment of textile wastewater, as the processes either remove the dyes, allowing for the re-use of the auxiliary chemicals used for dyeing, or concentrate the dyes and their auxiliary compounds producing purified water (Chakraborty *et al.,*2003). MBR technology is an attractive alternative to the conventional methods of treating textile wastewater, as no sludge is wasted and all the bacteria are retained within the reactor including specific bacteria capable of degrading the low degradable textile wastewater (You *et al.,* 2008; You *et al*., 2006).

## 1.2 Problem statement

Textile wastewater contains non-biodegradable constituents and therefore some companies do not meet the South African (SA) government discharge standards, causing detrimental problems if the wastewater is discharged into the sewerage system and into the environment. Unless the wastewater is properly treated before it is discharged, it may contaminate the natural water environment.

## 1.3 Research questions

- How efficient is a sidestream dual-stage MBR (dsMBR) in the treatment of textile wastewater?
- How robust is the dsMBR system?
- What degree of colour removal can be achieved by the system?
- What degree of mineralisation occurs in the process?
- Can textile wastewater be treated to within the required government discharge standards?
- Is it possible to re-use the water reclaimed from the treated textile wastewater? Or is subsequent treatment required before re-use is possible?
- Would the implementation of a full-scale MBR textile wastewater treatment system be economically feasible?

## 1.4 Aims and objectives

The aim of this study was to evaluate whether a 5 to10 $m^3$/d pilot plant sidestream dsMBR could render textile wastewater safe for discharge, recycling and/or re-use.

The specific objectives for this study were to:

- commission and operate the pilot plant and determine the treatment efficiency of the system to treat the textile wastewater to within the South African government's discharge standards or possibly even for re-use;

- remove residual colour after the dsMBR phase using treatment processes such as NF and RO, and determine the removal efficiency;
- evaluate the degree of overall colour removal from the textile wastewater;
- evaluate the degree of mineralisation; and
- propose a design and cost for a full-scale plant based on the pilot plant results.

## 1.5 Research design and methodology

A semi-automated pilot wastewater treatment plant using two 5.1 $m^2$ Norit X-flow airlift sidestream dsMBR modules were constructed and operated at a textile company located in the Western Cape, South Africa (SA). The industrial textile wastewater treatment system, which was robust and non-sterile, consisted of a series of tanks: 1) an anaerobic tank, which cleaved the azo bonds of the reactive dyes; 2) an anoxic tank containing reduced amounts of dissolved oxygen, in which denitrification occurred; and 3) an aerobic tank, in which nitrification, as well as mineralisation of the aromatic amines occurred. The sidestream crossflow dsMBR system would account for the removal of any solid organic material that may have been left in the water. The second phase was the treatment of the permeate from the ultrafiltration (UF)-MBR modules via NF for residual colour removal, alternated with RO for both colour and salt removal, to determine if the industry would be able to re-use the water.

Water samples collected from the sump, anaerobic-, anoxic- and aerobic-tanks, as well as permeate from the MBR and NF/RO systems were analysed on a daily basis. The following parameters were analysed: ammonium concentration ($NH_4$); biological oxygen demand (BOD); conductivity; dissolved organic carbon (DOC); nitrate concentration ($NO_3$); pH; temperature; total chemical oxygen demand (COD); total dissolved solids (TDS); total organic carbon (TOC); total phosphate concentration, total suspended solids (TSS); true colour; American dye manufacturing index (ADMI) colour; turbidity; and volatile suspended solids (VSS).

The following flow rates were monitored and recorded daily and adjusted accordingly to maintain constant liquid levels across the entire system:

- the sump outlet to the municipality;
- the equalisation tank to the anaerobic tank;
- the anaerobic tank to the anoxic tank;
- the anoxic tank to the aerobic tank;
- the recycle from the anoxic tank to the anaerobic tank; as well as
- the retentate recycle from the MBR system to the anoxic tank.

The following engineering aspects were covered during this study:

i. commissioning and operation of the pilot-scale dsMBR system (Chapter 3); calculation of the dosing requirements in order to obtain the required C:N:P ratio (Chapter 3); and operation of the pilot-scale NF/RO system (Chapter 3).
ii. material balances across each individual component of the pilot plant, as well as an overall material balance for the entire pilot plant (Chapter 3; Appendix D).
iii. calculating the oxygen requirements of the activated sludge within the biological section (i.e. aerobic tank) of the full-scale plant (Chapter 6).
iv. analysing the cost of the proposed full-scale plant (Chapter 6).
v. calculating the cost per unit water treated by the full-scale plant (Chapter 6).

## 1.6 Significance

The goal was to successfully treat industrial textile wastewater containing xenobiotic azo dyes that are aerobically recalcitrant to biodegradation. The wastewater treatment system was located on site and consequently afforded the opportunity for testing of an actual continuously changing industrial textile wastewater instead of normal laboratory prepared synthetic non-changing textile wastewaters.

This research provided SA textile industries which not only utilise large volumes of water, but also produce large quantities of wastewater containing azo dyes with the following options: 1) to reduce their water consumption, thereby utilising less of a valuable decreasing commodity; 2) to meet the SA government discharge standards and reduce their discharge costs; 3) re-using the treated wastewater to reduce their carbon footprint by utilising less water from the municipality, thus reducing their impact on the environment; and 4) to decrease their annual expenditure on water, since the treated water would be available for re-use.

## 1.7 Delineation

This pilot plant study was an industrial application and therefore did not include basic fundamental research on principles relating to the following:

i. modelling the hydrodynamics of the MBR system;
ii. determining the microbial population dynamics of the MBR system; or
iii. developing the mathematical model that best describes the kinetics of the wastewater treatment MBR.

It did not include testing of the following:

i. different membrane types;
ii. different membrane materials; or
iii. different membrane sizes.

Although cleaning-in-place (CIP) was performed when the membranes fouled, this study did not focus on the fouling (i.e. cause or removal) of the membranes.

It did not include salt recovery and recycling from the brine produced during the subsequent Phase 2 of operation with the NF/RO pilot-scale system.

This pilot plant study also did not include the optimisation of the treatment process since the purpose of the project was to determine whether the use of MBRs was a plausible method for textile wastewater treatment, recovery and re-use.

# CHAPTER 2

# LITERATURE REVIEW

## REVIEW: MEMBRANE BIOREACTORS (MBRS) FOR THE TREATMENT OF TEXTILE WASTEWATER AND THE SOUTH AFRICAN PERSPECTIVE

# CHAPTER 2

# REVIEW: MEMBRANE BIOREACTORS (MBRS) FOR THE TREATMENT OF TEXTILE WASTEWATER AND THE SOUTH AFRICAN PERSPECTIVE

## 2.1 Introduction

The textile industry is a water intensive industry (Brik *et al.,* 2006; Badani *et al.,* 2005; Chakraborty *et al*., 2003; Barclay & Buckley, 2002) producing wastewater containing varying concentrations of both organic and inorganic compounds (Libra & Sosath, 2003). Treating industrial textile wastewater is complicated due to the high levels of biological oxygen demand (BOD), chemical oxygen demand (COD), total dissolved solids (TDS) and non-biodegradable nature of the organic dyes present in the wastewater (Badani *et al.,* 2005; Kim *et al.,* 2002). When treating textile wastewater for re-use by the industry colour removal, as well as total suspended solids (TSS), BOD and COD reduction must be addressed in the primary treatment stage. The most commonly used treatment methods for textile wastewater are the following : 1) physico-chemical treatment using lime and ferrous/alum that generates large quantities of hazardous sludge and is ineffective in the removal of colour, TSS, BOD and COD; 2) biological treatment with aeration in order to reduce BOD and COD levels; 3) chlorination that successfully removes colour and reduces BOD and COD, though this method produces chloro-organic compounds that are potentially carcinogenic and therefore not eco-friendly; and 4) ozonation with or without ultraviolet irradiation (You & Teng, 2009; González-Zafrilla *et al.,* 2008; Badani *et al.,* 2005; Chakraborty *et al*., 2003; Kural *et al.,*2001; Gupta *et al*., 2000; Slokar & Le Marechal, 1998). The key factors affecting the reclamation of treated wastewater are suspended solids (SS), turbidity, colour and non-biodegradable substances. Therefore, advanced treatment processes are required to improve the quality of the treated wastewater up to re-use criteria (Feng *et al.,* 2010).

## 2.2 Treatment of azo dyes

### 2.2.1 Dye structure and decolourisation

Azo dyes are aromatic compounds that make up the largest and most diverse group of synthetic dyes (Kodam *et al.,* 2005) found in the wastewaters of dye utilising industries such as textile, paper printing, food and cosmetic industries (Pandey *et al.,* 2007; Żyłła *et al*., 2006; Chang *et al.,* 2001). Azo dyes make up 60% to 70% of the dyes used in the textile dyeing industry (Hai *et al*., 2011) and therefore are a major problem in the treatment of textile wastewater because they are recalcitrant to conventional wastewater treatment methods (Hai *et al*., 2011; Kodam *et al.,* 2005). The azo dyes commonly utilised in the textile industry include reactive, acid and direct dyes (You & Teng, 2009). Decolourisation of water polluted with organic colourants occurs when the –C=C- bonds, -N=N- bonds and the heterocyclic and aromatic rings are reduced (Slokar & Le Marechal, 1998). These dyes are electron deficient, xenobiotic compounds (Hai *et al*., 2011) because they possess one or more azo (-N=N-) groups, as well as sulfonic (-$SO_3$) electron withdrawing groups. This generates electron deficiency within the molecule, making the compound less susceptible to oxidative catabolism by bacteria (You & Teng, 2009). Therefore, for complete mineralisation of azo dyes, a combination of reductive (anaerobic) and oxidative (aerobic) steps are required (Dafale *et al.,* 2010; Kodam & Gawai, 2006; Kodam *et al.,* 2005).

The reactive azo dyes contain one to four azo bonds (You & Teng, 2009) which are easily reduced under anaerobic conditions. Van der Zee and Villaverde (2005) reported decolouration of textile wastewaters through reductive cleavage of the azo bonds. The disruption or cleavage of the azo bonds under anaerobic conditions (negative redox potential) (Żyłła *et al.,* 2006) is initiated by azoreductase (Kodam *et al.,* 2005), an enzyme many bacterial consortia possess. The products of this anaerobic degradation are colourless aromatic amines which may be cytotoxic, mutagenic or carcinogenic (Kodam & Gawai, 2006; Sponza & Işik, 2005). These aromatic amines, which are recalcitrant to biodegradation under anaerobic conditions, can be readily degraded via aerobic digestion (Dafale *et al.,* 2010; Kodam & Gawai, 2006; Kodam *et al.,* 2005; Sponza & Işik, 2005; Van der Zee & Villaverde 2005; Slokar & Le Marechal, 1998) by non-specific enzymes through hydroxylation and ring opening of the aromatic compounds (Sponza & Işik, 2005). Mineralisation of the azo dyes are complete when the aromatic amines have been biodegraded to carbon dioxide ($CO_2$), water ($H_2O$) and ammonia ($NH_3$) (Sponza & Işik, 2005). Figure 2.1 (Van der Zee & Villaverde, 2005; Carliell *et al.* 1996) and Equations (Eq.) 2.1 to 2.3 (Dafale *et al.,* 2010) illustrate the pathway of azo dye degradation via anaerobic digestion and the degradation of the resulting aromatic amine via aerobic digestion.

Step 1: Under anaerobic conditions, reduction of azo dye to the corresponding amine. A reaction catalysed by an enzyme azoreductase and an electron donor (Dafale *et al.,* 2010).

$$R_1 - N = N - R_2 + 2e^- + 2H^+ \rightarrow R_1 - NH - NH - R_2 \qquad \text{Eq. 2.1}$$

Step 2: Degradation of the resulting aromatic amine via a multi-step bioconversion under aerobic conditions (Dafale *et al.,* 2010).

$$R_1 - NH - NH - R_2 + 2e^- + 2H^+ \rightarrow R_1 - NH_2 + R_2 - NH_2 \qquad \text{Eq. 2.2}$$

Equation 2.3 represents the overall reduction of dye molecules through the cleavage and transfer of four electrons (Dafale *et al.,* 2010). $R_1$ and $R_2$ are aromatic constituents in the dye molecule (Anjaneyulu *et al.,* 2005).

$$(R_1 - N = N - R_2 + 2H^+) + 4e^- + 4H^+ \rightarrow R_1 - NH_2 + R_2 - NH_2 \qquad \text{Eq. 2.3}$$

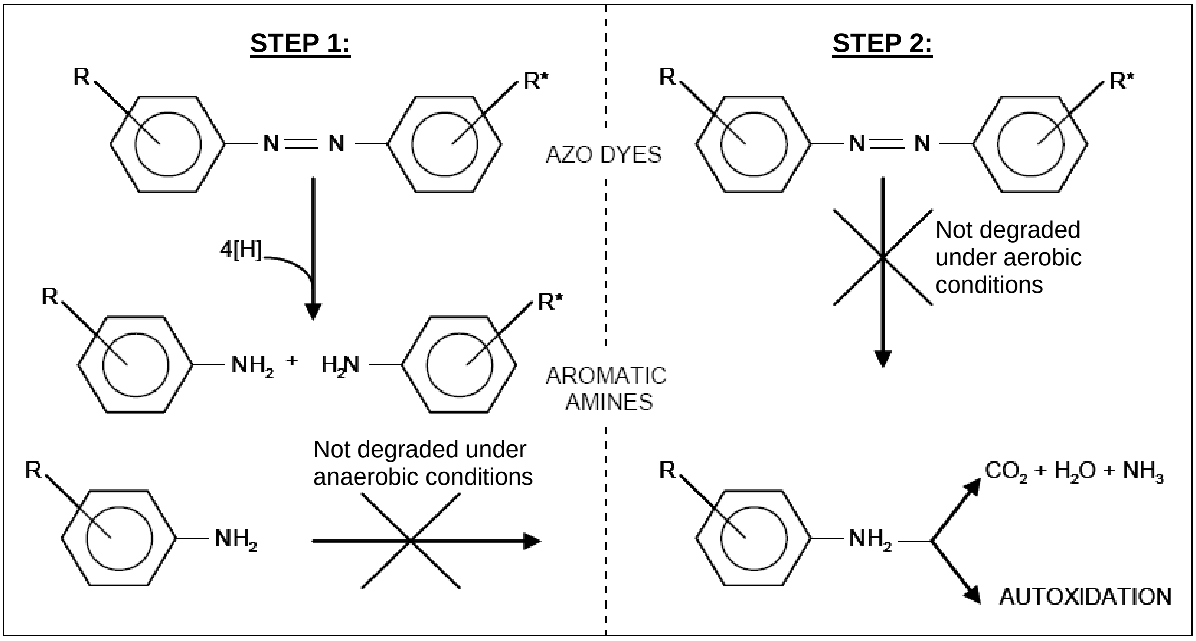


**Figure 2.1:** Anaerobic and aerobic degradation pathways of azo dyes and aromatic amines (Van der Zee & Villaverde, 2005)

The degradation of aromatic amines depends on their chemical structure. Simple aromatic amines can be mineralised under methanogenic conditions, while sulphonated aromatic amines are resistant and require specialised aerobic microbial consortia in order to be mineralised (Pandey *et al.,* 2007).

### 2.2.2 Biological treatment

Biological decolourisation to transform, degrade or mineralise azo dyes has been investigated over the past decades (Libra *et al.,* 2004; Slokar & Le Marechal, 1998). The two stage bacterial process (refer to Figure 2.1) is based on the observation that azo dyes can be anaerobically reduced into colourless aromatic amines, which then can be degraded aerobically. The use of bacterial strains to decolourise azo dyes is generally initiated by azoreductase-catalysed direct or indirect anaerobic reduction or cleavage via a four electron reduction at the azo bond (Sponza & Işik, 2005). The resulting aromatic amines are either aerobically or anoxically/anaerobically degraded by the use of a mixed or pure bacterial community. These operating conditions allow the reaction to be non-specific with respect to the bacteria as well as the dyes (Chang *et al.,* 2001; Libra *et al.,* 2004; Pandey *et al.,* 2007). Hu (1994) isolated and identified a bacterial strain of *Pseudomonas luteola* capable of efficiently decolourising a group of azo dyes, specifically reactive red 22 (Chang *et al.,* 2001). Several genera of *Basidiomycetes* have been identified as capable of mineralising azo dyes (Libra *et al.,* 2004).

## 2.3 Textile wastewater treatment methods

Conventional aerobic biological wastewater treatments, such as activated sludge processes, cannot efficiently decolourise wastewaters contaminated with azo dyes since these dyes are considered xenobiotic in nature and aerobically recalcitrant to biodegradation (Pandey *et al.,* 2007; Żyłła *et al*., 2006; Chang *et al.,* 2001). Most commercial dyes are toxic to the micro-organisms utilised in activated sludge processes resulting in sludge bulking (Kim *et al*., 2002; Ahn *et al.,* 1999; Hsu *et al.,* 1997; Lin & Peng, 1996; Gurnhan, 1965). Conventional treatment processes used in the treatment of textile wastewater (refer to Table 2.1) include physical methods (e.g. coagulation, flocculation, sedimentation, adsorption on activated carbon, biological sludge and silikagel, reverse osmosis, filtration); and chemical methods (e.g. reduction, compleximetric methods, ion exchange, neutralisation, chemical oxidation with UV/$O_3$, UV/$H_2O_2$ and Fenton reagent; advanced oxidation processes such as photocatalysis, electrochemical, sonolysis, ionising radiation) as well as biological methods which differ according to the presence or absence of oxygen (e.g. biological sludge, sequencing batch reactor) (You & Teng, 2009; González-Zafrilla *et al.,* 2008; Badani *et al.,* 2005; Chakraborty *et al*., 2003; Kural *et al.,*2001; Gupta *et al*., 2000; Slokar & Le Marechal, 1998).

**Table 2.1:** Conventional textile wastewater treatment processes (summarised from Dafale *et al.,* 2010; You & Teng, 2009; González-Zafrilla *et al.,* 2008; Badani *et al.,* 2005; Chakraborty *et al*., 2003; Kural *et al.,*2001; Ciardelli & Ranieri, 2000; Gupta *et al*., 2000; Slokar & Le Marechal, 1998)

| **Conventional treatment processes** | | | |
|---|---|---|---|
| Physical methods | Chemical oxidation | Advanced oxidation processes | Biological methods |
| 1. Coagulation<br>2. Adsorption (e.g. Activated carbon)<br>3. Ion exchange<br>4. Membrane filtration (e.g. reverse osmosis, nanofiltration) | 1. UV/$O_3$<br>2. UV/$H_2O_2$<br>3. Fenton reagent<br>4. Ozonation | 1. Photocatalysis<br>2. Electrochemical<br>3. Sonolysis<br>4. Ionising radiation | 1. Activated sludge (e.g. anaerobic, anoxic, aerobic)<br>2. Sequencing batch reactor |

The effectiveness of coagulation depends on the molecular structure, molecular mass, ionic character and auxiliary components of the dye (Zahrim *et al.,* 2010). A potentially powerful method for pollution control with high removal efficiencies is the advanced oxidation method of electrochemical wastewater treatment (Kim *et al.,* 2002).

Ozonation and electroflocculation are two oxidation techniques utilised in the treatment of textile wastewater. Electroflocculation combines an oxidation, flocculation and flotation process. Wet processes in the textile industry require water of a high quality, specifically with regards to dyes, detergents and suspended solids. Wastewater from textile industries is normally treated in chemical-physical plants, most commonly activated sludge biochemical plants. However, if the treated water is to be recycled and re-used, especially in dyeing processes, further treatment is still required.

In purification systems, oxidative treatments are effective in the oxidation of the chromophoric structures of dyes, as well as the removal of colour. The most common oxidation agents in wastewater treatment are: 1) chlorine and its derivatives (i.e. chlorine dioxide, sodium hypochlorite); 2) hydrogen peroxide; 3) ozone; 4) ultraviolet (UV) irradiation often combined with ozone or hydrogen peroxide; and 5) electrochemical cells (Dafale *et al.,* 2010; Ciardelli & Ranieri, 2000).

Ozonation and electroflocculation techniques were utilised in two pilot-scale plants in the treatment of dyeing and finishing textile wastewater for re-use (Ciardelli & Ranieri, 2000). The pilot plants closely resembled full-scale treatment to investigate the feasibility of scale-up. Prior to ozonation and electroflocculation, the textile wastewater was pre-treated in a biologically active sludge plant. The ozonation technique achieved 95% to 99% colour removal and the treated water was successfully re-used in dyeing, even with light colours. COD achieved a decrease of 60% (75 to 120 mg/L), which is normally considered too high for recycling, especially for dyeing with light colours. The electroflocculation technique achieved 80% to 100% colour removal and 70% to 90% COD removal. Biological pre-treatment and sand filtration is essential for successful ozonation while post-treatment (i.e. removal of flocculants) in the electroflocculation technique must be perfected in order to establish the correct cost-to-benefits ratio necessary for implementing these techniques at an industrial scale (Ciardelli & Ranieri, 2000).

Fenton oxidation ($FO_x$) is recognised as a valuable method to remove colour and increase the biodegradability of dyeing wastewater due to its high oxidative efficiency, non-selectivity and easy implementation. However, due to the high costs of reagents sole $FO_x$ is not economical (Feng *et al.,* 2010). Biotreatment is a more attractive option for the final purification process of wastewater. $FO_x$, based on the generation of highly reactive hydroxyl radicals, in combination with a submerged aerobic MBR composed of an activated sludge reactor and a hollow fibre membrane module, was utilised in the advanced treatment of wastewater from a dyeing wastewater treatment plant. Under optimal operating conditions, an initial pH of 5, ferrous iron ($Fe^{2+}$) concentration of 1.7 mmol/L and a hydrogen peroxide ($H_2O_2$) concentration of 17 mmol/L, Fenton's reagent reduced total organic carbon (TOC) and colour by 39.3% and 69.5%, respectively. Further purification with the MBR and an optimal hydraulic retention time (HRT) of 18 h yielded a 0.078 kg TOC/($m^3$.day). The Fenton process was thus effective in further enhancing the biodegradability of the wastewater (Feng *et al.,* 2010).

Often biological processes are combined with physical and chemical treatment processes (such as flocculation, precipitation and chemical coagulation), in order to better decolourise the wastewater and treat the wastewater of activated sludge (Dafale *et al.,* 2010; You *et al*., 2008). However, these physical and chemical treatment processes result in excess sludge requiring safe environmentally friendly disposal. Therefore, a demand exists for the development of natural, alternative, economic and environmentally friendly methods for the decolourisation of wastewater containing azo dyes (Pandey *et al.,* 2007; Chang *et al.,* 2001). Wastewater containing dyes can therefore be treated in two ways: 1) by using chemical or physical treatment methods to remove the dyes, referred to as 'decolouration', and 2) by biodegradation (Slokar & Le Marechal, 1998).

A pilot-scale combining various treatment methods including the fluidised biofilm process, chemical coagulation and electrochemical oxidation was investigated in the continuous treatment of textile wastewater from a synthetic textile dyeing factory (Kim *et al.,* 2002). The efficiency of the biological wastewater treatment process (i.e. fluidised biofilm process) was enhanced by including a supporting medium for the two isolated micro-organisms, *Aeromonas salmonicida* and *Pseudomonas vesicularis*. Using relatively low mixed liquor suspended solids (MLSS) and short sludge retention time, the COD and colour removal efficiency of the fluidised biofilm process was 68.8% and 54.5%, respectively. The inclusion of a supporting medium in the biological stage increased the COD and colour removal from 34.8% to 55.3% and 46.4% to 72.1%, respectively. The biological treatment stage was followed by chemical coagulation with the main purpose of decreasing the pollutant loading. The total COD and colour removal efficiency of the combined process was 95.4% and 98.5%, respectively (Kim *et al.,* 2002).

Biologically treated textile wastewater from a cotton thread factory was subjected to both direct nanofiltration (NF) and NF after pre-treatment with ultrafiltration (UF) to investigate the possibility of re-using the textile wastewater after NF treatment. For direct NF two membrane configurations were utilised: flat sheet membranes and spiral wound modules. In addition different pore sizes, namely NF90, NF200 and NF270 from Dow-Filmtec, were used for the flat sheet membranes operated at four different pressures in a pilot plant in order to select the most effective size. The parameters monitored were permeate flux, salt retention and COD removal. The NF90 flat sheet membrane modules yielded a COD reduction of 99% and the highest salt retention of 75% to 95%. After pre-treatment with UF the permeate flux from the NF increased by approximately 50% and the COD concentration in the feed was reduced by about 40% (González-Zafrilla *et al.* 2008). González-Zafrilla *et al.* (2008) and Arnal *et al.* (2008) concluded that the UF pre-treatment eliminated colloids and macromolecules responsible for most of the NF membrane fouling, thus reducing fouling and increasing total water recovery.

MBR technology is an attractive alternative and promising option for wastewater treatment and re-use (Guo *et al.,* 2008) compared to the conventional methods of treating wastewater. MBR technology is a biotreatment method involving a suspended growth activated sludge system and filtration on a porous-like membrane (i.e. microfiltration (MF) or UF) for solid/liquid separation. This leads to total retention of the biomass and improved biological reactor operation (Feng *et al.,* 2010; Guo *et al.,* 2008; Brik *et* al., 2006; Badani *et al.,* 2005; Van der Roest *et al.,* 2002). MBR systems have the potential to degrade residual organic matter and obtain high effluent quality, both for water re-use and additional purification steps (Feng *et al.,* 2010).

A fast emerging area of MBR applications is in the treatment of industrial trade wastewater. The first MBRs were introduced over 30 years ago in the 1970s by Dorr-Oliver (Hunter, 2007), with their main industrial application being wastewater treatment (Hai *et al.,* 2006; Schoeberl *et al*., 2005; Xu *et al.,* 2003; Voight *et al.,* 2001). However, in the 1980s the Japanese were quick to take up the technology (Hunter, 2007). Currently, the main influences affecting the MBR market worldwide are the following: 1) the more stringent legislation with regards to sewage treatment and the industrial wastewater discharge charge system; 2) the local water scarcity; 3) the implementation of an incentive reward system for improving wastewater technology for recycling and re-use; 4) decreasing investment costs; and 5) increasing confidence in MBR technology (Judd, 2011; Judd, 2006). The costs associated with the membranes and membrane processes have decreased (Judd, 2011; Judd, 2008; Judd, 2006) exponentially over the past 15 years (Judd, 2006). This reduction in costs is attributed to improved process designs and operation and maintenance schedules, as well as a greater membrane life span and the reduction in membrane cost (Judd, 2006).

MBRs are becoming the technology of choice since they provide effluent with high quality, low chemical pollutants and significant reduction in bacteria and viruses (Hunter, 2007). MBRs still tend to be more costly and energy intensive than conventional wastewater treatment processes despite the decrease in membrane costs, since the commercial implementation of immersed MBRs in 1990 (Judd, 2011). Membrane costs account for 53% to 64% of the total capital expenditure (CAPEX), while the other process equipment combined only accounts for 20% of the total CAPEX. However, recent developments suggest that membrane life of 10 years is not an unreasonable assumption (Judd, 2011). Since the system effluent can be recycled and re-used, the operating costs associated with MBR technology are significantly lower than those of conventional wastewater treatment processes, thereby reducing the volume of freshwater demand and wastewater disposal (Judd & Jefferson, 2003). Membrane techniques show great promise in the treatment of textile wastewater as they either remove the dyestuff allowing for re-use of the auxiliary chemicals used for dyeing or concentrate the dyestuffs and auxiliaries producing purified water (Chakraborty *et al.,*2003).

## 2.4 Membrane bioreactor technology

Advantages of MBR technology include the following: 1) high quality effluent free of suspended solids and macro-colloidal material; 2) complete removal of most micro-organisms and viruses, by increasing the solid retention time in order to increase the high biomass concentration and high organic loading rate capability; 3) high organics removal; 4) smaller footprint (Konsowa *et al.,* 2011; Feng *et al.,* 2010; Guo *et al.,* 2008; You *et al.,* 2008; Brik *et al.,* 2006; Hai *et al.,* 2006; You *et al.,* 2006; Schoeberl *et al.,* 2005; Stephenson *et al.,* 2000); as well as 5) decreased sludge production (Feng *et al.,* 2010; Guo *et al.,* 2008); 6) decreased total retention time; 7) enrichment of affected bacteria (Feng *et al.,* 2010); 8) enhanced nutrient removal stability (Guo *et al.,* 2008); and 9) lower sensitivity to contaminant peaks (Melin *et al.,* 2006).

The combination of activated sludge units and membrane filtration for biomass retention results in high effluent quality and compact plant configurations (Schoeberl *et al.,* 2005). Solid removal efficiencies of MBR technology is better than tertiary treatment effluent characteristics, resulting in high quality effluents with low TSS concentrations (Monclús *et al*., 2010).

Disadvantages of MBR technology include the following: 1) aeration limitations; 2) concentration polarisation (Cicek, 2003) and membrane fouling (You *et al.,* 2008; Charcosset, 2006; Melin *et al.,* 2006; Schoeberl *et al*., 2005); 3) high capital costs due to the expensive membranes even though membrane costs have decreased over time

(Judd, 2011); and 4) high energy costs due to the need to create a pressure gradient (Judd, 2011). The largest problem affecting MBR technology is membrane fouling, which results in reduced performance, a decline in flux or increase in transmembrane pressure (TMP), high energy consumption and frequent membrane cleaning or replacement. This leads to increased maintenance and operating costs (Konsowa *et al.,* 2011, Wang *et al.,* 2009). Techniques utilised to reduce fouling include 1) reduction of flux; 2) promotion of turbulence to limit the thickness of the boundary layer; 3) cleaning measures to remove the cake layer and foulants; and 4) relaxation and backwashing incorporated into MBR system designs to limit fouling (Kim *et al.,* 2011; Meng *et al.,* 2009; Remy *et al.,* 2009; Wu *et al.,* 2008; Le-Clech *et al.,* 2006; Schoeberl *et al*., 2005).

The application of MBRs in wastewater treatment and textile wastewater in particular will be discussed later under sections 2.4.5.1.

### 2.4.1 Membrane processes

Results from literature indicate that membrane systems within treatment-recycling systems reduce the cost of wastewater treatment through the recovery and re-use of chemicals and water (ElDefrawy & Shaalan, 2007). Membrane processes are categorised according to the pore size, molecular cut-off and pressure at which the membranes operate. These categories are inter-related, because as the pore size is reduced or the molecular cut-off decreases the pressure applied to the membrane, to maintain the same flux, increases (Van der Roest *et al.,* 2002). Membrane separation processes in which water forms the permeate can be categorised into four groups based on the pore size of the membranes. These categories, arranged from smallest to largest pore size, are reverse osmosis (RO), NF, UF and MF (Judd, 2011; Judd, 2006; Van Der Bruggen *et al.,* 2003; Mallia & Till*,* 2001*)*. Refer to Table 2.2 for a description and comparison of the four membrane processes. Current NF and RO membranes are made from thin film composite (TFC) membranes (Pearce, 2007a). The coarsest membrane, associated with MF, is capable of rejecting particulate matter, while the most selective membrane, associated with RO, can reject singly charged ions, like sodium ($Na^+$) and chloride ($Cl^-$) (Judd, 2006).

UF and MF are membrane separation processes that remove both large, dissolved solute molecules and suspended colloidal particles, as well as suspended solids, including micro-organisms (Charcosset, 2006; Judd and Jefferson, 2003; Van der Roest *et al.,* 2002). The term *membrane filtration* refers to the removal of particles from a feed stream as performed by UF and MF processes, while NF and RO processes remove dissolved species from the feed stream (Pearce, 2007b). NF selectively removes multivalent ions and certain charged or polar molecules, while RO removes inorganic ions (Judd & Jefferson, 2003). Therefore

**Table 2.2:** Comparison of the four membrane processes (Membrane processes, 2012; ElDefrawy *et al.,* 2007; Judd, 2006; Chakraborty *et al.,* 2003; Van Der Bruggen *et al.,* 2003; Wagner, 2001)

| | **Reverse osmosis** | **Nanofiltration** | **Ultrafiltration** | **Microfiltration** |
|---|---|---|---|---|
| **Membrane** | Asymmetrical | Asymmetrical | Asymmetrical | Symmetrical, Asymmetrical |
| **Pore size** | 0.0001 to 0.001 µm | 0.001 to 0.01 µm | 0.01 to 1 µm | 1 to 10 µm |
| **Rejection of** | High and low molecular weight components, sodium chloride, glucose, amino acids | High molecular weight components, mono-, di- and oligosaccharides, polyvalent negative ions | Macromolecules, proteins, polysaccharides vira | Particles, clay, bacteria |
| **Membrane materials** | Celulose acetate, Thin film | Celulose acetate, Thin film | Ceramic, PSO, PVDF, celulose acetate, Thin film | Ceramic, PP, PSO, PVDF |
| **Membrane module** | Tubular, spiral wound, plate-and-frame | Tubular, spiral wound, plate-and-frame | Tubular, hollow fibre, spiral wound, plate-and-frame | Tubular, hollow fibre |
| **Operating pressure** | 29 to 83 bar | 7 to 42 bar | 2 to 10 bar | 1 to 10 bar |
| **Permeability (L/h.m$^2$.bar)** | 0.05 to 1.5 | 1.5 to 30 | 10 to1,000 | > 1,000 |
| **Description/Application within wastewater treatment** | Suitable for the removal of ions and large species from dyebath wastewaters. RO systems have many applications in the recovery of dyes and hot water in continuous dyeing. RO is also utilised as a polishing step for UF/NF permeate. Ultrapure water and desalination. | Allows the separation of low molecular weight (< 1000) organic compounds and salts, with a softening effect. NF removes numerous pollutants such as COD, colour and non-bio-degradable contaminants. Removal of multi-valent ions and relatively small organics. | Used in the single step treatment of secondary textile wastewater. Removal of macromolecules, bacteria and viruses. | Separates suspended solids from water by sieving through macropores. Clarification, pre-treatment and removal of bacteria. |

membrane filtration processes UF and MF are often used as pre-treatment for NF and RO processes (Pearce, 2007b).

An application of UF in downstream processing is for product concentration, or in other words, the removal of a buffer or solvent. An application of MF is virus removal from cell cultures (Charcosset, 2006). In RO, separation occurs due to differing solubility and diffusion rates of solvents and solutes, while in NF separation occurs through a combination of charge rejection, solubility-diffusion and sieving through the membrane micropores (< 2 µm) (Judd, 2006). MF and UF-membrane separation processes are generally utilised in MBR concepts (Van der Roest *et al.,* 2002).

An alternative polishing method to RO is forward osmosis (FO), a concentration driven membrane process utilising the osmotic pressure difference across a selectively permeable membrane as the driving force (Wang *et al.,* 2010). In RO the driving force which facilitates mass transport through the membrane is the applied pressure (Cath *et al.,* 2006). FO has applications in the separation processes for wastewater treatment, food processing and desalination (Cath *et al.,* 2006) and has been used at bench-scale to treat industrial wastewater (Cath *et al.,* 2006; Wang *et al.,* 2010), to treat liquid foods in the food industry and to concentrate landfill leachate at pilot-scale. Research is currently occurring using FO to reclaim wastewater for potable re-use, desalinating seawater and for purifying water (Cath *et al.,* 2006; Wang *et al.,* 2010) in emergency relief situations (Cath *et al.,* 2006). Other unique research with regards to FO includes pressure-retarded osmosis for the generation of electricity from salt and fresh water (Cath *et al.,* 2006).

Advantages of FO include the following: 1) operating at low or no hydraulic pressures; 2) high rejection of a wide range of contaminants; and 3) possible lower membrane fouling than pressure driven processes (Cath *et al.,* 2006); Wang *et al.,* 2010). However, a number of technical barriers exist that prevent the use of FO in industrial applications: 1) the lack of an optimised membrane capable of producing a high flux comparable to commercial RO membranes (Wang *et al.,* 2010); and 2) the lack of robust membranes and membrane modules for FO (Cath *et al.,* 2006).

### 2.4.2 Membrane configurations

Membrane configuration refers to the geometry, the manner in which the membrane is mounted and its orientation in relation to the flow of water. Individual membrane units are housed together in “shells” to form modules, the units through which the water flows. For optimum efficiency a membrane should be configured to have 1) a high membrane area to module bulk volume ratio; 2) mass transfer on the feed side, promoted by a high degree of turbulence; 3) low energy requirements per unit volume of product water produced; (4) low costs per unit membrane area; 4) a design that allows easy cleaning; and 5) a design that permits modularisation (Judd, 2011; Judd, 2006).

Membrane systems take advantage of the inherent properties of high selectivity, high surface area per unit volume of reactor space (i.e. high packing density) and their potential for controlling the level of contact and/or mixing between two phases (Charcosset, 2006). The configuration of the membrane is important in determining the overall performance of the process. The principal membrane configurations based on either a planar or cylindrical geometry are pleated filter cartridge (FC), flat sheet (FS), hollow fibre (HF), multi-tubular (MT), capillary tube (CT), as well as spiral wound (SW) (Judd, 2006; Judd and Jefferson, 2003). Hollow fibre applies when the internal diameter is less than 0.5 mm and capillary applies when the internal diameter is larger than 1.0 mm (Pearce, 2007a).

However, for MBR technologies only FS, HF and MT are suitable since these configurations promote a high degree of turbulence and are easy to clean. MT modules operate with the flow passing from inside to outside (i.e. the feed enters the lumen during filtration (Pearce, 2007c); while HF modules operate with the flow passing from outside to inside (i.e. the feed is fed to the outside of the fibre during filtration (Pearce, 2007c; Judd, 2006; Judd, 2011). The main UF and MF products available for wastewater treatment are dominated by hollow fibre and capillary modules (Pearce, 2007c), while NF and RO are dominated by spiral wound elements.

### 2.4.3 Membrane system operation modes

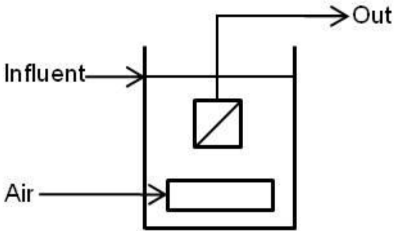
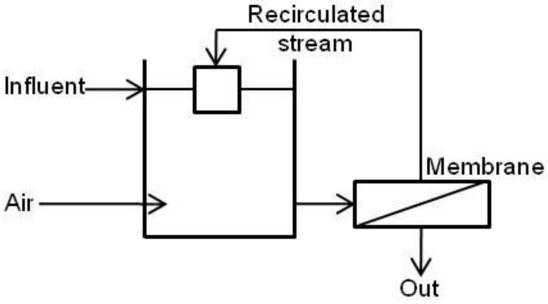
Crossflow and dead-end operation are two standard modes of operation in conventional pressure driven membrane processes (Judd, 2006; Judd, 2011). In crossflow the fluid being filtered flows parallel to the membrane surface and permeates through the membrane due to the existence of a pressure difference across the membrane (Charcosset, 2006). Crossflow operation is widely utilised in membrane technologies (Melin *et al.,* 2006). During crossflow operation, only a fraction of the feed water passing across the membrane is converted to permeate (Judd, 2006). During pressure driven membrane processes a permeate stream with a volume of 10% to 20% of the feed stream is produced (Van Der Bruggen *et al.,* 2003) when operated in crossflow operation. No retentate stream is found in dead-end operation (Judd, 2006), therefore this mode of operation is often applied in MF as the feed is forced through the membrane. In industrial applications, however, crossflow operation is preferred over dead-end operation as less fouling occurs (Judd, 2006). During dead-end operation the resistance to flux during filtration increases with an increase in the thickness of the cake formed on the surface of the membrane. With crossflow operations flux decline is lower and there is a variety of different crossflow modes such as co-current, counter-current and crossflow with perfect permeate mixing (Stanojević *et al.,* 2003). Disadvantages of crossflow operation in large scale applications is the size and cost of the pump required to create sufficient velocity and the pumping energy resulting from the high feed channel pressure drop. Direct-flow or semi-dead operation was developed as an alternative to crossflow as direct-flow relies on an intermittent backwash and is the operation mode of choice for wastewater treatment applications (Pearce, 2007b).

In a pressure driven module, the membranes are encapsulated within a shell which has a pressure rating allowing the module to be operated by a feed pump against a back pressure if necessary. Pressure driven systems are normally best suited for small to medium scale wastewater treatment applications with low amounts of suspended solids in the feed, or a large scale polishing application. In a submerged system (Table 2.3) the membranes are unencapsulated with the membranes immersed in an open tank, but connected to an off-take system, allowing a vacuum to be applied to the permeate side to draw the filtrate through the membrane. Submerged systems are best suited for large scale wastewater treatment applications with medium to high amounts of suspended solids in the feed (Pearce, 2007c).

### 2.4.4 Membrane bioreactor configurations and formats

MBR configuration refers to the MBR process, specifically how the membrane is integrated within the bioreactor, as well as the membrane module. The primary MBR process configurations are shown in Table 2.3: 1) the immersed MBR (iMBR), submerged or integrated (Hai *et al.,* 2011; Konsowa *et al.,* 2011; Tian *et al.,* 2010; Guo *et al.,* 2008; Wang *et al.,* 2008; Bernard, 2006; Charcosset, 2006; Judd, 2006; Hai *et al.,* 2006; McAdam & Judd, 2006; Meng *et al.,* 2006; Yang *et al.,*2006); and 2) the sidestream MBR (sMBR), recirculated or external (Konsowa *et al.,* 2011; Wang *et al.,* 2008; Bernard, 2006; McAdam & Judd, 2006), where the bioreactor and membrane modules are separate from each other. The different MBR formats are shown in Table 2.4: 1) extractive MBR (eMBR) (Wang *et al.,* 2008; McAdam & Judd, 2006; Yang *et al.,* 2006); 2) diffusive MBR (dMBR) (Yang *et al.,* 2006); 3) ion exchange MBR (IEMBR) (Table 2.3) (Wang *et al.,* 2008; McAdam & Judd, 2006; Yang *et al.*, 2006); 4) biofilm membrane bioreactor (BF-MBR) (Ivanovic & Leiknes, 2008; Leiknes & ∅degaard, 2007) which combines a biofilm reactor with the process of membrane separation of suspended solids

**Table 2.3:** Different MBR configurations

| **Configuration** | **Diagram** | **Reference** |
|---|---|---|
| Immersed/Submerged MBR (iMBR) |  | Hai *et al.,* 2011; Tian *et al.,* 2010; Bernard, 2006; Charcosset, 2006; Hai *et al.,* 2006; McAdam & Judd, 2006; Meng *et al.,* 2006; Yang *et al.,*2006 |
| Sidestream MBR (sMBR) |  | Bernard, 2006; McAdam & Judd, 2006 |

(Leiknes & ∅degaard, 2007); 5) biomass separation MBRs (BSMBR) (Wang *et al.,* 2008); 6) membrane aeration bioreactor (MAB) (Wang *et al.,* 2008; McAdam & Judd, 2006; Yang *et al.,* 2006); 7) floating media biofilter-crossflow microfiltration system (FMB-CFMF) (Guo *et al.,* 2008); 8) submerged membrane adsorption bioreactor (SMABR) (Guo *et al.,* 2008); and 9) a sponge-iMBR system (Guo *et al.,* 2008). MBR configurations can be operated in two hydraulic modes, either pumped or airlift; with one of three membrane configurations, either FS, HF or MT, utilised in commercial MBR technology (Judd, 2006). The MBR configuration applications for wastewater treatment and more specifically the textile wastewater treatment industry will be discussed later under section 2.4.5.2.

#### 2.4.4.1 Immersed and sidestream MBRs

Immersed MBRs (iMBRs) and sidestream MBRs (sMBRs) (Table 2.3) are conventional biomass rejection MBRs, and the driving force of this process is TMP. iMBRs are preferred to sMBRs or external loop mode due to significantly reduced energy consumption (Guo *et al.,* 2008; Rosenberger *et al.,* 2008). Membrane modules in a pumped sidestream crossflow utilises more energy due to the high pressures and volumetric flows (Judd, 2006). In iMBRs biocatalysts such as enzymes, micro-organisms and antibodies are immobilised on a membrane suspended in solution and compartmentalised in a reaction vessel. The separation of solids and liquid occurs inside the reactor. The influent is separated from the biomass to produce a clean, solid free permeate (Bernard, 2006; Charcosset, 2006). For sMBRs the bioreactor and membrane modules are separate from each other with separation occurring outside the reactor with a recirculation stream. Advantages of sMBRs include energy requirements that are almost equivalent to iMBRs, sludge retention, independent operation and optimisation of the biological and membrane systems, as well as easy chemical cleaning of the membranes (Judd, 2006). A modified sMBR known as an airlift external circulation MBR (AEC-MBR) has the advantages of both the sidestream and immersed MBRs. In the AEC-MBR the recirculation pump has been replaced by an H-type recycling pipe (Wang *et al.,* 2008).

Hai *et al.* (2006) developed a submerged MF- MBR implementing the white-rot fungus (WRF) *Coriolus versicolor.* WRF are capable of degrading a variety of recalcitrant organic pollutants, including various types of dyes, through the secretion of extracellular non-specific oxidative enzymes as secondary metabolites in response to carbon or nitrogen limitations. In submerged MBRs the membranes are housed within a casing (Hai *et al.,* 2006). MF is responsible for the significant volume reduction, as well as the separation and recycling of synthetic sizing agents and some types of intermediate chemicals and dyestuffs (ElDefrawy & Shaalan, 2007).

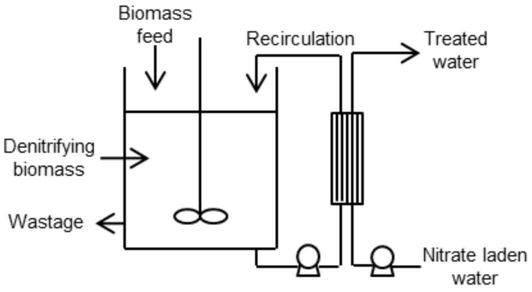
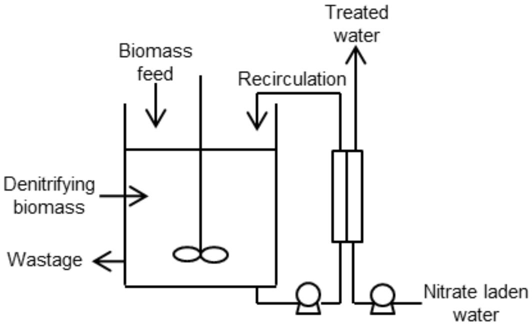
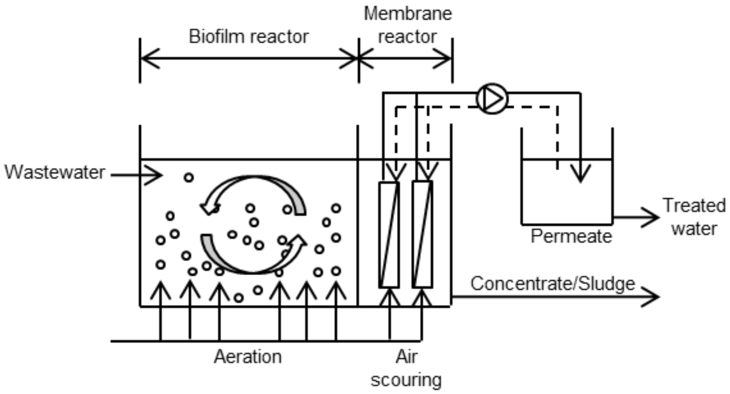
#### 2.4.4.2 Extractive and diffusive MBRs

Extractive MBR (eMBR) and diffusive MBR (dMBR) (Table 2.4) membrane process modes utilise membranes for a purpose other than separating biomass. The driving force for eMBRs and dMBRs are concentration and partial pressure gradients, respectively. In eMBRs, specific contaminants are extracted across a membrane of appropriate selectivity. On the permeate side of the membrane the contaminant is subjected to biotreatment, normally by the biofilm growing on the surface of the membrane. In dMBRs a gas permeable membrane is ultilised to introduce gas in the molecular form into the bioreactor, which reaches the biofilm growing on the membrane surface via the permeate. eMBRs are utilised in the treatment of toxic organic wastewaters, surface and drinking water (Wang *et al.,* 2008: Yang *et al.,* 2006). Diffusive systems are commonly referred to as membrane aeration bioreactors (MABs) due to the transfer of oxygen across a microporous surface, like a hollow fibre membrane. Often a biofilm will form on the surface of the membrane, protecting it from abrasion and grazing (Pankhania *et al.,* 1999). The major disadvantage of both eMBR and dMBR configurations is that neither process provides a barrier between the treated and untreated streams (Judd, 2006).

#### 2.4.4.3 Sequential anaerobic and aerobic MBRs

Due to simplicity and low cost, traditional aerobic/anaerobic activated sludge or aerobic biofilm processes are utilised worldwide for biodegradation. Disadvantages of this process include the following: 1) low biodegradability under aerobic conditions which limits the application of the aerobic biological process for the treatment of textile wastewater; and 2) longer hydraulic retention time or the requirement of a larger aerobic tank. Aromatic amines, metabolites, are more toxic than the dyes themselves; therefore a two-stage biological process is required for the treatment of textile wastewater, since the water contains reactive dyes. The two-stage biological process should consist of anaerobic and aerobic reactors in sequence, allowing the mineralisation of the xenobiotic azo-compounds (You & Teng, 2009; Żyłła *et al.,* 2006).

**Table 2.4:** Different MBR formats

| Format | Diagram | Reference |
|---|---|---|
| Extractive membrane bioreactor (eMBR) | <br> | Wang *et al.,* 2008; McAdam & Judd, 2006; Yang *et al.,* 2006 |
| Ion exchange membrane bioreactor (IEMBR) | <br> | Wang *et al.,* 2008; McAdam & Judd, 2006; Yang *et al.,et al.*, 2006 |
| Biofilm bioreactor (BF-MBR) | <br> | Ivanovic & Leiknes, 2008; Leiknes & ∅degaard, 2007 |

### 2.4.5 Applications of membrane bioreactors

The utilisation of membrane technology in the treatment of wastewater may be more appropriate and superior to existing treatment processes. However, there are situations where membrane technology can only be used to assist existing treatment processes (Mallia & Till, 2001).

In the past it was difficult to convince decision-makers that MBRs were an attractive reliable and relatively cost effective option. Today, however, it is easier to convince these decision-makers of the fact since there are a number of examples (Sheldon *et al.,* 2012; Judd, 2011; Monclús *et al.,* 2010; Dialynas & Diamadopoulos, 2009; Henkel *et al.,* 2009; Yigit *et al.,* 2009; Lesjean & Huisjes, 2008; Melin *et al.,* 2006; You *et al.,* 2006) where MBRs have been successfully implemented in wastewater treatment across a range of applications, including both municipal and industrial wastewater treatment (Judd, 2006). MBR technology has demonstrated sustained performance over several years with a reliable product quality, providing a clear cost benefit (Judd, 2006). The main drawback of MBR technology is the high cost associated with MBR systems, while membrane costs have decreased over the years since the commercialisation of immersed MBRs in 1990 (Judd, 2011; Kraume & Drews, 2010) leading to a decrease in capital costs, the energy demand associated with preventing and removing membrane fouling has become the main contributor to the overall operating costs (Kraume & Drews, 2010). According to Helble and Möbius (2009) the total operation costs for a submerged MBR with aerated tubular crossflow membranes amounted to € 0.22/m$^3$ treated water, while the cost for a MBR with classic tubular crossflow membranes amounted to € 0.33/m$^3$ due to the higher energy requirements (Helble and Möbius, 2009). The costs of installing and operating MBRs, with regard to a whole life basis, are comparable to conventional wastewater treatment plants provided the membranes have a long lifespan (i.e. ≥ 8 years) and have the added advantage of producing high quality effluent (Judd, 2011).

#### 2.4.5.1 MBR technology in wastewater treatment

MBRs in wastewater treatment are based upon the robust activated sludge process, where the membrane is used to separate the product stream from the mixed liquor effluent. MBRs are favoured when treating high-strength wastewater since the membrane area is determined on the hydraulic throughput and not the biological load. Therefore, when the strength of the wastewater increases the membrane area remains constant. In conventional wastewater treatment plants, as the strength of the wastewater increases, so does the required aeration volume and power (Hunter, 2007).

In MBR technology, the membrane replaces the sedimentation tank found in the activated sludge process. Therefore, when no sludge is wasted, all the micro-organisms are retained within the reactor including specific bacteria capable of degrading the low degradable textile wastewater (You *et al.,* 2008; You *et al*., 2006). An advantage of an MBR system to the conventional treatment methods is that the MBR system has low to zero sludge production. The reason for this is the long sludge age due to elevated sludge concentrations (Brik *et al*., 2006). High sludge ages cause the sludge yield to decrease due to emphasised endogenous metabolism (Brik *et al*., 2006). The retention of high biomass concentration within an MBR, which requires a small footprint, allows the process to be operated at a low food/micro-organism (F/M) ratio resulting in reduced excess sludge production (Hai *et al.,* 2006).

MBRs have been utilised in the treatment of various types of wastewater with COD concentrations and HRT ranging from 100 to more than 40,000 mg/L and 4 h to several days, respectively (Konsowa *et al.,* 2011). The use of MBRs technology in wastewater treatment was initially designed to achieve high organic matter removal (i.e. COD). However, this technology also demonstrated efficient nitrogen removal with high sludge retention times (SRT) and the addition of an anoxic zone. In systems with long SRT, biological phosphorus removal is limited due to the low net biomass growth, thereby limiting the incorporation of phosphate into new cell material (Monclús *et al*., 2010). The permeate stream from MBRs are normally free of bacteria (Jeffrey *et al.,* 1998; Dijk *et al.,* 1997), since most of the micro-organisms are retained by the membranes within the bioreactor. The HRT, then, becomes completely independent of the SRT (Muller & Stouthamber, 1995) and re-usable water can be recovered from the MBR permeate stream, thereby assisting industries producing wastewater in reducing water consumption and decreasing the amount of wastewater discharged (Konsowa *et al.,* 2011; Kim *et al.,* 2004b; Jiraratanaon *et al.,* 2000; Dhale & Mahajani, 1999). In MBRs contact time between activated sludge and non-biodegradable pollutants present in wastewater is elongated, with increased contact time increasing the degrading efficiency of the bioreactor and providing an optimum environment for the treatment of textile wastewater, characterised by strong colour and low $BOD_5$/COD ratio (Konsowa *et al.,* 2011).

An important feature of MBRs is the possibility of facilitating the growth of specialised micro-organisms due to the ability to employ high sludge ages, in this way promoting improved degradation of refractory organics (Brik *et al.,* 2006; Stephenson *et al.,* 2000). The advantage of a membrane reactor, as well as a flow reactor with recycle used to treat industrial wastewater and slurries, is to allow operation at lower residence times, increasing the throughput of wastewater (Nelson *et al*., 2008). The recycle in the continuous flow bioreactor allows the retention of a higher concentration of micro-organisms within the bioreactor, resulting in the reactor operating at greater flow rates and increasing its efficiency (Nelson *et al*., 2008).

#### 2.4.5.2 Various configurations and operation of MBRs in textile wastewater treatment

You and Teng (2009) utilised an anaerobic SBR (see Table 2.5) combined with an aerobic MBR (AMBR) in the treatment of azo dyes. *Lactobacillus casei* and *Lactobacillus lactis* were the anaerobic media and high colour degrading bacteria utilised in the anaerobic SBR. In the anaerobic activated sludge unit during the first stage, the azo bonds of the reactive dye were degraded, resulting in reduced colour and the production of toxic colourless aromatic amines. By adding suitable amounts of carbon and nitrogen the colour degrading performance of *L. casei* was improved (You & Teng, 2009). The aerobic activated sludge unit during the second stage further degraded/mineralised the aromatic amines (You & Teng, 2009). Table 2.6 indicates that the COD and colour removal efficiencies, 5.2% and 9.1%, respectively, of the aerobic MBR were not high. However, this stage is required in order to mineralise the amines into non-toxic metabolites and retain the particulate matter in the bioreactor.

You *et al.* (2008) found that the anaerobic tank of the anaerobic-oxic MBR (AOMBR) and AOMBR/RO (see Table 2.5) lowered the COD concentration. The membrane unit showed excellent performance with respect to the BOD concentration of the wastewater, while the anaerobic tank as well as the RO unit achieved high colour removal. The results in Table 2.5 indicate that all the processes with a membrane unit showed excellent efficiencies on SS and BOD removal, the presence of an anaerobic tank enhanced COD and true colour removal, and the RO unit further improved true colour removal (You *et al.,* 2008).

You *et al*. (2006) compared the performance of the MBR and SBR for the treatment of textile wastewater. Table 2.6 indicates 100% SS removal efficiency with the MBR, which, when compared to the 60% SS removal efficiency of the SBR, is excellent. The results in Table 2.6 reveal that the MBR shows higher performance in the treatment of textile wastewater than the SBR (You *et al.,* 2006).

**Table 2.5:** Different MBR formats utilised within the textile wastewater treatment industries

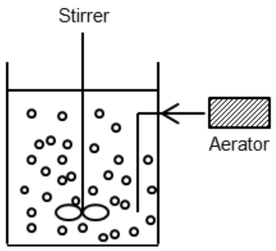
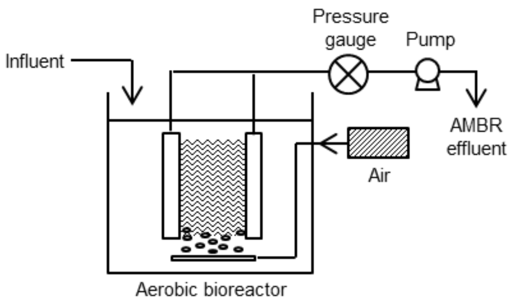
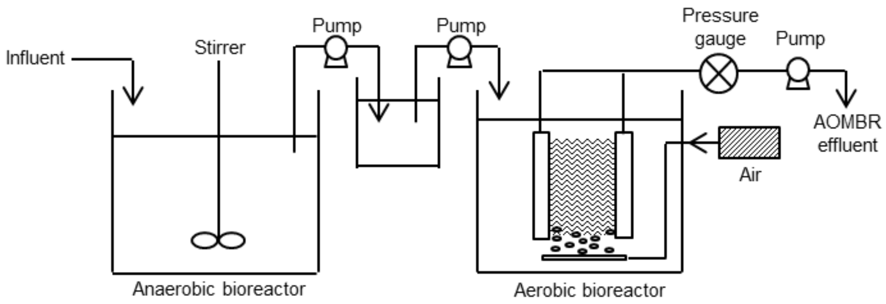
| Configuration | Diagram | Applications | Reference |
|---|---|---|---|
| Sequencing batch reactor (SBR) |  | Removal of colour, COD, BOD and TSS. | You *et al.,* 2006 |
| Aerobic MBR (AMBR) |  | Removal of colour, COD, BOD and TSS. | You *et al.,* 2006 |
| Anaerobic-oxic MBR (AOMBR) |  | Reduction of azo dyes and colour removal. | You & Teng, 2009; You *et al.,* 2006 |

**Table 2.5 (continued):** Different MBR formats utilised within the textile wastewater treatment industries

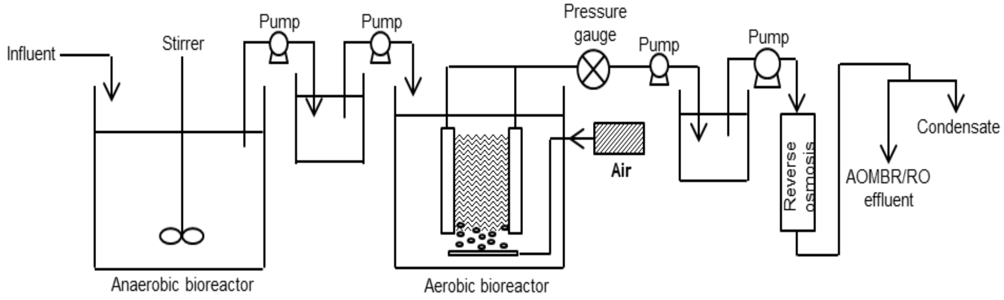
| **Configuration** | **Diagram** | **Applications** | **Reference** |
|---|---|---|---|
| Anaerobic-oxic MBR/Reverse osmosis (AOMBR/RO) |  | Removal of colour, COD, BOD and TSS. | You *et al.,* 2006 |

**Table 2.6:** Summary of COD, BOD, TOC, colour removal, true colour and SS removal capabilities of the various MBR set-ups

| **Set-up** | **COD concentration (mg/L)** | **COD removal** | **True colour (ADMI)** | **Colour removal** | **Reference** |
|---|---|---|---|---|---|
| Aerobic MBR | 1,280-5,600 (influent)<br>240 (effluent) | 96% | ni | 72% | Badani *et al*., 2005 |
| Aerobic MBR (activated sludge reactor connected to an external crossflow UF unit) | 1,380-6,033 (influent)<br>130-900 (effluent) | 60% - 95% | ni | 30% - 99.5% | Brik *et al.,* 2006 |
| Submerged microfiltration MBR | ni | ni | ni | 68.3% | Hai *et al.,* 2006 |
| Aerobic MBR (external UF module with plate and frame membranes) | 500-1,700 (influent)<br>40-60 | 93% (average) | ni | 70% - 80% | Lubello & Gori, 2004 |
| MBR (activated sludge reactor connected to an external crossflow UF unit) | 1,380-6,035 (influent)<br>225-350 (effluent) | 91.8%(maximum removal) | ni | ni | Schoeberl *et al.,* 2004 |
| MBR (submerged hollow fibre membrane module in an aeration tank) | 1,411 (influent) | ni | ni | ni | Yigit *et al.,* 2009 |
| Anaerobic SBR<br>Aerobic MBR | ni | 92.3%<br>5.2% | 264.2 ± 49.9<br>169.0 ± 22.4 | 74.6%<br>9.1% | You &Teng, 2009 |

**Table 2.6 (continued):** Summary of COD, BOD, TOC, colour removal, true colour and SS removal capabilities of the various MBR set-ups

| Set-up | COD concentration (mg/L) | COD removal | True colour (ADMI) | Colour removal | Reference |
|---|---|---|---|---|---|
| SBR | 133 (effluent) | ni | 548 | ni | You *et al.* 2008 |
| Aerobic MBR | 95 (effluent) | | 513 | | |
| AOMBR | 37 (effluent) | | 196 | | |
| AOMBR/RO | 38 (effluent) | | 32 | | |
| SBR | 133 (effluent) | 70% | 548 (effluent) | 51% | You *et al.,* 2006 |
| MBR | 95 (effluent) | 79% | 513 (effluent) | 54% | |
| MBR (with a gravity drain) | 128 – 321 (influent) | 80.3% (average) | 21 (dilution times) | 58.7% (average) | Zheng & Liu, 2006 |

ni – not indicated

### 2.4.6 Chemical oxygen demand (COD) removal using MBRs

Generally, textile wastewater is trademarked by its high COD, 3,500 to 37,000 mg/L (Judd, 2006), a measure of the oxygen required to oxidise both organic and inorganic compounds in wastewater. Large variations in the influent of MBRs treating textile wastewater is a common occurrence (Yigit *et al*., 2009; Zheng & Liu, 2006; Schoeberl *et al*., 2004; Badani *et al*., 2005), mainly due to the variations in the production programs of the textile industry and the break offs for cleaning and maintenance during weekends (Yigit *et al*., 2006).

Although there were large COD variations in the MBR influent, Brik *et al.* (2006) achieved a COD removal of greater than 90%; Badani *et al.* (2005) achieved an average COD removal of 96%; and Lubello and Gori (2004) achieved an average COD removal of 93%, (refer to Table 2.6 for the various MBR set-ups utilised).

When the influent COD values to the pilot-scale submerged MBR were at its maximum level (2,278 mg/L), Yigit *et al.* (2009) observed a COD removal that always exceeded 95% when treating industrial textile wastewater. Zheng and Liu (2006), utilising laboratory scale MBR with a influent COD value ranging from 128 to 321 mg/L and Schoeberl *et al.* (2004) utilising a crossflow UF-MBR system with a COD value of 1,380 to 6,035 mg/L present in the influent, both observed an average COD removal exceeding 80%. This indicates that the MBR systems responded very well to variations in the influent and are also able to buffer the changing influent composition. Therefore, it can be stated that MBR systems are very stable and that the outlet values are substantially independent from the inlet loads. Yigit *et al.* (2009) had a $BOD_5$/COD ratio of 0.32, suggesting the dominance of slowly biodegradable and/or biorecalcitrant organics in the textile wastewater. The stable and successful performance of Yigit *et al.* (2009) MBRs biological activity despite the low $BOD_5$/COD ratio, demonstrated the presence of a robust and specialised biomass mixture in the MBR that can respond to the sudden variations within the influent and degrade synthetic chemicals such as dyes. Another one of the key features underlined by this constant COD removal is that within the MBR, the insoluble components are rejected until they are susceptible to biodegradation or drawn out with surplus sludge.

### 2.4.7 Colour removal using membrane systems

Textile wastewater is characterised by being high in colour, mainly due to the dyes that are present in the water originating from the different printing and dyeing processes (You & Teng, 2009). The removal of colour from highly coloured textile wastewaters is both difficult and expensive (Gaydon & De Haas, 1998). The following methods have been suggested for colour removal: coagulation, activated carbon, sodium hypochlorite, ozone and electrochemical oxidation (Gaydon & De Haas, 1998). Adsorption, membrane separation and advanced oxidation processes are several physiochemical decolouration techniques used in the treatment of textile wastewaters. However, due to their high cost, low efficiency and limited versatility biodegradation is an environmentally friendly cost effective alternative (Hai *et al*., 2011). Removal of colour from textile wastewater is not a primary function of a MBR system; however, the MBR system does assist in removing some colour and in some cases very high colour removal was noted (Yigit *et al*., 2009). The colour removal function does vary considerably with time, though (Brik *et al*., 2006). The mechanisms for colour removal in an MBR include biodegradation and absorption onto biomass solids (Brik *et al*., 2006; Yigit *et al*., 2009). Brik *et al.* (2006) noted that an increasing sludge growth rate yielded higher colour removal efficiencies, because there was more biomass generated to adsorb the incoming colour.

Unsatisfactory colour reduction was noted by Schoeberl *et al.* (2004) using a sidestream MBR set-up, while emulsified lubricants were completely retained by the external polyacrylonitrile (PAN) tubular crossflow UF-membrane. Residual colour from the dyeing process was only partially eliminated. Removal efficiencies significantly dropped when the tested wastewater originated primarily from the dyeing process (Schoeberl *et al*. 2004). It is important to note that in the case of Schoeberl *et al.* (2004) that the system composed of an aerobic activated sludge tank connected to an external tubular crossflow UF unit and that the textile wastewater treated was from a polyester finishing industry. The effluent from the MBR process was treated with a spiral wound NF module in a post-treatment polishing step. However, Badani *et al.* (2005) operated an aerobic MBR system that treated mixed textile wastewater and obtained colour removal efficiencies averaging at 72%. The membrane used by Badani *et al.* (2005) was a tubular UF polyvinylidene fluoride (PVDF) module. Yigit *et al.* (2009) and Brik *et al.* (2006) both operated an aerobic MBR system and obtained colour removal efficiencies of > 97% and 80% respectively. Yigit *et al.* (2009) treated wastewater from a denim-producing textile industry while Brik *et al.* (2006) treated wastewater from polyester finishing factory. In the case of Schoeberl *et al.* (2009), the textile factory added a number of different substances to enhance the durability of the fabric produced, which, in conjunction with the short solid retention time, could explain the poor colour removal of the system. Brik *et al.* (2006) had the same type of wastewater, but a longer solid retention time was implemented. It can also be assumed that no substances were added to improve the robustness of the fabric because the end product was used for a different purpose.

If colour removal is a priority, NF and RO membranes should be utilised in the MBR system. Kim *et al.* (2004a) performed a study on the colour removal efficiencies of reactive dyes with a NF and RO combined MBR system. The results showed a > 90% removal of reactive black 5 and reactive blue 49 dyes and a 76% removal of the reactive blue 19 dye.

Konsowa *et al.* (2011) investigated the use of a laboratory scale aerated hollow fibre MBR for the treatment of wastewater polluted with the azo dye acid orange 7. The MBR process consisted of an activated sludge bioreactor and a submerged microfiltration hollow fibre membrane. In general, the longer the HRT, the more efficient the dye removal process (Zheng & Liu, 2006; Visvanathan *et al.* 2000). The biological process removed 60% - 80% of the COD and 94% of the acid orange 7 dye when present in low concentrations.

Baêta *et al.* (2012) investigated the use of a submerged anaerobic MBR (SAMBR) both with and without powdered activated carbon (PAC) for the treatment of industrial textile wastewater. The SAMBR containing PAC had removal efficiencies of 90% and 94%, while the SAMBR without PAC showed removal efficiencies of 79% and 86%, for COD and colour, respectively. Therefore, the SAMBR containing PAC produced an anaerobic effluent of high quality with regards to these two parameters.

A bioaugmented MBR with a granular activated carbon (GAC)-packed anaerobic zone was investigated for the treatment of textile wastewater containing structurally different azo dyes. In order to optimise the use of the developed MBR, a unique feeding strategy was employed with dye containing wastewater fed through the GAC-packed anaerobic zone and colourless wastewater fed through the aerobic zone, thereby keeping the waste streams separate as they emanated from the textile industry. The membrane module used was a microporous (0.4 µm), hydrophilically treated, polyethylene hollow fibre compact bundle. Four structurally different dyes - namely Acid orange II, Ploy S 119, Direct brilliant yellow and Reactive orange 16 - were fed in equal loading rates (i.e. 0.25 g/L.d) resulting in a total dye loading of 1 g/L.d. The average dye concentration after treatment in the anaerobic GAC-packed zone was 105 mg/L and 5 mg/L in the MBR permeate. TOC was 54 mg/L in the MBR permeate after treatment. Decolouration occurred in the GAC-packed anaerobic zone, while TOC removal occurred in the aerobic zone. When compared to conventional sequential anaerobic-aerobic processes where the aerobic stage has a limited role in decolouration, the aerobic zone in the developed MBR contributed significantly to decolouration at the higher dye loading rates (Hai *et al.,* 2011). Table 2.7 summarises various literature articles with regards to colour removal from industrial wastewater using various MBR configurations.

**Table 2.7:** Literature summary of case studies in the treatment of industrial wastewater

| **MBR configuration** | **Synthetic or industrial (type) wastewater** | **Average colour removal** | **Lab scale or pilot-scale** | **Plant size** | **Country** | **References** |
|---|---|---|---|---|---|---|
| Tangential flow sidestream MBR preceded by an equalisation tank (2-stage process) | Industrial mixed textile wastewater | 70%<br>(97.5% after post treatment with NF) | Lab and pilot-scale | 300 – 500 L (bioreactor) | Austria | Badani *et al.,* 2005 |
| Submerged anaerobic MBR (SAMBR) with and without powdered activated carbon (PAC) | Industrial textile wastewater | 94%<br>(SAMBR+PAC)<br>86%<br>(SAMBR-PAC) | Lab-scale | ni | Brazil | Baêta, et al., 2012 |
| Sidestream MBR consisting of an activated sludge reactor connected to an external tubular crossflow ultrafiltration (UF) unit (2-stage process) | Industrial textile wastewater from a polyester finishing mill | > 87% | Lab-scale | 20 L (aerobic reactor)<br>0.28m$^2$ (tubular membrane filter area) | Austria | Brik *et al.,* 2006 |
| Submerged MBR connected to a RO unit (2-stage process) | Industrial municipal wastewater | N/A | Pilot-scale | 80 000 p.e<br>2.3 L/min (maximum flow rate) through the UF-membrane | Greece | Dialynas & Diamadopoulos, 2009 |
| Submerged microfiltration MBR (1-stage process) | Synthetic textile wastewater | 68.3% | Lab scale | 12.5 L (bioreactor) | Japan | Hai *et al.*, 2006 |
| Submerged MBR (1-stage process) | Synthetic greywater | N/A | Lab and pilot-scale | 2600 L (bioreactor)<br>20m$^2$ (hollow fibre membrane module) | Germany | Henkel *et al.,* 2009 |

**Table 2.7 (continued):** Literature summary of case studies in the treatment of industrial wastewater

| MBR configuration | Synthetic or industrial (type) wastewater | Average colour removal | Lab scale or pilot-scale | Plant size | Country | References |
|---|---|---|---|---|---|---|
| Microfilter (MF) followed by 1) NF and 2) RO (2-stage process) | Synthetic textile wastewater (3 reactive dyes were investigated) | 97% | Lab scale | 2.5 L(bioreactor) | Republic of Korea | Kim *at el.,* 2004a |
| Submerged MBR (1-stage process) | Industrial greywater | N/A | Lab scale | 3 L (bioreactor)<br>0.04m$^2$ (hollow fibre UF-membrane module) | Morocco | Merz *et al.,* 2007 |
| Anaerobic, anoxic, aerobic reactor compartments followed by a submerged hollow fibre MBR and RO (5-stage process) | Industrial municipal wastewater | N/A | Pilot-scale | 316.4 L (anaerobic and anoxic tank volume)<br>519.8L (aerobic tank volume) | Spain | Monclús *et al*., 2010 |
| Sidestream membrane chemical reactor (1-stage process) | Synthetic greywater | N/A | Lab scale | ni | France | Pidou *et al.*, 2009 |
| Sidestream MBR consisting of an activated sludge reactor connected to an external tubular crossflow UF unit, followed by NF (3-stage process) | Industrial mixed textile wastewater from a polyester finishing plant | 80% after post treatment with NF | Lab scale | 20 L (aerobic reactor)<br>0.28 m$^2$ (tubular membrane filter area) | Austria | Schoeberl *et al*., 2004 |
| Submerged hollow fibre membrane module in a bioreactor (1-stage process) | Industrial mixed textile wastewater from a denim producing industry | > 97% | Pilot-scale | 230 L (bioreactor) | Turkey | Yigit *et al.,* 2009 |

**Table 2.7 (continued):** Literature summary of case studies in the treatment of industrial wastewater

| MBR configuration | Synthetic or industrial (type) wastewater | Average colour removal | Lab scale or pilot-scale | Plant size | Country | References |
|---|---|---|---|---|---|---|
| Anaerobic bioreactor followed by an aerobic membrane bioreactor (2-stage process) | Synthetic textile wastewater containing a reactive dye (Reactive Black 5) | 83.7% | Lab scale | 36 L (anaerobic bioreactor)<br>18 L (aerobic MBR bioreactor) | Taiwan | You & Teng, 2009 |
| Submerged aerobic MBR (1-stage process) | Industrial textile wastewater | 54% | Pilot-scale | 25 L (aerobic MBR bioreactor)<br>0.2 $m^2$ (hollow fibre membrane surface area) | Taiwan | You *et al.*, 2006 |
| Aerobic submerged MBR (1-stage process)<br>Anaerobic bioreactor followed by an aerobic submerged MBR (2-stage process)<br>Anaerobic bioreactor, aerobic submerged MBR followed by RO (3-stage process) | Synthetic textile wastewater containing Reactive Black 5 | 81% without post treatment with RO<br>97% after post treatment with RO | Lab scale | 25 L (anaerobic bioreactor)<br>25 L (aerobic bioreactor)<br>0.2 $m^2$ (hollow fibre membrane surface area) | Taiwan | You *et al.*, 2008 |

## 2.5 South African (SA) perspective on MBRs

In SA in the early 1990's an anaerobic MBR, the anaerobic digestor UF (ADUF) process based on a sidestream MBR configuration, in which biomass concentration was increased using tubular polyether-sulphone membrane, was commercialised by Weir Envig (Botha *et al.,* 1992) for the treatment of high-strength (i.e. 3,500 to 37,000 mg/L COD) industrial wastewater, such as maize processing wastewater (Judd, 2011). The ADUF process consisted of a 3 m long, 12 mm diameter MT polyethersulfone (PES) UF module with 0.1 µm pore size achieved > 90% removal depending on the biodegradability of the organic matter. The quality of the permeate produced is of a consistently high quality with regard to particulate material, bacteria and viruses (Judd, 2006).

The main drivers for the implementation of a large number of pilot-scale MBR trials in SA is the looming water shortage, which could see the Western Cape run out of water by 2016 (Gosling, 2010:4), resulting in the critical necessity to re-use water, the need to meet the waste discharge charge system (WDCS) standards to be implemented by the Department of Water and Environmental Affairs (DWEA) (South Africa, 2003), footprint limitations and the need to minimise waste. Currently, the MBR market in SA is in the initial stages. However, a few small MBR plants do exist (Judd, 2011) only two MBR plants exceed a capacity of 1 ML/d. The longest running MBR plant at the Illovo sugar plant at Sezela, commissioned in 2005, has a capacity of 1.2 ML/d and utilises flat plate membranes. The largest MBR plant in SA, with a capacity of 18 ML/d, was commissioned in early 2009. This plant, located at Zandvliet in the Western Cape, treats municipal wastewater to supplement the capacity of the conventional wastewater treatment works (Judd, 2011).

A number of municipal MBR wastewater treatment plants are planned in SA, namely 1) a 20 ML/d plant in Malmesbury, in the Western Cape, for eventual re-use of the water; 2) a 40 ML/d plant in Bellville, in the Western Cape, for water re-use; and 3) a 100 ML/d plant for water re-use in the Coega Industrial Zone in Port Elizabeth in the Eastern Cape. Both the planned Malmesbury and Bellville wastewater treatment plants were sent out to tender in July 2010, while the 100 ML/d plant planned for the Coega Industrial Zone was sent out to tender in 2011. In 2011, Umgeni Water, in Kwazulu Natal, conducted pilot-scale MBR trials at the Darville Wastewater Treatment Works (WWTW) to supplement the capacity of the Darville WWTW, trials to evaluate the stability and operability of external air lift Norit-, flat sheet Toray- and hollow fibre Pall-membranes under developing economy conditions (i.e. operational failures, electricity downtime and surges in feed quality) (Judd, 2011). Currently, Veolia Water is designing, building and commissioning the largest MBR in South Africa to date at Bellville WWTW in the Western Cape. The project involves upgrading the existing WWTW to treat an additional 20 ML/d high-strength wastewater, bringing the total treatment capacity to 70 ML/d. The raw municipal wastewater will pass through both coarse and fine screens before undergoing anaerobic, anoxic and aerobic treatment followed by UF and dewatering of the sludge (Bellville wastewater treatment plant, 2013). Veolia Water has also been contracted to design, build and commission a municipal wastewater treatment plant in Mosselbay to upgrade and re-use the treated wastewater. The project aims to meet the region’s short term water requirements and was designed to deliver 5 ML/d, with future expansion of 15 ML/d, reclaimed water to the Wolwedans dam which supplies PetroSA, an industrial client of the municipality. Membrane separation by means of RO and micro-screening UF have been utilised in the design (Mosselbay wastewater treatment works, 2013).

Figure 2.2 represents the drivers and restraints. The longer arrows indicate the factors which have a larger impact, while the arrows with the dotted lines indicate factors that are having a decreasing impact on the European wastewater treatment market (Judd, 2006). Numerous MBR pilot plant trials have been conducted in various industries in SA, including the paper and pulp (Sheldon *et al.,* 2012) and the textile industry. However, to date, none of the pilot plants have resulted in a full-scale MBR wastewater treatment plant. Some of the likely barriers to the implementation of a full-scale MBR wastewater treatment plant in SA is that the drivers for wastewater treatment in SA is limited to meeting the discharge standards only and delay in legislation. Some industries are able to meet these discharge standards by utilising existing chemical treatment processes. However, if these industries had to re-use their treated water they would be forced to implement full-scale MBR systems (Judd, 2011). Currently, the major drivers for the implementation of full-scale MBR wastewater treatment plants in SA are the following: 1) rising municipal costs; 2) water re-use due to the impending water shortage since the United Nations Environment Programme defines SA as a water stressed country currently and projects that it will be a water scarce country by 2020 (Esterhuizen, 2013); 3) waste minimisation; and 4) the need to meet water balances especially when industries discharge their wastewater into environmentally sensitive areas (Judd, 2011). However, the major implementation restraint in SA is delay in legislation. The WDCS (South Africa, 2003) was proposed in 2003; unfortunately however, it has yet to be implemented.

Currently, a research group at the University of the Western Cape is investigating the modification of polymeric membranes using nanostructures to produce low fouling membranes for MBRs. The Department of Chemical Engineering at Durban University of Technology is evaluating woven fabric flat sheet membranes claimed to be more robust than currently available commercial membranes, while the Pollution Research Groups at the University of Kwazulu Natal are investigating the integration of membranes into the

Decentralised Wastewater Treatment Systems (DEWATS) anaerobic baffled reactor (ABR) as a polishing step for possible agricultural use in the future (Judd, 2011).

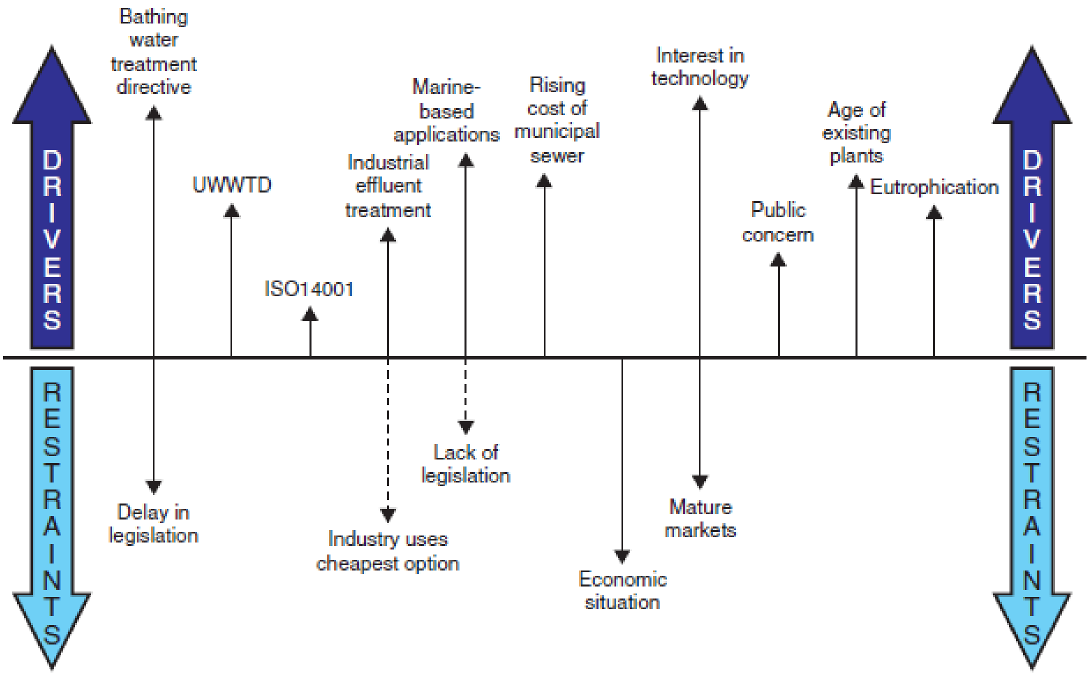


**Figure 2.2:** Factors influencing the MBR market both positively (i.e. drivers) and negatively (i.e. restraints) in Europe (Judd, 2006)

### 2.5.1 SA perspective on textile wastewater treatment works

The quality and availability of water in South Africa has become a challenge, one which will only become more complex in the future. To date, limited information has been published on textile wastewater treatment for re-use in South Africa. The information that is available focuses on conventional wastewater treatment methods such as biological systems and digestors.

During the 1970s the Department of Water Affairs and Forestry (DWAF) constructed the Hammarsdale Wastewater Treatment Works (HWWTW) to service the industrial hub in Hammarsdale, Kwazulu Natal. This industrial area was “rich” in textile industries. However, due to poor environmental planning for the expanding Hammarsdale industrial area, the water quality of the Sterkspruit River was declining and the organic capacity of the HWWTW was at its limit. The high-strength organic industrial wastewater from the textile industries together with the waste arising from the chicken abattoir overloaded the HWWTW resulting in the colouration of the Sterkspruit river (eThekwini, 2007:1-4). The COD was not as bio-degradable as domestic wastewater and the colour and electrical conductivity adversely affected the quality of the wastewater (Fennemore *et al.,* n.d.). The severity of the pollution was increased by the salts from the textile industries entering the river, as the HWWTW was not capable of removing the salts. Additional problems with screening arose and the wire screens utilised to remove excessive materials were producing 25 $m^3$ of waste per week, needing to be disposed of to a low hazard waste disposal site (Fennemore *et al.*, n.d.; eThekwini, 2007:1-4). The environmental impact on the Sterkspruit River included the following: 1) excessive screenings; 2) excessively high organic load; 3) intractable COD; 4) colour; and 5) electrical conductivity (Fennemore *et al.,* n.d.).

In 1982 Umgeni Water took over the HWWTW in an effort to minimise waste and encourage cleaner production. However, Umgeni Water was applying an effluent tariff, not accounting for effluent strength, at a flat rate. Since there was no legal or financial incentive to reduce effluent loads, there was little improvement in the water quality delivered to HWWTW. The factor leading to a reduction in the effluent load was the incorporation of Hammarsdale and the nearby township of Mpumalanga into the eThekwini municipality and the Water Services Act of 1997. eThekwini municipality chose to own and operate HWWTW with by-laws to support the collection of sewerage rates and additional charges for high-strength wastewater. This approach led to the accelerated development of waste minimisation, ensuring that the wastewater from the textile industry was at an acceptable standard. Gelvenor, an ISO 14001 accredited company, was chosen to install the waste minimisation process funded by the European Union and the Water Research Commission (WRC) (eThekwini, 2007:1-4).

The HWWTW, consisting of a modified extended aeration 5-stage Bardenpho process (Gaydon and De Haas, 1998), has a volumetric designed capacity of 27 ML/d with an influent COD of 750 mg $O_2$/L. After screening, the wastewater is processed through anaerobic-, anoxic-, aerobic-, anoxic-, and aerobic-stages. At HWWTW alum, ferric chloride and a polymeric coagulant were investigated for colour removal from the treated textile wastewater (Gaydon & De Haas, 1998). An alum dosing system was installed at the wastewater treatment works in 1989. Alum is added to the wastewater after secondary settling, before discharge into the Sterkspruit River. The sludge is either dewatered in a centrifuge or in sludge drying beds before being disposed of (Fennemore *et al.,* n.d.).

Co-operation between Umgeni Water, eThekwini municipality, the University of Kwazulu Natal in conjunction with DANIDA and the WRC, the Hammarsdale Industrial Conservancy, the Norwegian Government and the National Cleaner Production Centre industries in the Hammarsdale Industrial area set a path to save money, reduce waste loads and reduce the colouration of the Sterkspruit River (Fennemore *et al.,* n.d.).

A laboratory scale ABR was utilised to successfully treat synthetic dye water at the University of Natal in Durban, South Africa. The ABR is a high rate reactor containing 3 to 5 compartments in which the wastewater flows alternately up and down between the compartment partitions. The ABR removed an average of 86% of the colour as well as more than 90% of the COD from the wastewater (Bell & Buckley, 2003)

Carliell *et al.* (1996) investigated the effect of adding concentrated reactive dyebath wastewater, containing azo dyes, daily to an operating anaerobic sewage sludge digestor at the Umbilo Sewage Purification Works (USPW) in Pinetown, Kwazulu-Natal, over a 5-month trial period. The wastewater had an American dye manufacturing index (ADMI) ranging from 3110 to 36600 ADMI units due to the presence of hydrolysed reactive dyes and a pH of between 11.7 and 12.4. The USPW is divided into two sections: 1) the “old” plant which utilises biofilters for primary treatment; and 2) the activated sludge plant, commissioned in 1992. The USPW treats ~19 ML/d, of which approximately 10 ML is treated by the activated sludge plant. A laboratory scale digestor was operated as a control analysis. Unfortunately, due to the presence of residual turbidity, the ADMI could not be determined after treatment. However, visual comparison was made between the supernatant from the sludge from the full-scale digestor and the control digestor. Co-treatment of the exhausted reactive dyebath wastewater, in a ratio of 3:48 kL per day effluent to sludge, showed promising results (Carliell *et al.,* 1996).

The Precipitation and Crystallisation Unit at the University of Cape Town (UCT) has conducted research on using eutectic freeze crystallisation (EFC) to treat hypersaline brines and concentrates from mining and industrial operations, such as the textile industry, in order to recover potable water and pure salt(s) (Randall *et al.,* 2011). EFC operates at a eutectic point with lower energy consumption than evaporative crystallisation. EFC was used to recover 97% pure water, 98% pure calcium sulphate and 96.4% pure sodium sulphate from a multi-component hypersaline RO brine obtained from the eMalahleni Water Reclamation Plant (EWRP) (Randall *et al.,* 2011). Both NF and RO brines recovered after treating textile wastewater are hypersaline, with high conductivity and total dissolved solid concentrations from the large quantity of salts used during the dyeing processes.

## 2.6 Summary

MBR systems using various configurations have been utilised extensively in the rest of the world to treat textile wastewater at both lab and pilot-scale. In the SA textile industry, various pilot-scale MBR trials have been performed but have yet to be implemented at full-scale. In order to facilitate full-scale MBR implementation within SA industries, including the textile industry, re-use of the water treated by the industry *must* become a driver.

# CHAPTER 3

# MATERIALS AND METHODS

# CHAPTER 3
# MATERIALS AND METHODS

## 3.1 Background

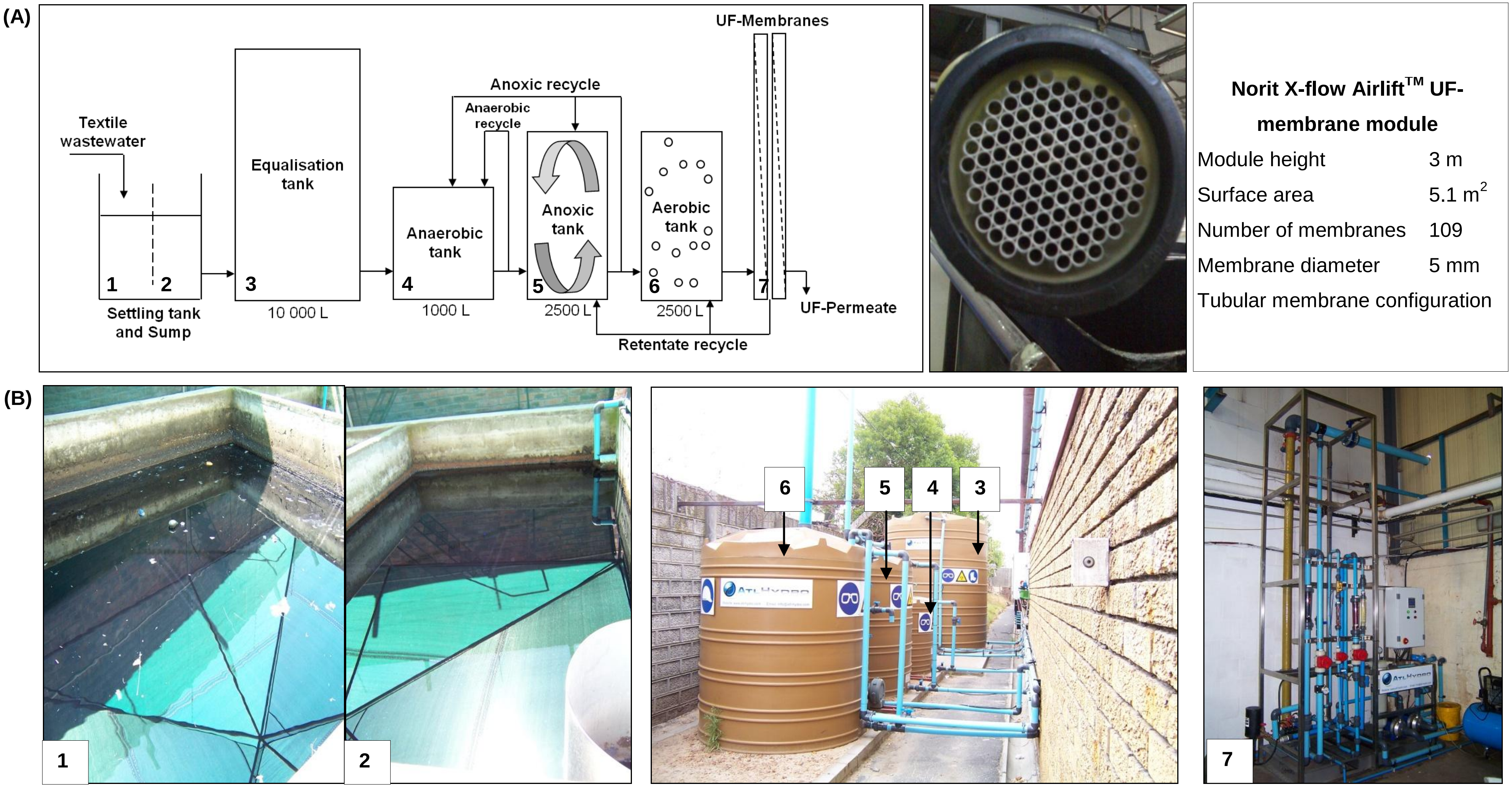
A textile industry located in the Western Cape was chosen as the industrial partner for the on-site evaluation of a semi-automated 5 to 10m$^3$/day membrane bioreactor (MBR) pilot plant incorporating Norit's sidestream X-flow Airlift$^{TM}$ membrane modules. The pilot plant was designed, constructed and operated on site for 250 days. The purpose of the piloting evaluation was to do a technology assessment on the effectiveness and efficiency of the MBR technology for the treatment of textile wastewater and propose a full-scale design to provide a complete solution to meet their current and future water needs.

## 3.2 Experimental

The textile wastewater generated by the industrial partner included a combination of water from the dyehouse processes, washing and drying processes, dye vat cleaning as well as rain and washwater run off from the processes areas. As shown in Figure 3.1, this wastewater was channelled into a settling tank where, via gravity separation, it entered an overflow sump before being discharged to the municipality. The pilot plant system design for the treatment of the wastewater discharged from the sump was based on a modified version of the traditional University of Cape Town (UCT) biological nutrient removal (BNR) conventional activated sludge (CAS) system (Du Toit *et al.,* 2010; ∅stgaard *et al.,* 1997). The process design aimed for optimal microbial community enrichment based on a pre-denitrification configuration coupled with enhanced biological phosphate removal (EBPR). The anaerobic-anoxic-aerobic process with recycles was designed to incorporate two primary purposes: 1) azo dye cleavage in a reducing anaerobic environment followed by oxidation of the resultant aromatic amines in an aerobic environment; and 2) biological nutrient removal by microbial consortia using nitrification, denitrification, and phosphate removal. As shown in Figures 3.1 and 3.5 it consisted of two phases: 1) the sidestream ultrafiltration (UF) dual-stage MBR (UF-dsMBR) followed by 2) nanofiltration (NF) alternated with reverse osmosis (RO) for residual colour and salt removal.

### 3.2.1 Phase 1: Pilot plant UF-dsMBR

As shown in Figure 3.1(A and B), the UF-dsMBR, which was robust and non-sterile, consisted of a series of tanks: 1) a 10 m$^3$ equalisation tank, which acted as a buffer tank and reduced variation of the textile wastewater composition entering the biological treatment tanks (the feed from the equalisation tank to the anaerobic tank occurred via gravity); 2) a 1 m$^3$ anaerobic tank, for the cleavage of the azo bonds of the reactive dyes; 3) a 2.5 m$^3$ anoxic



**Norit X-flow Airlift$^{TM}$ UF-membrane module**

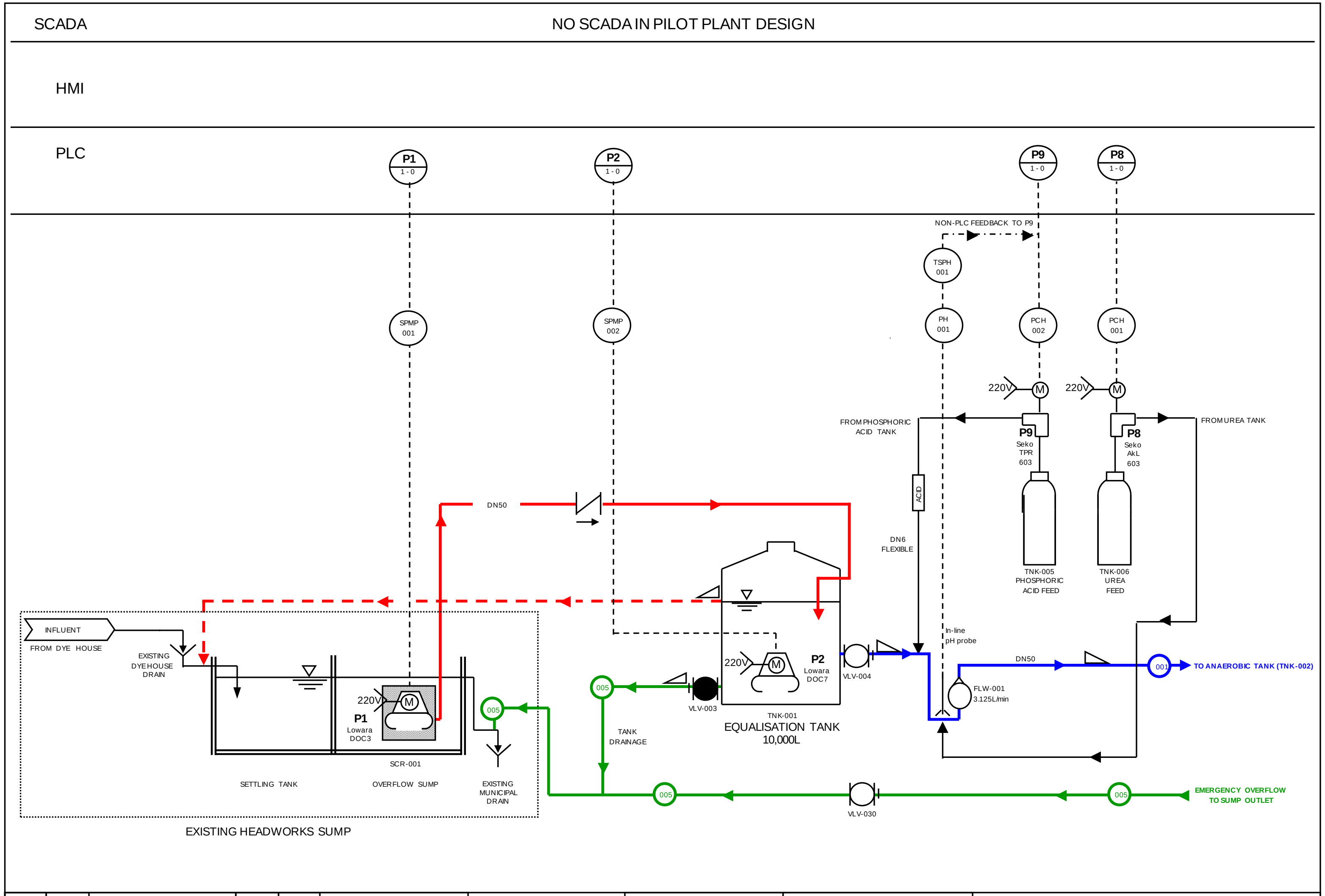
| | |
|---|---|
| Module height | 3 m |
| Surface area | 5.1 m$^2$ |
| Number of membranes | 109 |
| Membrane diameter | 5 mm |
| Tubular membrane configuration | |

**Figure 3.1:** Phase 1 – (A) Schematic diagram and (B) pictures showing the pilot-scale sidestream UF-dsMBR system used in the treatment of textile wastewater, where (1) represents the settling tank; (2) the sump; (3) the equalisation tank; (4, 5, and 6) the anaerobic, anoxic and aerobic tanks, respectively; and (7) the UF-membranes

tank with decreased levels of dissolved oxygen, in which denitrification occurred; and 4) a 2.5 m$^3$ aerobic tank, in which nitrification occurred, as well as mineralisation of the aromatic amines. The sidestream crossflow UF-membranes accounted for the removal of any solid organic material remaining after biological treatment. As shown in Figure 3.1 the mixed liquor, containing the acclimated microbial consortia, was recycled to the aerobic and anoxic tanks. Each 3 m high membrane module housed 109 tubular membranes with a membrane diameter of 5 mm and surface of ~0.046 m$^2$, with each membrane module having a total surface area of 5.1 m$^2$. The UF permeate was collected in a 1 m$^3$ UF permeate tank which was used as the backflush liquid and once full overflowed into the sump. The UF-membrane section of the pilot plant was operated using a programmable logic controller (PLC) human machine interface (HMI) (Appendix A) with the forward cycle set to 5 minutes and the backflush cycle to 30 seconds for the duration of the study. During the backflush cycle both the forward cycle and airscouring continued to operate. Only the permeate flow into the permeate tank was shutdown with the aid of a solenoid valve since the permeate line was used to feed the backflush liquid (i.e. UF permeate) into the lumen of the membranes.

The anaerobic, anoxic and aerobic tanks were inoculated with filtered 9 g/L activated sludge, with total suspended solids (TSS) of 1,330 mg/L, volatile suspended solids (VSS) of 958.13 mg/L and a COD of 5,815 mg/L obtained from the Bellville Wastewater Treatment Plant (Cape Town, South Africa). The sludge was filtered by hand through a large metal mesh sieve with a 1 mm pore size in 10 L batches. The anaerobic tank was inoculated with 200 L and the anoxic and aerobic tanks with 300 L of activated sludge. In addition to the activated sludge, 1 kg of urea was added to the anaerobic tank, while 2 kg of urea was added to both the anoxic and aerobic tanks. All the biological tanks were allowed to stabilise for 24 h before being taken off recycle. The pilot plant was operated at ambient temperature with a C:N:P ratio of 100:10:1 (Russell, 2006), achieved by dosing the system after the equalisation tank with 1.7 M urea and 0.5 M phosphoric acid to adjust the pH of the textile wastewater from ~10 to ~7 before entering the anaerobic tank. Refer to Appendix B for preparation of the dosing solutions.

Refer to the piping and instrumentations diagrams (P&ID) in Figures 3.2 to 3.4 for a detailed design of the textile wastewater treatment pilot plant.



| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 04-02-2011 | Project<br>TEXTILE EFFLUENT RE-USE |
|---|---|---|---|---|---|---|---|---|---|---|
| A | 07-09-2009 | Initial Drawing | D | 11-01-2010 | Final commissioning design amendments | Drawing number | DWG_CLI_007_09-004 | Author | D De Jager | |
| B | 30-09-2009 | Hardware & Equipment Specification | E | 02-02-2010 | Omission of sump recirculation line | Scale | N/A | Checked by | MS Sheldon | Drawing title<br>HEADWORKS, SUMP AND EQUALISATION TANK |
| C | 06-10-2009 | Equalisation tank recycle added | F | 04-02-2011 | PID modifications | Revision | F | Status: | DRAFT | |

**Figure 3.2:** P&ID representing the sump and equalisation tank of the of the UF-dsMBR textile wastewater treatment pilot plant (drawn using Microsoft Office Power Point 2007)

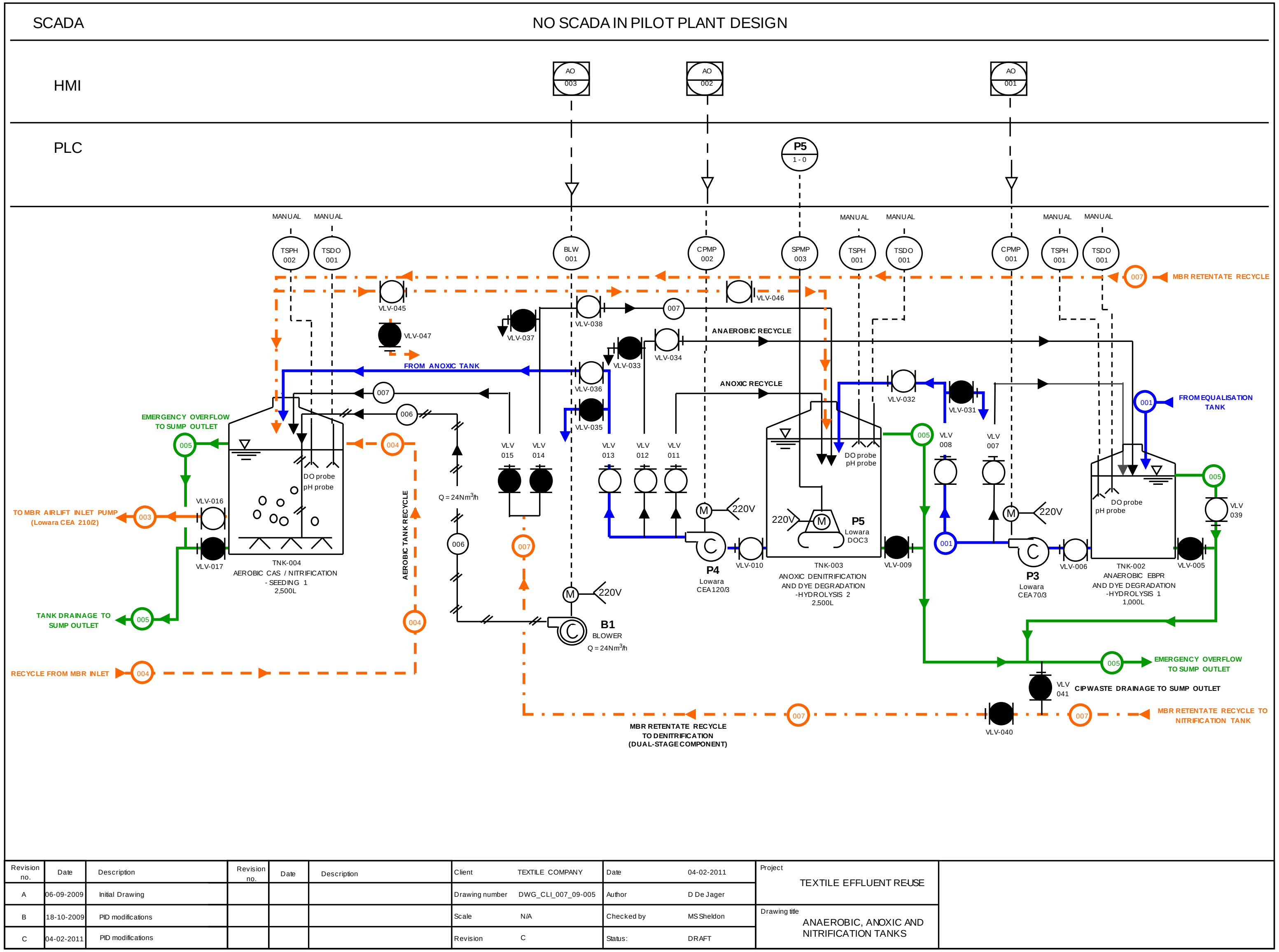


**Figure 3.3**: P&ID representing the biological system of the UF-dsMBR textile wastewater treatment pilot plant (drawn using Microsoft Office Power Point 2007)

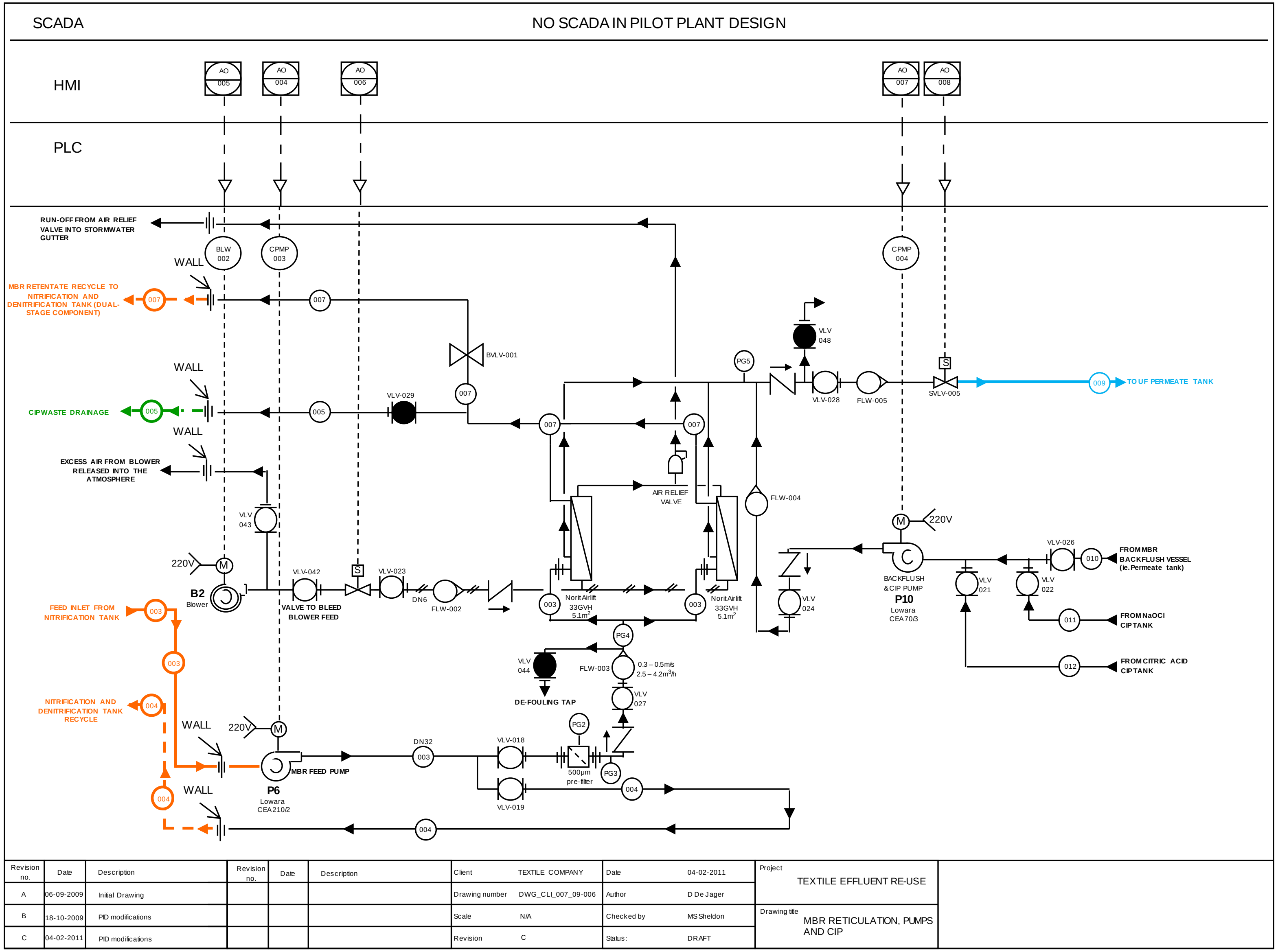


**Figure 3.4**: P&ID representing the membrane section of the UF-dsMBR textile wastewater treatment pilot plant (drawn using Microsoft Office Power Point 2007)

Cleaning-in-place (CIP) was performed on the UF-membrane modules approximately once every two weeks. During CIP the biological tanks were placed in recycle mode, while the membrane modules were flushed and soaked with 400 ppm sodium hypochlorite (NaOCl) for 24 h to remove any organic material and micro-organisms attached to the surface of the membranes, followed by 1% citric acid for 4 h, to remove any inorganic deposits, including scaling, from the membranes. Refer to Appendix B for the preparation of the CIP solutions.

### 3.2.2 Phase 2: NF/RO pilot plant set-up

The UF permeate, obtained after treatment with the UF-dsMBR pilot-scale system, was within the City of Cape Town (CCT) wastewater and industrial discharge standards, but not all the parameters of the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specifications. Therefore, the UF permeate could not be re-used for dyeing purposes by the industrial partner and was therefore subsequently treated with alternating NF and RO. Phase 2 involved feeding of the UF permeate, from Phase 1 (the pilot-scale UF-dsMBR system), collected in the 1 $m^3$ UF permeate tank into a pilot-scale NF/RO system (Figure 3.5). This phase was added towards the end of the study after analysis of the UF permeate showed the presence of residual colour and salt. The UF permeate tank was emptied and rinsed with clean water prior to connecting the NF/RO pilot plant. Therefore, the NF/RO pilot plant (shown in Figure 3.5) was fed with “fresh” UF permeate and not UF permeate that had been stored for a prolonged time period, during which the characteristics of the UF permeate could have changed. Refer to Table 3.1 for maximum operating conditions at which both the NF and RO membranes could be operated. During Phase 2 the spiral wound Desalogics DK2540 NF membrane with a molecular weight cut off (MWCO) of 150 to 300 Da was first operated in the system and then alternated with a spiral wound (XUS-SW30XHR-2540) RO membrane, in order to compare the treatment efficiency of both NF and RO on the UF permeate by comparing the NF and RO permeates obtained during operation. The feed to the NF/RO membrane was a combination of UF permeate and recycled brine produced by the NF/RO system, which had been returned to the NF/RO holding tank. Both streams were fed to the holding tank of the NF/RO system where mixing occurred prior to being fed to the NF/RO membrane. Prior to installing the RO membrane the system underwent CIP and the holding tank was drained and rinsed with clean water to ensure the feed to the RO membrane was a combination of UF permeate and RO brine only with no residual NF brine present. CIP of the NF and RO membranes was performed using 30 L of 1% sodium hydroxide (NaOH) for 1 h and 30 min when required. Refer to Appendix B for preparation of the CIP solution.

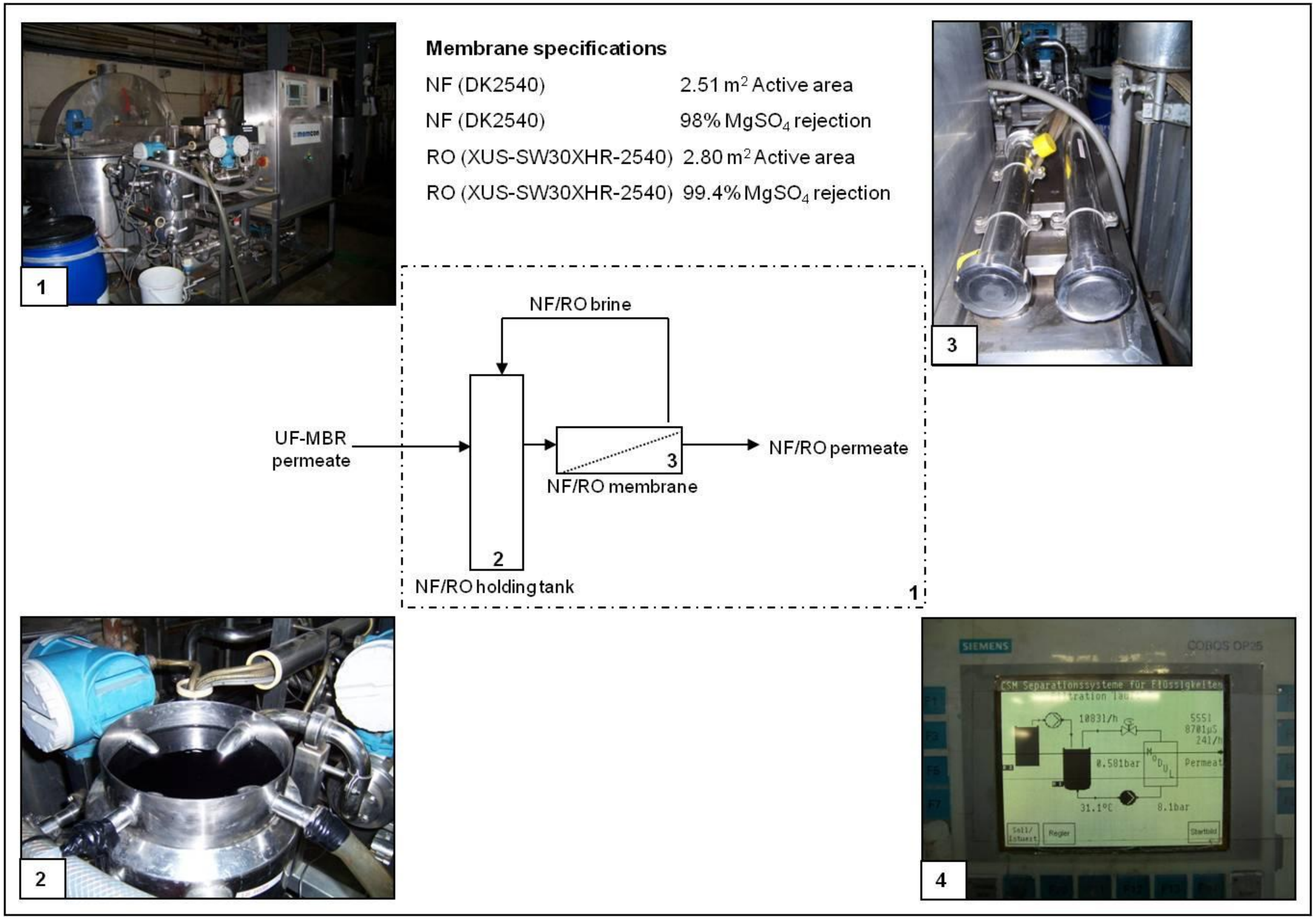
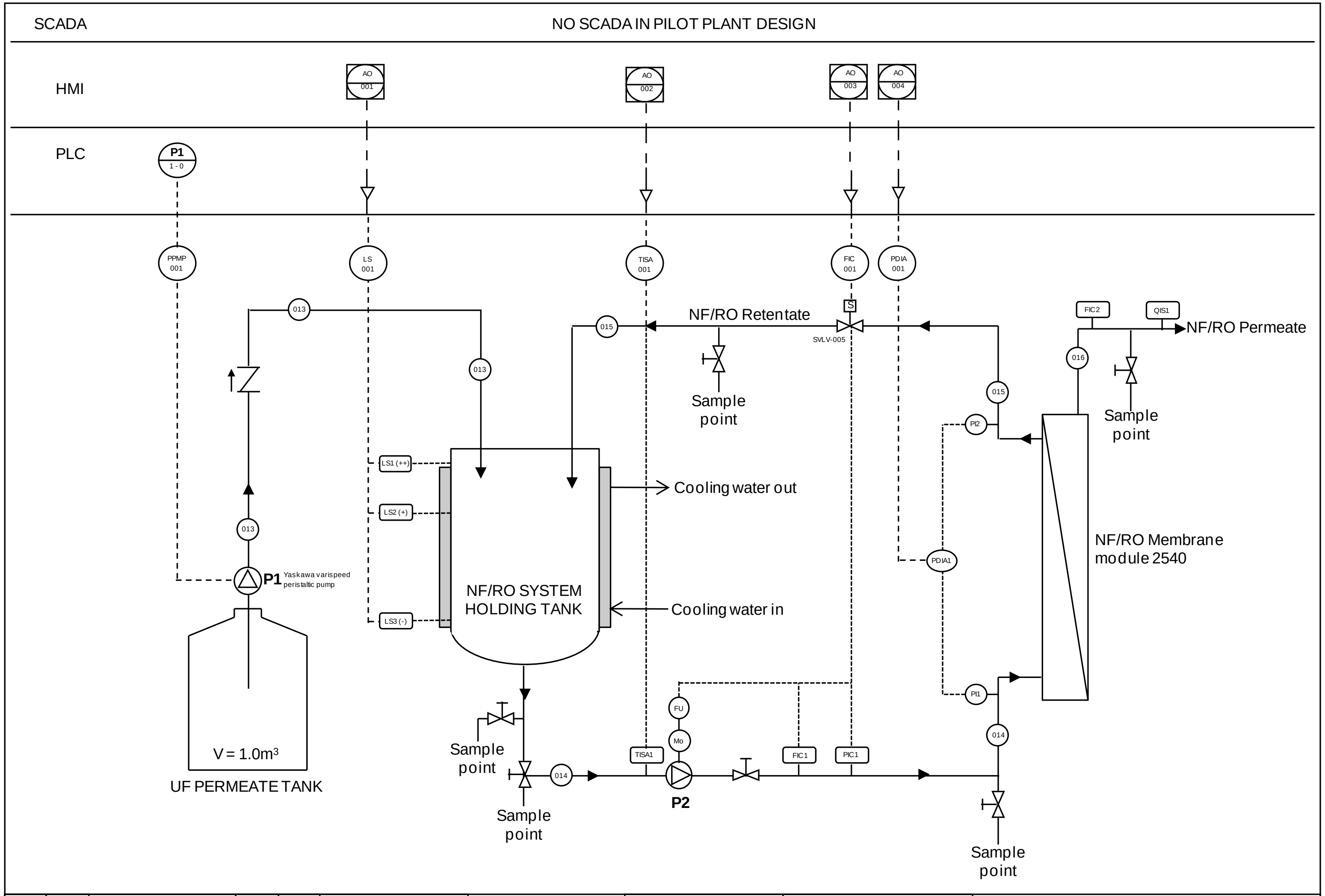


**Figure 3.5:** Phase 2 - Schematic diagram and pictures of the pilot-scale NF and RO system, where: (1) represents the pilot-scale NF/RO system; (2) the holding tank; (3) the NF/RO membrane modules; and (4) the PLC screen



| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 04-02-2011 | Project<br>TEXTILE EFFLUENT RE-USE | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A | 06-09-2009 | Initial Drawing | | | | Drawing number | DWG_CLI_007_09-006 | Author | D De Jager | | |
| B | 18-10-2009 | PID modifications | | | | Scale | N/A | Checked by | MS Sheldon | Drawing title<br>MBR RETICULATION, PUMPS AND CIP | |
| C | 04-02-2011 | PID modifications | | | | Revision | C | Status: | DRAFT | | |

**Figure 3.6:** P&ID representing the pilot-scale NF and RO system as supplied by MEMCON (Pty) Ltd. (drawn using Microsoft Office Power Point 2007)

Refer to the P&ID in Figure 3.6 for a detailed design of the NF/RO pilot plant. The entire system was automated by a programmable logic controller (PLC) and human machine interface (HMI).

**Table 3.1:** NF/RO membrane operating specifications for maximum operation

| Membrane type | Model | Active area ($m^2$) | Permeate flow rate ($m^3$/d) | Maximum pressure drop (bar) | % $MgSO_4$ rejection |
|---|---|---|---|---|---|
| **NF** | DK2540 | 2.51 | 2.27 | 0.6 | 98 |
| **RO** | XUS-SW30XHR-2540 | 2.80 | 2.6 | 1.0 | 99.4 |

The combination of UF permeate and NF brine mixed in the NF/RO system holding tank was fed to the NF membrane at an average flow rate of 0.94 $m^3$/h, with an average feed pressure of 11.1 bar, 0.45 bar differential pressure, a crossflow velocity (CFV) of 5.43 m/s and an average flux of 14.3 L/$m^2$h. While the combination of UF permeate and RO brine mixed in the holding tank of NF/RO system was fed to the RO system at an average flow rate of 0.8 $m^3$/h, with an average feed pressure of 12.6 bar, 0.8 bar differential pressure, a CFV of 4.62 m/s and an average flux of 5 L/$m^2$h.

The industrial partner utilised two salts in their dyeing processes, namely fine salt (NaCl) for cotton fabrics and Glaubers salt ($Na_2SO_4$) for wool and acrylic fabrics. Both of these salts were therefore present in the textile wastewater that was fed to the UF-dsMBR pilot plant system from the sump and therefore also present in the resulting NF/RO brine. Various techniques exist for salt recovery including: evaporation and cooling (i.e. distillation); membrane separation; electrodialysis; ion exchange; eutectic freeze crystallisation (EFC); chemical processes such as calcinations; as well as hybrid systems combining these techniques (Kim *et al.,* 2011). However, the costs associated with these techniques are expensive and since the industrial partner was utilising two types of salt in their dyeing processes it is currently more cost effective to buy the salts from a chemical supplier (i.e. ZAR 166.00 for 5 kg of NaCl and ZAR 63.00 for 500 g) than to try and recover it. Research (Kim *et al.,* 2011; Randall *et al.,* 2011) is currently being undertaken to make salt recovery more efficient and cost effective. However, the recovery of salt from the NF and RO brines did not form part of this study.

## 3.3 Operating procedure

The UF-dsMBR pilot plant was operated consecutively in modes 1 to 9, as indicated in Table 3.2, for different time periods. The UF-dsMBR system was designed to operate with a feed flow rate ranging from 56 to 403 L/h. During the start-up of the system it was noted that it was easier to maintain a constant low feed flow rate (i.e. 100 L/h) to the anaerobic tank than a high feed flow rate (i.e. 403 L/h) due to the feed from the equalisation tank being via

**Table 3.2:** Average flow rates through the UF-dsMBR pilot plant system for the 9 consecutive modes of operation

| **Parameter** | **Mode 1** | **Mode 2** | **Mode 3** | **Mode 4** | **Mode 5** | **Mode 6** | **Mode 7** | **Mode 8** | **Mode 9** |
|---|---|---|---|---|---|---|---|---|---|
| **Duration (days)** | 6 | 18 | 21 | 13 | 8 | 23 | 87 | 31 | 32 |
| **Anaerobic tank (1 $m^3$)** | | | | | | | | | |
| **Anaerobic feed (L/h)** | 403 | 288 | 134 | 56 | 187 | 217 | 100 | 87 | 100 |
| **Recycle from the anoxic tank (L/h)** | 83 | 63 | 42 | 42 | 42 | 42 | 42 | 42 | 42 |
| **Anoxic tank (2.5 $m^3$)** | | | | | | | | | |
| **Anoxic feed (L/h)** | 486 | 351 | 176 | 98 | 229 | 259 | 142 | 129 | 142 |
| **Recycle from the UF-membrane modules (L/h))** | 417 | 365 | 208 | 104 | 260 | 313 | 156 | 156 | 156 |
| **Aerobic tank (2.5 $m^3$)** | | | | | | | | | |
| **Aerobic feed (L/h)** | 820 | 653 | 342 | 160 | 447 | 530 | 256 | 243 | 256 |
| **Recycle from the UF-membrane modules (L/h)** | 3,733 | 2,955 | 2,282 | 3,216 | 3,060 | 3,008 | 2,334 | 2,334 | 2,334 |
| **UF-membrane modules** | | | | | | | | | |
| **Feed to UF-membrane modules ($m^3$/h)** | 4,550 | 3,610 | 2,620 | 3,380 | 3,510 | 3,540 | 2,590 | 2,580 | 2,590 |
| **UF permeate flow rate (L/h)** | 403 | 288 | 134 | 56 | 187 | 217 | 100 | 87 | 100 |
| **Flux (L/$m^2$h)** | 39.51 | 28.24 | 13.14 | 5.49 | 18.33 | 21.27 | 9.80 | 8.53 | 9.80 |
| **Air scouring (L/h)** | 1.45 | 1.35 | 1.61 | 1.14 | 1.07 | 3.80 | 3.32 | 4.65 | 4.60 |

gravity. The decreasing liquid level in the equalisation tank resulted in a decreasing feed flow rate to the anaerobic tank, which resulted in the operators constantly having to adjust the valves to ensure the set flow rate was maintained. While, at lower feed flow rates this was easier to manage. This also resulted in a variation between the number of days the different modes were operated.

Table 3.3 provides the average operating parameters for the NF and RO membranes, respectively. The pilot plant was first operated with the NF membrane for 4 days. However, analysis of the NF permeate indicated large amounts of residual salt present with average conductivity and total dissolved solids (TDS) of 3,543 µS/cm and 2,652 mg/L, respectively. The NF membrane was then replaced with the RO membrane and operated for 13 days.

**Table 3.3:** Average operating parameters for the NF and RO membrane, respectively

| **Parameter** | **NF membrane** | **RO membrane** |
|---|---|---|
| **Duration (days)** | 4 | 13 |
| **Feed pressure (bar)** | 11.1 | 12.6 |
| **Differential pressure** | 0.45 | 0.80 |
| **Temperature (°C)** | 31.3 | 32.1 |
| **Feed flow rate (L/h)** | 940 | 800 |
| **Permeate flow rate (L/h)** | 50.1 | 14.1 |
| **Flux (L/m$^2$h)** | 14.28 | 5.01 |

### 3.3.1 Daily operational parameter measurements and troubleshooting

Figure 3.7 indicates how the permeate flux decreased over the course of the UF-dsMBR operation. Each month indicates the average flux for that particular month with standard deviation bars. A problem observed in June 2010, after 57 days of operation, was that the permeate flow rate did not remain at the set flow rate following a backflush cycle. After a 30 second backflush cycle the permeate flow rate would increase to the set flow rate and then steadily decrease until a stable flow rate was reached and would remain at that flow rate for the remainder of the forward cycle.

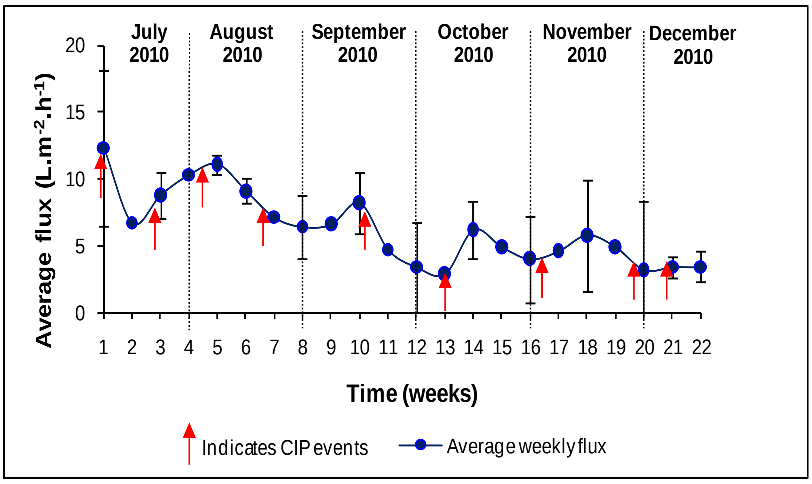


**Figure 3.7:** Decline in UF permeate flux from July till December 2010

In Figures 3.20 to 3.25 no data points are available for day 3; this was due to the system being shutdown in order to CIP before replacing the NF membrane with the RO membrane. During operation of the RO membrane, on day 11 the system shutdown automatically when the maximum pressure differential (1 bar) was exceeded. In order to solve this problem the system was CIPed, therefore no temperature point was recorded in Figures 3.20 to 3.25. On day 12 the temperature exceeded the maximum operating conditions (50°C) also resulting in automatic shutdown of the system. The last temperature recorded by the NF/RO pilot-scale system prior to shutdown was 45.5°C. This was due to a summer heat wave with temperatures in excess of 30°C, together with a large amount of additional heat released by the dye vats in the dyehouse. The same weather conditions were not experienced during operation of the NF membrane. On the ninth day of operation with the RO membrane (i.e. day 13) the feed flow rate was decreased in order to reduce the crossflow velocity (CFV) (Figure 3.8) and thus lower the operating temperature of the system and increase the flux (Figure 3.9).

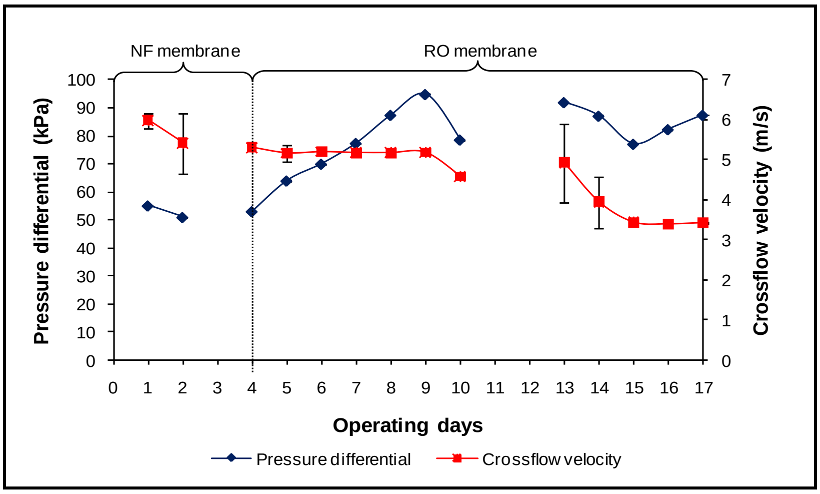


**Figure 3.8:** Crossflow velocity was decreased on day 13

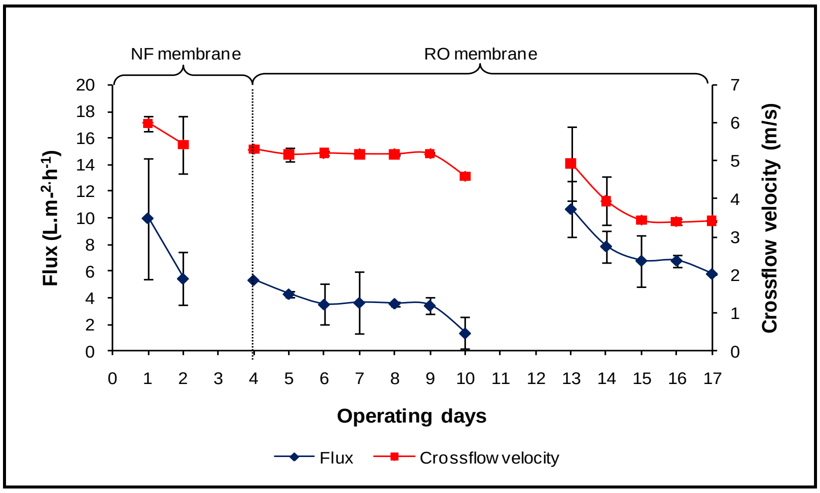


**Figure 3.9:** Increased flux after decreasing the crossflow velocity

### 3.3.2 Experimental timeline

Phase 1 (UF-dsMBR) was designed, constructed, commissioned and operated on-site for 250 days. However, the results averages and percentage removals for Phase 1 were calculated based on data collected after the system stabilised (i.e. 220 days).

Phase 2 (NF/RO system) was only operated for the last 17 days. This was due to logistical challenges with regard to transportation of the NF/RO pilot plant and financial restrictions with regard to the cost of hiring the system. Hence, all the results averages and percentage removals for this system were calculated based on data during this period. An analysis for the colour removal over both Phase 1 and 2 was done over the last two weeks of the study when the NF/RO system was in operation.

All parameters measured for the UF, NF and RO permeates were compared to: 1) the City of Cape Town (CCT) wastewater and industrial effluent discharge standards by-law (South Africa (Western Cape), 2006) in order to ensure that if discharged directly to the municipality all standards are met; 2) the SANS 241 drinking water specification (South Africa. Department of Water Affairs, 2011; South Africa. Department of Water Affairs and Forestry, 2005) in order to ensure the resulting permeate, especially from NF and RO, was re-usable by the industrial partner; and 3) the potable water of the industrial partner in order to compare the quality of the UF, NF and RO permeates obtained to the quality of the current water used in the dyeing processes – if the UF, NF and RO permeates are of the same if not better quality than the potable water currently utilised in dyeing processes then the permeate obtained after treatment can be re-used by the industrial partner.

## 3.4 Plant measurements and analytical methods

All analyses for the duration of the study were measured in duplicate. An average for each parameter was then calculated for each day from the data obtained. Throughout the study randomly selected water samples that had been collected for analysis were sent to a South African National Accreditation System (SANAS) accredited laboratory, in order to verify/validate the results obtained in the Cape Peninsula University of Technology (CPUT) research laboratory where the analyses were performed.

### 3.4.1 Daily on-site pilot plant analyses and checks

Water samples collected from the sump, anaerobic-, anoxic- and aerobic-tanks, the UF, NF and RO permeates, NF and RO brines and the NF/RO holding tank were taken three times a day (at 9:00 am, 12:00 pm and 15:00 pm) and analysed for the following physico-chemical parameters indicated in Table 3.4. See Appendix C for detail of analysis.

Figures 3.10 to 3.32 indicate the daily analysis and measurements recorded as indicated in Table 3.4. Figures 3.10 to 3.19 are for 220 days of the UF-dsMBR system and Figures 3.20 to 3.25 represent the NF/RO pilot plant for 17 days. All figures include standard deviation error bars. Data points missing between days 150 and 191 in Figures 3.10 to 3.19 were due to the hand held multiparameter PCSTestr 35 breaking and waiting for a replacement to arrive.

**Table 3.4:** Physico-chemical parameters measured daily

| Physico-chemical parameters | |
|---|---|
| **Parameter** | **Daily plant checks (thrice daily)** |
| Water usage | X |
| Flow rates | X |
| Pressures | X |
| pH | X |
| Temperature | X |
| Conductivity | X |
| Total dissolved solids (TDS) | X |

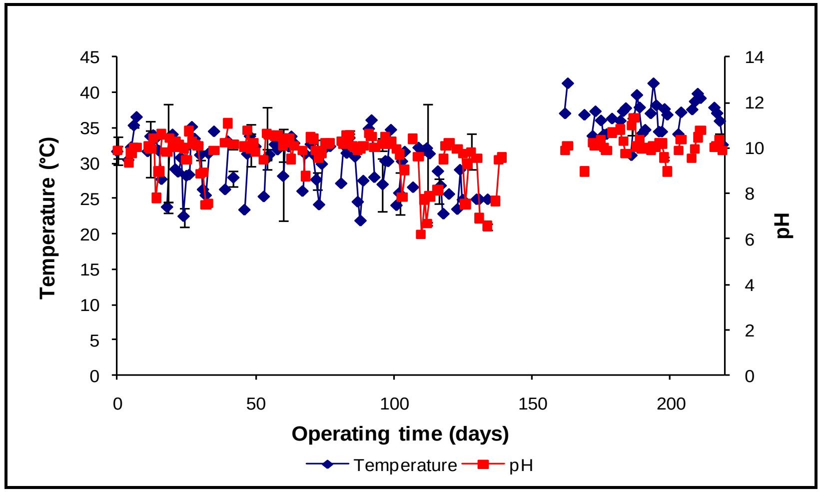


**Figure 3.10:** Average temperature and pH of the textile wastewater exiting the sump

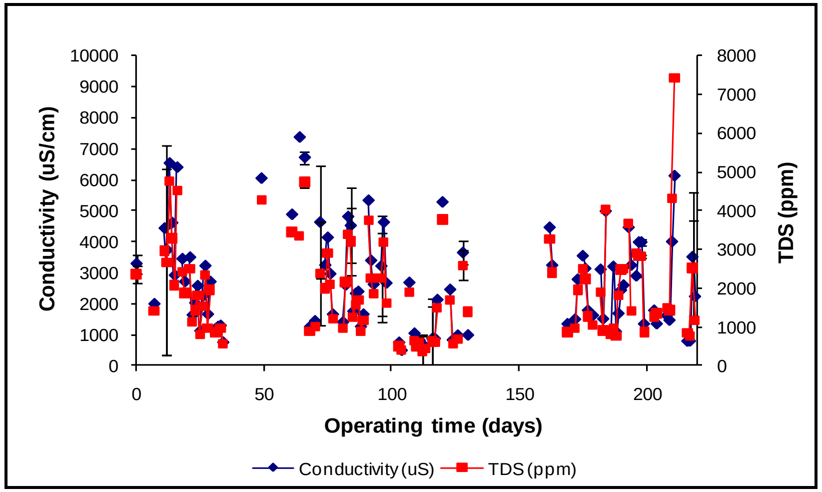


**Figure 3.11:** Average conductivity and total dissolved solids present in the textile wastewater exiting the sump

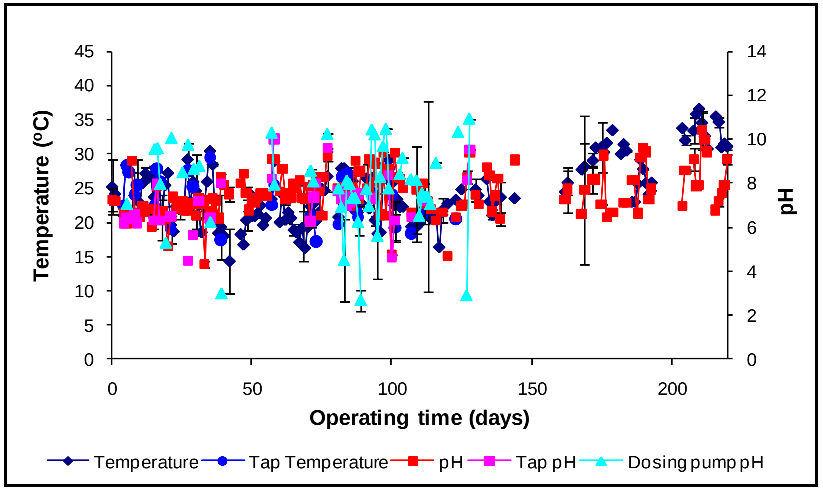


**Figure 3.12:** Average temperature and pH of the anaerobic tank

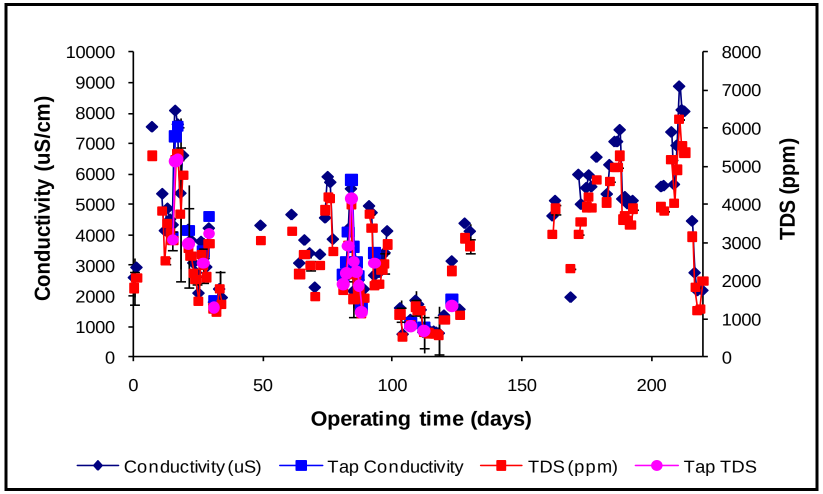


**Figure 3.13:** Average conductivity and total dissolved solids of the anaerobic tank

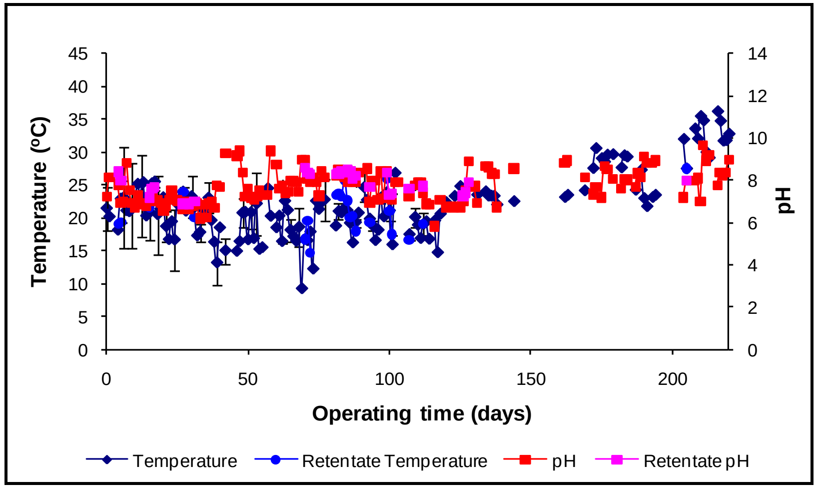


**Figure 3.14**: Average temperature and pH of the anoxic tank

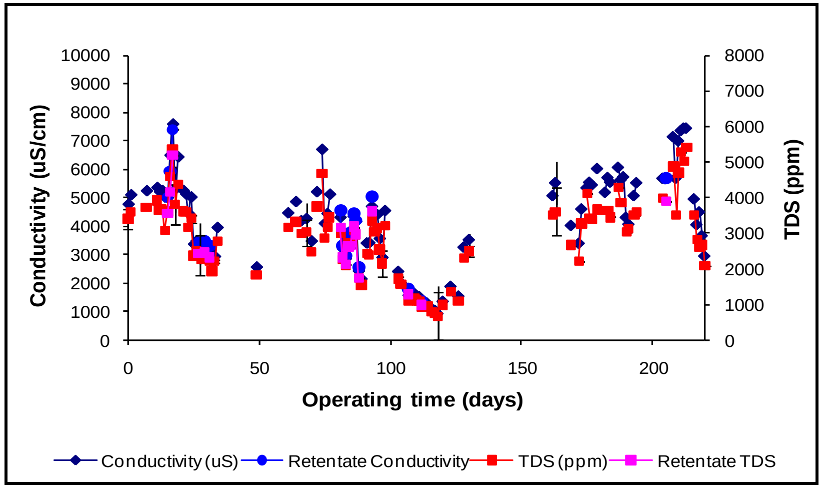


**Figure 3.15:** Average conductivity and total dissolved solids of the anoxic tank

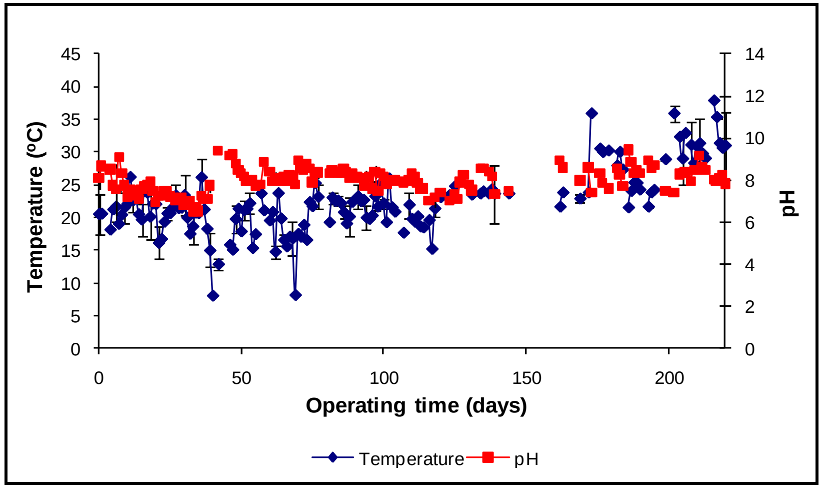


**Figure 3.16**: Average temperature and pH of the aerobic tank

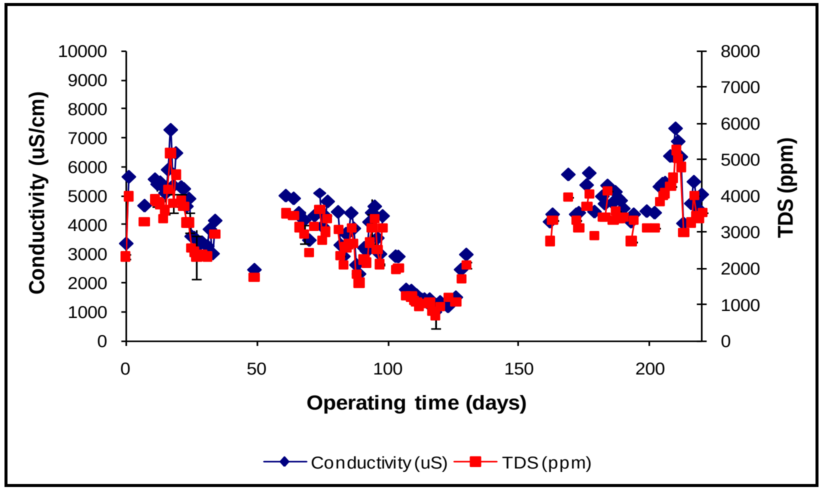


**Figure 3.17**: Average conductivity and total dissolved solids of the aerobic tank

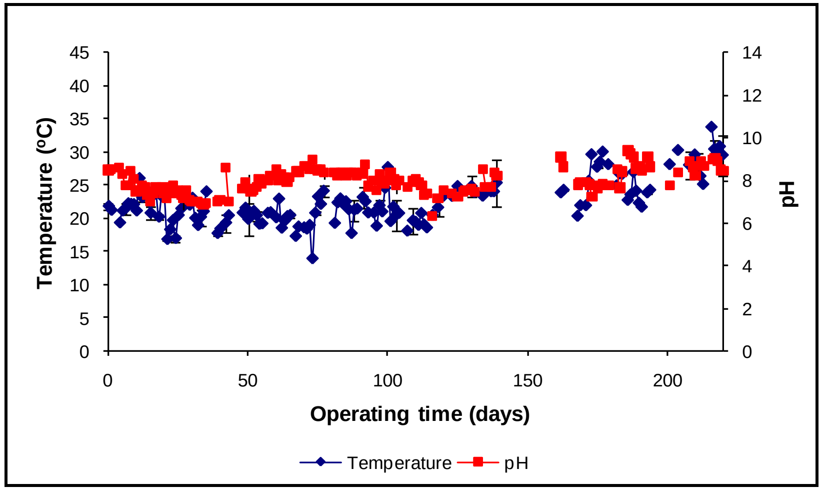


**Figure 3.18:** Average temperature and pH of the UF permeate

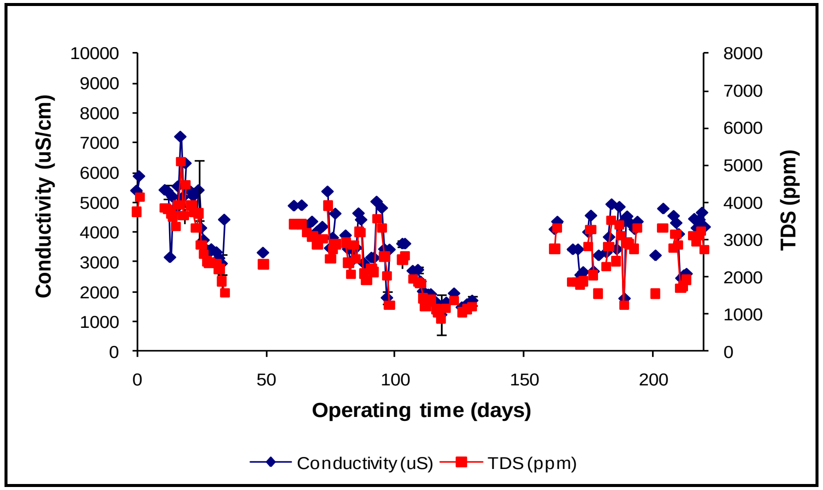


**Figure 3.19:** Average conductivity and total dissolved solids of the UF permeate

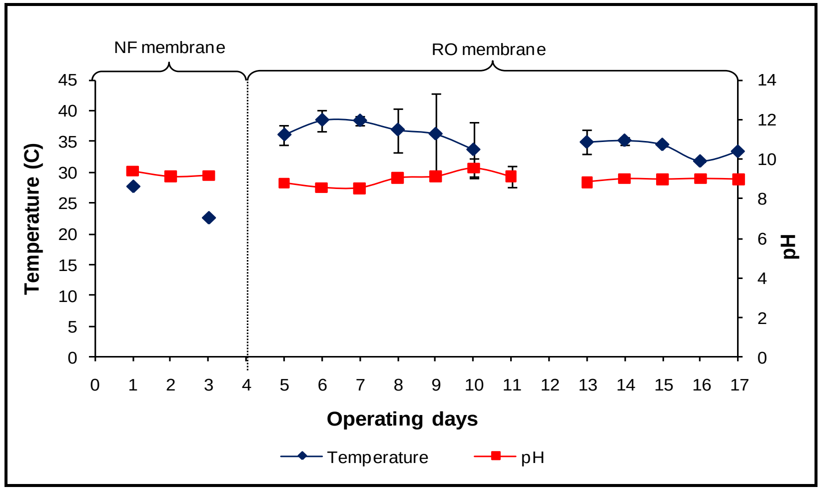


**Figure 3.20:** Average temperature and pH of the NF/RO brine

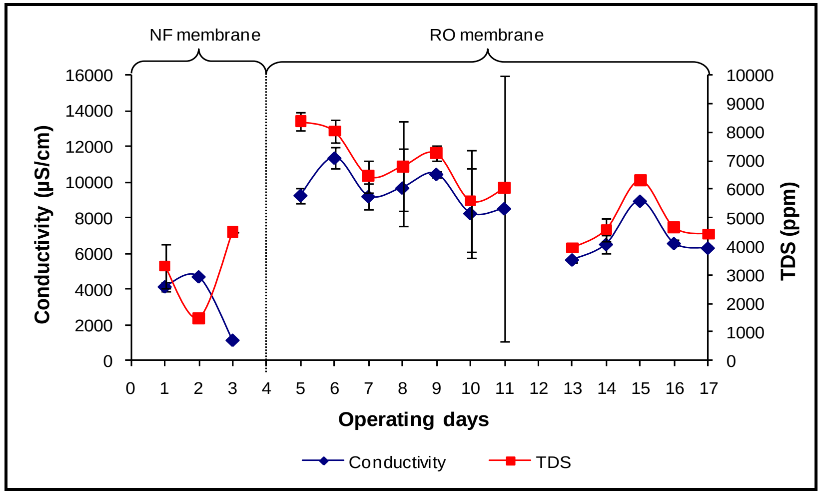


**Figure 3.21:** Average conductivity and total dissolved solids of the NF/RO brine

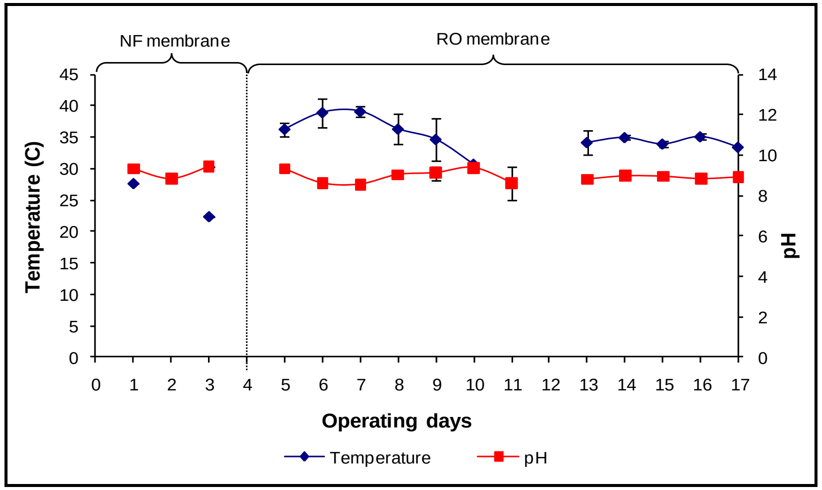


**Figure 3.22:** Average temperature and pH of the NF/RO holding tank

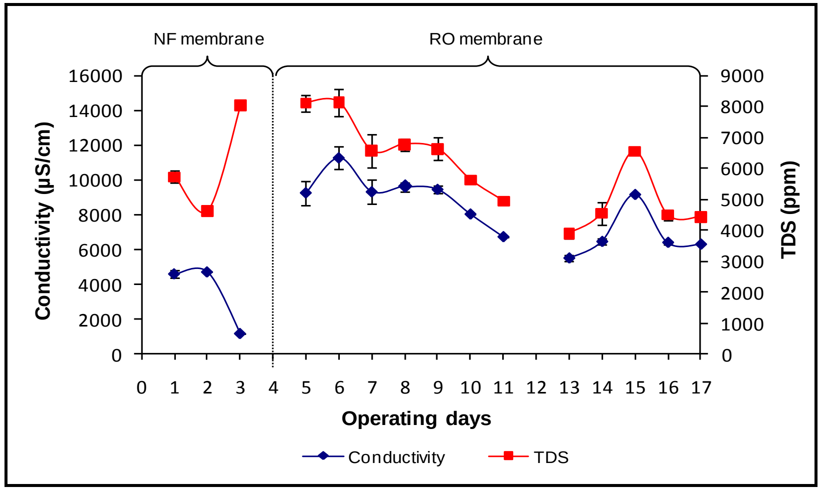


**Figure 3.23:** Average conductivity and total dissolved solids of the NF/RO holding tank

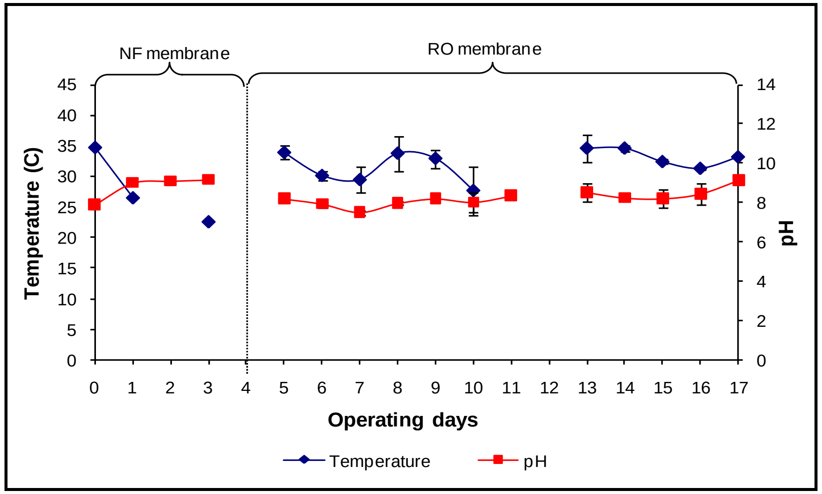


**Figure 3.24:** Average temperature and pH of the NF/RO permeate

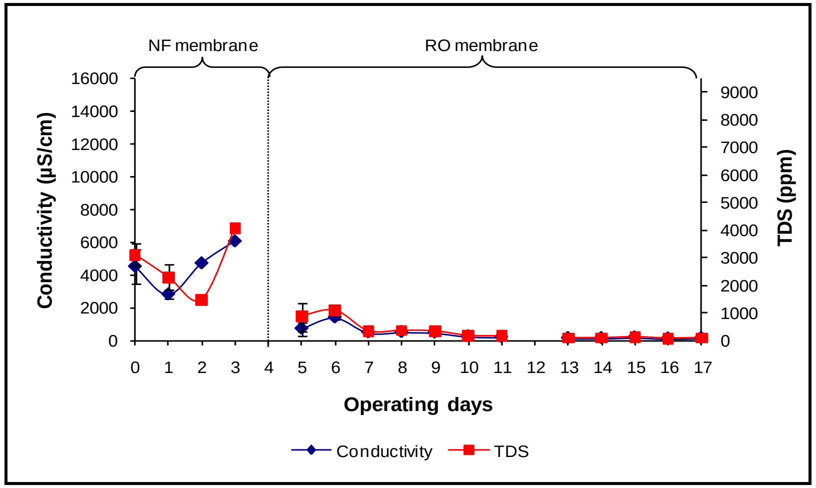


**Figure 3.25:** Average conductivity and total dissolved solids of the NF/RO permeate

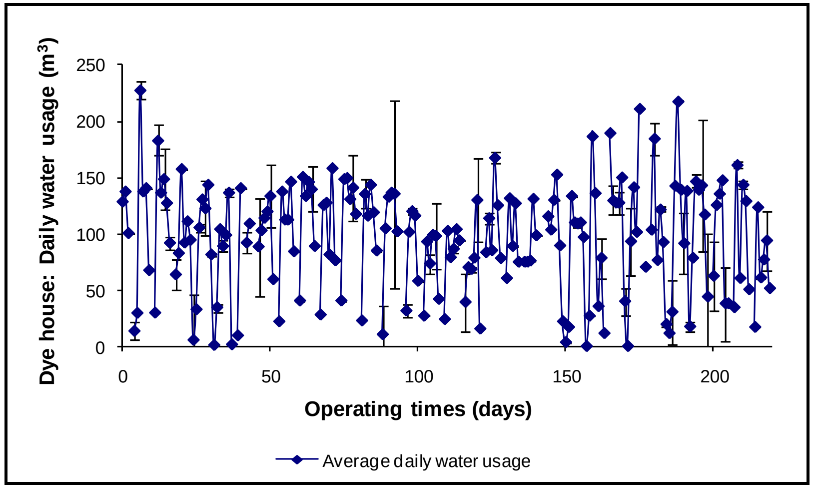


**Figure 3.26:** Daily water usage by the dyehouse

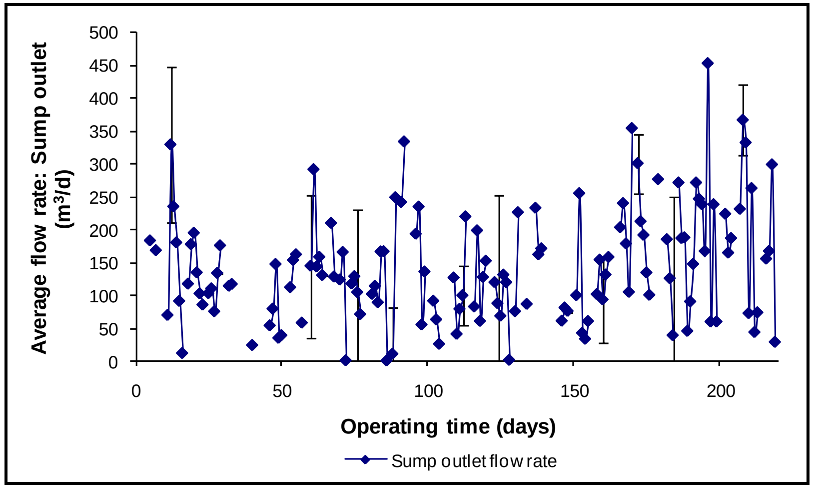


**Figure 3.27:** Average daily flow rate from the sump to the municipality

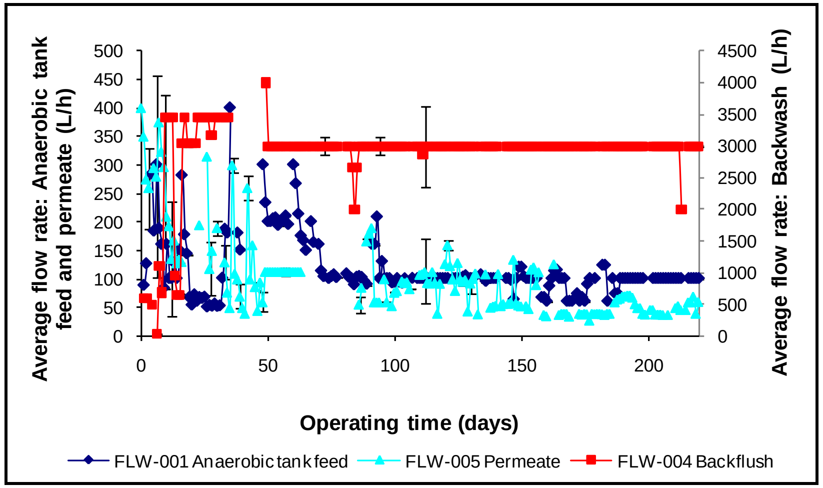


**Figure 3.28:** Average flow rate into the anaerobic tank, UF permeate and membrane backflush

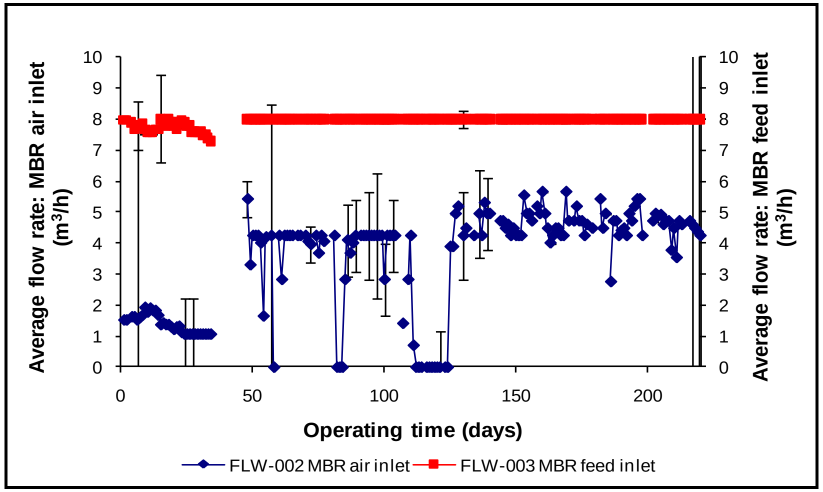


**Figure 3.29:** Average airflow rate through the lumen and feed flow rate to the UF-membrane modules

The following flow rates were monitored and recorded three times daily and adjusted accordingly to ensure constant tank levels were maintained within the biological tanks:

- daily water usage by the dyehouse (Figure 3.26);
- the sump outlet to the municipality (Figure 3.27);
- the equalisation tank to the anaerobic tank, as well as the permeate flow rate and backflush flow rate (Figure 3.28);
- the anaerobic tank to the anoxic tank (Appendix D) ;
- the anoxic tank to the aerobic tank (Appendix D);
- the recycle from the anoxic tank to the anaerobic tank (Appendix D);
- the retentate recycle from the UF-membrane modules to both the anoxic and aerobic tanks (Appendix D);
- the air flow rate through the lumen of the membrane modules (Figure 3.29);
- the feed inlet to the UF-membrane modules from the aerobic tank (Figure 3.29); as well as
- the flux across the membrane modules (Figures 3.30).

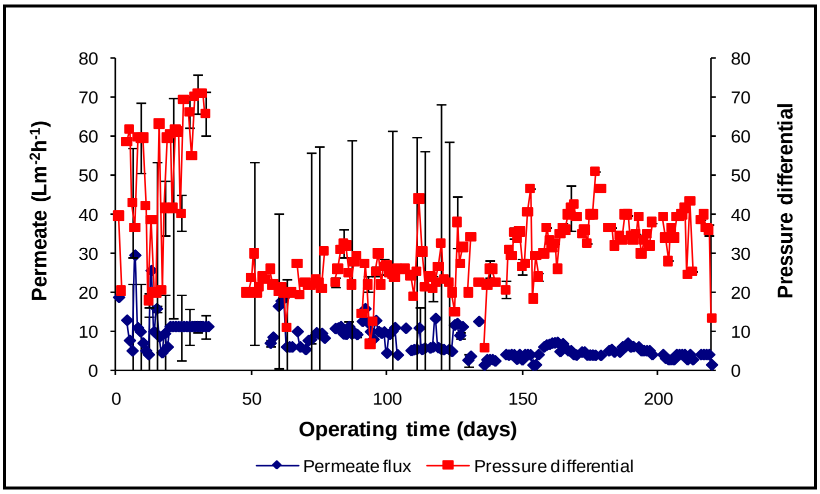


**Figure 3.30:** Permeate flux and pressure differential across the membrane modules

The following pressures on the pilot plant were also recorded thrice daily:

- the inlet and outlet pressure of the 500 µm in-line pre-filter (PG2 and PG3 in Figures 3.4 and 3.31);
- the inlet pressure to the UF-membrane modules (PG4 in Figures 3.4 and 3.32); and
- the outlet permeate pressure (PG5 in Figures 3.4 and 3.32).

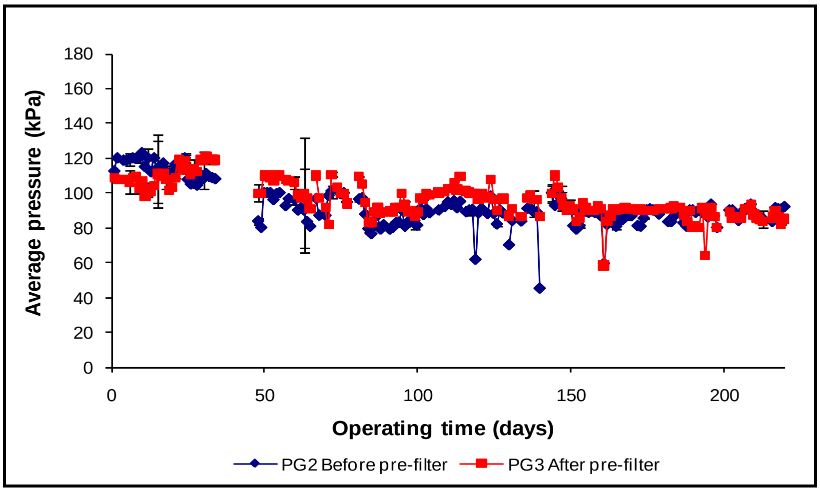


**Figure 3.31:** Inlet and outlet pressure of the in-line pre-filter to the UF-membrane modules

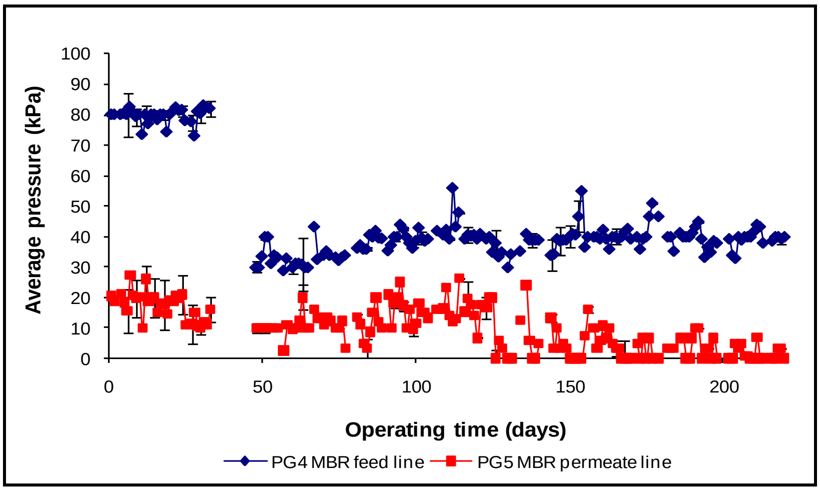


**Figure 3.32:** Inlet and outlet pressure of the UF-membrane modules and UF permeate, respectively

### 3.4.2 Laboratory analysis

The daily water samples collected from the sump, anaerobic-, anoxic- and aerobic-tanks, the UF permeate, as well as permeate from the NF and RO membranes were analysed every second day (Table 3.5), in duplicate, for:

- pH, temperature, conductivity and total dissolved solids (TDS) using a calibrated PCSTestr 35 multiparameter (Wirsam Scientific and Precision Equipment (Pty) Ltd) (Appendix C).
- ammonium ($NH_4$) (Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001) (Appendix E);
- COD (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 F);
- nitrate ($NO_3$) (Merck Spectroquant Nitrate cell test; Cat. No. 1.14773.0001) (Appendix G);

- phosphate ($PO_4$) (Merck Spectroquant Phosphate cell test for orthophosphate and total phosphorus, Cat. No. 1.14543.0001) (Appendix H);
- total suspended solids (TSS) (ESS Method 350.2) (Appendix I);
- turbidity (TN-100 turbidimeter, ISO 7027 compliant nephelometric method) (Appendix J).

**Table 3.5:** Physico-chemical and organic parameters measured every second day

**Physico-chemical parameters**

| **Parameter** | **Lab analysis** |
|---|---|
| pH | X |
| Temperature | X |
| Conductivity | X |
| Total dissolved solids (TDS) | X |
| Total suspended solids (TSS) | X |
| Volatile suspended solids (VSS) | X |
| Chemical oxygen demand (COD) | X |
| Turbidity | X |

**Inorganic composition**

| **Parameter** | **Lab analysis** |
|---|---|
| Phosphate | X |
| Nitrate | X |
| Ammonium | X |

### 3.4.3 Colour analyses on hydraulic retention time (HRT) samples

Five sample sets taking the hydraulic retention time (HRT) into account, for a constant feed flow rate of 100 L/h into the anaerobic tank, were analysed for colour. The corresponding average HRT for each biological stage was 7.1 $h^{-1}$ (anaerobic tank), 5.6 $h^{-1}$ (anoxic tank) and 13.4 $h^{-1}$ (aerobic tank). The HRTs were determined based on the flow rates entering each biological tank at the time each sample was taken. The HRT of each biological stage remained relatively constant with only the textile wastewater composition changing. Each sample set consisted of a sump, anaerobic feed, anaerobic tank, anoxic tank, aerobic tank, UF permeate and NF/RO permeate sample. These five sample sets were labelled HRT1, HRT2, HRT3, HRT4, and HRT5. For the sample set labelled HRT1 no NF/RO permeate sample was taken as at that stage the NF/RO pilot-scale system was not in operation yet.

These five sample sets were analysed, in duplicate, for (Table 3.6):

- true colour using the Hazen method (Platinum-cobalt standard method analogous to APHA 2120B, DIN53409) (Hongve & Åkesson, 1996) (Appendix K);
- American dye manufacturing index (ADMI) (Greenberg *et al*., 1985) (Appendix L);
- total organic carbon (TOC) and dissolved organic carbon (DOC) (both determined using Merck Spectroquant TOC cell tests, Cat. No. 1.14878.0001) (Appendix M); as well as
- all the physic-chemical and inorganic parameters indicated in Table 3.3 in section 3.4.2.

**Table 3.6:** Physico-chemical parameters determined for all HRT sample sets

| **Physico-chemical parameters** | |
|---|---|
| **Parameter** | **Dye analysis** |
| Colour (ADMI) | X |
| True colour (Colour Hazen) | X |
| Total organic carbon (TOC) | X |
| Dissolved organic carbon (DOC) | X |

# CHAPTER 4

# RESULTS

## MEMBRANE BIOREACTOR APPLICATION WITHIN THE TREATMENT OF TEXTILE WASTEWATER

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# CHAPTER 4

# MEMBRANE BIOREACTOR APPLICATION WITHIN THE TREATMENT OF TEXTILE WASTEWATER

## 4.1 Introduction

The textile industry not only consumes large quantities of water (Brik *et al.,* 2006; Chakraborty *et al*., 2003; Barclay and Buckley, 2002), but it also produces large volumes of toxic, low biodegradable, highly coloured wastewater, which without suitable treatment would spoil the natural water environment (You & Teng, 2009; You *et al.*, 2008; You *et al.,* 2006; Badani *et al.,* 2005; Kim *et al.,* 2004). Textile wastewater contains high concentrations of slow or non-biodegradable organic substances and inorganic chemicals (Badani *et al.,* 2005; Lubello & Gori, 2004), such as dyes, pigments and often heavy metals (Feng *et al.,* 2010; ElDefrawy & Shaalan, 2007), as well as a range of contaminants, including: salts, enzymes, surfactants, as well as oxidising and reducing agents. These contaminants result in suspended solids, fluctuating pH, high temperature, strong colour (Feng *et al.,* 2010; Kim *et al.,* 2002) and influence the chemical oxygen demand (COD) and biological oxygen demand (BOD) of the wastewater (Badani *et al.,* 2005; Kim *et al.,* 2002). The quality of textile wastewater depends on the dyestuffs, accompanying chemicals and the process utilised by the textile company (Brik *et al*., 2006; Chakraborty *et al*., 2003).

Dyehouse wastewaters are complex and consist of concentrated waste process water, which contains a wide and varied range of dyes and other products (Fersi *et al*., 2005; Chakraborty *et al*., 2003; Barclay & Buckley, 2002; Robinson *et al.,* 2001). Unless the wastewater is properly treated before it is discharged into the environment, it may have serious long-lasting consequences, which include (You & Teng, 2009; Barclay & Buckley, 2002):

- Solid waste, which are unsightly and may result in anaerobic sludge layers in receiving streams.
- Many organic contaminants, such as dyes, synthetic sizes and detergents, are relatively non-biodegradable.
- Other organic compounds with very high BOD can cause anaerobic conditions in natural water sources resulting in death of the aquatic fauna and flora.
- The presence of inorganic salts, acids or alkalis in high concentrations, will make the receiving water unsuitable for most industrial and municipal purposes.

With the implementation of the Waste Discharge Charge System (WDCS) by the Department of Water and Environmental Affairs (DWEA), municipalities and wastewater producing industries will be pressurised to find innovative ways to treat industrial wastewater at its source; since companies who do not comply will be severely penalised, while companies who do will be rewarded (Mazema *et al*. 2008; South Africa, 2003).

Conventional treatment processes used in the treatment of textile wastewater, includes physical and chemical methods (e.g. coagulation, activated carbon adsorption, ion exchange, reverse osmosis); chemical oxidation (e.g. UV/$O_3$, UV/$H_2O_2$ Fenton reagent); advanced oxidation processes (e.g. photocatalysis, electrochemical, sonolysis, ionising radiation) as well as biological (e.g. activated sludge, sequencing batch reactor) (You & Teng, 2009; González-Zafrilla *et al.,* 2008; Badani *et al.,* 2005; Chakraborty *et al*., 2003; Kural *et al.,* 2001). MBR technology, which combines a biological process with membrane separation (Brik *et* al., 2006; Badani *et al.,* 2005), is an attractive alternative to the conventional methods of treating textile wastewater; as they either remove the dyestuff allowing re-use of the auxiliary chemicals used for dyeing or concentrate the dyestuffs and auxiliaries producing purified water (Chakraborty *et al.,* 2003). A major advantage is that MBR plants operate effectively at mixed liquor suspended solid (MLSS) concentrations of 8,000 to 12,000 mg/L (i.e. 0.8% to 1.2%) (Helble & Möbius, 2009; Sutherland, 2007) and it has even been demonstrated to operate successfully at 3%, unlike conventional activated sludge (CAS) plants that operate at MLSS concentrations of 2,000 to 3,000 mg/L. The high MLSS assists MBR systems in dealing effectively with strong industrial wastewaters (Sutherland, 2007). The latest MBR technology has overcome the high construction and maintenance costs associated with first generation MBRs. MBRs are becoming the technology of choice since they provide effluent with high quality; low chemical pollutants; a small footprint; and significant reduction in bacteria and viruses (Brik *et* al., 2006; Hai *et al.,* 2006; Schoeberl *et al.,* 2005).

## 4.2 Objectives

The objectives of this section of the study were to: 1) treat the wastewater to within the Western Cape effluent discharge standards to render the water safe for discharge; and 2) evaluate the possible re-use/recycle of the treated water by the industrial partner.

## 4.3 Materials and methods

A textile industry located in the Western Cape was chosen as the industrial partner for the on-site evaluation of the 5 to 10 pilot plant. The system design was based on a modified version of the traditional UCT-configured biological nutrient removal (BNR) CAS system (∅stgaard *et al.,* 1997; Du Toit *et al.,* 2010) and consisted of two stages: 1) the sidestream UF-dsMBR, followed by 2) nanofiltration (NF) alternated with reverse osmosis (RO) after 4 days to facilitate re-use of the water.

### 4.3.1 Phase 1: UF-dsMBR pilot plant set-up

A pilot-scale dsMBR system, was designed, constructed, commissioned and operated on-site for 250 days. However, all results are based on 220 days of operation after stabilisation of the UF-dsMBR system. The system was robust and non-sterile with the textile wastewater treated in a series of tanks: a 10 $m^3$ equalisation tank; a 1 $m^3$ anaerobic tank; a 2.5 $m^3$ anoxic, and a 2.5 $m^3$ aerobic tanks, followed by two 3 m high 5.1 $m^2$ Norit X-flow Airlift$^{TM}$ UF-membrane modules. Refer to Figure 3.1 in Chapter 3 for a schematic diagram of the UF-dsMBR pilot plant system. The anaerobic tank was designed to incorporate anaerobic cleavage of the azo bonds of the reactive dyes, at the start of the biological process, into aromatic amines, upstream of conventional anoxic denitrification, and aerobic nitrification as well as mineralisation of the aromatic amines. The UF-membrane modules removed any organic material remaining in the wastewater after biological treatment. Recycle of the mixed liquor containing the acclimated microbial consortia to the aerobic and anoxic tanks was facilitated by the UF-membrane modules. The UF permeate was fed continuously to the final polishing treatment step, a pilot-scale NF/RO system.

#### 4.3.1.1 Inoculation and operation

The anaerobic, anoxic and aerobic tanks were inoculated with filtered activated sludge, with total suspended solids (TSS) of 1,330 mg/L, volatile suspended solids (VSS) of 958.13 mg/L and a chemical oxygen demand (COD) of 5,815 mg/L, obtained from the Bellville wastewater treatment plant. The anaerobic tank was inoculated with 200 L of activated sludge and the anoxic and aerobic tanks with 300 L of activated sludge. In addition to the activated sludge, 1 kg of urea was added to the anaerobic tank, while 2 kg of urea was added to both the anoxic and aerobic tanks. The pilot plant was operated at ambient temperature with a C:N:P ratio of 100:10:1 (Russell, 2006), which was achieved by dosing the system after the equalisation tank with 1.7 M urea and 0.5 M phosphoric acid to adjust the pH of the textile wastewater from ~10 to ~7 before entering the anaerobic tank. The flow rates between the equalisation, anaerobic, anoxic and aerobic tanks, as well as the UF-membrane modules were monitored daily.

The UF-dsMBR system was designed and operated in 9 consecutive recycle modes and hydraulic retention times (HRT). An average flux of only 9.5 L/$m^2$h was obtained. Over the course of the study the flux steadily declined (Figure 3.7 in section 3.3.1 of Chapter 3)and was measured at 3.4 L/$m^2$h just before shutdown of the pilot plant. Therefore, the flow rate entering the anaerobic tank was adjusted based on the permeate flow in order to ensure the mass balance over the system was maintained.The cause for the decline in flux was attributed to the low MLSS (1,329 mg/L) and sludge volume index (SVI) (method explained in Appendix N) determined to be a minimum of 1.6 ml/g, a maximum of 27.3 ml/g, with an average of 15.8 ml/g in the aerobic tank which resulted in membrane fouling, since overtime slower MLSS thickening results in fouling at low MLSS concentrations (Trussell technologies, 2012). At lower MLSS concentrations progressive pore blocking (i.e. fouling), to potentially protect the membranes, are created by colloids and particles (Judd, 2011). The common range of the SVI for a conventional activated sludge plant should be between 50 and 150 ml/g (Toprak Home Page, 2006). However, at high MLSS concentrations (i.e. > 12,000 mg/L) results in membrane 'sludging'or rapid fouling due to the formation an excessive cake layer which reduces the membrane permeability (Trussell technologies, 2012).

#### 4.3.1.2 Cleaning-in-place (CIP)

CIP of the membrane modules occurred approximately once every two weeks. During CIP the biological system was placed on recycle, while the membrane modules were flushed and soaked with 400 ppm sodium hypochlorite (NaOCl) for 24 hours, to remove any organic material and micro-organisms; followed by 1% citric acid for 4 hours, to remove any inorganic deposits, including scaling, from the membranes.

### 4.3.2 Phase 2: Subsequent nanofiltration/reverse osmosis (NF/RO) treatment

After Phase 1 the UF permeate still contained residual dye (i.e. colour) and salt, removal of which was imperative for successful reclamation of the wastewater for re-use, especially in dyeing processes. The UF-dsMBR permeate was therefore treated using a pilot-scale automated NF/RO system. For the first 4 days the system was operated with a NF membrane (Table 3.1 in Chapter 3) with a feed pressure of 11.1 bar, 0.45 bar differential pressure, a crossflow velocity of 5.43 m/s and an average flux of 14.3 L/m$^2$h. However, due to the low percentage conductivity and TDS rejection (i.e. 30.4% and 32.5%, respectively), the membrane was replaced with an RO membrane (Table 3.1 in Chapter 3).

The RO membrane was operated with a feed pressure of 12.6 bar, 0.8 bar differential pressure, a crossflow velocity of 4.62 m/s and an average flux of 5 L/m$^2$h. The RO membrane had to be operated at a lower feed pressure than the NF membrane, because at 13 bar the temperature produced by the RO system together with the high temperature in the dyehouse, caused the plant to exceed its maximum operating temperature and shut down. Therefore, the average flux obtained for both membranes could not be compared.

### 4.3.3 Analytical methods

Samples collected from the sump, anaerobic-, anoxic-, and aerobic-tanks, as well as the UF- and NF/RO permeate, were analysed daily for pH, conductivity and TDS using a calibrated

PCSTestr 35 multiparameter (Wirsam Scientific and Precision Equipment (Pty) Ltd) (Appendix C); and every second day for:

- turbidity (TN-100 turbidimeter, ISO 7027 compliant nephelometric method) (Appendix J);
- COD (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 F);
- ammonium ($NH_4$) (Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001) (Appendix E);
- nitrate ($NO_3$) (Merck Spectroquant Nitrate cell test, Cat. No. 1.14773.0001) (Appendix G);
- total suspended solids (TSS) (ESS Method 340.2) (Appendix I);
- phosphate ($PO_4$) (Merck Spectroquant Phosphate cell test for orthophosphate and total phosphorus, Cat. No. 1.14543.0001) (Appendix H); and
- colour (Appendices K and L).

Two methods were utilised to determine the amount of colour present, namely the colour Hazen method (Platinum-cobalt standard method analogous to APHA 2120B, DIN53409) (Hongve & Åkesson, 1996), which measured the true colour of samples; and the American dye manufacturing index (ADMI) method (Greenberg *et al*., 1985) which, measures the colour in ADMI units.

## 4.4 Results and discussion

The minimum, maximum and average values of all the parameters measured during the study with regard to Phase 1 and Phase 2 are summarised in Tables O.1 to O.12 in Appendix O. Standard deviation error bars have been included in all the figures.

### 4.4.1 Wastewater composition analysis

The wastewater stream was characterised by a COD range of between 45 to 2,820 mg/L (Table 4.1) and an average BOD of 192.5 mg/L. The maximum conductivity and TDS measured, 8,500 μS/cm and 6,260 ppm, respectively, did not meet the City of Cape Town (CCT) wastewater and industrial effluent discharge standards by-law (South Africa (Western Cape), 2006). However, the biggest problem facing the industry was the amount of colour present in the wastewater, a maximum of 427 mg Pt/L and an average of 131 mg Pt/L.

### 4.4.2 Phase 1: UF-dsMBR treatment

The amount of COD removed from the textile wastewater remained relatively stable after ~100 days of operation (Figure 4.1). During Phase 1 the minimum amount of COD removed was 19% and the maximum 97%, with an average of 75% (Table 4.2). The COD value for the treated textile wastewater (191 mg/L) was well within the CCT wastewater and industrial discharge standards for COD (≤ 5,000 mg/L). The average overall removal of TSS, turbidity, and phosphate was 19.6%, 94% and 14.5%, respectively. An increase in the amount of ammonium was observed during this phase due to mineralisation occurring in the final aerobic biological tank prior to the wastewater entering the UF-membrane modules. The nitrification of ammonia released during mineralisation of the aromatic amines, in the aerobic tank, added to the increased nitrate level in the UF permeate. The increase in the amount of conductivity (4,528 mg/L) and TDS (3,657 mg/L) noted in the UF permeate, when compared to the composition of the wastewater entering the UF-dsMBR system, was due to dosing the system with urea and phosphoric acid to achieve the desired C:N:P ratio.

**Table 4.1:** Wastewater composition fed to Phase 1 over the 220 day result period of pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 12.3 | 29.6 | 22.8 |
| **pH** | - | 5.3 | 12.2 | 9.8 |
| **Conductivity** | µS/cm | 495 | 8,500 | 2,716 |
| **TDS** | ppm | 169 | 6,260 | 1,973 |
| **COD** | mg/L | 45 | 2,820 | 763 |
| **Ammonium** | mg/L | 1.1 | 28.5 | 9.0 |
| **TSS** | mg/L | 3.0 | 430 | 54 |
| **Turbidity** | NTU | 14.6 | 575 | 45 |
| **Phosphate** | mg/L | 0.2 | 4.0 | 1.5 |
| **Nitrate** | mg/L | 0.7 | 6.8 | 3.0 |
| **True colour** | mg Pt/L | 31 | 427 | 131 |
| **ADMI** | ADMI units | 195 | 2,070 | 659 |

However, all parameters measured in the treated wastewater were within the wastewater discharge standards, but could not be re-used by the textile company. According to the ADMI colour determination method there was a 28.6% removal of colour from the treated

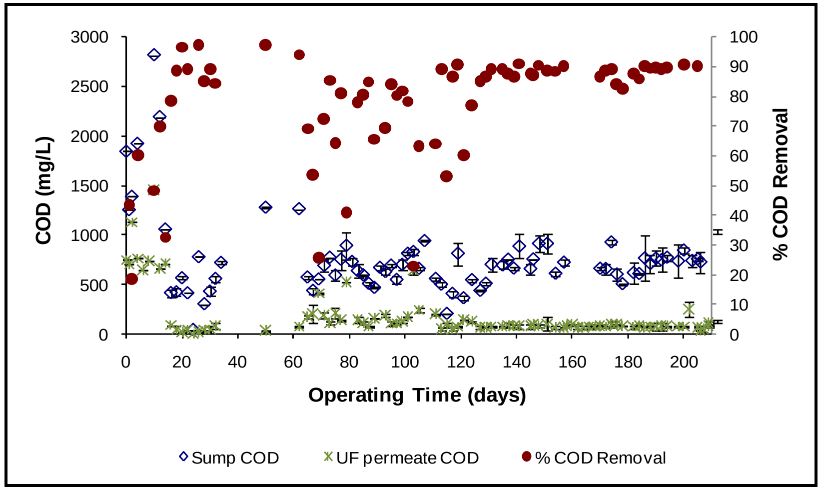


**Figure 4.1:** Percentage chemical oxygen demand removed within the UF-dsMBR

**Table 4.2:** Average water quality of Phase 1 UF-dsMBR permeate and percentage overall average removal compared to the CCT discharge standards

| Parameter | Units | Textile wastewater | UF-dsMBR Permeate | % Removal (UF-membrane) | CCT Wastewater discharge standards* |
|---|---|---|---|---|---|
| **Temperature** | $^{0}$C | 22.8 | 22.2 | N/A | 0-40 |
| **pH** | - | 9.8 | 8.4 | N/A | 5.5-12 |
| **Conductivity** | µS/cm | 2,716 | 4,302 | - | ≤ 5,000 |
| **TDS** | ppm | 1,973 | 3,657 | - | 4,000 |
| **COD** | mg/L | 763 | 191 | 75 | ≤ 5,000 |
| **Ammonium** | mg/L | 9.0 | 25.3 | - | ni |
| **TSS** | mg/L | 53.7 | 43.1 | 19.6 | 1,000 |
| **Turbidity** | NTU | 45.1 | 2.7 | 94 | ni |
| **Phosphate** | mg/L | 1.5 | 1.3 | 14.5 | 25.0 |
| **Nitrate** | mg/L | 3.0 | 3.2 | - | ni |
| **True colour** | mg Pt/L | 131 | 129 | 1.3 | ni |
| **ADMI** | ADMI units | 659 | 471 | 28.6 | ni |

ni – not indicated

*City of Cape Town: Wastewater and industrial effluent by-law (South Africa (Western Cape), 2006) wastewater samples (471 ADMI units) during the UF-dsMBR phase. However, the UF permeate (129 mg Pt/L) did not meet the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specification (Table 4.3) for true colour (20 to 50 mg Pt/L).

### 4.4.3 Phase 2: NF/RO treatment

The permeate obtained after treatment with the UF-dsMBR was within the CCT wastewater and industrial discharge standards, but not all the parameters of the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specifications. The UF permeate exceeded the textile company's potable water levels for conductivity (4,528 µS/cm), TDS (3,657 ppm), ammonium (25.3 mg/L), turbidity (2.7 NTU) and true colour (129 mg Pt/L). Therefore, the UF permeate could not be re-used for dyeing purposes by the industrial partner. Subsequent treatment with NF and RO membranes removed most of the salts remaining in the UF-dsMBR permeate. Table 4.3 compares the analysed parameters of the UF, NF and RO permeate to the potable water currently used by the industrial partner, as well as the water discharge and the drinking water standards. All percentage removals were calculated based on the average wastewater parameters entering the system during the last 17 days of operation for the NF and RO membranes only. Based on the UF permeate composition fed, the maximum conductivity and TDS removal with the NF membrane, was 58% and 59.6%, respectively. The minimum rejection rate was 27.7% and 32.2% for conductivity and TDS, with an average removal rate of 30.4% and 32.5% (Table 4.3 and Figure 4.2A and B), respectively. Based on the UF permeate composition fed, the average conductivity and TDS removal with the RO membrane was 97.5% and 97.4%, respectively (Table 4.3 and Figure 4.2A and B). The minimum removal rate was 79.7% and 80.8% for conductivity and TDS, respectively with a maximum of 98.7% for both.

As expected under these operating conditions higher salt rejection was obtained using the RO membrane than the NF membrane due to the fact that RO membranes are the most selective membrane, out of MF, UF, NF and RO membranes, and can reject monovalent ions, such as sodium ($Na^+$) and chloride ($Cl^-$), with hydraulic diameters of less than 1 nm (Judd, 2011; Judd, 2006).

The average ammonium removal for both NF and RO was 36.6% and 8.3%, respectively, and did not bring the treated wastewater to within the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specifications. However, it did bring the ammonium level to below that of the potable water received by the textile company (Table 4.3). NF and RO treatment showed 66.7% and 88.8%, respectively for TSS removal, and 89.1% and 4.9% removal, respectively for turbidity. The phosphate and nitrate levels of 0.40 and 2.4 mg/L for NF, respectively, were below the textile company's potable water levels for phosphate (1.3 mg/L) and within range for nitrate (2.3 mg/L). For RO 1.6 and 1.7 mg/L was within range of the textile company's potable water levels for phosphate (1.3 mg/L) and below for nitrate (2.3 mg/L), respectively. Both the NF and RO permeate contained nitrate levels within the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specification.

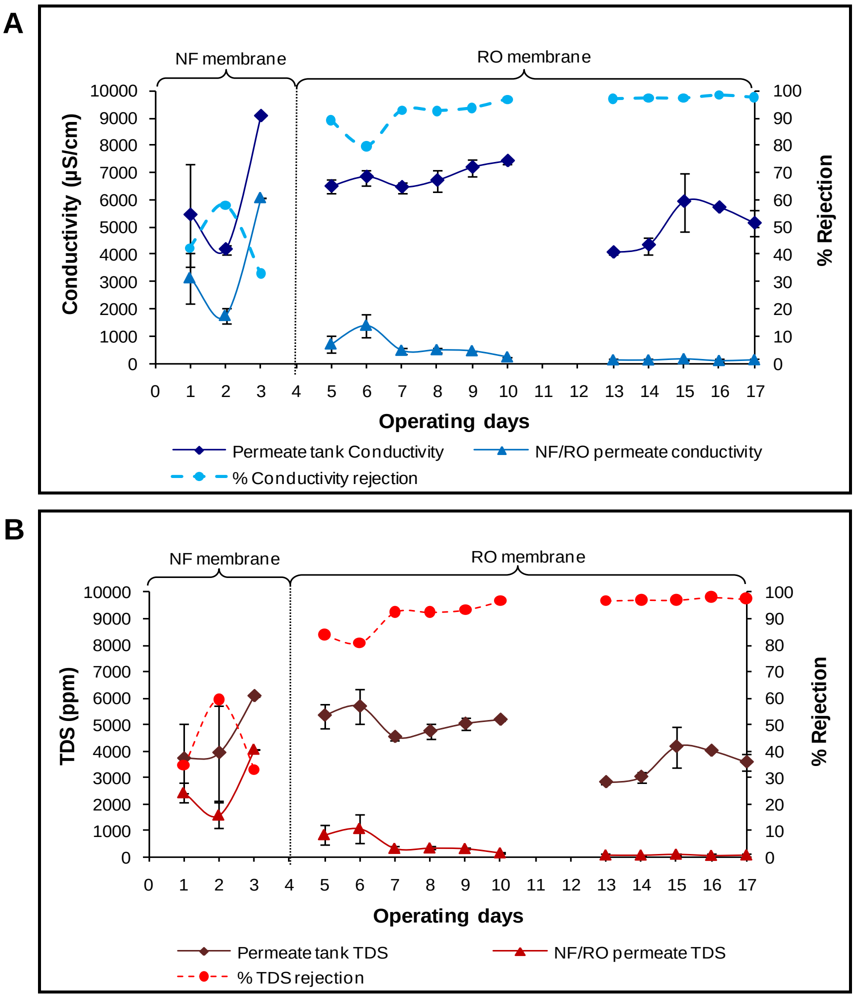


**Figure 4.2:** (A) Conductivity and (B) TDS removal through the NF/RO system (the missing data was explained in Chapter 3 section 3.3)

When comparing the NF permeate results for conductivity (6,077 µS/cm), TDS (4,077 ppm), COD (113 mg/L) and nitrate (2.4 mg/L) to the RO permeate results (Table 4.3) it was observed that the RO system produced permeate with lower conductivity (204 µS/cm), TDS (146 ppm) and nitrate (1.7 mg/L). Therefore, it was concluded that the permeate from the RO system was preferred to the permeate from the NF system. With the exception of ammonium (11 mg/L) all the other parameters analysed within the RO permeate were within the SANS 241:2005 (South Africa. Department of Water Affairs and Forestry, 2005) drinking water specifications. When compared to the potable water of the textile company, with the

**Table 4.3:** Average water quality of the NF/RO permeate and percentage removal (with regard to the average UF permeate entering the NF/RO pilot-scale system for the 4 days of NF and 13 days of RO operation) compared to the textile company's received potable water and the SANS 241:2005 drinking water specifications

| **Parameter** | **UF permeate entering the NF/RO system while operating** | | **NF permeate** | **% Removal (NF membrane)** | **RO permeate** | **% Removal (RO membrane)** | **Textile company potable water** | **SANS 241:2005 Drinking water specification*** | **SANS 241-1:2011 Drinking water specification**** |
|---|---|---|---|---|---|---|---|---|---|
| | **NF membrane** | **RO membrane** | | | | | | | |
| **Temperature ($^0$C)** | 24.9 | 24.8 | 22.6 | N/A | 24.7 | N/A | 22.2 | ni | ni |
| **pH** | 9.4 | 9.5 | 9.2 | N/A | 9.3 | N/A | 8.7 | 4.0-10 | ≥ 5 - ≤ 9.7 |
| **Conductivity (µS/cm)** | 8,734 | 8,215 | 6,077 | 30.4 | 204 | 97.5 | 92.6 | 1,500-3,700 | ≤ 1,700 |
| **TDS (ppm)** | 6,043 | 5,688 | 4,077 | 32.5 | 146 | 97.4 | 66.3 | 1,000-2,400 | ≤ 1,200 |
| **COD (mg/L)** | 36.8 | 77.0 | 113 | - | 81.6 | - | 58.9 | ni | ni |
| **Ammonium (mg/L)** | 12.3 | 12.0 | 7.8 | 36.6 | 11.0 | 8.3 | 11.2 | 1.0-2.0 | ≤ 1.5 |
| **TSS (mg/L)** | 13.5 | 16.4 | 4.5 | 66.7 | 1.8 | 88.8 | 1.5 | ni | ni |
| **Turbidity (NTU)** | 1.1 | 0.41 | 0.12 | 89.1 | 0.39 | 4.9 | 1.5 | 1.0-5.0 | ≤ 5.0 |
| **Phosphate (mg/L)** | 0.55 | 1.1 | 0.40 | 27.3 | 1.6 | - | 1.3 | ni | ni |
| **Nitrate (mg/L)** | 1.6 | 2.0 | 2.4 | - | 1.7 | 15 | 2.3 | ni | ni |
| **True colour (mg Pt/L)** | 148 | 153 | 13.0 | 91.2 | 14.1 | 90.8 | 22.7 | 20-50 | ≤ 15 |
| **ADMI (ADMI units)** | 463 | 503 | 11.9 | 97.4 | 19.9 | 96.0 | 16.0 | ni | ni |

ni – not indicated

*SANS 241:2005 Drinking water specification (South Africa. Department of Water Affairs and Forestry, 2005)

**SANS 241-1:2011 Drinking water specification (South Africa. Department of Water Affairs, 2011)

**Table 4.4:** Average water quality of the UF-dsMBR/NF/RO permeate, and the overall percentage removal compared to the industrial partner's potable water and the SANS 241-1:2011 drinking water specifications

| Parameter | UF-dsMBR permeate | Overall % removal (UF-membrane) | NF permeate | Overall % removal (NF membrane) | RO permeate | Overall % removal (RO membrane) | Textile company potable water | SANS 241-1:2011 Drinking water specification* |
|---|---|---|---|---|---|---|---|---|
| **Temperature ($^0$C)** | 22.2 | N/A | 22.6 | N/A | 24.7 | N/A | 22.2 | ni |
| **pH** | 8.4 | N/A | 9.2 | N/A | 9.3 | N/A | 8.7 | ≥ 5 - ≤ 9.7 |
| **Conductivity (µS/cm)** | 4,302 | - | 6,077 | - | 204 | 84.8 | 92.6 | ≤ 1,700 |
| **TDS (ppm)** | 3,657 | - | 4,077 | - | 146 | 85.2 | 66.3 | ≤ 1,200 |
| **COD (mg/L)** | 191 | 75 | 113 | 85.8 | 81.6 | 90.3 | 58.9 | ni |
| **Ammonium (mg/L)** | 25.3 | - | 7.8 | 14.3 | 11.0 | 18.6 | 11.2 | ≤ 1.5 |
| **TSS (mg/L)** | 43.1 | 19.6 | 4.5 | 81.2 | 1.8 | 94.5 | 1.5 | ni |
| **Turbidity (NTU)** | 2.7 | 94 | 0.12 | 99.5 | 0.39 | 98.1 | 1.5 | ≤ 5.0 |
| **Phosphate (mg/L)** | 1.3 | 14.5 | 0.40 | 57.7 | 1.6 | - | 1.3 | ni |
| **Nitrate (mg/L)** | 3.2 | - | 2.4 | - | 1.7 | 7.96 | 2.3 | ni |
| **True colour (mg Pt/L)** | 129 | 1.3 | 13.0 | 91.0 | 14.1 | 90.8 | 22.7 | ≤ 15 |
| **ADMI (ADMI units)** | 471 | 28.6 | 11.9 | 98.7 | 19.9 | 97.7 | 16.0 | ni |

ni – not indicated

*SANS 241-1:2011 Drinking water specification (South Africa. Department of Water Affairs, 2011) exception of conductivity and TDS, all the other parameters analysed were below or close to the values recorded for the same parameters in the potable water.

### 4.4.4 Overall water treatment efficiency

During the current study the UF-dsMBR treatment system showed an average overall COD removal of 75% (Table 4.4). However, all the COD readings were well within the CCT wastewater and industrial effluent discharge standards. When comparing the NF permeate results to the RO permeate results for this study (refer to Table 4.4) it was observed that the RO system produced permeate with lower conductivity (204 µS/cm), TDS (473 ppm), COD (81.6 mg/L), TSS (1.8 mg/L) and nitrate (1.7 mg/L). During the current study an average overall colour removal of 98.7% and 97.2% (Table 4.4) for ADMI and 91.0% and 90.8% for true colour was obtained for the NF and RO membranes, respectively; while the UF-dsMBR system was only able to obtain 28.6% overall removal. The ADMI was reduced from an average of 659 ADMI units in the wastewater entering the sump to ~12 ADMI units in the NF permeate and ~20 ADMI units in the RO permeate, a lower ADMI and colour compared to the received potable water. A slightly higher colour removal was obtained with the NF membrane than the RO membrane due to NF membranes achieving separation through a combination of charge rejection, solubility-diffusion and sieving through micropores (Judd, 2011), and most of the dyes used by the industrial partner are anionic.

Comparison of the NF and RO permeate results to that of the potable water received and currently being used by the industrial partner, showed that most of the parameters, with the exception of conductivity, TDS and COD, were below or close to that of the industrial partner. Optimising the efficiency of the NF/RO system adjusting the cross flow velocity to increase the hydraulic retention time (HRT) of the UF permeate within the system would decrease the conductivity and TDS, as seen in Figure 4.2(A and B). Days 13 to 17 showed improved conductivity and TDS removal efficiencies after decreasing the cross flow velocity on day 13. A decrease in conductivity, TDS and COD would bring the parameters analysed to below or close to the values recorded for the industrial partner’s potable water and therefore make both the NF and RO permeate re-usable. However, if colour removal from textile wastewater is the primary objective, then an MBR coupled with NF is the treatment method to utilise; if re-use if the primary objective then treatment of the UF permeate with RO would successfully remove both the residual colour and salts which is imperative for reclamation of the wastewater for re-use, especially in dyeing processes.

The percentage removal for COD and colour achieved by the UF, NF and RO membranes for the current study were compared to values obtained from literature (Table 4.5) for various types of textile wastewaters. Higher percentage COD removals (i.e. 91.8%, 92.3%, 93%,

**Table 4.5:** Summary of COD and colour removal of various MBR set-ups compared to the pilot-scale UF-dsMBR and NF/RO system

| Set-up | COD removal | Colour removal | Reference |
|---|---|---|---|
| MBR (activated sludge reactor connected to an external crossflow UF unit) | 91.8% | Not indicated | Schoeberl *et al.,* 2004 |
| Aerobic MBR (external UF module with plate and frame membranes) | 93% (average) | 70% - 80% | Lubello and Gori, 2004 |
| Aerobic MBR | 96% | 72% | Badani *et al*., 2005 |
| SBR | 70% | 51% | You *et al.,* 2006 |
| MBR | 79% | 54% | |
| Submerged microfiltration MBR | Not indicated | 68.3% | Hai *et al.,* 2006 |
| Aerobic MBR (activated sludge reactor connected to an external crossflow UF unit) | 60% - 95% | 30% - 99.5% | Brik *et al.,* 2006 |
| UF pretreatment followed by NF | Not indicated | 95% | Fersi & Dhahbi, 2008 |
| Anaerobic SBR | 92.3% | 74.6% | You & Teng, 2009 |
| Aerobic MBR | 5.2% | 9.1% | |
| ***Dual-stage UF-MBR followed by an NF/RO polishing step*** | ***50% - 97% (Average 75%) (UF)***<br>***86% (NF)***<br>***90% (RO)*** | ***29% (UF)***<br>***98% (NF)***<br>***97% (RO)*** | ***Current study*** |

96%) were obtained in the comparative studies. The highest colour removal obtained by the comparative literature was in a range of 30% to 99%, using an aerobic MBR as the treatment method.

## 4.5 Conclusions

The UF-dsMBR system reduced the COD and turbidity of the wastewater by an average of 75% and 94%, respectively, to concentrations of 191 mg/L for COD and 2.7 NTU for turbidity. However, the UF-dsMBR system only removed 28.6% of the colour present in the wastewater. After treatment of the UF permeate with the pilot-scale NF/RO system it was concluded that higher residual colour removal was achieved with NF, 97.4%, than RO, 96.0%. However, treatment with RO achieved higher conductivity and TDS removal, 97.5% and 97.4%, respectively; while NF treatment only achieved removal of 30.4% and 32.5%, respectively. It was therefore concluded that treatment of the UF permeate with RO was preferred to NF, since treatment with RO successfully removed both residual colour and salt from the UF permeate. While treatment with NF removed residual colour, significantly less salts were removed. With the exception of the conductivity, TDS and COD levels in the RO permeate, which were above the potable water values of the industrial partner, all these parameters, with the exception of ammonium were within the drinking water standards and therefore made the RO permeate re-usable in the dyehouse processes.

## 4.6 Summary

The 5 to 10 $m^3$/d pilot plant sidestream dsMBR system, evaluated on site at the industrial partner, successfully treated the continuously changing textile wastewater to within the CCT industrial wastewater discharge standards. Due to the presence of residual colour, as well as high concentrations of TDS and conductivity present after treatment, the UF permeate was treated with NF which was then alternated with RO to evaluate the degree of residual colour and salt removal by these treatment processes individually. Evaluation of colour removal by subsequent NF and alternating RO treatment will be covered in Chapter 5.

# CHAPTER 5

# RESULTS

## COLOUR REMOVAL FROM TEXTILE WASTEWATER USING A PILOT-SCALE UF-DSMBR AND SUBSEQUENT NF/RO SYSTEM

# CHAPTER 5

# COLOUR REMOVAL FROM TEXTILE WASTEWATER USING A PILOT-SCALE UF-DSMBR AND SUBSEQUENT NF/RO SYSTEM

## 5.1 Introduction

The textile industry not only utilises large quantities of water, but produces highly coloured wastewaters polluted with dyes, textile auxiliaries and other chemicals that are generally toxic and resistant to biological treatment methods (El-Gohary & Tawfik, 2009; Brik *et al.,* 2006; Badani *et al.,* 2005; Chakraborty *et al.,* 2003; Barclay & Barclay, 2002; Ledakowicz *et al.,* 2001). Textile wastewater is characterised by significant fluctuations in the COD and biological oxygen demand (BOD) concentrations, pH, colour and salinity (Dos Santos *et al.,* 2007) due to the composition of the dye wastewater varying with the textile produced (O'Neill *et al.,* 1999b). The release of textile wastewater into the environment is undesirable since the dyes and their breakdown products 1) affect the aesthetics of aquatic environments due to highly visible colour; 2) have an acute and/or chronic effect on the organisms exposed to them depending on the length of exposure; 3) interfere with the growth of bacteria that degrade water impurities as the dyes adsorb and reflect the sunlight entering the water, effecting the transparency and gas solubility in water bodies; and 4) are toxic and mutagenic or carcinogenic to life (Firmino *et al.,* 2010; Dos Santos *et al.,* 2007; Slokar & Le Marechal, 1998).

Azo dyes are the largest chemical class of synthetic dyes (Kodam & Gawai, 2006; Kodam *et al.,* 2005) accounting for 60 to 70% of the dyes used in the textile dyeing industry. These dyes are electron deficient, xenobiotic compounds that are recalcitrant to aerobic degradation and are therefore a major problem in the treatment of textile wastewater (Firmino *et al.,* 2010; Hai *et al.,* 2006; Kodam *et al.,* 2005). The azo dyes commonly utilised in the textile industry include reactive, acid and direct dyes. The reactive azo dyes contain one to four azo bonds that are reduced under anaerobic biological conditions (You & Teng, 2009; Kodam & Gawai, 2006; Sponza & Işik, 2005; Libra & Sosath, 2003), resulting in the decolouration of the azo dye (Kodam & Gawai, 2006; Bonakdarpout *et al.,* 2011). The products of this anaerobic degradation are colourless aromatic amines that can be readily degraded (i.e. mineralised) via aerobic digestion (Dos Santos *et al.,* 2007; Kodam & Gawai, 2006; Sponza & Işik, 2005; Van Der Zee & Villaverde, 2005).

Textile wastewater can be treated in two ways: either by 1) chemical or physical methods for dye removal (coagulation, flocculation, sedimentation, activated carbon adsorption, ion exchange, reverse osmosis filtration for example), or by 2) biodegradation (activated sludge, sequencing batch reactor for example) (You & Teng, 2009; Badani *et al.,* 2005; Chakraborty *et al.,* 2003; Gupta *et al.,* 2000; Kural *et al.,* 2001; O'Neill *et al.,* 1999c; Slokar & Le Marechal, 1998). The treatment of textile wastewater includes the reduction of COD, BOD, total organic carbon (TOC), adsorbable organic halide (AOX), temperature and pH, as well as the reduction of dye concentrations (Slokar & Le Marechal, 1998). Therefore, it is necessary to find an effective treatment method capable of removing both strong colour and the toxic organic compounds from textile wastewater (Ledakowicz *et al.,* 2001). Strong colour is the hardest component to treat because the modern textile dyes have a high degree of chemical and photolytic stability, colour fastness, and resistance to degradation (Firmino *et al.,* 2010; O'Neill et al., 1999b; O'Neill *et al.,* 1999c). However, the non-biodegradable compounds (i.e. xenobiotic compounds) cannot be removed solely by biological treatment (Ledakowicz *et al.,* 2001).

Membrane techniques show great promise in the treatment of textile wastewater, as they either remove the dyestuff allowing for re-use of the auxiliary chemicals used for dyeing or concentrate the dyestuffs and auxiliaries producing purified water (Chakraborty *et al.,* 2003). Ultrafiltration (UF) effectively removes particles and macro molecules from textile wastewater (Marcucci *et al.,* 2001). However, the removal of polluting substances such as colour is normally between 31 and 76% (Allegre *et al.,* 2006). Nanofiltration (NF) separates low molecular weight (< 1,000) organic compounds, divalent salts or large monovalent ions such as hydrolysed reactive dyes and dyeing auxiliaries from textile wastewater, while reverse osmosis (RO) removes all mineral salts, hydrolysed reactive dyes, chemical auxiliaries, ions and larger species from the wastewater (Allegre *et al.,* 2006; Chakraborty *et al.,* 2003; Marcucci *et al.,* 2001). However, the higher the salt concentration of the wastewater, the more important osmotic pressure becomes when using RO membranes and the greater the energy requirements (Allegre *et al.,* 2006). Successful treatment of textile wastewater would allow water re-use, reduced pollution of surface waters and reduced bioaccumulation of dyes and other dyeing chemicals within the environment (Slokar & Le Marechal, 1998). Membrane bioreactors (MBRs), which combine a biological process with membrane separation (Brik *et al.,* 2006; Badani *et al.,* 2005) are becoming the technology of choice since they provide effluent with high quality, low chemical pollutants, a small footprint, and significant reduction in bacteria and viruses (Brik *et al.,* 2006; Hai *et al.,* 2006; Schoeberl *et al.,* 2005). The use of MBRs for the treatment of textile wastewater has not been applied at industrial scale in South Africa and is still in need of further research. Therefore, this section of the study investigated the removal of colour and recovery clean water from industrial textile wastewater containing azo dyes using a pilot-scale dual-stage sidestream MBR (UF-dsMBR) for the treatment of continuously changing industrial textile wastewater instead of laboratory prepared synthetic non-changing wastewater.

## 5.2 Objectives

The objectives of this study were to: 1) determine the removal efficiencies (i.e. treatment efficiency) for all the parameters measured during this study but with a focus on colour removal; 2) evaluate the cleavage of the azo bonds during the anaerobic stage; 3) establish the presence of dye mineralisation within the aerobic environment; and 4) determine whether the RO treated water is re-usable by the industrial partner. The above objectives were met by taking the hydraulic retention time (HRT) of both the UF-dsMBR and NF/RO pilot plants into account while operating the NF/RO system with the RO membrane, which was not covered in Chapter 4.

## 5.3 Materials and Methods

As was previously explained the system design was based on a modified version of the traditional UCT-configured biological nutrient removal (BNR) conventional activated sludge (CAS) system (Du Toit *et al.,* 2010; ∅stgaard *et al.,* 1997) and consisted of two phases: 1) the sidestream UF-dsMBR, followed by 2) UF permeate treated with NF for residual colour removal and then UF permeate treated with RO for salt removal, imperative for the successful reclamation of the wastewater for re-use of the water, particularly in dyeing processes.

### 5.3.1 Phase 1: UF-dsMBR pilot plant set-up and operation

The pilot-scale dsMBR system (refer to Figure 3.1 in Chapter 3), was designed, constructed, commissioned and operated on-site for 250 days. The colour removal part of the study was conducted over the last 17 days of operation. The system was robust and non-sterile with the textile wastewater treated in a series of tanks followed by two 3 m high 5.1 m$^2$ Norit X-flow Airlift$^{TM}$ UF-membrane modules. The wastewater was pumped from the sump to the equalisation tank, which acted as a buffer tank. Therefore, the colour of the wastewater in the sump was not the same as the colour of the wastewater stored in the equalisation tank.

The system was designed to incorporate anaerobic cleavage of the azo bonds upstream of conventional anoxic denitrification and aerobic nitrification and mineralisation of the aromatic amines with COD reduction. The UF-membrane modules removed any organic material remaining in the wastewater after biological treatment. Recycle of the mixed liquor, via a Lowara CEA 210/2 centrifugal pump (refer to Figure 3.4 in Chapter 3), containing the acclimated microbial consortia to the aerobic and anoxic tanks was facilitated by the UF-modules. Each module consisted of 109 tubular UF-membranes with a diameter of 5 mm and surface area of ~0.046 m$^2$ each. Treated and filtered UF permeate was fed to the polishing final treatment step (Phase 2), a pilot-scale NF/RO system for the removal of residual colour, salts and total suspended solids (TSS).

#### 5.3.1.1 Inoculation and operation

The UF-dsMBR system was designed to operate with a feed flow rate, ranging from 56 to 403 L/h, into the anaerobic tank and corresponding hydraulic retention times (HRT) for each of the biological stages in the systems. The average feed and permeate pressures to the UF-membrane modules were maintained at 49 and 11 kPa, respectively, with an average pressure difference of about 34 to 38 kPa across the membranes. In order to ensure permeation occurred from the lumen to the shell-side, across the membranes, the transmembrane pressure (TMP) was maintained at a pressure greater than atmospheric pressure (i.e. 101.3 kPa).

For the duration of this section of the study the feed flow rate into the anaerobic tank was set at 100 L/h. The corresponding average HRT for each biological stage was 7.1 $h^{-1}$ (anaerobic tank), 5.6 $h^{-1}$ (anoxic tank) and 13.4 $h^{-1}$ (aerobic tank). The HRT of each biological stage remained constant, therefore, the only parameter changing during this study was the feed composition of the textile wastewater fed into the anaerobic tank, which was dependent on what the industrial partner was producing. Five sample sets (each set consisting of an anaerobic feed, anaerobic tank, anoxic tank, aerobic tank, UF permeate, RO permeate sample), were collected at the set feed flow rate, over a three week period. The samples were taken at times corresponding to the HRT of each stage. The sample sets were subsequently labelled HRT1, HRT2, HRT3, HRT4, and HRT5. For the sample set labelled HRT1 no RO permeate sample was taken as at that stage the NF/RO pilot-scale system was not in operation yet.

### 5.3.2 Phase 2: Subsequent reverse osmosis (RO) treatment

The UF permeate was continuously fed into a holding tank and was combined with the brine from the RO system. During this section of the study only the spiral wound (XUS-SW30XHR-2540) RO membrane was in use. The UF permeate combined with RO brine was fed to the RO system at an average flow rate of 0.8 $m^3/h$, with an average feed pressure of 12.6 bar, 0.8 bar differential pressure, a CFV of 4.62 m/s and an average flux of 5 $L/m^2h$.

### 5.3.3 Analytical methods

The representative samples, taking the HRT into account, of each biological treatment stage, as well as permeate from the UF and NF/RO systems were analysed to quantify the colour of the combination of dyes in the samples using two spectrophotometric methods: 1) the colour Hazen method (Platinum-cobalt standard method analogous to APHA 2120B, DIN53509) (Hongve & Åkesson, 1996), measuring the true colour of samples (Appendix K); and 2) the American dye manufacturing index (ADMI) method (Greenberg *et al.,* 1985), measuring the colour in ADMI units (Appendix L).

For the colour Hazen method, the samples were first filtered through a 0.22 μm syringe filter before using a NOVA 60 Spectroquant to read the true colour of the samples at 350 nm (Hongve & Åkesson, 1996). All true colour readings were measured using a 10 mm glass quartz cuvette.

Describing textile wastewater in terms of absorbance and ADMI instead of dye concentration is useful when describing a pollutant since different dyes result in different intensities and colours. ADMI and absorbance changes with the type of dye used. However, there is no direct relationship between absorbance, ADMI and the dye concentrations (O'Neill *et al.,* 1999c). There are a number of sub-divisions for the ADMI tristimulus filter method used for the analysis of true colour in coloured aqueous wastewaters, namely: 1) the weighted ordinate method (Allen *et al.,* 1973); 2) the 10 or 30 ordinate method (O'Neill *et al.,* 1999c; Greenberg *et al.,* 1985); 3) the 4/6 wavelength method (Yu *et al.,* 2006); and 4) the 3/31 wavelength method (Kao *et al.,* 2001). The 10 ordinate method is fairly accurate, however for increased accuracy all 30 ordinates are used (Greenberg *et al.,* 1985). The 10 ordinates form part of all 30 ordinates. For this study, the 10 ordinate method (Greenberg *et al.,* 1985) was used for determination of ADMI true colour values. The samples were filtered to remove excess quantities of suspended solids using a glass gooch filtering crucible fitted to a flask connected to a vacuum pump. Glass fibre filter paper was used as the filtration medium since polymer membranes absorbed the dye. Once filtered, the samples were analysed using a Cary 300 Bio UV-VIS spectrophotometer. The photometric scan determined the absorbance of each sample at 10 different predetermined wavelengths (i.e. 10 ordinate method), indicated in Table 5.1, for the columns X, Y and Z. From the absorbance values obtained the % transmittance, tristimulus values for X, Y, and Z, Munsell values, Adams Nickerson colour difference (DE) and ADMI were calculated (Greenberg *et al.,* 1985).

The samples were also analysed daily for pH, total dissolved solids (TDS) and conductivity using a calibrated PCSTestr 35 multiparameter (Appendix C), and every second day for the following:

- ammonium ($NH_4$) (Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001) (Appendix E);
- COD (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 F);
- nitrate ($NO_3$) (Merck Spectroquant Nitrate cell test; Cat. No. 1.14773.0001) (Appendix G);
- phosphate ($PO_4$) (Merck Spectroquant Phosphate cell test for orthophosphate and total phosphorus, Cat. No. 1.14543.0001) (Appendix H);
- TSS (ESS Method 350.2) (Appendix I); and

- turbidity (TN-100 turbidimeter, ISO 7027 compliant nephelometric method) (Appendix J).

**Table 5.1:** Selected ordinates for spectrophotometric colour determinations (Greenberg *et al.,* 1985)

| Wavelength No. | X | Y | Z |
|---|---|---|---|
| **1** | 435.5 | 498.5 | 422.2 |
| **2** | 461.2 | 515.2 | 432.0 |
| **3** | 544.3 | 529.8 | 438.6 |
| **4** | 564.1 | 541.4 | 444.4 |
| **5** | 577.4 | 551.8 | 450.1 |
| **6** | 588.7 | 561.9 | 455.9 |
| **7** | 599.6 | 572.5 | 462.0 |
| **8** | 610.9 | 584.8 | 468.7 |
| **9** | 624.2 | 600.8 | 477.7 |
| **10** | 645.9 | 627.3 | 495.2 |

Mineralisation was determined using dissolved organic carbon (DOC) and $NH_4$. In order to verify mineralisation, a decrease in DOC below the DOC value of the wastewater being treated must be shown (Bonakdarpour *et al.,* 2011).

- TOC and DOC (both determined using Merck Spectroquant TOC cell tests, Cat. No. 1.14878.0001). TOC is a more direct expression of the total organic content of wastewater than either BOD or COD. DOC is the fraction of TOC that passes through a 0.45 µm filter (Greenberg *et al.,* 1985) (Appendix M).

## 5.4 Results and Discussion

The average values for all the parameters measured for the HRT sample sets (i.e. anaerobic feed, anaerobic tank, anoxic tank, aerobic tank, UF permeate, and RO permeate) are summarised in Tables O.13 to O.18 in Appendix O. Standard deviation error bars have been included in all the figures.

### 5.4.1 Wastewater composition analysis

**Table 5.2:** Wastewater composition over the period when the HRT sample sets were taken (last 13 days of operation)

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **ADMI** | ADMI units | 195 | 2,070 | 659 |
| **True colour** | mg Pt/L | 63.7 | 288 | 129 |
| **Ammonium** | mg/L | 7.8 | 25.1 | 11.4 |
| **COD** | mg/L | 728 | 1,033 | 802 |
| **Conductivity** | μS/cm | 995 | 2,295 | 1,583 |
| **DOC** | mg/L | 78.4 | 108 | 90.3 |
| **Nitrate** | mg/L | 1.2 | 2.3 | 1.8 |
| **pH** | - | 7.8 | 10.4 | 9.7 |
| **Phosphate** | mg/L | 0.38 | 1.3 | 0.89 |
| **TDS** | ppm | 963 | 1,632 | 1,119 |
| **Temperature** | °C | 17.2 | 25.9 | 23.6 |
| **TOC** | mg/L | 90.6 | 108 | 101 |
| **TSS** | mg/L | 16.0 | 46.0 | 30.2 |
| **Turbidity** | NTU | 16.4 | 26.9 | 22.9 |

The biggest problem facing the industry was the amount of colour, measured as true colour and ADMI colour, present in the wastewater and influencing the re-usability of textile wastewater. The wastewater analysed over the sampling period for this study reached a maximum colour of 288 mg Pt/L with an average of 129 mg Pt/L. No value is provided for colour in the City of Cape Town (CCT) wastewater discharge standards, but the SANS 241-1:2011 (South Africa. Department of Water Affairs, 2011) drinking water specification is ≤ 15 mg Pt/L. The composition of the wastewater stream for this part of the study (Table 5.2) was also characterised by a COD range of 728 to 1,033 mg/L and an average BOD of 192.5 mg/L. The maximum conductivity and TDS measured, 2,295 μS/cm and 1,632 ppm, respectively, did not meet the CCT wastewater and industrial effluent discharge standards (South Africa (Western Cape), 2006).

The composition of the wastewater (Table 5.2) discharged into the sump was continually changing depending on the process being utilised in the dyehouse of the textile company. The discharged wastewater varied from “light” to “medium” to “dark” colour due to the erratic trends in dye consumption mainly dependent on trends in the supply and demand of the textile fabric. The wastewater colour discharged from the equalisation tank to the anaerobic tank was classified based on ADMI values (Table 5.3). For this study, wastewater with an ADMI value below 500 ADMI units was classified as light in colour; 500 to 1,500 ADMI units was classified as medium in colour; and above 1,500 ADMI units was classified as dark in colour. The ADMI colour and true colour of 5 sample sets (HRT1, HRT2, HRT3, HRT4 and HRT5 each containing an anaerobic feed, anaerobic tank, anoxic tank, aerobic tank, UF permeate and RO permeate) was determined.

**Table 5.3:** Colour classification based on the ADMI values of the wastewater fed from the equalisation tank to the anaerobic tank

| Samples | ADMI of equalisation discharge (ADMI units) | | Colour classification of the wastewater |
|---|---|---|---|
| **HRT1** | 1,027 | | Medium colour |
| **HRT2** | 1,649 | Average: 1,956 | Dark colour |
| **HRT3** | 1,829 | | Dark colour |
| **HRT4** | 2,388 | | Dark colour |
| **HRT5** | 290 | | Light colour |

### 5.4.2 Colour removal

It should be noted that the percent reduction (efficiency of colour removal) was divided into two: 1) the individual reduction, based on the percent decrease in ADMI from stage to stage; and 2) the overall reduction, based on the total percent decrease from the initial ADMI of the wastewater discharged from the equalisation tank (i.e. anaerobic feed) up to the respective stages. The individual reduction indicates the amount of residual colour removed during each stage, while the overall reduction indicates the overall removal with regard to the initial colour of the wastewater entering the system. Therefore, the difference in colour removal between stages indicates how much colour was removed during a respective stage.

**Table 5.4:** ADMI reduction for the respective treatment stages of: (A) dark colour (average of HRT2, HRT3 and HRT4); (B) medium colour (HRT1); and (C) light colour (HRT5)

| | Treatment stages | ADMI | % Reduction efficiency | |
|---|---|---|---|---|
| | | | Individual | Overall |
| **A** | **Anaerobic feed** | 1,956 | - | - |
| | **Anaerobic tank** | 1,014 | 48.2 | 48.2 |
| | **Anoxic tank** | 602 | 40.6 | 69.2 |
| | **Aerobic tank** | 657 | - | 66.4 |
| | **UF Permeate** | 572 | 12.9 | 70.8 |
| | **RO Permeate** | 18 | 96.9 | 99.1 |
| **B** | **Anaerobic feed** | 1,027 | - | - |
| | **Anaerobic tank** | 372 | 63.7 | 63.7 |
| | **Anoxic tank** | 484 | - | 52.9 |
| | **Aerobic tank** | 409 | 15.6 | 60.2 |
| | **UF Permeate** | 463 | - | 54.9 |
| **C** | **Anaerobic feed** | 290 | - | - |
| | **Anaerobic tank** | 581 | - | - |
| | **Anoxic tank** | 406 | 30.2 | - |
| | **Aerobic tank** | 387 | 4.6 | - |
| | **UF Permeate** | 423 | - | - |
| | **RO Permeate** | 28 | 93.4 | 90.4 |

#### 5.4.2.1 Dark colour

The initial wastewater (i.e. anaerobic feed) for HRT2, HRT3 and HRT4 sample sets were all classified as a dark colour. The average ADMI value was 1,956 ADMI units (Tables 5.3 and 5.4A) and therefore above 1,500 ADMI units. The highest individual residual colour removal, 96.9%, occurred during the RO stage followed by 48.2% residual colour removal during the anaerobic biological stage (Table 5.4A). The least amount of residual colour, 12.9%, was removed during the UF stage. The highest overall colour removal for the biological treatment occurred during the anaerobic stage with 48.2% overall reduction in colour, while only 21% overall colour removal occurred during the anoxic stage (Table 5.4A). The amount of colour present in the aerobic stage showed an increase (i.e. 2.8%) due to the recycle of mixed liquor, containing residual colour, from the UF-membrane modules. The RO stage showed an overall removal of 29.7%, while collectively an overall removal of 99.1% was observed. The measured ADMI and true colour values were plotted graphically for the various treatment stages (Figure 5.4A). Even though two different methods were utilised to determine the colour during the different treatment stages, the same trend for colour reduction was observed. Figure 5.1 is a visual representation of colour removal through the various treatment stages for a dark coloured wastewater treated by the semi-automated UF-dsMBR pilot plant system and subsequent NF/RO system.

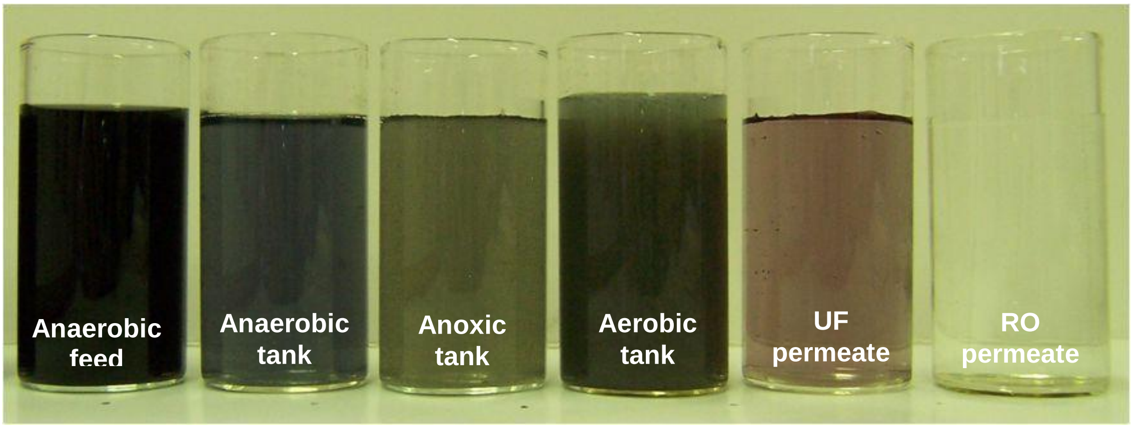


**Figure 5.1:** Visual representation of colour removal for a dark colour

#### 5.4.2.2 Medium colour

The initial wastewater (i.e. anaerobic feed) for the HRT1 sample set was classified as a medium colour since the ADMI value was 1,027 ADMI units (Tables 5.3 and 5.4B). The highest individual and overall colour removal of 63.7% occurred during the anaerobic biological stage (Table 5.4B). During the anaerobic stage, cleavage of the azo bonds occurred that resulted in aromatic amines. Individually the least amount of residual colour was removed during the aerobic stage (15.6%). The colour removal noticed during the aerobic stage was a combination of the removal of dyes that did not contain azo bonds and the mineralisation of the aromatic amines formed during the anaerobic stage. Collectively an overall colour removal of 54.9% was observed. The measured ADMI and true colour values were plotted graphically with respect to the various treatment stages (Figure 5.4B). Again, the same trend for ADMI and colour Hazen reduction was observed. Figure 5.2 is a visual representation of colour removal through the various treatment stages for a medium coloured wastewater treated by the semi-automated UF-dsMBR pilot plant system.

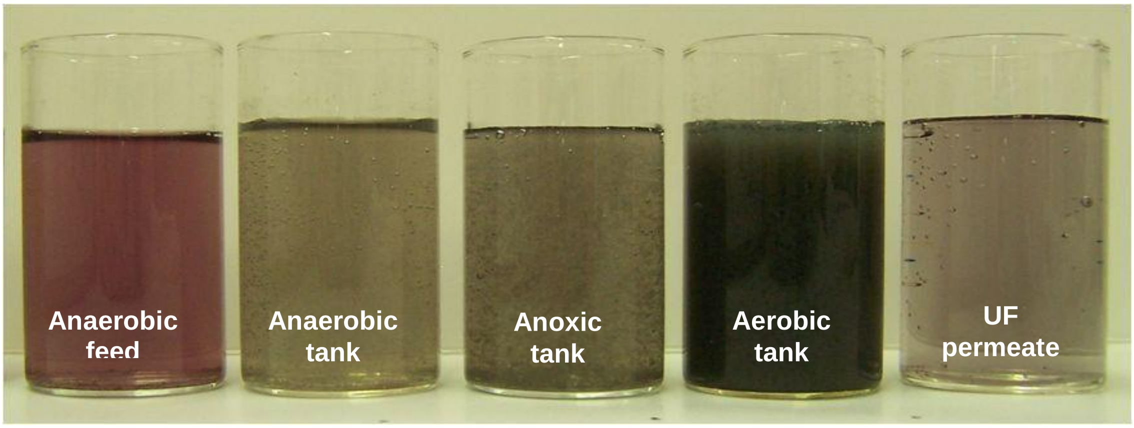


**Figure 5.2:** Visual representation of colour removal for a medium colour

#### 5.4.2.3 Light colour

The initial wastewater (i.e. anaerobic feed) for the HRT5 sample set was classified as a light colour since the ADMI value was 290 ADMI units (see Tables 5.3 and 5.4C) and therefore below 500 ADMI units. An increase in ADMI value was observed between the anaerobic feed and the anaerobic tank; therefore the percentage removal was negative (Table 5.4C). Due to the effect of residence time distribution in non-plug flow reactors this increase in colour was attributed to the presence of a dark colour (i.e. HRT4) in the anaerobic tank prior to the light colour being discharged. Therefore, with the exception of the RO stage, all the treatment stages showed a negative overall colour reduction. The highest individual residual colour removal of 93.4% was observed during the RO stage with a collective overall colour removal of 90.4%. However, the biological stage with the highest residual colour reduction was the anoxic stage, during which a colour reduction of 30.2% was observed. The amount of colour present in the aerobic stage showed an increase (i.e. 25.6%) due to the recycle of mixed liquor, from the UF-membrane modules, containing darker residual colour. Figure 5.3 is a visual representation of colour removal through the various treatment stages for a light coloured wastewater treated by the semi-automated UF-dsMBR pilot plant system and subsequent NF/RO system.

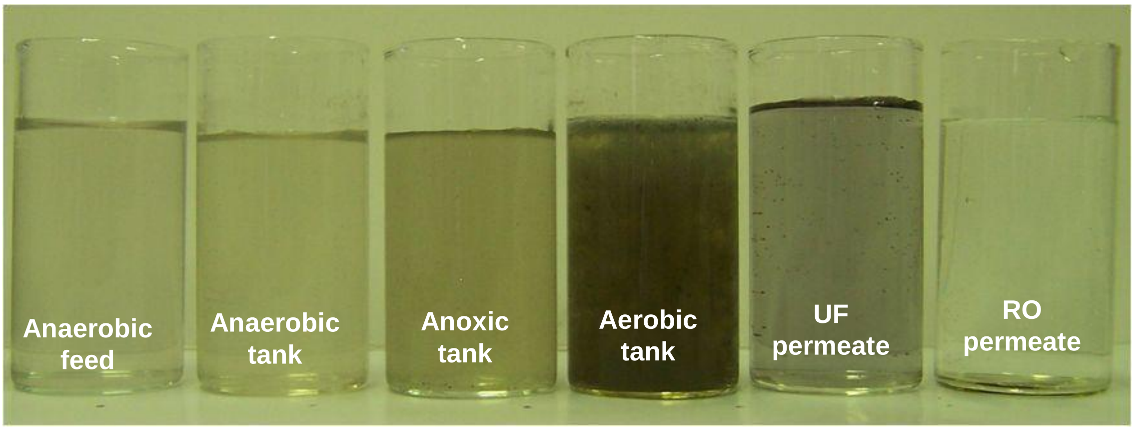


**Figure 5.3:** Visual representation of colour removal for a light colour

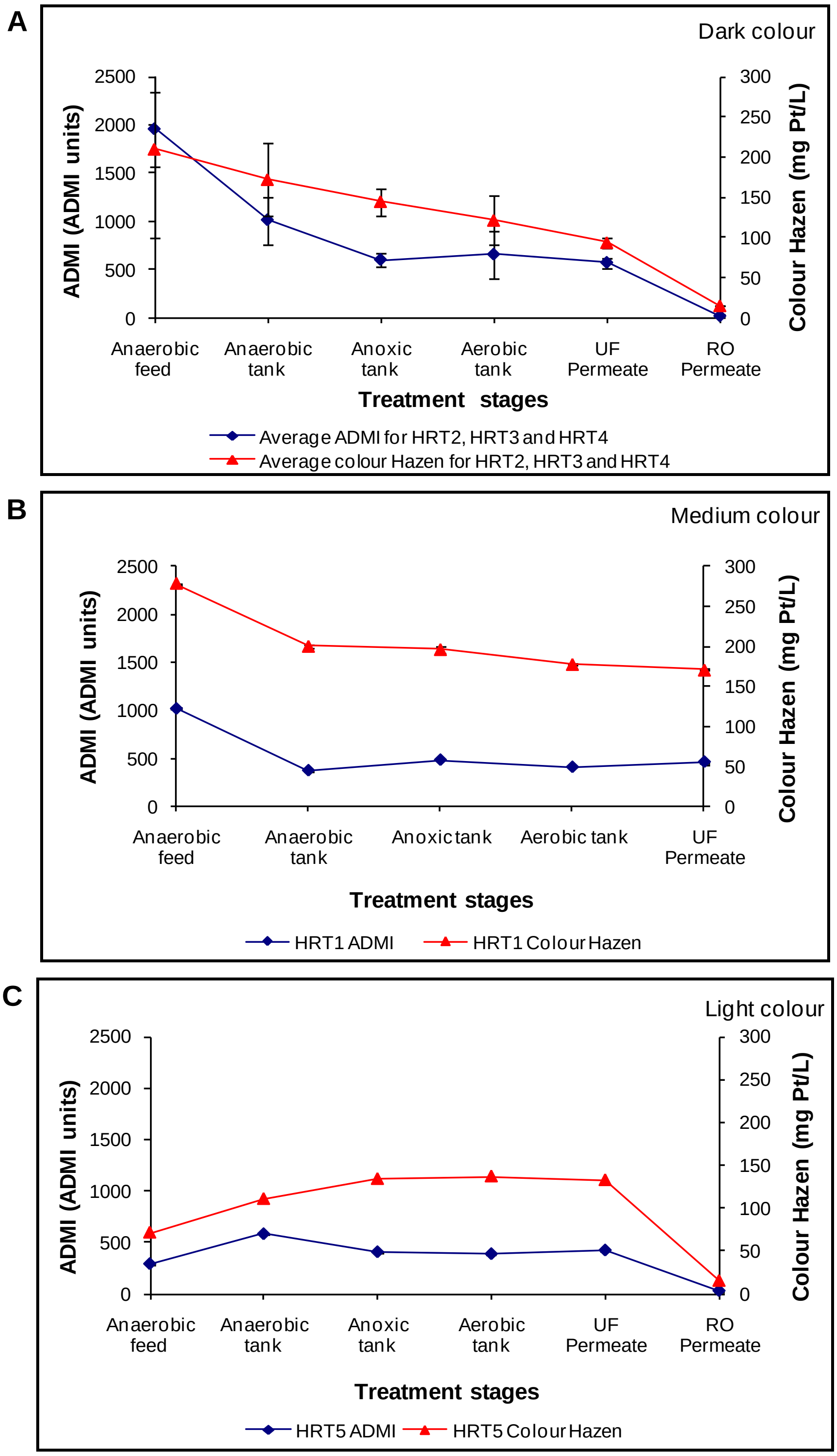


**Figure 5.4:** ADMI and colour Hazen values during the different treatment stages for: (A) dark colour (average of HRT2, HRT3 and HRT4); (B) medium colour (HRT1); and (C) light colour (HRT5)

The measured ADMI and true colour values were plotted graphically with respect to the various treatment stages (Figure 5.4C). Again, with the exception of the colour Hazen value obtained for the anaerobic stage, a similar trend for colour reduction was observed.

### 5.4.3 Colour removal efficiency

From Figures 5.4A to 5.4C it was clear that the respective biological treatment stages exhibit some degree of dye removal and hence a reduction in colour, with the exception of the light coloured wastewater being discharged after a dark coloured wastewater. From the ADMI results obtained for dark, medium, and light coloured wastewaters the following was observed: 1) colour removal did occur during the UF-dsMBR stage of the treatment system, however, the RO stage removed a higher percentage of colour; 2) for both medium and dark coloured discharged wastewater the maximum individual colour removal occurred during the anaerobic biological stage. This indicated the presence of azo dyes in the textile wastewater, since the purpose of the anaerobic tank was the degradation of azo bonds found in azo dyes resulting in decolouration of the azo dye (Bonakdarpour *et al.,* 2011; Kodam & Gawai, 2006). Therefore, the higher the concentration of azo dyes in the wastewater, the higher the colour removal in the anaerobic stage.

An independent t-test was done between the ADMI values obtained for both the UF permeate and RO permeate of the dark, medium and light coloured HRT sample sets (Table 5.5). The t-test measures the significant difference between two sets of data, either dependently or independently, by comparing the means of the two points. The t-value, which can be negative as absolute values are not used when calculating the t-value, and p-value take both the sample size and standard deviation into account. The t-test is interpreted using p-values.

**Table 5.5:** Results for the t-test

| t-test between ADMI values of the: | t-value | p-value |
|---|---|---|
| **Dark and medium coloured UF permeate** | 2.50 | 0.05 |
| **Dark and light coloured UF permeate** | 3.47 | 0.02 |
| **Medium and light coloured UF permeate** | 4.58 | 0.04 |
| **Dark and light coloured RO permeate** | -3.08 | 0.02 |

A p-value smaller than and equal to 0.05 ($p \leq 0.05$) was interpreted as significant as it indicated a probability of 5 % or less difference between the ADMI results obtained for the different coloured sample sets and treatment stages. The p-values, in Table 5.5, indicate that the difference between the ADMI values of the UF permeate for the dark, medium and light coloured sample sets was significant, as the p-values were below 0.05 or equal to 0.05. The p-values also indicate that the difference between the ADMI values of the RO permeate for the dark and light coloured sample sets was significant, as the p-values were below 0.05. Therefore, even though different coloured wastewater was fed to Phase 1 the results obtained, with regard to colour removal in both the UF and RO permeate was significant and could be compared.

Ferraz *et al*. (2011) operated an upflow anaerobic sludge bed (UASB) combined with a submerged aerated biofilter (SAB) system in the treatment of textile wastewater from a jeans factory. Using a combination of various HRT and organic loading rates (OLR) the individual colour removal efficiency for the UASB ranged from 50% to 64%, with an overall colour removal range of 61% to 86% after the SAB. In the current study individual colour removal efficiencies were obtained ranging from 5% to 97% depending on the colour of the wastewater discharged and the colour of the wastewater already in the system. Brik *et al.* (2006) (tubular UF-membrane with a 0.28 $m^2$ filter area and 15 kDa cut-off); Badani *et al.* (2005) (tubular UF-membrane with 25 nm mean pore size), and Schoeberl *et al.* (2004) (tubular crossflow UF-membrane with a 0.28 $m^2$ filter area and 15 kDa cut-off) all utilised sidestream MBR systems to treat industrial mixed textile wastewater (refer to Table 2.7 in section 2.4.7 of Chapter 2). Badani *et al.* (2005) and Brik *et al.* (2006) obtained colour removal of 70% and > 87% with the side stream MBR system, respectively. Kim *et al.* (2004) performed a study on the colour removal efficiencies of reactive dyes with a NF and RO combined MBR system. The results showed a > 90% removal of reactive black 5 and reactive blue 49 dyes and a 76% removal of the reactive blue 19 dye. Nowak and Winnicki (1986) reported 97% dye, > 780 molecular weight, removal, with a tubular cross flow UF-membrane. During the current study the sidestream UF-dsMBR system used to treat industrial textile wastewater containing reactive, vat, 1:2 metal complex and dispersed dyes achieved an overall colour removal (Table 5.6) of 99.1% and 90.4% in the dark and light HRT sample set, respectively and 54.9% for the medium coloured HRT sample set that stopped at the UF permeate. Therefore, although the composition of the feed wastewater was continuously changing during the study period the results obtained show that the UF-and RO permeate composition remained consistent.

The overall removal results (refer to Table 5.6) obtained for the HRT sample sets, accounting for the flow rates and tank volumes, provide a more accurate representation of the treatment process since both the flow rate and tank volume influences the amount of time the wastewater spends in a treatment stage, which has a direct impact on the efficiency of the treatment process. Taking all the samples of a sample set simultaneously only indicates that moment in the treatment process.

**Table 5.6:** Overall percentage removal of the hydraulic retention time sample sets

| Parameter | Dark coloured wastewater (HRT2, HRT3 and HRT4): | | | Medium coloured wastewater (HRT1): | | | Light coloured wastewater (HRT5): | | | SANS 241-1:2011 Drinking water specification* |
|---|---|---|---|---|---|---|---|---|---|---|
| | Anaerobic feed | RO permeate | % Overall Removal | Anaerobic feed | UF permeate | % Overall Removal | Anaerobic feed | RO permeate | % Overall Removal | |
| **Temperature ($^0$C)** | 24.9 | 25.0 | N/A | 16.2 | 21.3 | N/A | 26.2 | 24.2 | N/A | ni |
| **pH** | 11.1 | 8.8 | N/A | 11.1 | 8.7 | N/A | 7.4 | 9.5 | N/A | ≥ 5 - ≤ 9.7 |
| **Conductivity (µS/cm)** | 3,848 | 305 | 92.1 | 1,079 | 6,805 | - | 3,012 | 175 | 94.2 | ≤ 1,700 |
| **TDS (ppm)** | 7,710 | 215 | 97.2 | 759 | 6,730 | - | 2,020 | 125 | 93.8 | ≤ 1,200 |
| **COD (mg/L)** | 579 | 78.3 | 86.5 | 738 | 80.0 | 89.2 | 715 | 87.0 | 87.8 | ni |
| **Ammonium (mg/L)** | 11.8 | 8.8 | 25.4 | 10.3 | 10.3 | 0 | 18.7 | 11.0 | 41.3 | ≤ 1.5 |
| **TSS (mg/L)** | 40.0 | 0.33 | 99.2 | 51.0 | 15.0 | 70.6 | 34.0 | 0.0 | 100 | ni |
| **Turbidity (NTU)** | 24.3 | 0.58 | 97.6 | 43.1 | 0.74 | 98.3 | 30.5 | 0.0 | 100 | ≤ 5.0 |
| **Phosphate (mg/L)** | 1.4 | 0.92 | 36.3 | 1.9 | 1.1 | 40.5 | 1.6 | 1.2 | 23.7 | ni |
| **Nitrate (mg/L)** | 1.9 | 1.9 | 0 | 2.0 | 1.8 | 12.5 | 1.7 | 1.3 | 26.7 | ni |
| **TOC (mg/L)** | 107 | 14.0 | 6.9 | 107 | 104 | 2.5 | 103 | 8.8 | 91.5 | ≤ 15 |
| **DOC (mg/L)** | 92.8 | 11.3 | 87.8 | 78.4 | 57.0 | 27.3 | 62.7 | 11.5 | 81.7 | ni |
| **True colour (mg Pt/L)** | 399 | 15.4 | 96.1 | 278 | 171 | 38.4 | 68.0 | 13.0 | 80.9 | 20-50 |
| **ADMI (ADMI units)** | 1,956 | 18.3 | 99.1 | 1,027 | 436 | 54.9 | 290 | 27.8 | 90.4 | ni |

*SANS 241-1:2011 Drinking water specification (South Africa. Department of Water Affairs 2011)

### 5.4.4 Overall treatment efficiency for the hydraulic retention time (HRT) samples

The pilot plant UF-dsMBR design was based on a modified version of the traditional UCT-configured BNR CAS system. Therefore, denitrification occurred in the anoxic tank and nitrification occurred in the aerobic tank. When denitrification occurs, the nitrate concentration decreases as nitrate is reduced to nitrogen gas (Judd, 2006), while the ammonium concentration decreases and nitrate concentration increases during the process of nitrification in the aerobic tank. However, during this study in addition to nitrification occurring in the aerobic tank the separate process of mineralisation of the resulting aromatic amines from the cleavage of the azo dyes was also occurring. During the current study in the aerobic tank instead of the ammonium concentration decreasing and the nitrate concentration increasing, as is expected during nitrification, the ammonium concentration increased (Table 5.7). Mineralisation of the azo dyes are complete when the aromatic amines have been biodegraded to carbon dioxide ($CO_2$), water ($H_2O$) and ammonia ($NH_3$) (Sponza & Işik, 2005). Therefore, the occurrence of mineralisation was confirmed in the aerobic tank. However, an increase in the ammonium concentration was also observed between the sump and anaerobic tank, this was due to the 1.7 M urea that was dosed into the anaerobic tank feed line during operation of the pilot plant. Due to the increase in ammonium concentration observed in the anoxic tank it was concluded that mineralisation also occured during this treatment stage. This can be attributed to recycle lines from both the aerobic tank and UF-membrane modules feeding into the anoxic tank.

**Table 5.7:** Average ammonium and nitrate present in the different biological treatment stages for the duration of the study

| Treatment stage | Average ammonium (mg/L) | Average nitrate (mg/L) |
|---|---|---|
| **Sump** | 9.0 | 3.0 |
| **Anaerobic tank** | 21.5 | 3.0 |
| **Anoxic** | 28 | 3.9 |
| **Aerobic** | 31 | 3.9 |

The overall percentage DOC removal (i.e. mineralisation) (Bonakdarpour *et al.,* 2011) was 87.8%, 27.3% and 81.7%, respectively for the dark, medium and light coloured HRT sample sets (Table 5.6). The medium coloured HRT sample set showed a lower percentage removal since this sample set did not include Phase 2 (i.e. the RO membrane). These results, together with the increased ammonium concentration in the aerobic tank therefore, lead to the conclusion that mineralisation of the aromatic amines occurred in the aerobic tank.

O'Neill *et al.* (1999b) noted that following the treatment of a simulated textile wastewater, with an inclined tubular anaerobic digester and upflow anaerobic sludge blanket reactor followed by aerobic treatment, the ADMI of a pH-adjusted sample was lower than a non pH-adjusted sample. This trend was also observed during operation of the pilot plant in this study when dosing the textile wastewater entering the anaerobic tank with 0.5 M phosphoric acid to decrease the pH from ~10 to ~7. Randomly selected wastewater samples showed a decrease in ADMI from an average value of 951 ADMI units to an average of 701 ADMI units (an average reduction of 26.3%). It was also observed that during phosphoric acid dosing the total organic carbon (TOC) values decreased from 106.8 to 90.6 mg/L and 105.5 to 76.3 mg/L for two random samples, while the dissolved organic carbon (DOC) subsequently increased from 78.4 to 86.9 mg/L and 75.8 to 81.6 mg/L for the same two samples.

Even though HRT1 was classified as a medium colour and HRT3 as a dark colour both HRT samples sets showed a significant decrease in DOC during the anaerobic stage, of 16.6% and 41.8%, respectively (Figure 5.5A). Data collected and correlated to the dyehouse processes, not included due to a non-disclosure agreement with the industrial partner, occurring during the operation of Phase 1 indicated that for both HRT1 and HRT3 samples the same vat dyes, specifically basic dyes used to dye acrylic fabric were being used. Therefore, it appears that most of the vat dye mineralisation occurs during the anaerobic stage of the biological treatment system. In Figure 5.5B a decrease in DOC, of 12.5%, 27.2% and 23.1% for HRT2, HRT4 and HRT5, respectively, was observed during the anoxic stage. Both HRT2 and HRT4 were classified as a dark colour, while HRT5 was classified as light in colour. For HRT2, HRT4 and HRT5 the same reactive dyes, specifically 1:2 metal complex dyes used to dye nylon or wool, and vinylsulphone reactive dyes used to dye cotton were being used by the dyehouse. Therefore, it would appear that for reactive dyes most of the dye mineralisation occurs during the anoxic stage. According to a study by Slokar and Le Marechal (1998), the best colour removal achieved using biodegradation occurred when disperse dyes were present in the textile wastewater. More than 50% of the disperse dyes were decolourised within one day using anaerobic and aerobic biodegradation techniques; 74% of the reactive dyes were decolourised using biodegradation, longer time periods of four days were required. However, DOC reduction never exceeded 50%.

Nowak and Winnicki (1986) reported 50% to 60% TOC removal, with a tubular crossflow UF-membrane. During the current study TOC removal of 4.5%, 2.5% and 1.6% was obtained during the UF-dsMBR treatment stage for the dark, medium and light coloured textile wastewaters, respectively. An overall TOC removal of 86.9% and 91.5% was obtained for the dark and light coloured HRT sample sets.

The HRT sample sets which included the NF/RO system (i.e. the dark and light coloured sample sets) showed an overall removal of above 80% for all the parameters measured with the exception of ammonium, phosphate and nitrate. However, all the parameters met the SANS 241-1:2011 (South Africa. Department of Water Affairs, 2011) drinking water specifications.

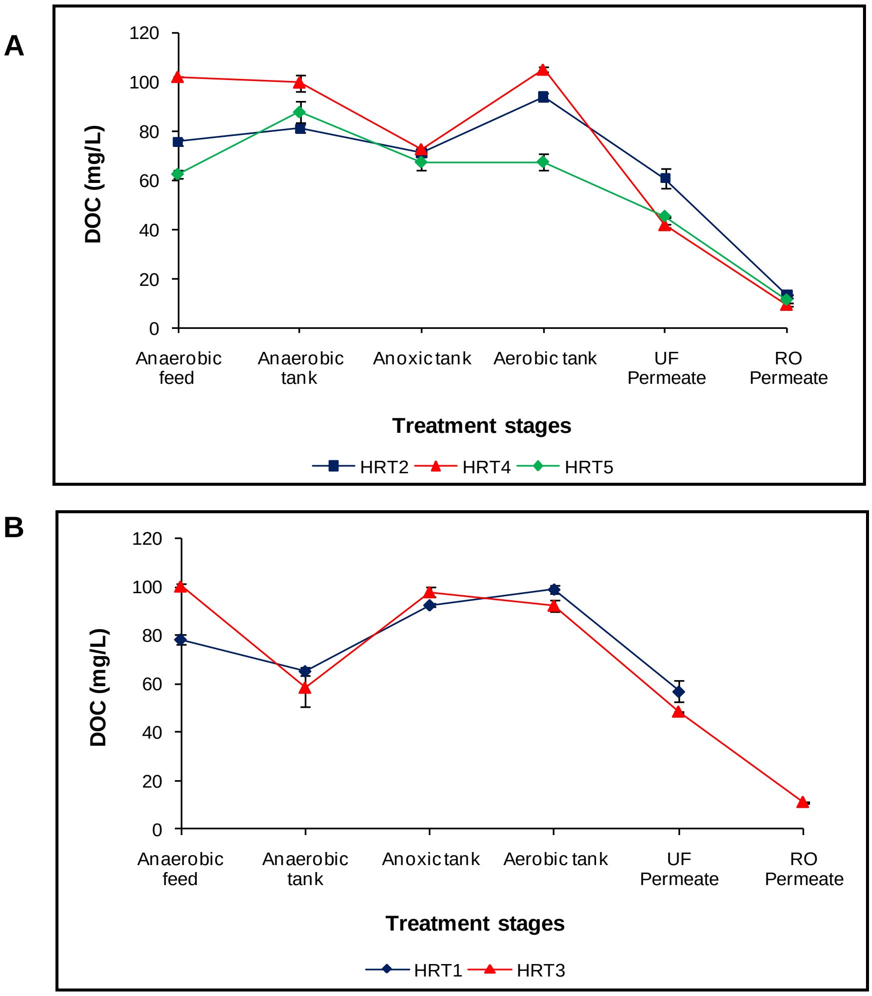


**Figure 5.5:** Dissolved organic carbon trends for different dye classifications: (A) vat dyes, and (B) reactive dyes

## 5.5 Conclusions

After treating the textile wastewater of the industrial partner with the sidestream UF-dsMBR system, the treated water met the City of Cape Town (CCT) industrial wastewater standards, but could not be re-used within the dyeing processes (Chapter 4) due to the presence of residual colour and salt in the UF permeate. All parameters measured for the HRT sample sets which included the NF/RO post treatment system met the SANS 241-1:2011 drinking water specifications and with the exception of ammonium, phosphate, nitrate and TOC showed overall removal efficiencies in excess of 80%. Overall colour removal efficiencies of 99.1% and 90.4% was recorded for the dark and light coloured HRT sample sets, which included the RO post treatment stage, respectively while an overall colour removal efficiency of 54.9% was recorded for the medium coloured HRT sample set which excluded the post treatment stage.

The cleavage of azo bonds was indicated by removal efficiencies of 48.2% and 63.2% for the dark and medium coloured HRT sample sets in the anaerobic treatment stage. Overall DOC removal efficiencies of 87.8%, 27.3% and 81.7%, respectively for the dark, medium and light coloured HRT sample sets together with increased ammonium concentration in the aerobic tank indicated mineralisation of the resulting aromatic amines.

Although the composition of the feed wastewater was continuously changing during the study, consistent reduction of the colour and the other parameters measured in the incoming wastewater was evident in the composition of the UF and RO permeates. From the results obtained it may be concluded that the combined UF-dsMBR and RO membrane in the NF/RO system can successfully treat industrial textile wastewater to within drinking water specifications and may therefore be re-used by the industrial partner in dyeing processes.

It is important to note that the above study was done under limited time and therefore the number of samples taken were not enough for such a complex plant and hence the results obtained not necessarily repeatable. The results therefore only relate to particular conditions and are not of a general application. The author therefore recommends that this be expanded in a future study as it was not possible to do so at this stage due to the pilot plant already being decommissioned.

## 5.6 Summary

Based on the results obtained from operating the pilot-scale UF-dsMBR and NF/RO plants, a proposed full-scale design, together with cost consideration, will be covered in Chapter 6.

# CHAPTER 6

# RESULTS

## PROPOSED DESIGN OF A FULL-SCALE MEMBRANE BIOREACTOR WASTEWATER TREATMENT SYSTEM FOR INDUSTRIAL TEXTILE WASTEWATER

# CHAPTER 6

# PROPOSED DESIGN OF A FULL-SCALE MEMBRANE BIOREACTOR WASTEWATER TREATMENT SYSTEM FOR INDUSTRIAL TEXTILE WASTEWATER

## 6.1 Introduction

This study investigated the application and suitability of a pilot-scale dual-stage sidestream MBR (UF-dsMBR) for the continuous on-site treatment and recovery of variable concentration industrial textile wastewater containing azo dyes, with the aim of re-using the treated wastewater. The design of the pilot-scale dsMBR was geared towards optimal microbial community enrichment with the anaerobic-anoxic-aerobic process designed to incorporate both wastewater azo dye cleavage in a reducing environment followed by oxidation of the resultant aromatic amines, as well as biological nutrient removal through enrichment of the associated microbial consortia using nitrification and denitrification in the aerobic and anoxic tanks, respectively. The dsMBR system served as pre-treatment for the reduction of the wastewater pollution load so that downstream nanofiltration (NF) followed by reverse osmosis (RO) could be incorporated to facilitate a zero liquid discharge strategy, as well as wastewater re-use for the industrial partner. This partner, who currently spends in excess of ZAR 400,000 per annum on water usage and wastewater discharge, calculated from the dyehouse water usage over the 220 days of pilot plant operation in 2010 and the 2010 water rate of ZAR 23.42 per kL of water, for water usage in excess of 50 kL/d.

## 6.2 Objectives

The objective of this chapter was to utilise the data accumulated from the pilot plant over the 220 day period of operation to identify critical scale-up criteria and develop a thorough cost analysis, including capital (CAPEX) and operational (OPEX) expenditure. For a full-scale treatment facility to meet both their current and future water requirements.

## 6.3 Experimental: pilot plant summary

The data collected from the pilot plant over the 220 day operational period at the textile processing plant increased the understanding of the functioning of the treatment system for the given raw wastewater characteristics and the environmental conditions under which it was operated. Refer to Table 4.1 in Chapter 4, section 4.4.2 for a full compositional analysis of the wastewater.

### 6.3.1 Motivation for installing a full-scale textile wastewater treatment plant

The textile processing plant consumes an average of 2,913 kL/month of potable water and discharges approximately 95% (i.e. 2,767 kL/month) of this as wastewater to the municipal sewer. In South Africa since June 2008 till September 2011, the costs of potable water and the wastewater discharge rate increased by 42.9% and 35%, respectively (South Africa, 2011). These tariff increases translated into an increase from ZAR 7.59/kL to ZAR 9.93/kL for potable water and ZAR 5.99/kL to ZAR 7.63/kL for wastewater discharge, an overall cost-to-company increase in total water charges of 31% (ZAR 33,700 to ZAR 44,100 per month) between May 2009 and April 2011. If these trends remain consistent, without wastewater treatment and wastewater re-use, the textile company's water consumption and wastewater discharge will cost approximately ZAR 4.7 million over the next 54 months. The increase in the cost of potable water has pressurised this textile company to find innovative ways to treat their wastewater as they are being severely penalised financially.

## 6.4 Full-scale design

The aim of the proposed full-scale design encompassed the following: 1) remove fine suspended matter (UF-membranes); 2) reduce the chemical oxygen demand (COD) (biological units); 3) remove colour (NF membranes); and 4) remove salt and soluble matter (RO membranes) from the textile wastewater being treated in order for the industrial partner to be able to re-use the reclaimed water in their dyeing processes.

During pilot plant operation the NF and RO membranes were alternated in order to compare the treatment efficiency of both membranes by comparing the quality of the NF and RO permeates obtained during operation However, after analysing the pilot plant experimental results it was decided to design the full-scale plant to incorporate consecutive NF and RO units for colour and excess salt removal, respectively, so that the industrial partner could re-use the treated water. NF was also incorporated as a pre-treatment system for RO.

### 6.4.1 Full-scale design assumptions

The full-scale design was based on the following assumptions:

1. The full-scale plant was designed to have a total operating capacity of 160 $m^3$/d (8 $m^3$/h), even though it will only operate at an average of 97 $m^3$/d (4.05 $m^3$/h). Water consumption by the dyehouse exceeded 200 $m^3$/d (i.e. 200 kL/d) only on three occasions during the 9-month pilot plant operation. Therefore, should the wastewater production by the dyehouse exceed 160 $m^3$/d, the excess will 'overflow' down the drain to the municipality, which is how the wastewater is currently disposed.

2. The current sump and settling tank will serve as the equalisation tank, since the wastewater from the dyehouse first enters the settling tank before passing into the sump; by the time the wastewater enters the sump all pH and colour spikes have been absorbed and the wastewater exiting the sump has an attenuated composition.
3. The dyehouse would be operating continuously with no downtime, since an MBR system is a biological system, it requires a continuous feed supply containing a sufficient carbon source (i.e. COD). During downtime (i.e. maintenance) when the UF, NF, and RO plants, as well as the flocculation chamber and filter press are shut down, the biological tank system will by operating on recycle with the agitator, blower and recycle lines in operation.
4. Wastewater quality on average of 763.4 mg/L COD, 192.5 mg/L BOD, 2,713 µS/cm conductivity, 1,973 mg/L TDS and 2070 ADMI units colour.
5. Based on previous results, the NF system would ensure that sufficient colour removal occurred from the UF permeate, while the RO system would remove the residual salts in the NF permeate allowing re-use of the RO permeate by the industrial partner.

### 6.4.2 Full-scale design options

The proposed full-scale system design is based on the UF-dsMBR pilot plant design and the results obtained from the extensive 220 day pilot plant operation. Figures 6.1 and 6.2 are block flow diagrams (BFD) suggesting configurations for the design of the proposed full-scale textile wastewater treatment plant. The proposed full-scale wastewater treatment plant will receive wastewater from the existing discharge infrastructure and treat this further before being channelled into a holding tank for re-use in the processing plant. The equalisation tank used in the pilot plant to eliminate contaminant spikes was removed from the full-scale design and the existing settling tank and sump was used as the equalisation step instead. Two full-scale design options were considered in Figures 6.1 and 6.2. Figure 6.1 is a direct scale-up of the pilot plant operated utilising three free standing tanks in series for the biological phase (i.e. anaerobic, anoxic and aerobic tanks), while in Figure 6.2 the full-scale biological treatment stage utilises a single biological reactor containing three treatment chambers (i.e. anaerobic, anoxic and aerobic chambers). With the exception of the biological treatment phase, the remainder of the two full-scale designs is the same.

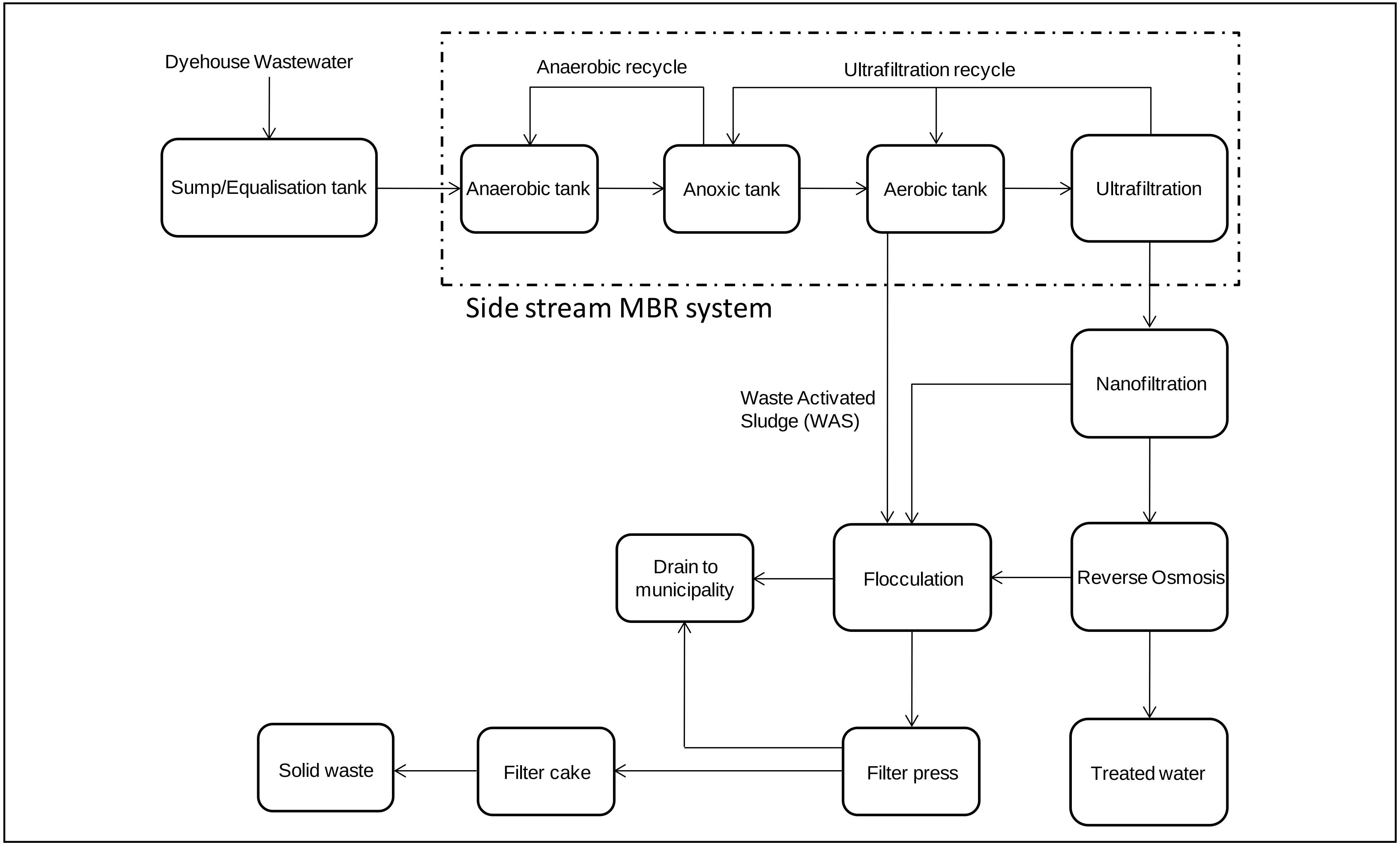


**Figure 6.1:** Full-scale textile wastewater treatment plant BFD using separate anaerobic-anoxic-aerobic biological tanks

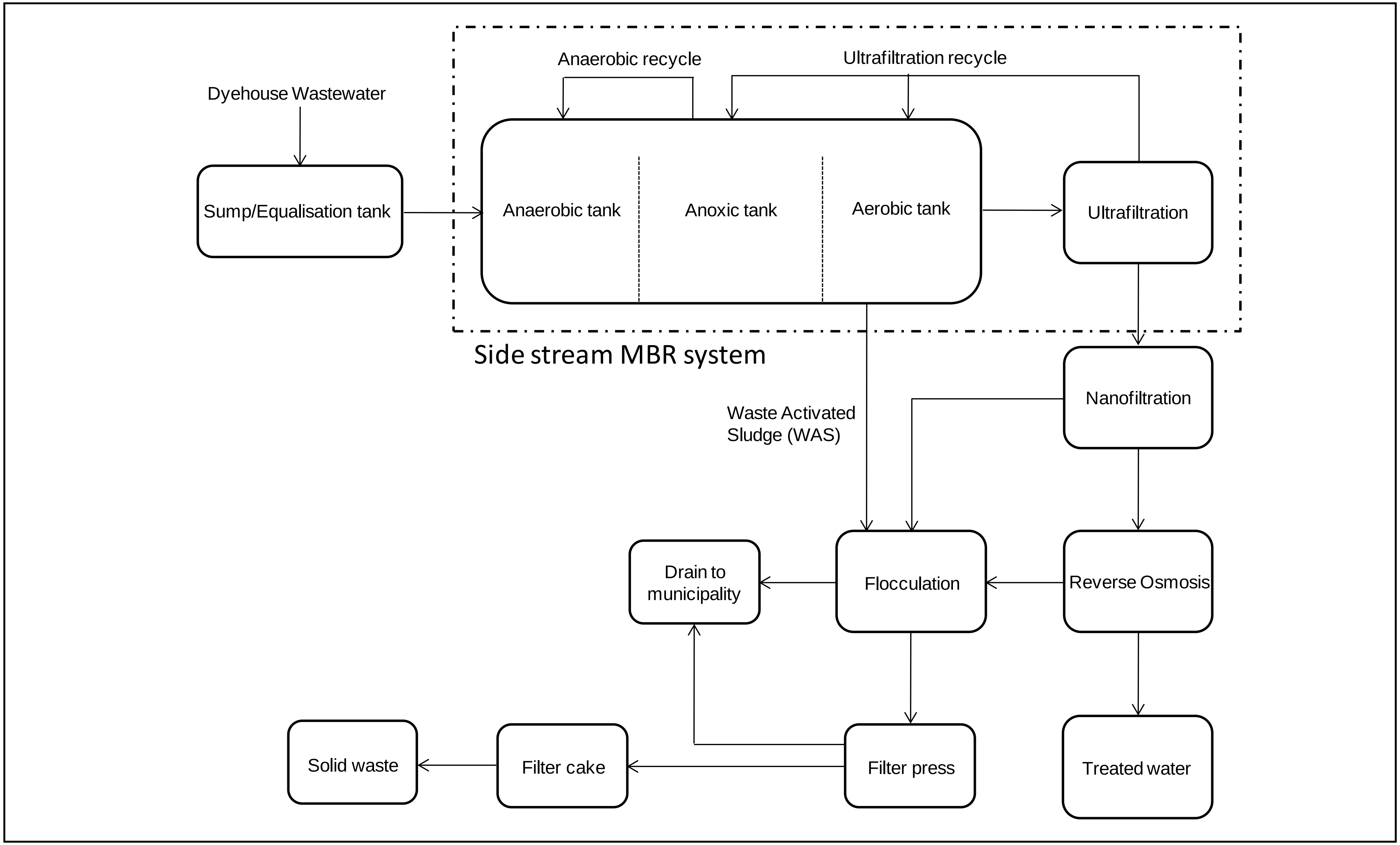


**Figure 6.2:** Full-scale textile wastewater treatment plant BFD using a single biological reactor with three chambers

### 6.4.3 Full-scale design option chosen

The decision to utilise a single biological reactor containing three chambers was chosen for a number of reasons as listed below:

1. The cost associated with excavating and building a single bioreactor containing three chambers (dimensions: 2 m by 2.63 m by 17.11 m) - including excavation; compaction; building the walls with 190 mm blocks; throwing the floor (100 mm thick); back filling and plastering the walls for water resistance - will cost approximately ZAR 100,000 to build. The single bioreactor will be divided into a 15 $m^3$ anaerobic tank (dimensions: 2 m by 2.63 m by 2.85 m), 37.5 $m^3$ anoxic tank (dimensions: 2 m by 2.63 m by 7.13 m) and 37.5 $m^3$ aerobic tank (dimensions: 2 m by 2.63 m by 7.13 m).
2. The cost associated with the three free standing tanks in series are ZAR 17,982 for the anaerobic tank, excluding transport costs from Johannesburg, South Africa, with the 37.5 $m^3$ anoxic and aerobic tanks being custom-made from 3.16 Stainless steel to prevent corrosion due to the high salt content of the wastewater, at a cost of approximately ZAR 50,000 each. Bunding walls must be erected around each free standing tank to protect the environment should one of the free standing tanks leak. Therefore, the estimated total cost of the free standing tanks alone, excluding value added tax (VAT), transport to the site and the bunding walls would be ZAR 115,773, which already costs more than building the single bioreactor with three chambers
3. Additional reasons for choosing the proposed design in Figure 6.2 include the facts that the industrial partner has limited space for the placement of three free standing tanks and for ease of operation. During the operation of the pilot plant maintaining the levels of the biological tanks without any of them overflowing or running empty was a full time duty.

Figure 6.3 is a process flow diagram (PFD) of the proposed full-scale textile wastewater treatment plant. The textile wastewater will be fed from the existing sump into the anaerobic tank. From the anaerobic tank the wastewater will be fed to the anoxic tank followed by the aerobic tank. From the aerobic tank the wastewater is supplied into the UF unit, where the fine solids (i.e. colloids, oils and particles) are separated from the soluble matter. The solids in the UF retentate are returned to, and split between, the anoxic and aerobic tanks. Any waste activated sludge (WAS) from the aerobic tank will be sent to a flocculation chamber. The UF permeate is then further treated by the NF system, used for the removal of low molecular weight components, such as the colour and dye molecules, from the wastewater. Monovalent ions that pass through the NF membrane are polished by the RO system, the permeate of which is high quality with low TDS and free of suspended matter, allowing re-use in the dyehouse. The concentrates (i.e. brines) produced by the NF and RO systems are

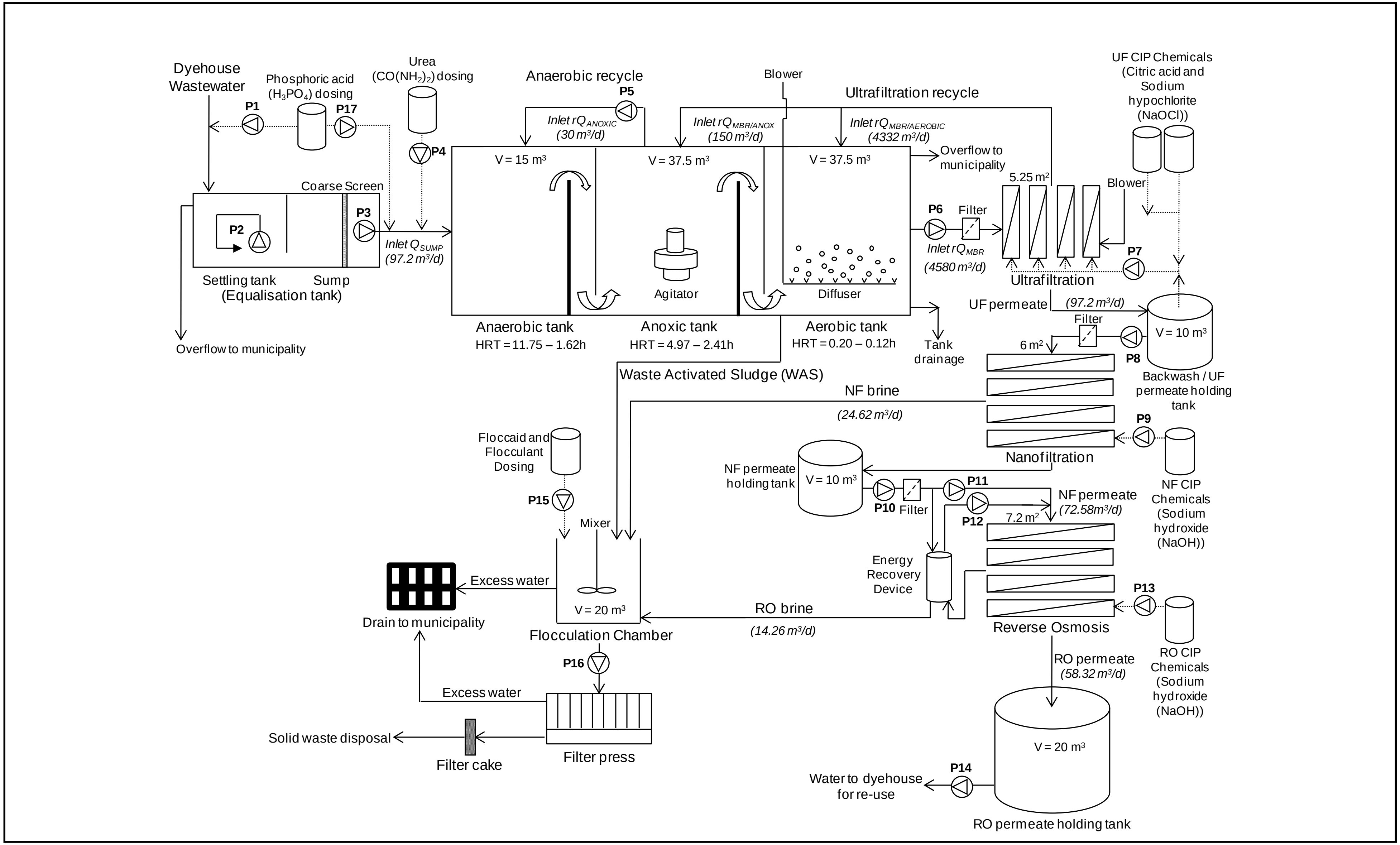


**Figure 6.3:** PFD of the full-scale textile wastewater treatment plant incorporating a single biological reactor with three chambers

collected in a flocculation chamber. A portion of the RO brine is used in an energy recovery device (ERD) attached to the RO system to lower the energy requirements of the system. With the addition of a flocculating agent to the flocculation tank, colloids and other suspended particles present in the NF and RO brines will become insoluble and form aggregates (i.e. flocs) which will settle out. The settled matter is then transferred to a filter press where the solids are dried, while the excess water, free of colour from both the flocculation chamber and filter press is sent down the drain to the municipality. The resulting filter cakes will be disposed of as solid waste, which will be the responsibility of the partner. It is not suggested that the filter cakes be used in a furnace, since the salt content of the brine and therefore the resultant filter will cause the furnace to corrode. The entire system is automated by a programmable logic controller (PLC) and human machine interface (HMI). The operator will be in charge of starting the filtration processes; managing the consumables (the cleaning-in-place (CIP) chemicals, Flocculant, Floccaid and antiscalant), charging and emptying the filter press

#### 6.4.3.1 C:N:P ratio adjustment for the anaerobic process

In the settling tank and sump, as observed during the operation of the pilot plant, the wastewater entering the settling tank was alkaline with an average pH of 9.8. Since biological processes operate optimally at near-neutral pH values, a pH monitoring probe and acid dosing pump, using a feed-backward mechanism, is integrated into the settling tank at the point where the wastewater enters the settling tank. A pH probe located in the anaerobic tank will also monitor the pH of the anaerobic tank. Should the pH not be neutral, a second dosing pump located on the line entering the anaerobic tank will adjust the pH of the wastewater. Dosing with phosphoric acid will not only neutralise the feed to the anaerobic tank, but will also assist in achieving the optimal C:N:P ratio of 100:10:1 (Russell, 2006) along with the installation of a dosing pump for urea located on the anaerobic tank feed line. An average COD value of 763 mg/L requires the ammonium and organically bound nitrogen, as well as phosphorous concentrations in the wastewater to be 76.3 mg/L and 7.63 mg/L, respectively, to maintain a C:N:P ratio of 100:10:1.

Based on the actual average ammonium (9 mg/L) and nitrate (3 mg/L), as well as phosphorous (1.5 mg/L) concentrations present in the wastewater, the actual C:N:P ratio is 100:0.16:0.19. Therefore, in order to obtain the optimal C:N:P ratio of 100:10:1 for an average influent feed of 4.05 $m^3$/h it is necessary to have an average supplemental loading of 1.17 kg $NH_4$/d and 0.15 kg $PO_4$/d. An optimal C:N:P ratio will ensure a food/micro-organism (F/M) ratio that will result in an optimal MBR mixed liquor suspended solids (MLSS) in the range of 10,000 to 12,000 mg/L (Helble & Möbius, 2009). The organic loading rate (OLR) of the UF-dsMBR system was determined by the amount of COD present in the wastewater.

For an average COD of 763 mg/L the OLR was determined to be 0.76 kg COD/m$^3$/d. Therefore, the average loading rate of the proposed full-scale wastewater treatment system was determined to be 74.2 kg COD/d. A submerged recirculation pump located in the sump will provide sufficient mixing of the wastewater to avoid stratification. A second submerged recirculation pump in a screened mesh inlet will transport the wastewater to the anaerobic tank of the MBR. Should the MBR system not be in operation, the wastewater being discharged into the settling tank from the dyehouse will "overflow" into the existing municipal sewer line attached to the sump.

### 6.4.3.2 Flow rates for the proposed wastewater treatment system

Table 6.1 indicates the hydraulic retention times (HRT) calculated for the minimum (0.021 m$^3$/h), maximum (8 m$^3$/h) and average flow rate (4.05 m$^3$/h) (refer to Tables 6.2 and 6.3) at which the proposed wastewater treatment system can operate, as well as the total time the wastewater will spend in the biological section of the system.

**Table 6.1:** Hydraulic retention times (h$^{-1}$) determined for the minimum, maximum and average flow rates of the proposed wastewater treatment system

| **Biological tank** | **Minimum flow rate** | **Maximum flow rate** | **Average flow rate** |
|---|---|---|---|
| **Anaerobic** | 11.75 | 1.62 | 2.83 |
| **Anoxic** | 4.97 | 2.41 | 3.24 |
| **Aerobic** | 0.20 | 0.12 | 0.20 |
| **Total HRT (h$^{-1}$)** | 16.92 | 4.15 | 6.27 |

Tables 6.2 and 6.3 indicate the flow rates (m$^3$/d) for the various streams within the proposed wastewater treatment system when operated at the minimum (min), maximum (max) and average (ave) flow rates (m$^3$/h) for the UF-MBR, and NF, RO and flocculation chamber.

The UF, NF, RO, flocculation chamber and filter press design and costing was outsourced to a company specialising in membrane systems, which supplied the specifications (i.e. membrane surface area) for each respective plant used in later calculations, such as determining the crossflow velocities. Only the bioreactor plant, then, was designed and costed during this study. Refer to Appendix P for pump and blower specifications of the bioreactor plant.

**Table 6.2:** Minimum, maximum and average flow rates through the biological section of the proposed wastewater treatment system

| **Feed flow rate ($m^3$/h)** | **Sump to anaerobic tank ($m^3$/d)** | **Anaerobic tank to anoxic tank ($m^3$/d)** | **Anaerobic recycle from anoxic tank ($m^3$/d)** | **Anoxic tank to aerobic tank ($m^3$/d)** | **Aerobic tank to UF plant ($m^3$/d)** | **UF recycle to anoxic tank ($m^3$/d)** | **UF recycle to aerobic tank ($m^3$/d)** |
|---|---|---|---|---|---|---|---|
| **0.021 (Min)** | 0.5 | 31 | 30 | 151 | 0.5 | 6 | 4332 |
| **8 (Max)** | 192 | 222 | 30 | 343 | 192 | 278 | 7319 |
| **4.05 (Ave)** | 97 | 127 | 30 | 248 | 4580 | 150 | 4332 |

**Table 6.3:** Minimum, maximum and average flow rates ($m^3$/d) through the NF, RO and flocculation chamber of the proposed wastewater treatment system

| **Feed flow rate ($m^3$/h)** | **UF plant to NF plant ($m^3$/d)** | **NF brine to flocculation chamber ($m^3$/d)** | **NF plant to RO plant ($m^3$/d)** | **RO product ($m^3$/d)** | **RO to flocculation chamber ($m^3$/d)** |
|---|---|---|---|---|---|
| **0.021 (Min)** | 0.5 | 0.13 | 0.37 | 0.3 | 0.07 |
| **8 (Max)** | 192 | 46 | 134 | 108 | 26 |
| **4.05 (Ave)** | 97 | 25 | 73 | 58 | 14 |

### 6.4.3.3 Actual oxygen required (AOR) in the aerobic tank to support biomass

Aerobic conditions in the treatment of wastewater are required for the process of nitrification during which ammonium is oxidised to nitrate (Christopher, 2007). When designing an aerobic biological system, the rate of aeration is a critical component in the effectiveness of the aerobic system (Shammas & Wang, 2000). The conversion of ammonium to nitrate, via oxidation, is an oxygen intensive process requiring 4.57 mg $O_2$/mg $NH_3$-N oxidised (Christopher, 2007). The required dissolved oxygen (DO) for optimal biological activity within an aerobic system is between 1.5 and 2 mg/L (Christopher, 2007). Table 6.4 indicates the constants needed to calculate the flow of air required for an aerobic system.

The oxygen transfer rate ($OTR_{Nitrification}$) required for 60% nitrification was determined using Equation 6.1 (Bolles, n.d.).

$$OTR_{Nitrification} = Con.N \times R \times N_{Effective} \times Q \qquad \text{Eq. 6.1}$$

Where, $Con.N$ represents the amount of oxygen required for nitrification of ammonium to nitrate (4.57 kg $O_2$/kg ammonium oxidised); $R$ represents the universal gas constant (8.413 J.K $^{-1}$.mol); $N_{Effective}$ represents 60% of the average ammonium (9 mg/L) in the wastewater; and $Q$ the average volumetric flow rate (97 $m^3$/d) entering the proposed wastewater treatment system.

The ratio of actual oxygen required ($AOR$) to oxygen required under standard conditions ($SOR$) was determined by utilising Equation 6.2 (Bolles, n.d.).

$$\frac{AOR}{SOR} = \frac{\alpha \times \theta \times (T - T_{ref})}{\left(\frac{C_{S,T} - C_L}{C_S}\right) \times \beta \times \tau \times \omega} \qquad \text{Eq. 6.2}$$

Where, $\alpha$ is the transfer coefficient for oxygen from potable water to wastewater (dimensionless); $\theta$ is the Arrhenius constant used to correct the effects of temperature (°C$^{-1}$); $T$ is the inlet temperature of aerobic tank (°C); $T_{ref}$ is the ambient temperature (°C); $C_{s,T}$ is the oxygen saturation concentration corrected for temperature and altitude (kg $O_2$/L); $C_L$ is the designed dissolved oxygen concentration (kg $O_2$/L); $C_S$ is the saturation concentration of water (kg/L); $\beta$ is the wastewater factor that inhibits oxygen transfer (dimensionless); $\tau$ is the temperature correction factor for saturation concentration of water (dimensionless); and $\omega$ is the pressure correction factor for saturation concentration of water (dimensionless)

(Bolles, n.d.). The reference temperature was taken to be 20°C and the temperature of the feed into the MBR system (25.19°C) was used for $T_{ref}$. The constants required to calculate the $AOR/SOR$ ratio can be found in Table 6.4. These constants are based on the use of a coarse bubble diffuser.

**Table 6.4:** Constants required in the air rate calculations

| Constants | Value | Units | Reference |
|---|---|---|---|
| $\alpha$ | 0.8 | dimensionless | Bolles, n.d. |
| $\theta$ | 1.024 | °$C^{-1}$ | Bolles, n.d. |
| $C_S$ | 9.02 | kg/L | Bolles, n.d. |
| $C_{s,T}$ | 8.19 | kg $O_2$/L | Bolles, n.d. |
| $C_L$ | 2 | kg $O_2$/L | Bolles, n.d. |
| $\beta$ | 0.95 | dimensionless | Bolles, n.d. |
| $\tau$ | 0.91 | dimensionless | Sinnott, 1999 |
| $\omega$ | 0.98 | dimensionless | Ryan, 2007 |
| $\rho_{Air}$ | 1.205 | kg/$m^3$ | Sinnott, 1999 |
| $Con.N$ | 4.75 | kg $O_2$/kg ammonium | Christopher, 2007 |
| % Oxygen in air (23%) | 0.23 | dimensionless | Sinnott, 1999 |
| $R$ | 8.413 | J.$K^{-1}$.mol | Bolles, n.d |

The $AOR$ (kg $O_2$/h) for the aerobic system was determined using Equation 6.3 (Bolles, n.d.).

$$AOR = \frac{\frac{AOR}{SOR}}{OTR_{Nitrification}} \qquad \text{Eq. 6.3}$$

In order to determine the air flow rate into the aerobic system (i.e. the actual air required ($AAR$), via coarse diffusers, to deliver the required oxygen for nitrification, Equation 6.4 was utilised. This equation was used in order to account for the ratio of oxygen present in air (Table 6.5).

$$AAR = \frac{AOR}{\rho_{Air} \times 0.23} \qquad \text{Eq. 6.4}$$

The air flow rate that the blower must provide to the aerobic tank to provide actual oxygen required of 0.0087 kg $O_2$/h was determined to be 37.3 L/h. All the results determined from Equations 6.1 to 6.4 are summarised in Table 6.5. Note that all calculations were based on the average flow rate (4.05 $m^3$/h) at which the proposed full-scale wastewater treatment system can operate (refer to Table 6.2). Refer Appendix Q for all calculations and values used.

**Table 6.5:** Calculated oxygen requirement

| Determined Parameters | Value | Units |
|---|---|---|
| $OTR_{Nitrification}$ for 60% nitrification | 839.1 | kg/h |
| $AOR/SOR$ | 7.31 | dimensionless |
| Actual oxygen required ($AOR$) | 0.0087 | kg $O_2$/h |
| Actual air required ($AAR$) | 31.43 | L/h |

### 6.4.3.4 UF, NF, RO and concentrate plants

The UF-membrane crossflow velocity of the proposed wastewater treatment system was determined to be 0.038 m/s for both the minimum and average flow rates, and 0.065 m/s for the maximum flow rate. The solid-liquid separation UF, liquid-liquid separation NF and RO plants were designed with total operating capacities of 192 $m^3$/d (8.0 $m^3$/h), 180 $m^3$/d (7.5 $m^3$/h) and 5.6 $m^3$/h (135 $m^3$/d). While the total dissolved solids (TDS) liquid concentrate plant had a total operating capacity of 84 $m^3$/d (3.5 $m^3$/h). The 5.25 $m^2$ UF plant will consist of four polypropylene membrane modules with 0.2 µm pore size, and an airlift control system with blower, to assist in fouling reduction within the UF-membrane modules. The 6 $m^2$ NF plant will have six PN40 pressure casings composed of fibreglass reinforced plastic (FRP), each pressure casing containing 24 NF (Type 8040) spiral membranes. While the 7.2 $m^2$ RO plant will consist of three PN40 FRP pressure casings each containing 15 RO (Type 8040) spiral membranes. The filter press has 30 plates and a holding capacity of 80 kg.

The approximate power requirements for the UF, NF and RO plants was supplied as 44 kW, 30 kW and 20 kW by the supplier of the plants. However, an isobaric (i.e. pressure equalising) ERD which transfers energy from the membrane reject or brine stream directly to the membrane feed stream was fitted to the RO plant in order to reduce the energy consumption by the RO plant (Stover, 2007). ERDs can be classified as either centrifugal type or positive displacement (PD) type. The centrifugal type of ERD, such as a Pelton turbines, turbochargers and reverse running pumps, utilises the centrifugal principle to "hydraulic energy to mechanical power to hydraulic energy", while the PD type of ERD, such as piston-type work exchangers and rotary PX Pressure Exhcanger$^{TM}$ devices transfers "hydraulic energy to hydraulic energy" and consequently their efficiency is high (Stover, 2007; Sun *et al.,* 2007). The RO brine driven by the circulation pump flows to the ERD. The ERD replaces the brine with feed water. The feed water pressurised in the ERD merges with the feed water pressurised by the high pressure pump, these combined streams feed the RO membrane (Stover & Blanco, 2009). The addition of a direct, positive displacement pressure ERD can result in a net transfer efficiency of up to 97%. This allows improvement in the performance of an RO plant by reducing the energy consumption by as much as 60% when compared to RO systems without ERDs (Stover, 2007). An ERD also offers benefits to the plant designers and operators, including unlimited capacity, reduced high pressure pump costs, high efficiency and operational flexibility (Stover, 2007). Figure 6.4 illustrates how an ERD would be incorporated to transfer energy from the brine to the RO feed (Energy Recovery Inc., 2012).

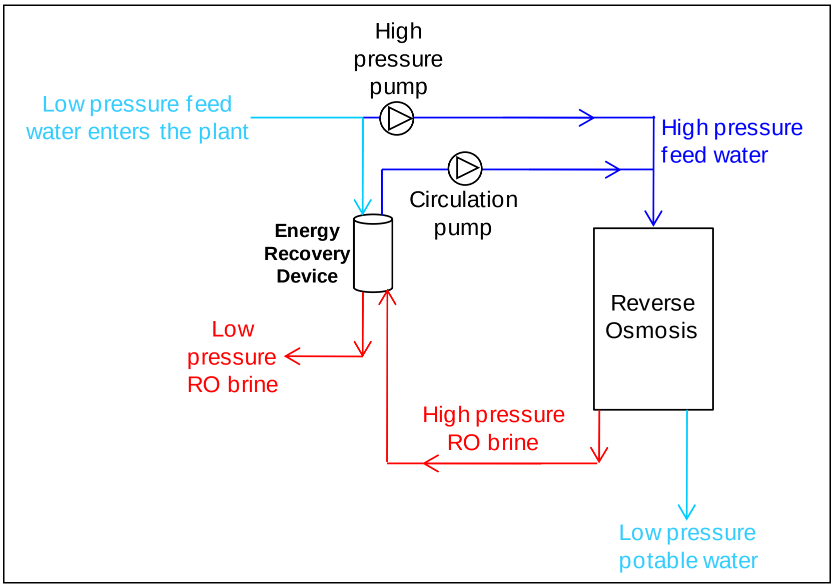


**Figure 6.4:** Schematic diagram of an energy recovery device

Therefore, the power requirements for the RO plant as stated by the supplier of 20 kW will actually be approximately 8 kW due to the approximately 60% reduction in energy consumption due to the ERD fitted to the RO plant.

Tables 6.6 to 6.9 list all the equipment included in the UF, NF, RO and concentrate plants. Included with the UF, NF and RO plants are the following:

- the automation and visualisation equipment (motor control centre (MCC), programmable logic controller (PLC) and human machine interface (HMI));
- all relevant PLC field instrumentation (gauges, sensors and transmitters);
- all relevant automatic and manual valves;
- pipe work (316 stainless steel) and fittings;
- the entire plant mounted on one skid;
- field wiring and field pneumatic installation;
- basic and detail engineering;
- project management;
- transport to site;
- installation on-site;
- commissioning and training of operators;
- preliminary and general (P&G) costs; and
- all documentation including manuals.

The TDS concentrate plant includes the following:

- all relevant PLC field instrumentation (gauges, sensors and transmitters);
- all relevant automatic and manual valves;
- pipe work and fittings (polyvinyl chloride (PVC));
- the entire plant mounted on one skid;
- field wiring and field pneumatic installation;
- basic and detail engineering;
- project management;
- quality management;
- transport to site;
- installation on-site;
- commissioning and training of operators;
- preliminary and general (P&G) costs; and
- all documentation including manuals.

**Table 6.6:** Equipment included in the 8.0 $m^3$/h (192 $m^3$/d) solid-liquid separation UF plant

| **Quantity** | **UF plant equipment** |
|---|---|
| 1 | Feed pump (Variable speed drive (VSD) operated) |
| 1 | Loop pump |
| 1 | Filter bag unit |
| 4 | Polypropylene membrane modules (0.2 µm) |
| 1 | UF holding/cleaning-in-place (CIP) tank |
| 1 | Chemical dosing unit |

**Table 6.7:** Equipment included in the 7.5 $m^3$/h (180 $m^3$/d) liquid-liquid separation NF plant

| **Quantity** | **NF plant equipment** |
|---|---|
| 1 | Feed pump (VSD operated) |
| 1 | Loop pump |
| 1 | Filter bag unit |
| 6 | Pressure casing PN40 (material fibreglass reinforced plastic (FRP)) |
| 24 | NF spiral membrane module (type 8040) |
| 1 | NF holding/CIP tank |
| 1 | Chemical dosing unit |

**Table 6.8:** Equipment included in the 5.6 $m^3/h$ (135 $m^3/d$) liquid-liquid separation RO plant

| Quantity | RO plant equipment |
|---|---|
| 1 | Feed pump (VSD operated) |
| 1 | High pressure pump |
| 1 | Circulation pump |
| 1 | Loop pump |
| 1 | Filter bag unit |
| 3 | Pressure casing PN40 (material FRP) |
| 15 | RO spiral membrane module (type 8040) |
| 1 | RO holding/CIP tank |
| 3 | Chemical dosing unit |

**Table 6.9:** Equipment included in the 3.5 $m^3/h$ (84 $m^3/h$) high TDS liquid concentrate plant

| Quantity | Concentrate plant equipment |
|---|---|
| 1 | Flocculation chamber |
| 1 | Floccaid dosing unit |
| 1 | Flocculant dosing unit |
| 1 | Filter press supply pump (air driven diaphragm pump) |
| 1 | Filter press (30 plates, cake holding capacity 80 kg) |

Refer to the piping and instrumentations (P&ID) diagrams in Figures 6.5 to 6.9 for a detailed design of the proposed full-scale textile wastewater treatment plant. Even though the full-scale plant has a total treatment capacity of 160 $m^3/d$, all the values mentioned in the P&IDs are for an average wastewater flow rate of 97 $m^3/d$ (4.05 $m^3/h$).

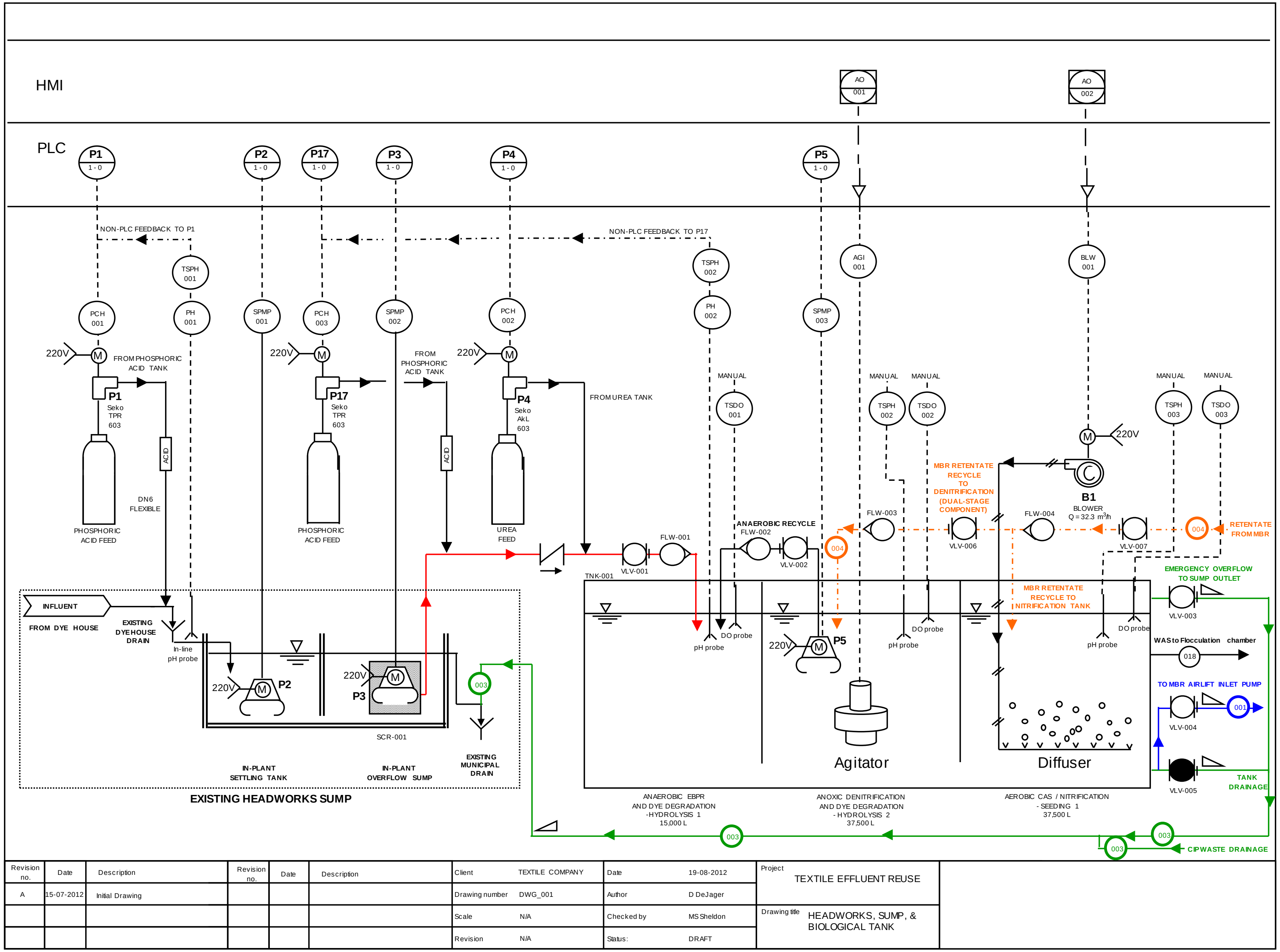
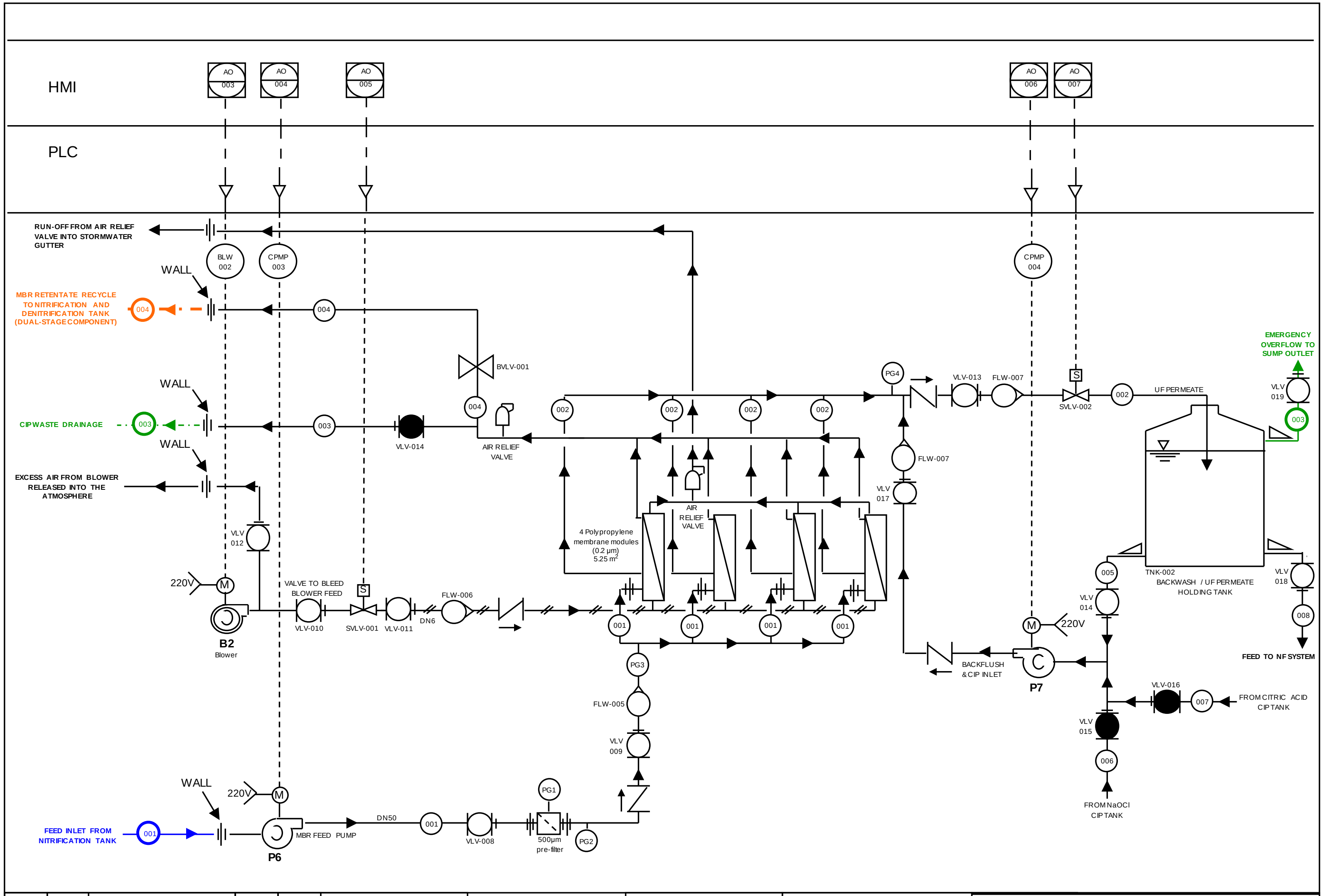
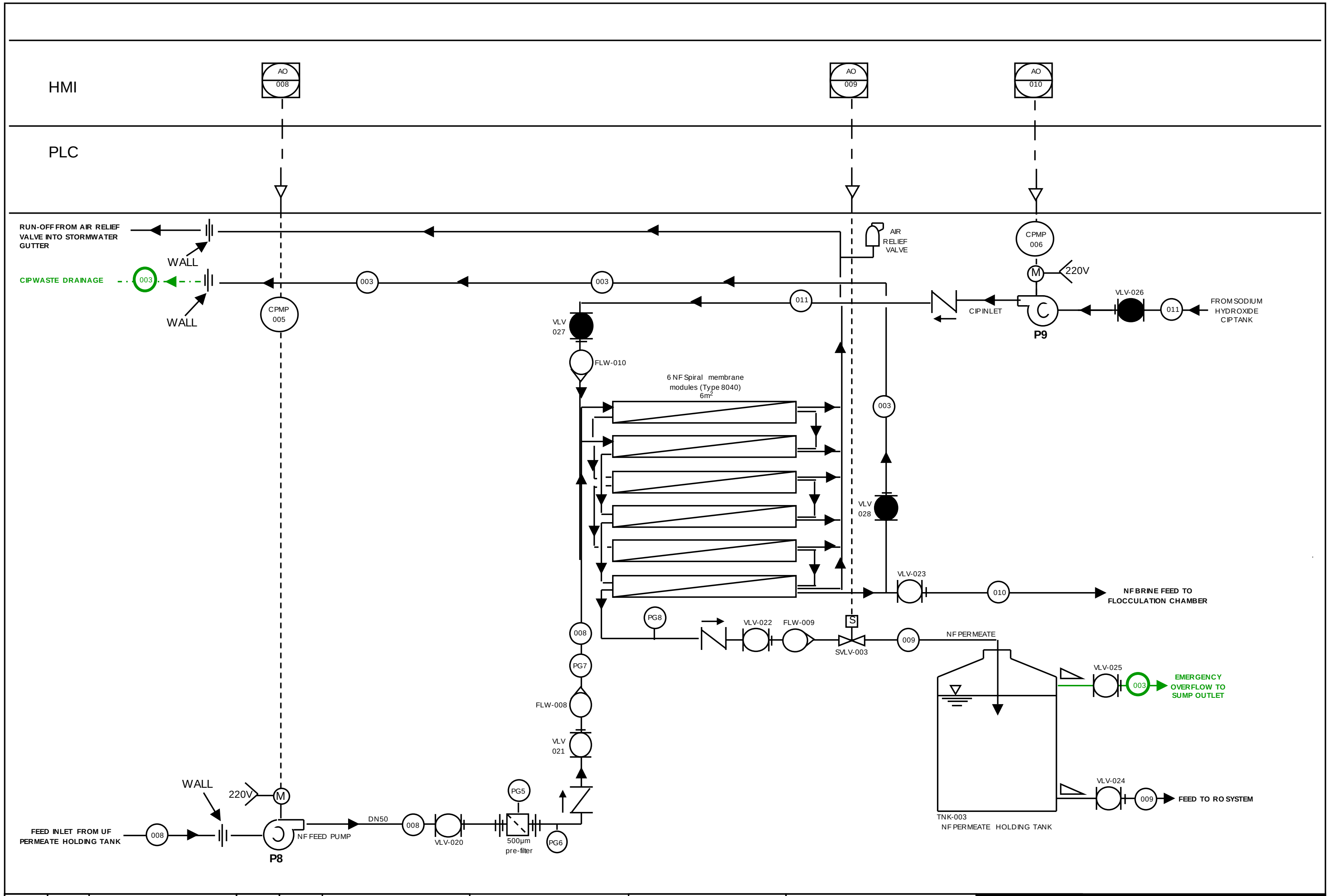


**Figure 6.5:** P&ID representing the biological system of the UF-dsMBR section of the proposed full-scale wastewater treatment plant (drawn using Microsoft Office Power Point 2007)



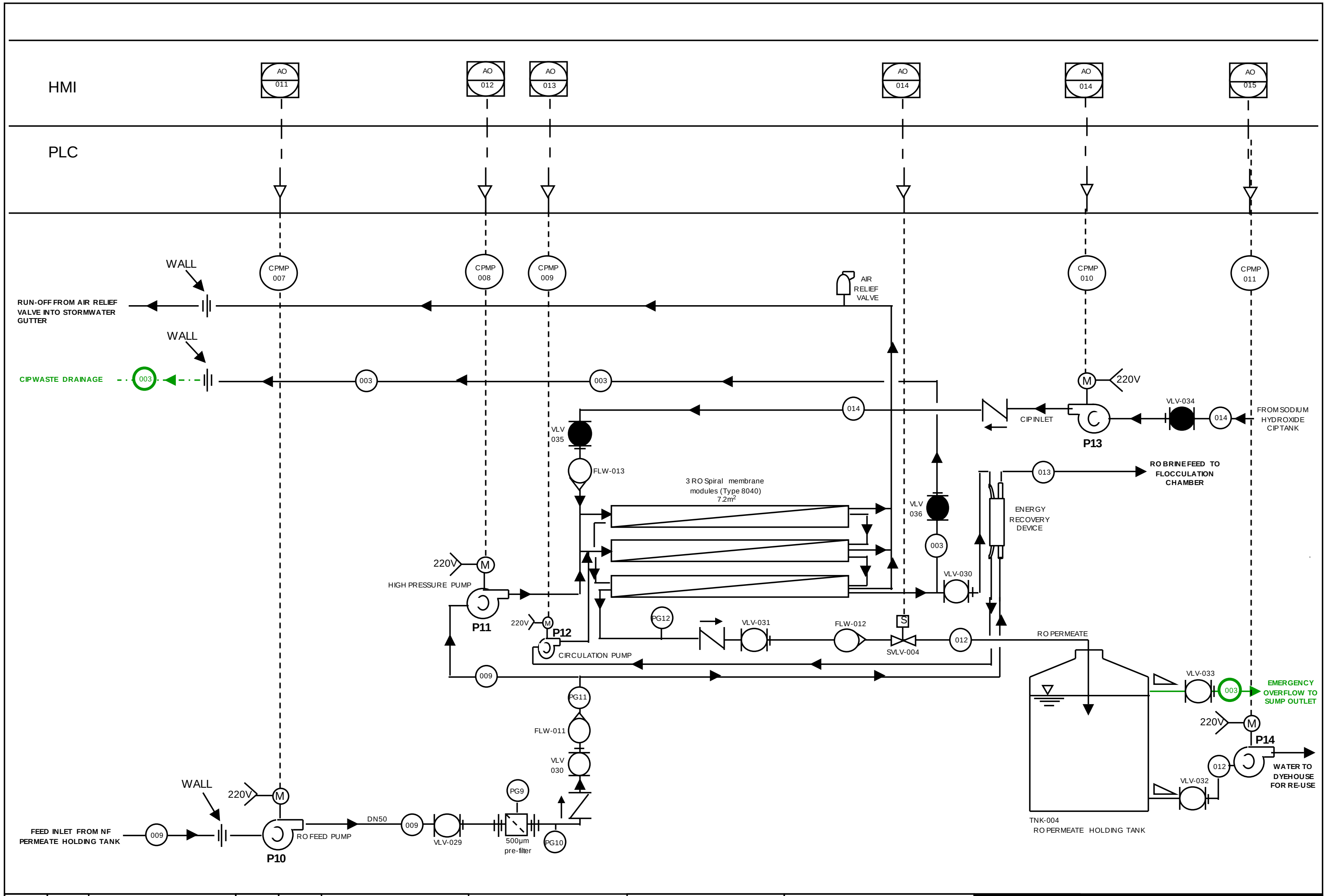
| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 19-08-2012 | Project<br>TEXTILE EFFLUENT REUSE | |
| A | 15-07-2012 | Initial Drawing | | | | Drawing number | DWG_002 | Author | D DeJager | | |
| | | | | | | Scale | N/A | Checked by | MS Sheldon | Drawing title<br>MBR RETICULATION, PUMPS, & CIP | |
| | | | | | | Revision | N/A | Status: | DRAFT | | |

**Figure 6.6:** P&ID representing the liquid-solid separation UF section of the proposed full-scale wastewater treatment plant (drawn using Microsoft Office Power Point 2007)



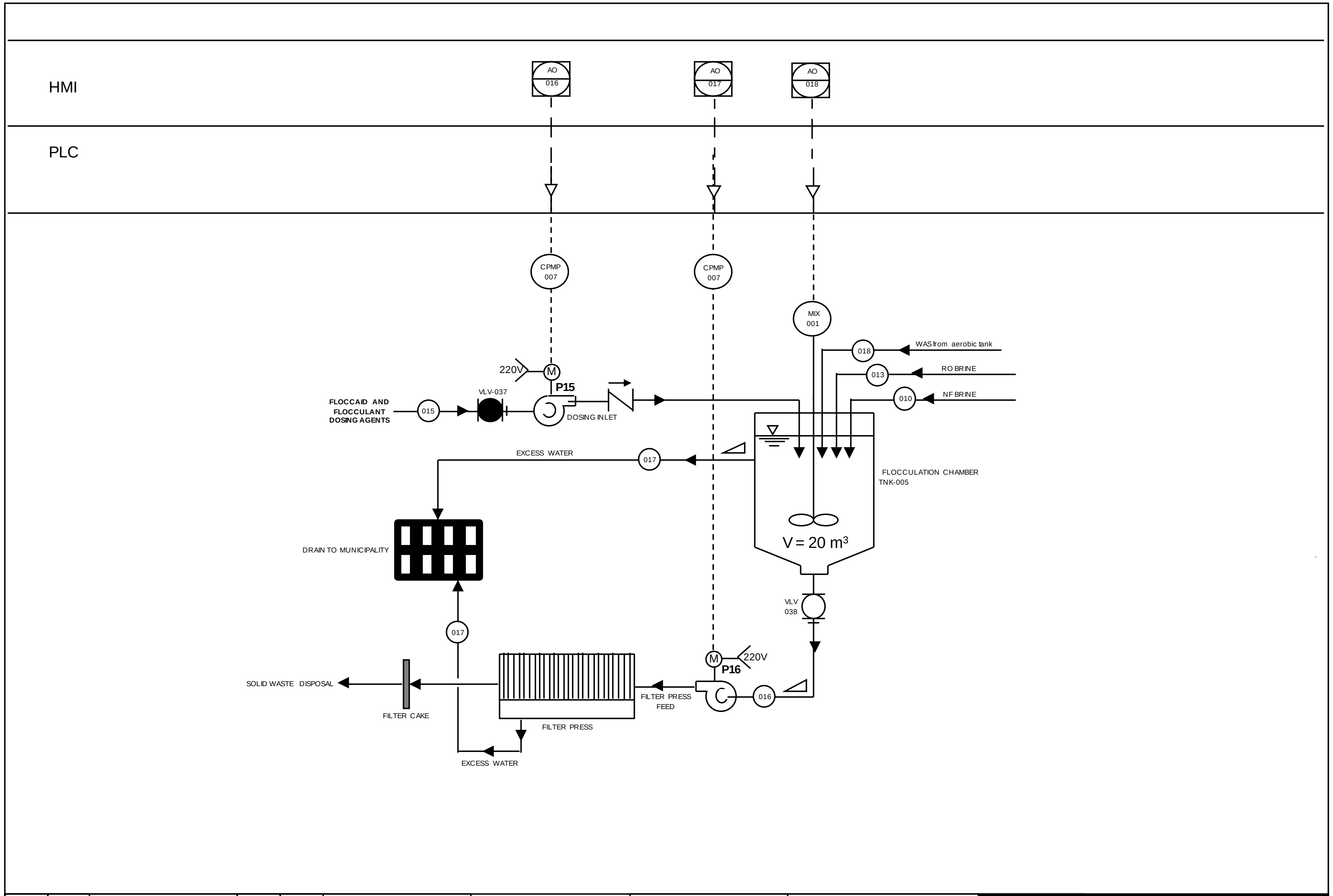
| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 19-08-2012 | Project<br>TEXTILE EFFLUENT REUSE |
| A | 15-07-2012 | Initial Drawing | | | | Drawing number | DWG_003 | Author | D DeJager | |
| | | | | | | Scale | N/A | Checked by | MS Sheldon | Drawing title<br>NF SYSTEM, PUMPS, & CIP |
| | | | | | | Revision | N/A | Status: | DRAFT | |

**Figure 6.7:** P&ID representing the liquid-liquid separation NF section of the proposed full-scale wastewater treatment plant (drawn using Microsoft Office Power Point 2007)



| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 19-08-2012 | Project TEXTILE EFFLUENT REUSE |
|---|---|---|---|---|---|---|---|---|---|---|
| A | 15-07-2012 | Initial Drawing | | | | Drawing number | DWG_04 | Author | D DeJager | |
| | | | | | | Scale | N/A | Checked by | MS Sheldon | Drawing title RO SYSTEM, PUMPS, & CIP |
| | | | | | | Revision | N/A | Status: | DRAFT | |

**Figure 6.8:** P&ID representing the liquid-liquid separation RO section of the proposed full-scale wastewater treatment plant (drawn using Microsoft Office Power Point 2007)



| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Revision no. | Date | Description | Revision no. | Date | Description | Client | TEXTILE COMPANY | Date | 19-08-2012 | Project<br>TEXTILE EFFLUENT REUSE |
| A | 15-07-2012 | Initial Drawing | | | | Drawing number | DWG_003 | Author | D DeJager | |
| | | | | | | Scale | N/A | Checked by | MS Sheldon | Drawing title<br>RO SYSTEM, PUMPS, & CIP |
| | | | | | | Revision | N/A | Status: | DRAFT | |

**Figure 6.9:** P&ID representing TDS liquid concentrate plant section of the proposed full-scale wastewater treatment plant (drawn using Microsoft Office Power Point 2007)

### 6.4.4 Environmental considerations

The following environmental concerns must be considered with the design and implementation of the proposed full-scale plant: 1) all emissions to land, air and water; 2) waste management; 3) smells emitted by the treatment plant; 4) noise; 5) visual impact; and 6) the environmental friendliness of the product (Sinnott, 1999).

Two waste products would be produced during operation of the proposed full-scale wastewater treatment plant, namely solid filter cakes and liquid filtrate from the filter press. These by-products would require environmentally acceptable disposal methods. The solid filter cake would be removed and disposed of at a solid waste disposal site, while the liquid filtrate would be sent to the municipality for further treatment. The liquid filtrate from the filter press should contain a negligible salt concentration since most of the salts would have settled out in the flocculation chamber. Negligible smells will be emitted by the treatment plant, since only the anaerobic plant would emit any biogases. Noise levels will remain low as the UF, NF and RO plants would be containerised and therefore most of the noise from the pumps and blowers would be contained. Most of the noise emitted would come from the bioreactor plant. Visually the full-scale treatment system would be environmentally friendly as the bioreactor plant would be located underground and the remainder of the plant containerised. The product produced would meet the SAN 241-1:2011 (South Africa. Department of Water Affairs, 2011) drinking water standards.

### 6.4.5 OPEX and CAPEX

The OPEX was determined for both the 4.5 m$^3$/d pilot plant and the 160 m$^3$/d proposed full-scale wastewater treatment plant. The annual estimated operational and maintenance costs for the first year of operation of the proposed full-scale plant was determined to be ZAR 1,316.53/d (Table 6.10) for each 160 m$^3$ treated. Consequently, it would cost the industrial partner ZAR 8.23 to treat 1 m$^3$. Of the ZAR 1,316.53 the electrical power required to operate the plant accounted for 75.2%; equipment repairs and replacement accounted for 2.7%; chemical cleaning accounted for 0.43%; the chemical costs associated with disinfection accounted for 0.29%; the possible replacement of the diffuser accounted for 0.14%; while membrane replacement cost estimates (based on a lifespan of 8 years) and labour accounted for 4.0% and 17.3% of the daily OPEX determined.

The total CAPEX of the 160 m$^3$/d proposed full-scale wastewater treatment plant was determined to be ZAR 2,228,720 (Table 6.11). The capital expenditure being ZAR 237,200 for the 192 m$^3$/d (8.0 m$^3$/h) bioreactor plant; ZAR 452,600 for the 192 m$^3$/d (8.0 m$^3$/h) UF plant; ZAR 431,520 for the 180 m$^3$/d (7.5 m$^3$/h) NF plant; ZAR 403,000 for the 5.6 m$^3$/h (135 m$^3$/h) RO plant; and ZAR 704,400 for the 84 m$^3$/d (3.5 m$^3$/h) liquid concentrate plant.

**Table 6.10:** OPEX for the proposed 160 $m^3$/d full-scale plant

| **Item/Description** | **Percentage of OPEX** | **Full-scale (160 $m^3$/d)** |
|---|---|---|
| Electrical power for the wastewater treatment process/Miscellaneous | 75.2 | 989.9 |
| Equipment repairs/replacements | 2.7 | 35.6 |
| Chemical cleaning | 0.43 | 5.6 |
| Chemical cost of disinfection | 0.29 | 3.8 |
| Diffuser replacement | 0.14 | 1.9 |
| Membrane replacement | 4.0 | 52.5 |
| Labour | 17.3 | 227.23 |
| **Annual Estimated Operational and Maintenance costs for the first year of operation (ZAR/d/160 $m^3$ treated)** | **100** | **1,316.53** |

**Table 6.11:** CAPEX for the proposed 160 $m^3$/d full-scale plant

| **Item/Description** | **Price (ZAR)** |
|---|---|
| 192 $m^3$/d (8.0 $m^3$/h) Bioreactor plant | 237,200 |
| 192 $m^3$/d (8.0 $m^3$/h) UF plant | 452,600 |
| 180 $m^3$/d (7.5 $m^3$/h) NF plant | 431,520 |
| 5.6 $m^3$/h (135 $m^3$/h) RO plant | 403,000 |
| 84 $m^3$/d (3.5 $m^3$/h) liquid concentrate plant | 704,400 |
| **Total CAPEX budget** | **2,228,720** |

The 2011 water rate of ZAR 25.72 per $m^3$ of water, for water usage in excess of 50 $m^3$/d indicated that the industrial partner spent ZAR 2459.89 for an average daily water usage of 97.1 $m^3$, and ZAR 73,902.81 per month for an average monthly water consumption of 2,913 $m^3$ during 2011. However, it would cost the industrial partner ZAR 798.97 and ZAR 23,969.14 to treat 97.1 $m^3$ and 2,913 $m^3$ of textile wastewater, respectively per day and per month. Therefore, by installing the proposed full-scale wastewater treatment plant, the industrial partner would be saving ZAR 1,664.46 per day and ZAR 49,933.7 per month on potable water cost since the treated water could be re-used in dyeing processes. The industrial partner would also be saving on wastewater discharge costs, normally ZAR 43,341.07 per month, since the only wastewater being discharge would be the excess water from the flocculation chamber and the filtrate from the filter press. Indicating a total cost saving of ZAR 93,274.39 per month. Since the increase in water cost was known together with the industrial partner's 1) water consumption; 2) wastewater discharge costs; and 3) the cost of the proposed full-scale textile wastewater treatment plant the return on investment could be calculated for the industrial partner. Therefore, based on the calculated CAPEX, the industrial partner should see return on investment within 23.9 months (i.e. ~2 years).

## 6.5 Conclusions

The proposed full-scale plant was designed to treat 97.1 $m^3$/d. However, should the industrial partner ever scale-up its dyeing processes thereby increasing the quantity of wastewater it produces, the proposed full-scale wastewater treatment plant could treat a maximum capacity of up to 160 $m^3$/d. With the implementation of the full-scale wastewater treatment plant the industrial partner would decrease its annual operating expenditure by: 1) reducing the wastewater discharge tariffs they currently pay, since the only wastewater being sent to the municipality would be the filtrate from the filter press; and 2) reducing the potable water intake costs as the industrial partner would be able to re-use the treated water in their dyeing processes. Unfortunately the industrial partner will not be able to achieve the status of Zero Liquid Discharge (ZLD) due to the filtrate from the filter press being sent down the drain to the municipality. However, with the implementation of the proposed full-scale wastewater treatment plant, the industrial partner would be saving ZAR 49,933.7 and ZAR 43,341.88 per month on potable water and wastewater discharge costs, respectively, and see a return on investment within 23.9 months.

## 6.6 Summary

The results obtained from the pilot plant (refer to Chapters 4 and 5) assisted in scaling-up and designing the full-scale plant. The proposed full-scale wastewater treatment plant closely resembled the pilot plant and therefore 1) the feasibility of scale-up could be investigated

(Chapter 6); 2) technology assessment of the sidestream dsMBR system in the treatment of textile wastewater could occur during the pilot plant operation (Chapters 4 and 5); and 3) results from the pilot plant could be used support the design of the full-scale MBR system.

# CHAPTER 7

# OVERALL RESULTS AND DISCUSSION

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## 7.1 Introduction

This chapter aims to collate the results from Chapters 4 and 5 and compare them with literature reviewed in Chapter 2 for similar membrane bioreactor (MBR) systems treating textile wastewater.

## 7.2 Results and discussion

The wastewater stream was characterised by a chemical oxygen demand (COD) range between 45 to 2,820 mg/L and an average biological oxygen demand (BOD) of 192.5 mg/L. The ultrafiltration (UF)-dsMBR achieved an average overall COD reduction of 75% with an overall maximum of 97% over the 220 day test period. The COD concentration obtained after ultrafiltration (UF) dual-stage (ds) sidestream membrane bioreactor (MBR) treatment averaged at 191 mg/L, well below the ≤ 5,000 mg/L City of Cape Town wastewater and effluent the discharge standard. Subsequent treatment of the UF permeate with nanofiltration (NF) alternated with reverse osmosis (RO) removed both the residual colour and remaining salt, since the UF-dsMBR pilot plant only showed an overall reduction of 28.6% in colour and no salt removal. The American dye manufacturing index (ADMI) was reduced from an average of 660 ADMI units in the UF permeate to below 20 ADMI units by the RO membrane, a lower ADMI and colour compared to the potable water the industrial partner was utilising in their dyeing processes. An average conductivity rejection of 97.5% was achieved with conductivity being reduced from an average of 8,215 to 204 µS/cm and the total dissolved solids (TDS) reduced from an average of 5,688 to 146 mg/L, which facilitated an average TDS rejection of 97.4%, with the RO membrane**.**

Ciardelli and Ranieri (2000) used the ozonation technique to treat textile wastewater and achieved 95% to 99% colour removal. The treated water was successfully re-used in dyeing, even with light colours. A decrease in COD of 60% (75 to 120 mg/L) was observed. Using the electroflocculation technique, Ciardelli and Ranieri (2000) achieved 80% to 100% colour removal and 70% to 90% COD removal. During the current study an overall colour removal of 98.7% and 97.7% was achieved with post treatment NF and RO, respectively, while the UF-dsMBR system showed an average COD removal of 75% (763 to 191 mg/L).

Kim *et al*. (2002) combined various treatment methods including the fluidised biofilm process, chemical coagulation and electrochemical oxidation to investigate the continuous treatment of textile wastewater from a synthetic textile dyeing factory. The system used a relatively low mixed liquor suspended solids (MLSS) and short sludge retention time. The fluidised biofilm process showed a COD and colour removal efficiency of 68.8% and 54.5%, respectively. The inclusion of a supporting medium in the biological stage increased the COD and colour removal from 34.8% to 55.3% and 46.4% to 72.1%, respectively. The biological treatment stage was followed by chemical coagulation. The total COD and colour removal efficiency of the combined process was 95.4% and 98.5%, respectively. The current study, which also had a low MLSS (i.e. 1.3 g/L) showed higher COD (i.e. 85.8% for NF and 90.3% for RO) and colour removal (i.e. 97.4% and 96.0%) efficiencies than Kim *et al.* (2002) observed during both the fluidised bed process and after a supporting medium was included in the biological stage.

Brik *et al.* (2006) using an aerobic MBR with an external crossflow UF unit; Badani *et al.* (2005) using an aerobic MBR and Lubello and Gori (2004) using an aerobic MBR external UF module with plate and frame membranes achieved a COD removal of > 90%; 96% and 93%, respectively, even though there were large COD variations in wastewater entering the MBR. Zheng and Liu (2006), utilising a laboratory scale MBR with a gravity treated wastewater with a COD value ranging from 128 to 321 mg/L and Schoeberl *et al.* (2004), utilising a crossflow UF-MBR system with a COD value of 1,380 to 6,035 mg/L in the wastewater, both observed an average COD removal exceeding 80%. The MBR system used in the current study responded well to fluctuations in the wastewater and was able to buffer the continuously changing pH and wastewater composition with a COD value ranging from 45 to 2,820 mg/L, since the COD removal rate remained relatively stable (i.e. an average of 75%) and unaffected once the MBR system had acclimatised (i.e. 70% to 90% COD removal efficiency after acclimatisation at ~100 days of operation), indicating the robust nature of the treatment system.

Yigit *et al.* (2009) observed a COD removal that exceeded 95% when treating industrial textile wastewater with a pilot-scale MBR using submerged hollow fibre membrane modules in an aeration tank. Yigit *et al.* (2009) had a $BOD_5$/COD ratio of 0.32, suggesting the dominance of slowly biodegradable and/or biorecalcitrant organics in the textile wastewater. The $BOD_5$/COD ratio of the MBR system used in the current study was 0.25 indicating that the wastewater was slowly biodegradable as reported by Yigit *et al.* (2009). Even though the $BOD_5$/COD ratio of the textile wastewater of the industrial partner indicated low biodegradability the pre-treatment UF-dsMBR stage assisted in the overall treatment efficiency of the combined UF-dsMBR and NF/RO pilot plant when looking at both the individual and overall removal efficiencies obtained during this study. Wastewater with a $BOD_5$/COD ratio of 0.4 to 0.8 is normally considered substantially biodegradable. However, in order to increase the biodegradability of textile wastewater the $BOD_5$/COD ratio has to be increased to 0.4 when total decolouration of the wastewater is obtained. One method that can be used is VUV photolysis which enhances the biodegradability of textile dyes by increasing the $BOD_5$/COD ratio. Using this method industrial textile wastewater with a $BOD_5$/COD ratio of 0.0 has been enhanced to 0.22 (Al-Momani *et al.,* 2002).

Feng *et al.,* (2010) observed that during Fenton oxidation, Fenton's reagent reduced the total organic carbon (TOC) and colour by 39.3% and 69.5%, respectively. Brik *et al*. (2006) and Schoeberl *et al.* (2004) both operated lab scale sidestream MBRs for the treatment of industrial textile wastewater. Brik *et al.* (2006) reported colour removal of > 87% without a subsequent NF or RO polishing step, while Schoeberl *et al.* (2004) achieved colour removal of 80% only after post treatment with NF. The UF-dsMBR coupled with RO, used during the current study, showed a TOC removal of 6.9%, 2.5% and 91.5% for the dark, medium and light coloured hydraulic retention sample sets, respectively with colour removal of 99.1%, 54.9% and 90.4%, respectively..

This research provides textile industries, which are water intensive industries, both in SA and internationally with the following options: 1) to reduce their water consumption, thereby utilising less of a valuable decreasing commodity; 2) to meet the SA government discharge standards and reduce their discharge costs; 3) to reduce their carbon footprint by using less waster, thus reducing their impact on the environment; and 4) to decrease their annual expenditure on water, since the treated water would be available for re-use. This research also fills the "gap" that existed with regard to the use of sidestream MBR systems for the treatment of textile wastewater in SA.

## 7.3 Summary

A comparison of the results obtained from the pilot-scale sidestream UF-dsMBR with subsequent polishing (i.e. NF/RO) utilised during this study, with similar MBR systems for the treatment of textile wastewater, showed higher colour removal efficiencies during the post treatment polishing step. While similar COD removal results were recorded. The results obtained during the current study, if not comparable, were improved when compared to the results reported in literature using various MBR configurations and treatment methods for textile wastewater.

# CHAPTER 8

# CONCLUSIONS AND RECOMMENDATIONS

# CHAPTER 8

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## 8.1 Conclusions

The study successfully evaluated the use of the 5 to 10 $m^3$/d pilot-scale sidestream dual-stage (ds) membrane bioreactor (MBR) for the on-site treatment of textile wastewater at an industrial partner. Textile wastewater contains non-biodegradable constituents. Therefore, some companies do not meet the South African (SA) government discharge standards, causing detrimental problems if the wastewater is discharged into the sewerage system and into the environment. The solution to this problem was to design, construct and operate an appropriate treatment process combining membranes and biological processes that would successfully treat textile wastewater to within the SA government industrial wastewater discharge standards, with subsequent treatment providing the potential for re-use. The results obtained from the pilot plant were utilised to design and propose a full-scale MBR wastewater treatment plant to provide a complete solution to meeting the industrial partners' current and future water requirements.

The UF-dsMBR system efficiently reduced the COD and turbidity of the wastewater by an average of 75% and 94%, respectively, to concentrations of 191 mg/L for COD and 2.7 NTU for turbidity. Due to the robustness of the UF-dsMBR pilot plant the wastewater treatment system was able to maintain a consistent average removal rate, with regard to COD, over the 220 days of operation even though the composition of the incoming wastewater was continuously fluctuating (i.e. COD range of 45 to 2,820 mg/L). However, the UF-dsMBR system only removed 28.6% of the colour present in the wastewater. After treating the textile wastewater with the UF-dsMBR system, the parameters measured in the ultrafiltration (UF) permeate were within the South African (SA) discharge standards. However, not all the parameters measured met the potable water standards of the industrial partner. NF and RO were alternated to subsequently polish the UF permeate, in order to optimise the overall treatment efficiency. Coupling the dsMBR pilot system with NF successfully removed colour from the industrial textile wastewater bringing all the parameters measured during the study to within the CCT wastewater discharge standards. After treatment with the pilot-scale NF/RO system, a higher residual colour removal was achieved with NF (97.4%) than RO (96.0%). Therefore, if colour removal from textile wastewater is the primary objective, then an MBR coupled with NF is the treatment method to utilise. However, coupling the dsMBR system with RO not only successfully removed the residual colour from the wastewater; RO also had the ability to efficiently reduce the salts (i.e. conductivity and total dissolved solids (TDS)) present in the dsMBR permeate to similar values to that of the potable water of the industrial partner. Treatment with RO achieved conductivity and TDS removals of 97.5% and 97.4%, respectively; while NF treatment only achieved removal of 30.4% and 32.5%, respectively. Therefore, RO removed both the residual colour and salts, imperative for successful reclamation of the wastewater. All the parameters measured in the HRT sample sets which included the NF/RO post treatment system met the SANS 241-1:2011 drinking water specifications and with the exception of ammonium, phosphate, nitrate and TOC showed overall removal efficiencies in excess of 80%.

The cleavage of azo bonds was indicated by the presence of dissolved organic carbon (DOC) in the HRT sample sets during the anaerobic treatment stage. DOC removal together with the increased ammonium concentration observed in the aerobic tank indicated the mineralisation of the resulting aromatic amines from azo dye cleavage.

The proposed full-scale plant was designed to treat 97.1 $m^3$/d. However, should the industrial partner scale-up in the future resulting in an increase in the quantity of wastewater produced, the proposed full-scale wastewater treatment plant can treat a maximum capacity of 160 $m^3$/d. With the implementation of the proposed full-scale wastewater treatment plant, the industrial partner would be saving ZAR 93,274.39 per month and see a return on investment within 23.9 months.

It may therefore be concluded that the combined UF-dsMBR pilot plant and NF/RO pilot-scale system can successfully treat industrial textile wastewater to within the SANS 241-1:2011 drinking water specifications and may therefore be re-used by the industrial partner, thereby: 1) significantly reducing their discharge costs; 2) reducing the wastewater load sent to the municipal treatment works; 3) reducing the volume of water used by the textile company thus decreasing their water dependence on the municipality; and 4) assisting in reducing their impact on the environment (i.e. carbon footprint).

## 8.2 Recommendations

After operating the pilot plant for 250 days the following recommendations are suggested from challenges experienced and results obtained:

1. Do not use gravity to feed the wastewater from the equalisation tank to the anaerobic tank, since the decreasing liquid level in the equalisation tank has a direct impact on the flow rate entering the anaerobic tank. This flow rate has to then be monitored continuously, which proves difficult when the wastewater treatment plant is operating continuously (i.e. overnight). It is suggested that a centrifugal pump be utilised instead of gravity, as the pump will not be affected by decreasing liquid levels.

2. Do not use ball valves to throttle the flow rates entering the biological tanks, as this is not accurate and incorrect flow rates entering and exiting the biological tanks results in tanks either running empty or overflowing, further resulting in decreased total suspended solids (TSS) and mixed liquor suspended solids (MLSS). Rather install robust flow meters capable of feeding wastewater containing activated sludge.
3. Install level controllers in the biological tanks to help maintain the desired tank levels, especially if the plant is running overnight.
4. When collecting samples, especially when treating “real” industrial wastewater with a continuously changing composition, take the hydraulic retention time (HRT) into consideration since HRT samples provide an accurate representation of the system as a whole, especially when calculating the efficiency of the system. The removal efficiency cannot accurately be calculated for a set samples collected at the same time, since the wastewater entering the system would have a different composition to the wastewater that was entering for the water that would currently be present in the aerobic tank. HRT samples account for both the flow rates entering a “stage” and the volume of the stage, essentially accounting for the time the “sample” spends within a stage.
5. Future work required before implementing, commissioning and constructing the proposed full-scale design, include lab scale studies on the re-usability of the treated water within the textile industry.
    - The treated water must be evaluated for re-use in dyeing processes, using various dyeing recipes to confirm that the same end results are obtained as when potable water is used in the recipes.
    - HRT sample sets, over a sufficient time period, accounting for the UF-dsMBR system combined with subsequent NF treatment must be collected and evaluated in order to compare the results obtained in Chapter 5 when a RO membrane was combined with the UF-dsMBR system. This would provide the information required when deciding whether to implement a UF-dsMBR combined with NF or RO depending on what the objective of the project would be. Unfortunately, due to time and money constraints this was not possible during the current study.
    - Piloting trials must be conducted in order to optimise the system.
6. Implementation of the full-scale wastewater treatment plant should be done using a modular approach in order to meet the expansion requirements of the industrial partner should it be necessary.
7. Cleaner production and waste minimisation audits, to optimise the raw material usage, energy requirements and production manufacturing, should be conducted in order to capitalise on additional long-term savings potential of the industrial partner.

8. Should the industrial partner (i.e. a textile company) not operate continuously and have periods of down time over weekends and public holidays the proposed full-scale system would not be ideal. Biological systems, such as the UF-dsMBR system requires continuous wastewater feed in order to supply the activated sludge with nutrients and thus operate efficiently. With non-continuous wastewater production by the industrial the system would have many periods of unstable treatment efficiency while the activated sludge stabilised after nutrient starvation. In this case the deal wastewater treatment system would include UF, NF and RO without the pre-treatment biological tanks as suggested in the proposed full-scale plant. The absence of the biological tanks would make it easy to start-up and shut down the wastewater treatment system in accordance with the wastewater production of the industry partner without being detrimental or affecting the efficiency of the treatment processes. However, the treatment efficiency of this treatment system for textile wastewater would have to be tested using a lab or pilot-scale system, as done during the current study, in order to evaluate the quality of the treated water before recommending this combination as a possible full-scale solution.

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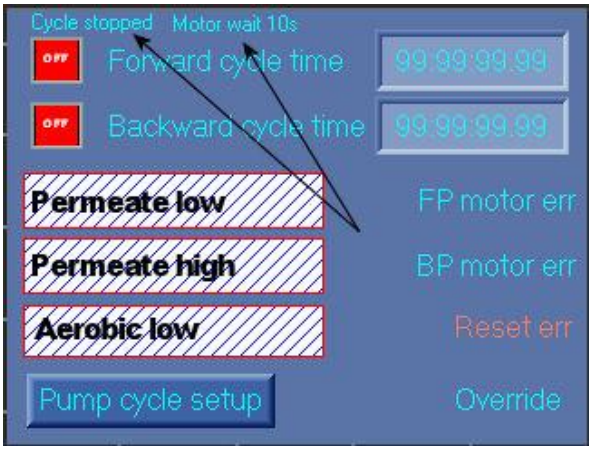
# APPENDICES

# APPENDIX A: Pilot plant programmable logic controller (PLC) human machine interface (HMI) operation

Operating the programmable logic controller (PLC) human machine interface (HMI) screen when setting the forward and backward (i.e. reverse) cycles of the pilot plant.

## Description of PLC HMI Screens

Cycle stopped and Motor wait 10s



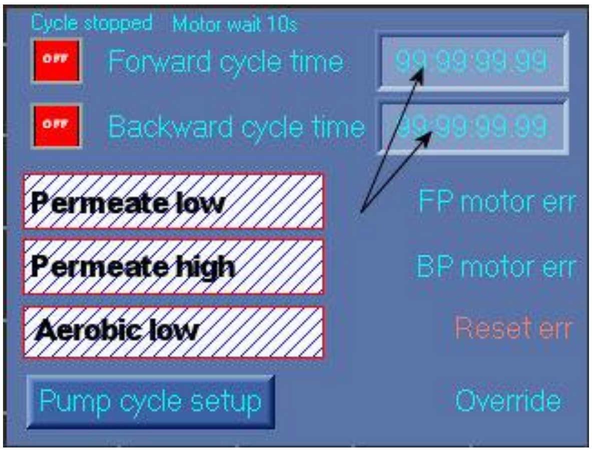
"Cycle stopped": Indicates when the normal cycle is not running.

This occurs when:

- the process is stopped with the red button on the panel; or
- if the backflush cycle was disabled and the forward cycle timer has expired; or
- if the forward cycle was disabled and the backward cycle timer has expired.

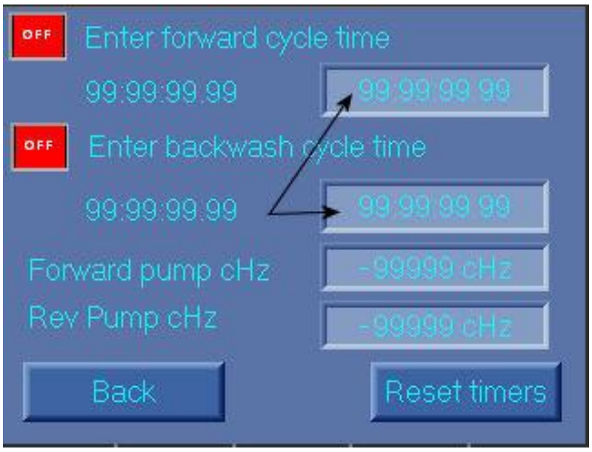
"Motor wait 10s": Indicates when the motor is waiting between cycles before starting up

## Forward and backward time indicators

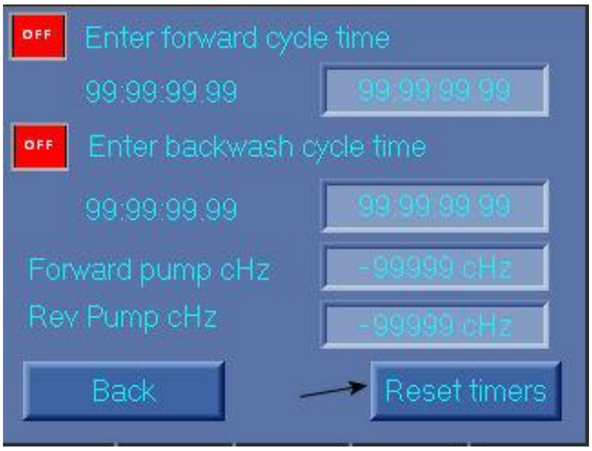


The two time indicators (i.e. forward cycle time and backward cycle time) show how much time is remaining in a forward or backward cycle. Ensure that the forward cycle is set to “always enabled” when setting the time. The forward cycle will therefore remain running even when the backward cycle runs.

## Setting the forward and backward time indicators

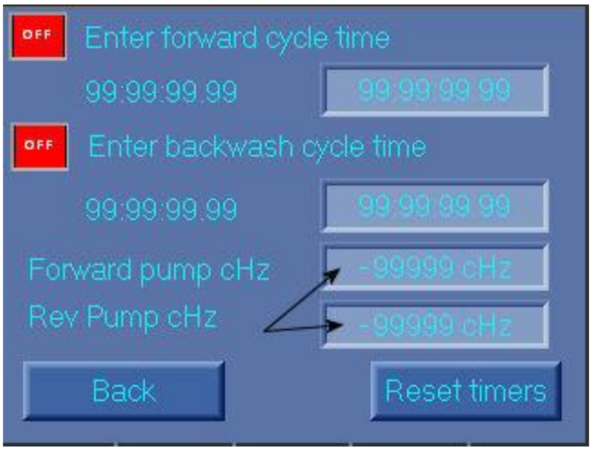


The time for the forward and backward cycles can be set with these input fields.

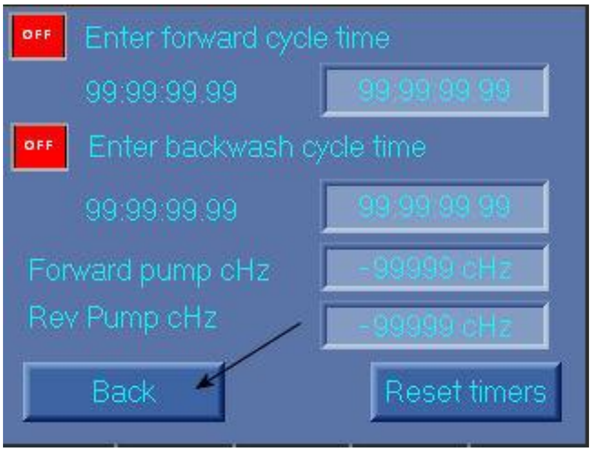


The forward and backward timers remember their settings. To force the timers to restart and to input new forward and backward time settings push the "Reset timers" button.

## Setting the pump frequency

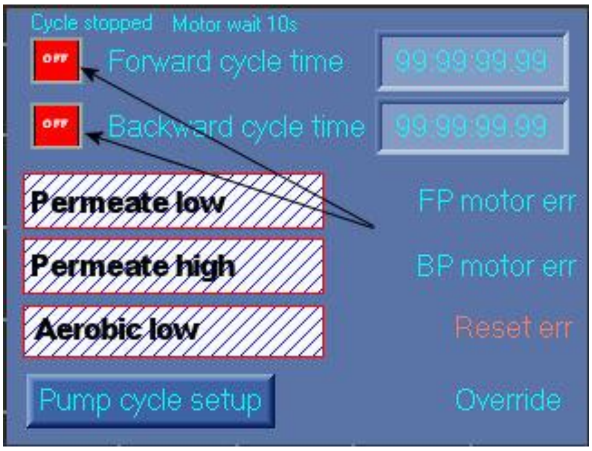


These inputs set the forward and backward pump frequency. The default setting for both the forward and backward pumps are 5000 cHz (50 $\text{Hz}_{\text{pump}}$). The pump speeds can be increased and decreased if needed by changing these values.



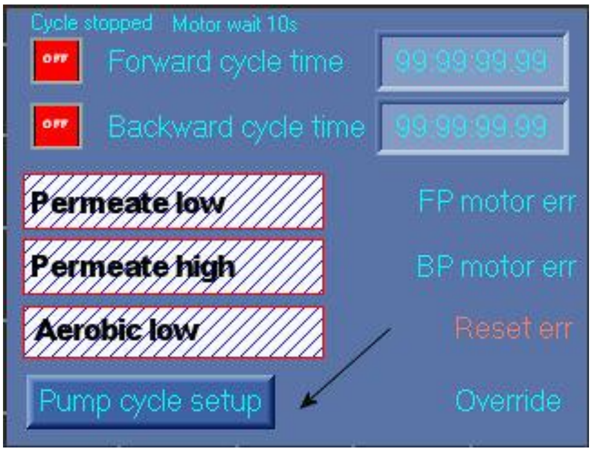
Press "Back" to return to the main screen.

## Enabled and disabled indicators

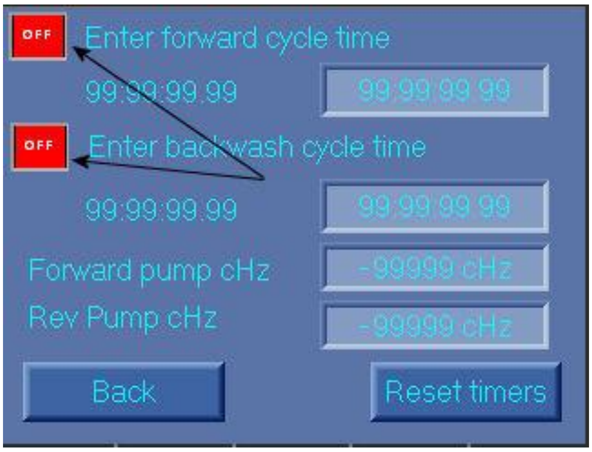


These are indicators show whether the forward or backward cycles have been disabled or enabled. Enabling and disabling of these cycles are done on the "Pump cycle setup" screen. The forward cycle will always be enabled (i.e. the indicator will be green and state “ON”) when the system is operating normally, and will be disabled when CIP is performed.

## Pump cycle setup screen

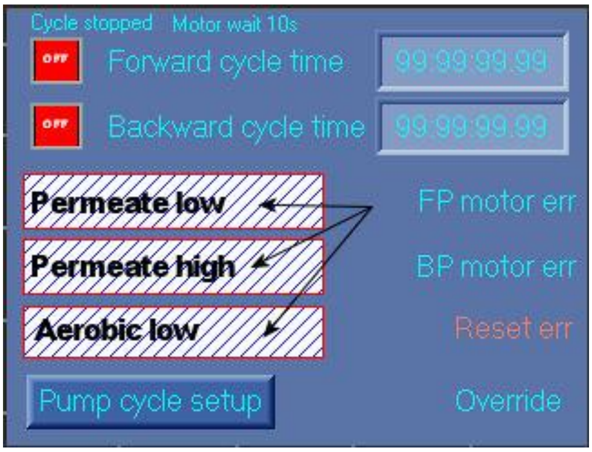


Pushing this button takes you to the "Pump cycle setup" screen.

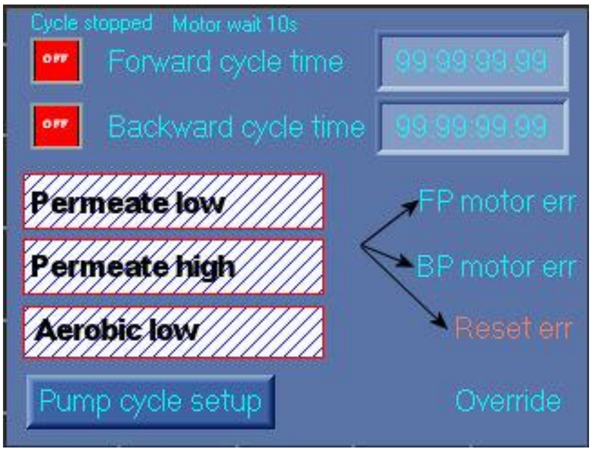


These can be pushed to enable and disable the respective forward and backward cycles of the normal operating cycle. If a cycle is disabled the other cycle will run by default. Cycles can only be enabled/disabled if the normal cycle is not in operation (i.e. the system must not be running).

## Tank level warnings and safety features

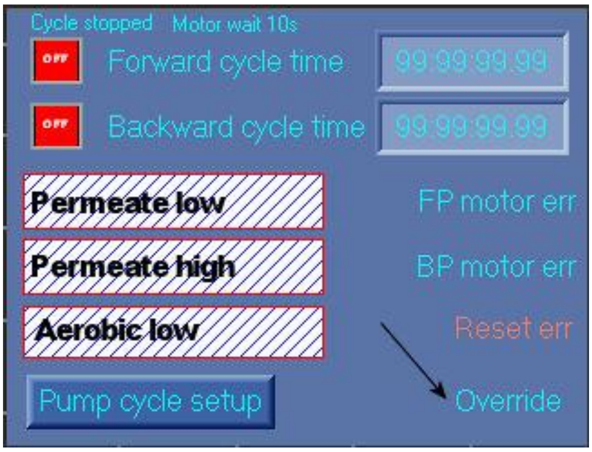


- When the level in the permeate tank is high the forward pump will automatically stop during the normal operating cycle.
- When the level in the aerobic tank is low the forward pump will automatically stop during the normal operating cycle.
- When the level in the permeate tank is low the backward pump will automatically stop during the normal operating cycle.
- In all three the above instances an “error” message will be displayed on the PLC screen indicating the reason for pump stoppage.



## Motor error warnings

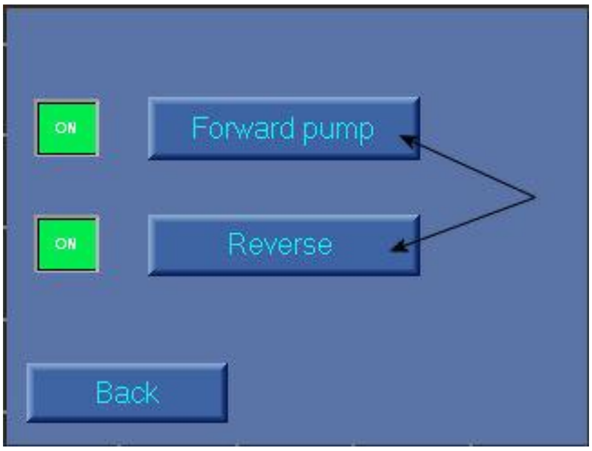
- "FP motor err" indicates the forward pump has an error.
- "BP motor err" indicates the backward pump has an error.
- "Reset err" can be pushed on the PLC screen to reset the error. When the message “Reset err" disappears from the PLC screen the system and pumps can be restarted.



This label is only visible when the normal cycle is disabled.

Pushing this label takes you to the over ride screen.

## Over ride screen



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

Preparing the chemical solutions of NaOCl and citric acid for cleaning in place (CIP), urea and phosphoric acid for dosing, as well as sodium metabisulphite for long-term UF-membrane storage.

## Steps for preparing 130 L of 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%, therefore $C_1$ = 125 g/L and $V_1$ = unknown volume
3. 130 L 400 ppm NaOCl is required, therefore $C_2$ = 0.4 g/L and $V_2$ = 130 L
4. $C_1V_1 = C_2V_2$
5. (125 g/L) x $V_1$ = (0.4 g/L) x (130 L)
6. $V_1$ = 0.416 L per 130 L water
7. Therefore, in order to prepare 130 L of 400 ppm NaOCl, 0.416 L (416 ml) of 12.5% NaOCl must be added to 129.58 L of water and mixed well.

## Steps for preparing 100 L of 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%, therefore $C_1$ = 125 g/L and $V_1$ = unknown volume
3. 100 L 400 ppm NaOCl is required, therefore $C_2$ = 0.4 g/L and $V_2$ = 100 L
4. $C_1V_1 = C_2V_2$
5. (125 g/L) x $V_1$ = (0.4 g/L) x (100 L)
6. $V_1$ = 0.32 L per 100 L water
7. In order to prepare 100 L of 400 ppm NaOCl, 0.32 L (320 ml) of 12.5% NaOCl must be added to 99.68 L of water and mixed well.

## Steps for preparing 130 L of 1% citric acid

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

2. Therefore, $\frac{10g}{1L} = \frac{xg}{130L}$

$$\mathbf{x} = \left(\frac{10}{1}\right) x\, 130$$

$$\mathbf{x} = 1300g$$

3. In order to prepare 130 L of 1% citric acid, 1300 g (1.3 kg) of citric acid must be added to 130 L of water and mixed well.

### Steps for preparing 100 L of 1% citric acid

1. 1% citric acid = 1 g/ 100 ml

   = 10 g /L

2. Therefore, $\frac{10g}{1L} = \frac{xg}{100L}$

   $$\boldsymbol{x} = \left(\frac{10}{1}\right) x\,100$$

   $$\boldsymbol{x} = 1000g$$

3. In order to prepare 100 L of 1% citric acid, 1000 g (1.0 kg) of citric acid must be added to 100 L of water and mixed well.

### Steps for preparing 100 L of 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

3. Concentration of undiluted $H_3PO_4$ ($C_1$) = 80%, therefore $C_1$ = 800 g/L and $V_1$ = unknown volume
4. 100 L 0.5 M $H_3PO_4$ is required, therefore $C_2$ = 48.98 g/L and $V_2$ = 100 L
5. $C_1V_1 = C_2V_2$
6. (800 g/L) x $V_1$ = (48.98 g/L) x (100 L)
7. $V_1$ = 6.12 L per 100 L water
8. Therefore, in order to prepare 100 L of 0.5 M $H_3PO_4$, 6.12 L (6120 ml) of $H_3PO_4$ must be added to 93.88 L of water and mixed well.

### Steps for preparing 100 L of 1.7 M urea

1. Molecular weight of urea (Mw(urea)) = 60 g/mol
2. 1.7 M = 1.7 mol/L x Mw(urea)

   = 1.7 x 60

   = 102 g/L

3. Therefore, $\frac{102g}{1L} = \frac{xg}{100L}$

   $$\boldsymbol{x} = \left(\frac{102}{1}\right) x\,100$$

$$\boldsymbol{x} = 10200g$$

4. Therefore, in order to prepare 100 L of 1.7 M urea, 10200 g (10.2 kg) of urea must be added to 100 L of water and mixed well.

## Steps for preparing 130 L of 1% sodium metabisulphite

1. 1% sodium metabisulphite = 1 g/ 100 ml

   = 10 g /L

2. Therefore, $\frac{10g}{1L} = \frac{\boldsymbol{x}g}{130L}$

$$\boldsymbol{x} = \left(\frac{10}{1}\right) x\, 130$$

$$\boldsymbol{x} = 1300g$$

3. Therefore, in order to prepare 130 L of 1% sodium metabisulphite, 1300 g (1.3 kg) of sodium metabisulphite must be added to 130 L of water and mixed well.

## Steps for preparing 100 L of 1% sodium metabisulphite

1. 1% sodium metabisulphite = 1 g/ 100 ml

   = 10 g /L

2. Therefore, $\frac{10g}{1L} = \frac{\boldsymbol{x}g}{100L}$

$$\boldsymbol{x} = \left(\frac{10}{1}\right) x\, 100$$

$$\boldsymbol{x} = 1000g$$

3. Therefore, in order to prepare 100 L of 1% sodium metabisulphite, 1000 g (1.0 kg) of sodium metabisulphite must be added to 100 L of water and mixed well.

## APPENDIX C: PCSTestr 35 multiparameter for pH, temperature, conductivity and total dissolved solids (TDS) determination

### Calibration of the PCSTestr 35:

- The PCSTestr 35 must be calibrated before-use.
- Switch the PCSTestr 35 on.
- Place 100 ml of distilled water in a 250 ml beaker and place the PCSTestr probe in the distilled water for 2 minutes.
- Take PCSTestr 35 out of distilled water and pat dry on the tissue paper. Do not rub, since rubbing can cause static electricity which may damage the PCSTestr 35 probe.

### pH Calibration:

1. Press the mode button on the PCSTestr 35 until the pH screen is reached.
2. Press the CAL button on the PCSTestr 35. The calibration screen is opened on the digital display and the bottom row flashes 4.01, 7.00 and 10.00.
3. Place the PCSTestr 35 probe in the pH 4 buffer solution and wait. The bottom reading will display the correct buffer value.
4. Keep the PCSTestr 35 in the pH 4 buffer solution until the top reading on the digital display stabilises.
5. Press the MODE/ENT button. The pH 4 buffer calibration is completed.
6. Rinse the PCSTestr 35 probe in the distilled water and pat dry.
7. Place PCSTestr 35 probe in the pH 7 buffer solution and allow the top reading on the digital display to stabilise.
8. Press the MODE/ENT button. The pH 7 buffer calibration is completed.
9. When the calibration is finished, press the CAL key to exit the pH calibration mode. Rinse the PCSTestr 35 probe in the distilled water and pat dry

### TDS and Conductivity calibration:

1. Press the MODE button on the PCSTestr 35 till the TDS screen is reached.
2. Place the PCSTestr 35 probe in the 300 ppm TDS buffer solution and press the CAL button to start the calibration.
3. Press the HOLD button to increase the value in the top digital display screen and the CAL button to decrease the value in the top digital display screen until the value is set to the known concentration of the buffer (i.e. 300 ppm).
4. When the desired value is reached, press the CAL button to confirm the calibration and return to the main screen.
5. The above procedure is repeated for the conductivity calibration using the 1,413 µS/cm conductivity buffer solution.

## Determination of pH, TDS and Conductivity

1. When taking reading, approximately 50ml to 100ml of the required sample is placed in a 250ml beaker.
2. The PCSTestr 35 is switched on and the MODE button pressed until the desired variable for measuring is reached (i.e. pH, TDS or conductivity).
3. The front 3 cm of the PCSTestr 35 probe is submerged in the 250 ml beaker containing the sample.
4. Keep the PCSTestr 35 probe submerged in the sample until the required reading has stabilised.
5. The measurement of each variable is done in duplicate.
6. This procedure is followed for each measured variable.

# APPENDIX D: Flow rate determination and mass balance over the pilot plant

## Steps for determining the flow rate from the 1000 L anaerobic tank (TNK002) to the anoxic tank (TNK003)

1. Ensure that P3 is on and set to a maximum speed of 10.
2. Throttle VLV-007 and VLV-008.
3. Place bucket below the sampling tap; close VLV-032 and open VLV-031 to flush liquid.
4. Place the measure cylinder under the tap and record the time (using the stopwatch) and volume of the liquid (using the measuring cylinder).
5. Use a calculator to determine the flow rate obtained.
6. Adjust the flow rate to correspond to the mass balance of the chosen recycle mode by throttling VLV-007 and VLV-008.

## Steps for determining the flow rate of the recycle stream from the 2500 L anoxic tank (TNK003) to the 1000 L anaerobic tank (TNK002)

1. Ensure that P4 is on and set at a maximum speed of 10.
2. Throttle VLV-011 and VLV-012.
3. Place the bucket below the sampling tap.
4. Close VLV-034 and open VLV-033 to flush out liquid.
5. Place the measure cylinder under the tap and record the time (using the stopwatch) and volume of the liquid (using the measuring cylinder).
6. Use a calculator to determine the flow rate obtained.
7. Adjust the flow rate to correspond to the mass balance of the chosen recycle mode by throttling VLV-011 and VLV-012.

## Steps for determining the flow rate from the 2500 L anoxic tank (TNK003) to the 2500 L aerobic tank (TNK004)

1. Ensure that P4 remains on and set to a maximum speed of 10.
2. Throttle VLV-013.
3. Place the bucket below the sampling tap.
4. Close VLV-036 and open VLV-035 to flush out liquid.
5. Place the measure cylinder under the tap and record the time (using the stopwatch) and volume of the liquid (using the measuring cylinder).
6. Use a calculator to determine the flow rate obtained.
7. Adjust the flow rate to correspond to the mass balance of the chosen recycle mode by throttling VLV-013.

### Steps for determining the flow rate of the MBR retentate recycle to the anoxic tank (TNK003)

1. Ensure that the MBR unit is running and VLV-045 is throttled to achieve the desired flow rate.
2. Place bucket below the sampling tap.
3. Close VLV-046 and open VLV-047 to flush out liquid.
4. Place the measure cylinder under the tap and record the time (using the stopwatch) and volume of the liquid (using the measuring cylinder).
5. Use a calculator to determine the flow rate obtained.
6. Adjust the flow rate to correspond to the mass balance of the chosen recycle mode by throttling VLV-045.

### Procedure

1. Set the varispeed centrifugal pumps P3 and P4 to a maximum speed of 10 in the circuit box located on the wall opposite the biological tanks.
2. Use the mass balances in Figures D.1 to D.9 A to obtain the required mode of operation by throttling VLV-007, VLV-008, VLV-011, VLV-012, VLV-013, VLV-014 and VLV-015.
3. Check that the correct recycle mode (Figures D.1 to D.9) is obtained by measuring the flow rates and ensuring the flow rates correspond to the flow rates indicated Tables D.1 and D.2.

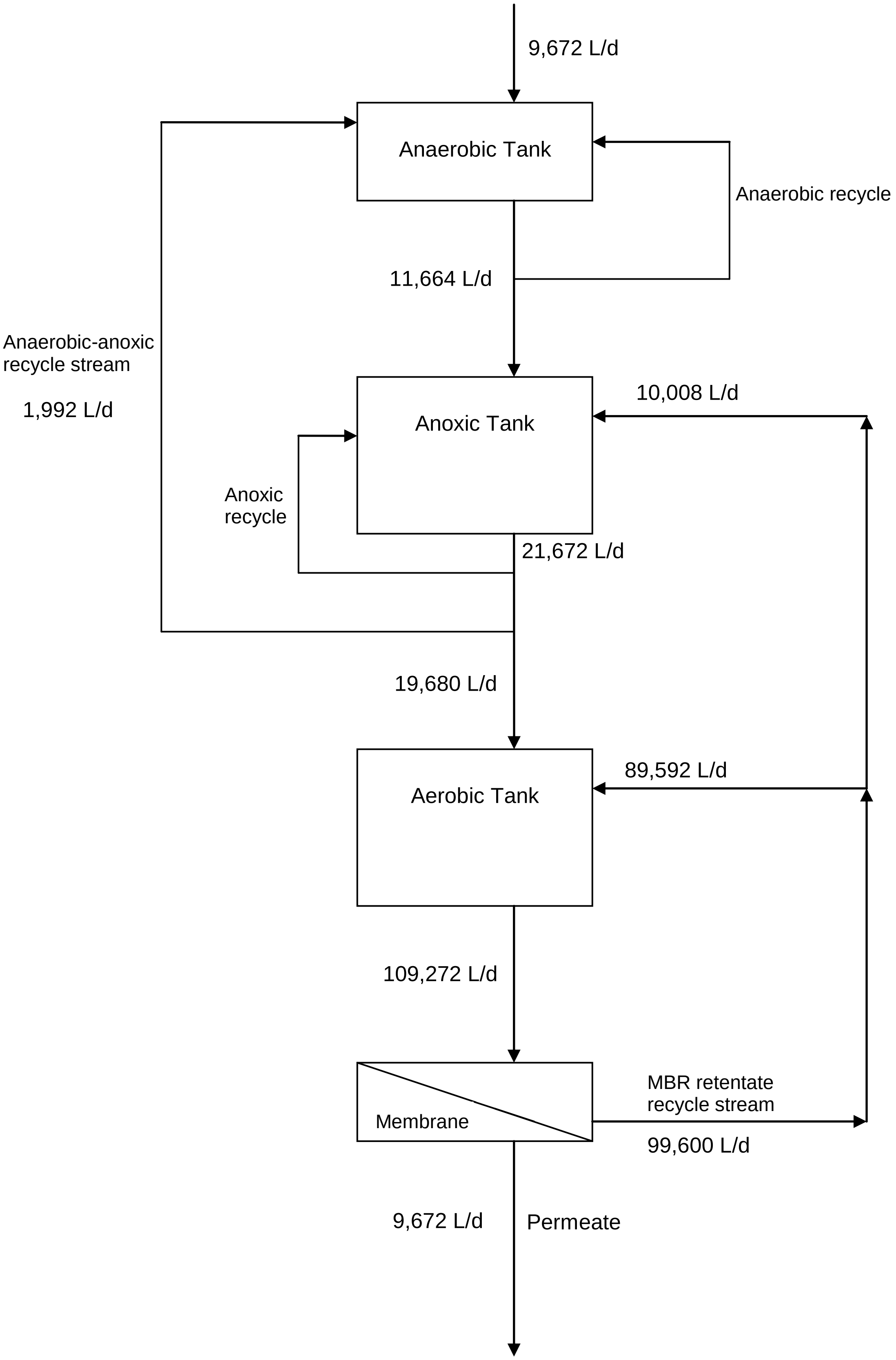


**Figure D.1:** Block flow diagram of mode 1 of operation

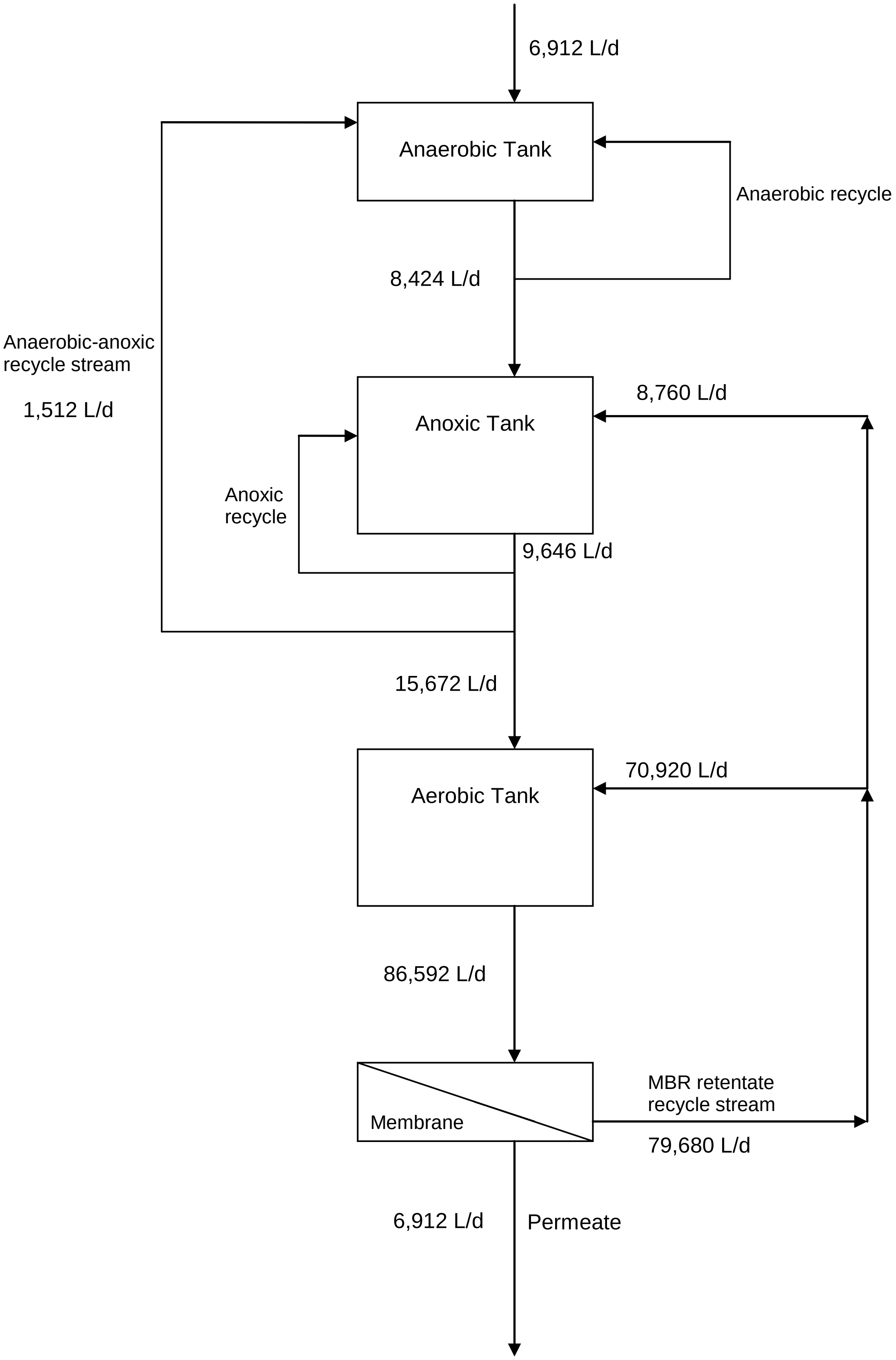


**Figure D.2:** Block flow diagram of mode 2 of operation

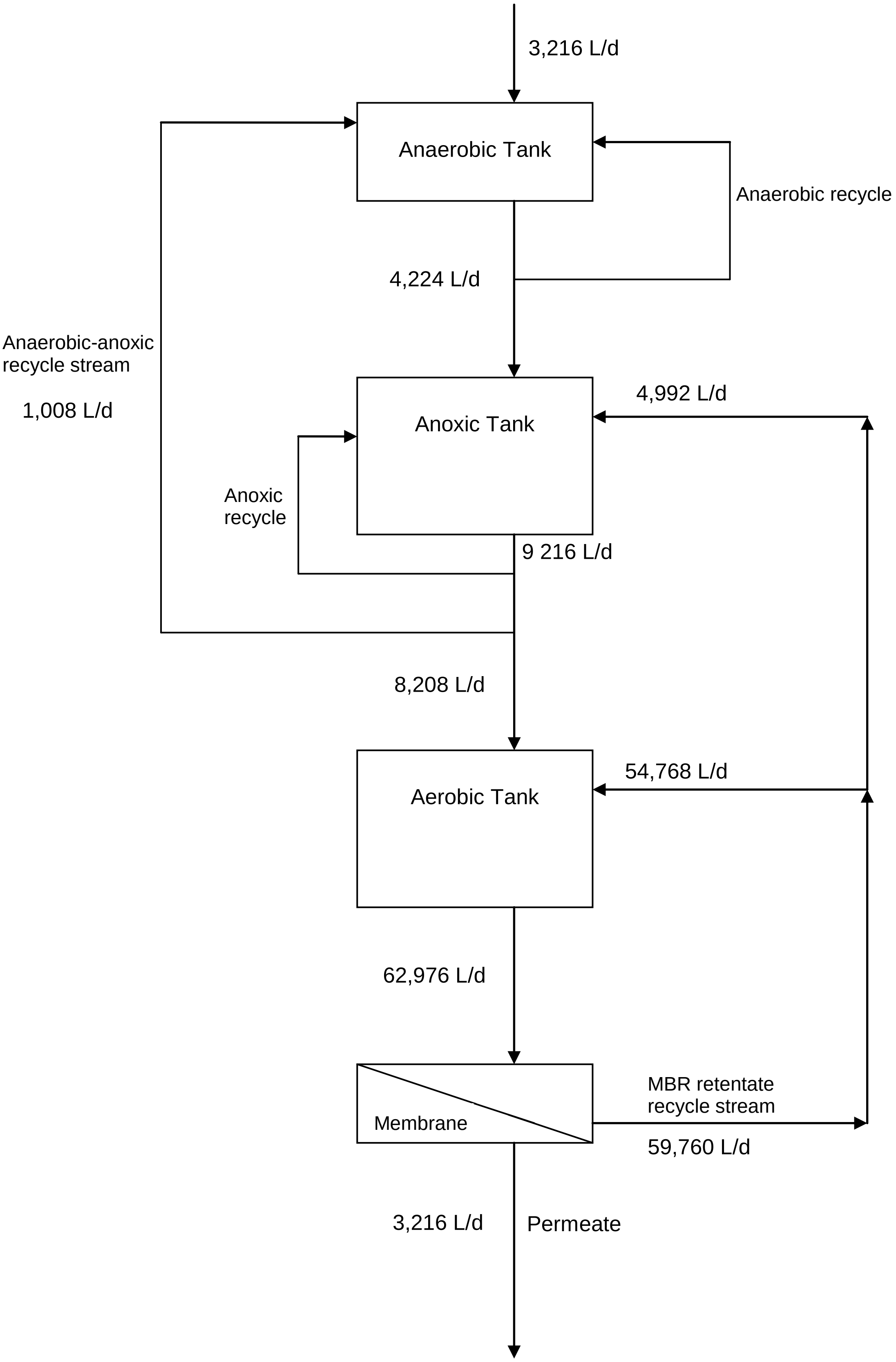


**Figure D.3:** Block flow diagram of mode 3 of operation

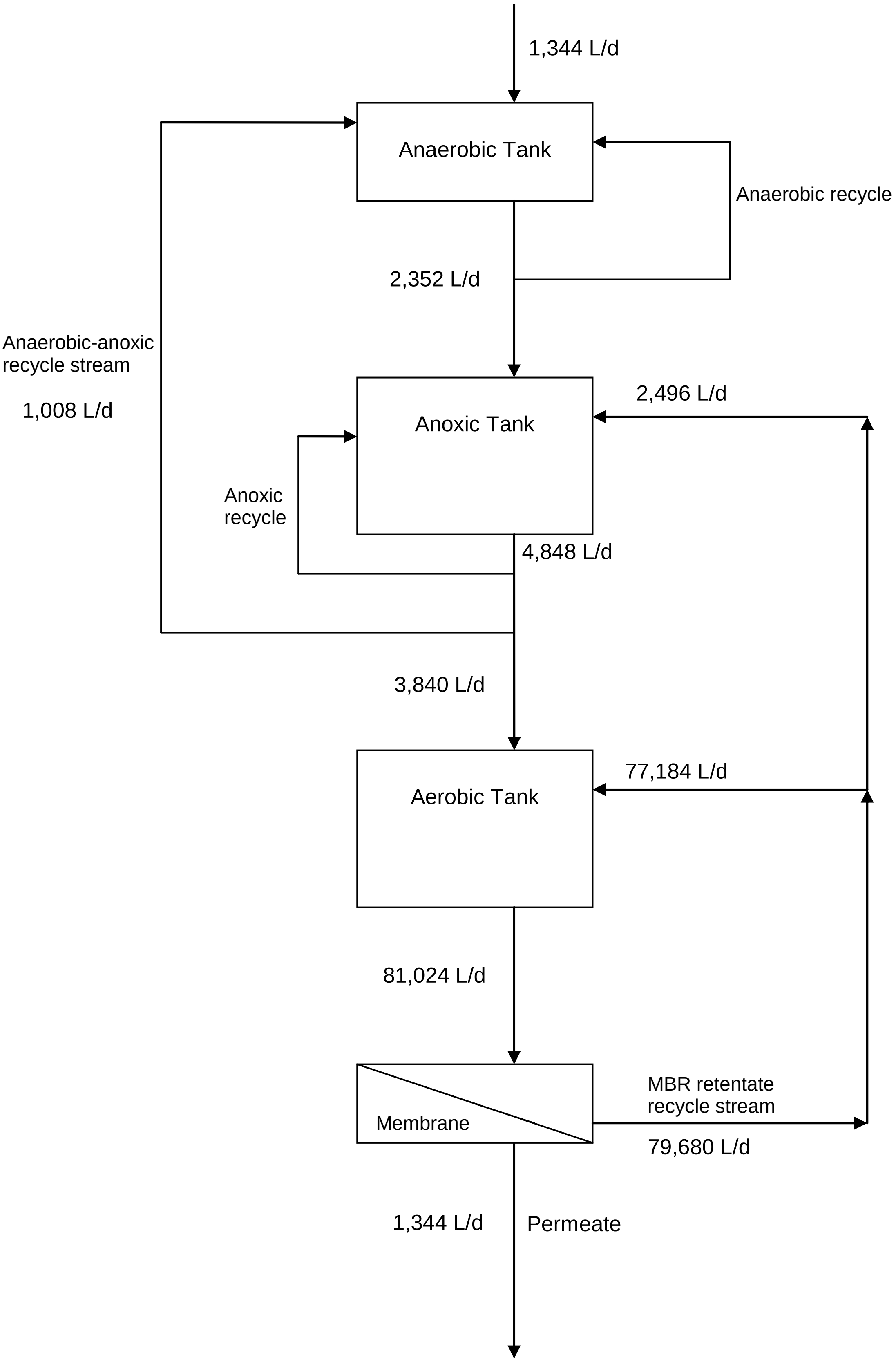


**Figure D.4:** Block flow diagram of mode 4 of operation

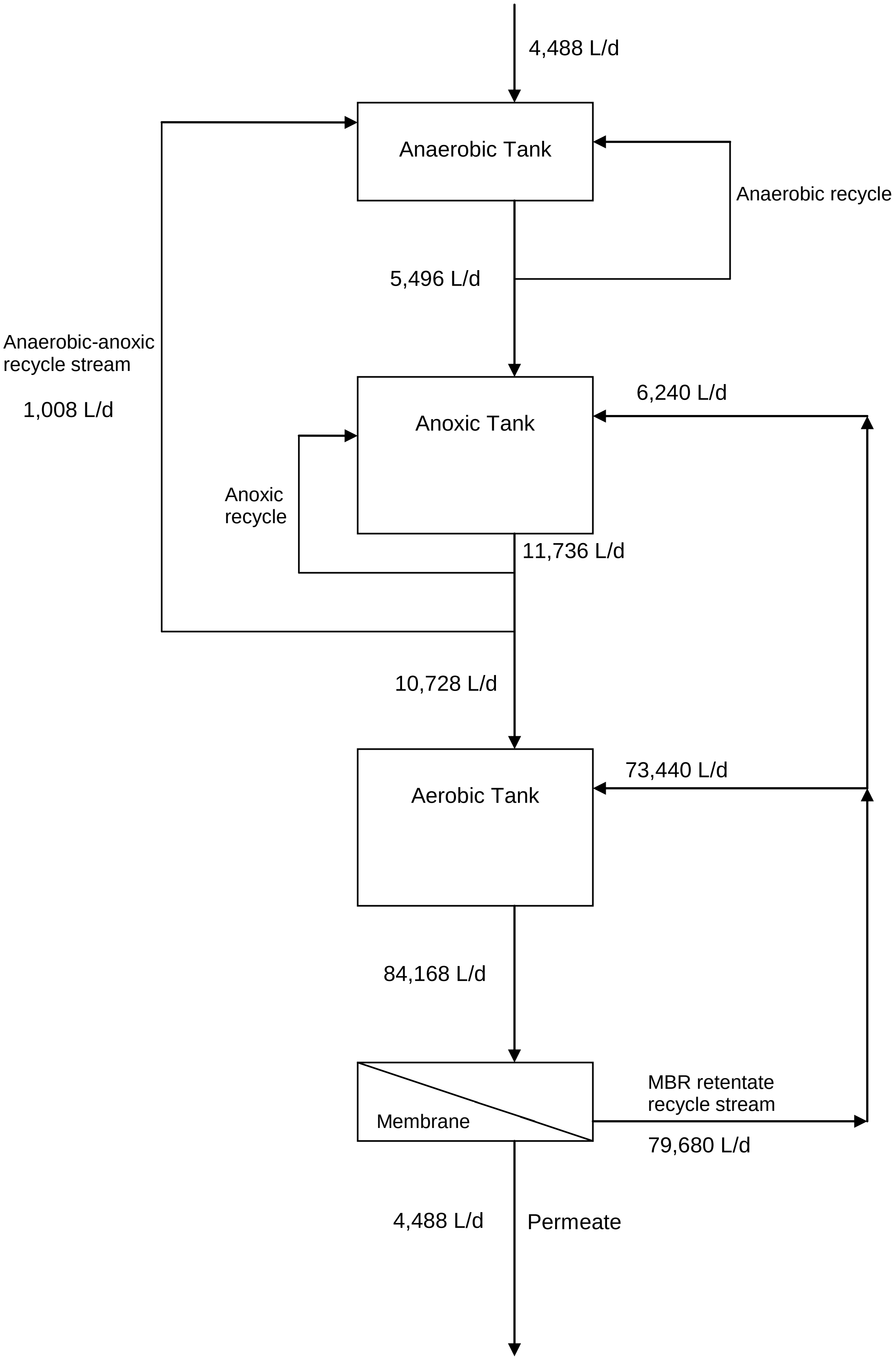


**Figure D.5:** Block flow diagram of mode 5 of operation

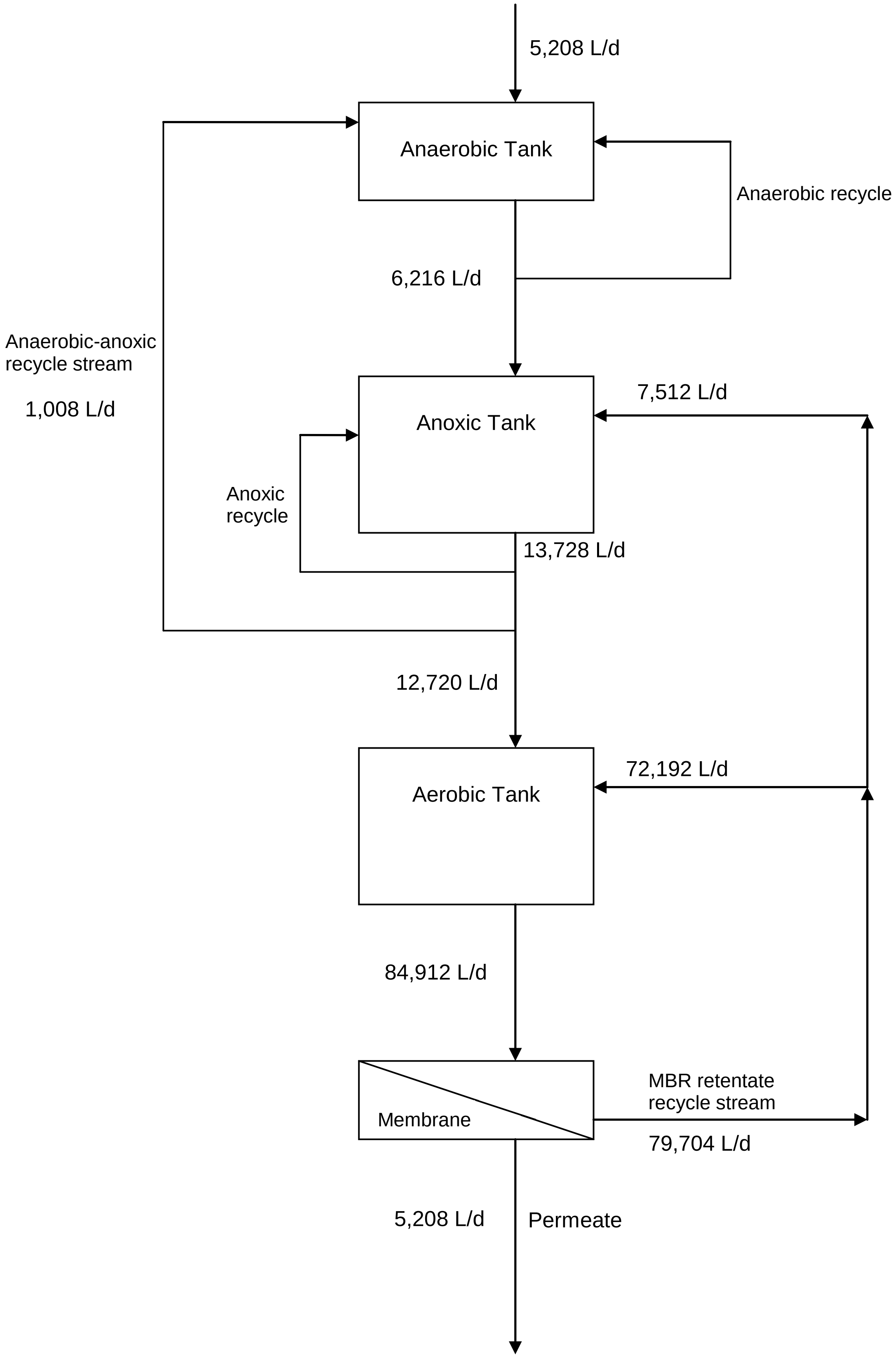


**Figure D.6:** Block flow diagram of mode 6 of operation

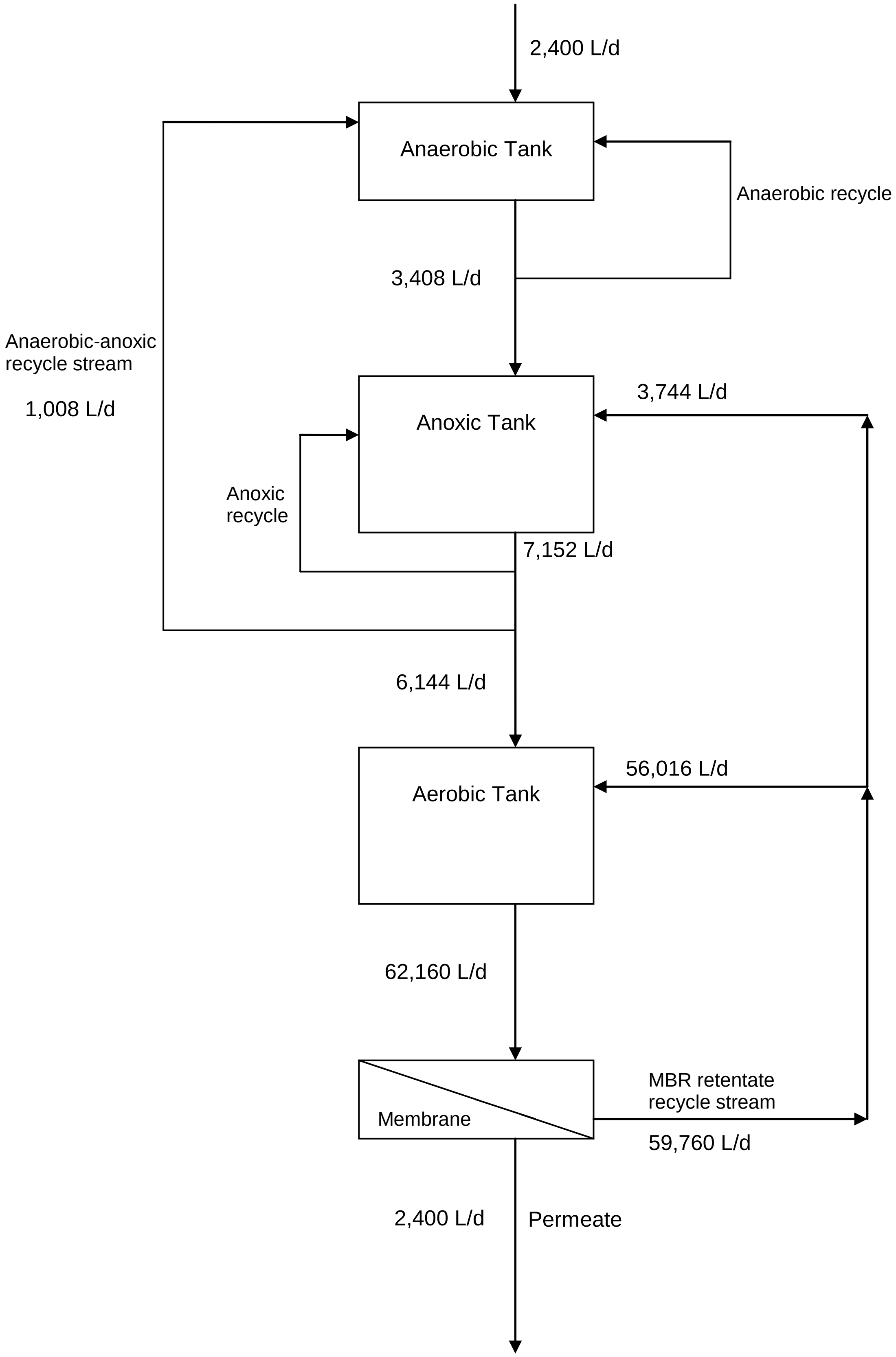


**Figure D.7:** Block flow diagram of mode 7 of operation

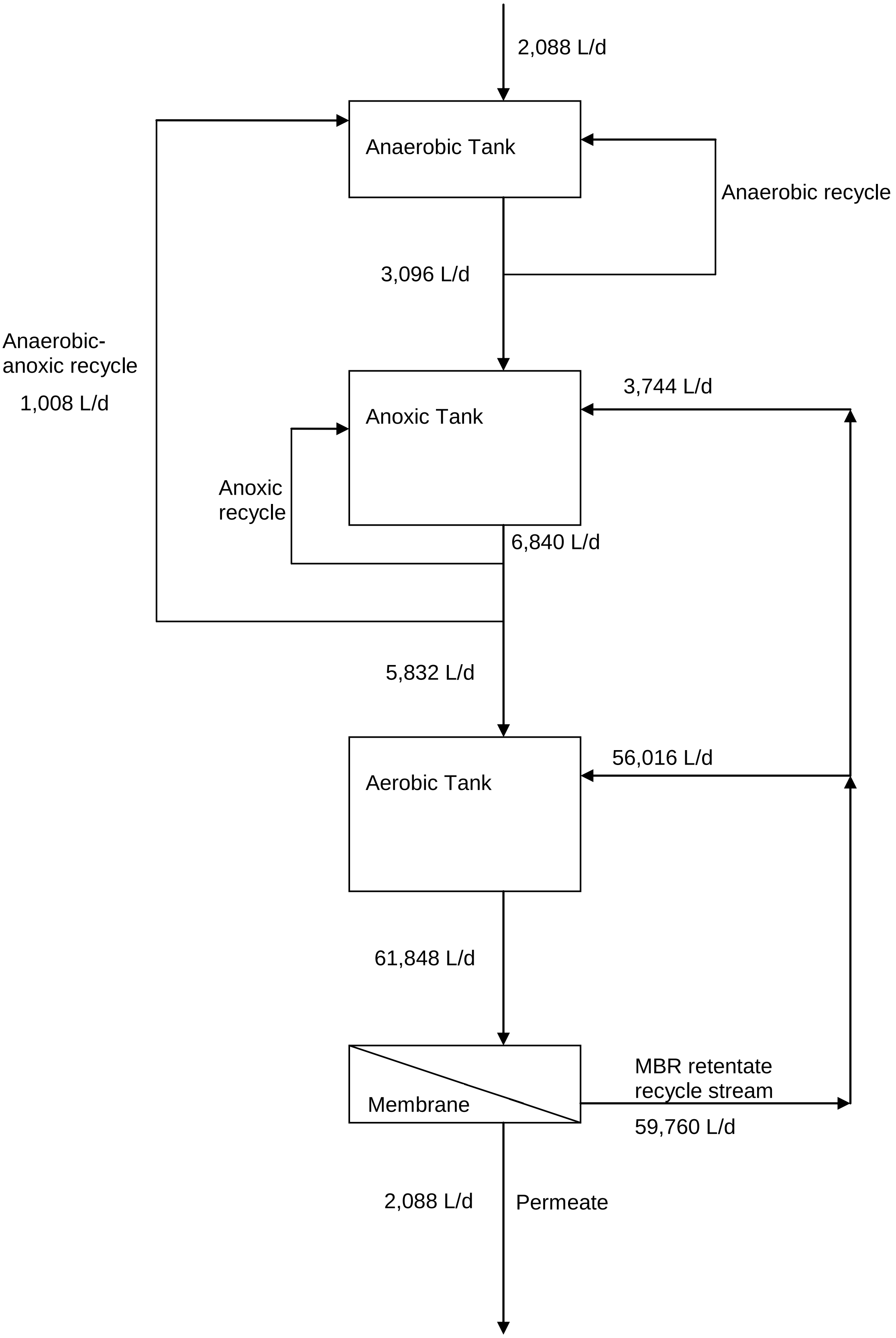


**Figure D.8:** Block flow diagram of mode 8 of operation

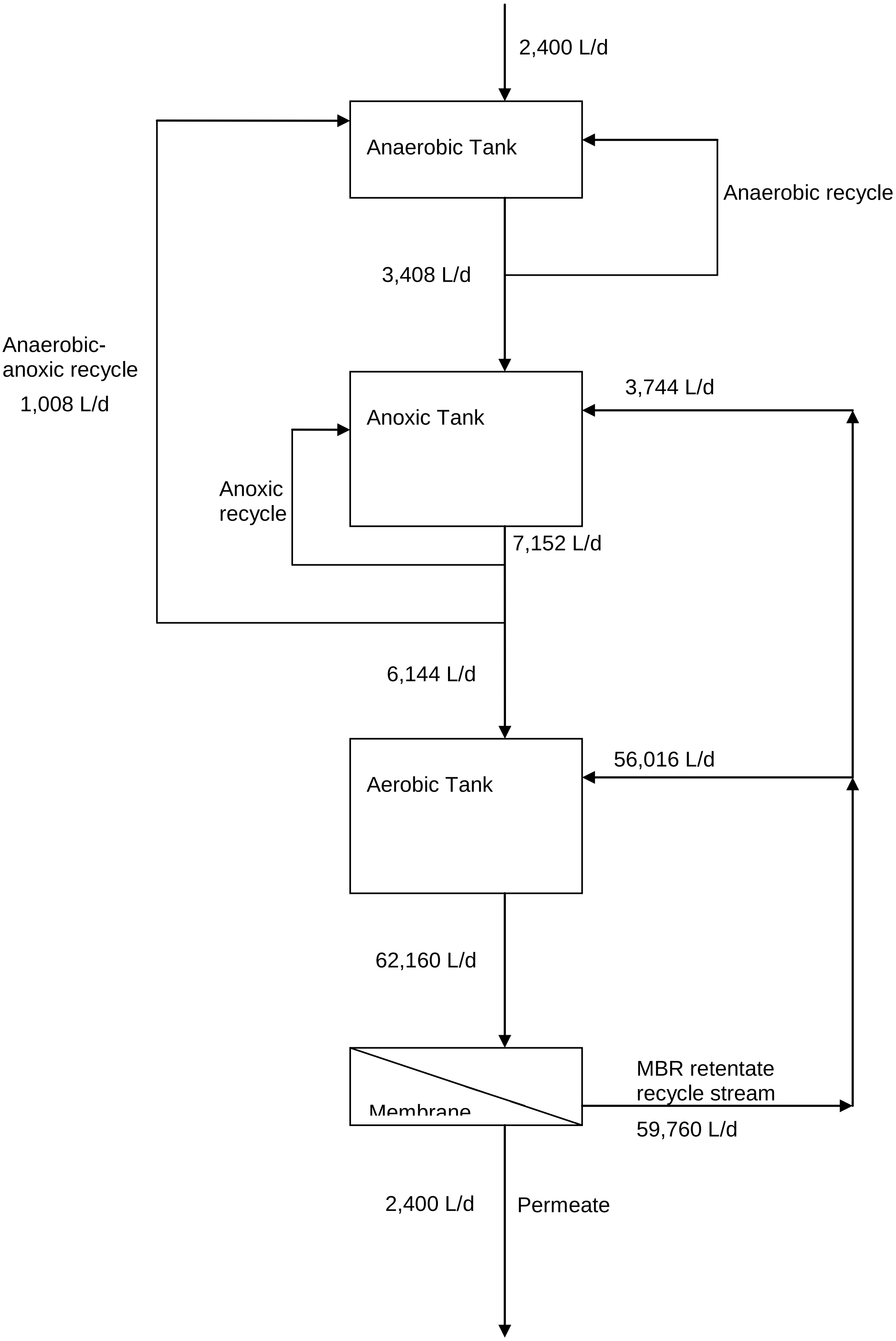


**Figure D.9:** Block flow diagram of mode 9 of operation

**Table D.1:** Operating parameters for modes 1 to 5 of operation

| Pump Tag | Min. m$^3$/d | Max. m$^3$/d | Min. L/h | Max. L/h | Parameter | Mode 1 | Mode 2 | Mode 3 | Mode 4 | Mode 5 |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Membrane area (m$^2$) | 10.2 | 10.2 | 10.2 | 10.2 | 10.2 |
| **VARIABLE (5 - 40L/m$^2$/h)** | | | | | J (flux (L/m$^2$/h) | 39.51 | 28.24 | 13.14 | 5.49 | 18.33 |
| | | | | | Volumetric flux (L/d) | 9,672 | 6,912 | 3,216 | 1,344 | 4,488 |
| | | | | | Influent flow rate Q (L/d) | 9,672 | 6,912 | 3,216 | 1,344 | 4,488 |
| | | | | | COD mg/L (average) | 570 | 2,471 | 1,481 | 424 | 642 |
| | | | | | COD mg/L (minimum) | 155 | 2,363 | 418 | 45 | 561 |
| | | | | | COD mg/L (maximum) | 828 | 2,580 | 2,820 | 780 | 723 |
| | | | | | $OLR_{Avg}$ (kg COD/m$^3$/day) | 0.57 | 2.47 | 1.48 | 0.42 | 0.64 |
| | | | | | $OLR_{Min}$ (kg COD/m$^3$/day) | 0.16 | 2.36 | 0.42 | 0.05 | 0.56 |
| | | | | | $OLR_{Max}$ (kg COD/m$^3$/day) | 0.83 | 2.58 | 2.82 | 0.78 | 0.72 |
| | | | | | Loading rate (kg COD/day) avg. | 5.51 | 17.08 | 4.76 | 0.57 | 2.88 |
| | | | | | Loading rate (kg COD/day) min. | 1.50 | 16.33 | 1.34 | 0.06 | 2.52 |
| | | | | | Loading rate (kg COD/day) max. | 0.47 | 17.83 | 9.07 | 1.05 | 3.24 |
| | | | | | BOD mg/L (average) | 192.5 | 192.50 | 192.50 | 192.50 | 192.50 |
| | | | | | $NH_4$ mg/L (average) | 10 | 13.4 | 15 | 4.4 | 2.1 |
| | | | | | Loading rate (kg $NH_4$/day) | 0.097 | 0.093 | 0.049 | 0.006 | 0.009 |
| | | | | | $PO_4$ mg/L (average) | 8.0 | 1.5 | 3.7 | 3.4 | 1.5 |
| | | | | | Loading rate (kg $PO_4$/day) | 0.077 | 0.010 | 0.012 | 0.005 | 0.007 |
| | | | | | C:N:P (additional N required) | 100:0.18:1.4 | 100:0.05:0.06 | 100:0.10:0.25 | 100:0.10:0.80 | 100:0.03:0.23 |
| | | | | | $Vol._{EQUALISATION}$ (L) | 10,000 | 10,000 | 10,000 | 10,000 | 10,000 |
| | | | | | Inlet $Q_{SUMP}$ (L/day) | 10,672 | 7,912 | 4,216 | 2,344 | 5,488 |
| | | | | | Outlet $rQ_{SUMP}$ (L/day) | 1,000 | 1,000 | 1,000 | 1,000 | 1,000 |
| **GRAVITY** | **9.67** | **5.21** | **403** | **217** | **Outlet $Q_{TOTAL}$ (L/day)** | **9,672** | **6,912** | **3,216** | **1,344** | **4,488** |
| | | | | | $HRT_{EQUALISATION}$ (h) | 24.81 | 34.72 | 74.63 | 178.57 | 53.48 |
| **ANAEROBIC TANK** | | | | | | | | | | |
| | | | | | $Vol._{ANAEROBIC}$ (L) | 1,000 | 1,000 | 1,000 | 1,000 | 1,000 |

**Table D.1 (continued):** Operating parameters for modes 1 to 5 of operation

| Pump Tag | Min. m$^3$/d | Max. m$^3$/d | Min. L/h | Max. L/h | Parameter | Mode 1 | Mode 2 | Mode 3 | Mode 4 | Mode 5 |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Inlet $Q_{EQUAL}$ (L/day) | 9,672 | 6,912 | 3,216 | 1,344 | 4,488 |
| **VARIABLE (100-200%)** | | | | | Inlet $rQ_{ANOXIC}$ (% x Vol.$_{ANAEROBIC}$) | 200 | 150 | 100 | 100 | 100 |
| | | | | | Inlet $rQ_{ANOXIC}$ (L/day) | 2,000 | 1,500 | 1,000 | 1,000 | 1,000 |
| | | | | | Inlet $rQ_{ANOXIC}$ (L/h) | 83 | 63 | 42 | 42 | 42 |
| | | | | | Inlet $Q_{TOTAL}$ (L/day) | 11,672 | 8,412 | 4,216 | 2,344 | 5,488 |
| **P3** | **5.40** | **10.80** | **225** | **450** | **Outlet $Q_{TOTAL}$ (L/day)** | **11,672** | **8,412** | **4,216** | **2,344** | **5,488** |
| | | | | | $HRT_{ANAEROBIC}$ (h) | 2.06 | 2.85 | 5.69 | 10.24 | 4.37 |
| **ANOXIC TANK** | | | | | | | | | | |
| | | | | | Vol.$_{ANOXIC}$ (L) | 2,500 | 2,500 | 2,500 | 2,500 | 2,500 |
| | | | | | Inlet $Q_{ANAEROBIC}$ (L/day) | 11,672 | 8,412 | 4,216 | 2,344 | 5,488 |
| **VARIABLE (100-400%)** | | | | | Inlet $rQ_{MBR/ANOX}$ (% x Vol.$_{ANOXIC}$) | 400 | 350 | 200 | 100 | 250 |
| | | | | | Inlet $rQ_{MBR/ANOX}$ (L/day) | 10,000 | 8,750 | 5,000 | 2,500 | 6,250 |
| | | | | | Inlet $rQ_{MBR/ANOX}$ (L/h) | 417 | 365 | 208 | 104 | 260 |
| | | | | | Inlet $Q_{TOTAL}$ (L/day) | 21,672 | 17,162 | 9,216 | 4,844 | 11,738 |
| | | | | | Outlet $rQ_{ANOXIC}$ (% x Vol.$_{ANAEROBIC}$) | 200 | 150 | 100 | 100 | 100 |
| | | | | | Outlet $rQ_{ANOXIC}$ (L/day) | 2,000 | 1,500 | 1,000 | 1,000 | 1,000 |
| **P4** | **6.90** | **20.00** | **288** | **833** | **Outlet $Q_{TOTAL}$ (L/day)** | **19,672** | **15,662** | **8,216** | **3,844** | **10,738** |
| | | | | | $HRT_{ANOXIC}$ (h) | 3.05 | 3.83 | 7.30 | 15.61 | 5.59 |
| **AEROBIC TANK** | | | | | | | | | | |
| | | | | | Vol.$_{AEROBIC}$ (L) | 2,500 | 2,500 | 2,500 | 2,500 | 2,500 |
| | | | | | Inlet $Q_{ANOXIC}$ (L/day) | 19,672 | 15,662 | 8,216 | 3,844 | 10,738 |
| | | | | | Inlet $rQ_{MBR}$ (% of total recycle) | 11.16 | 12.34 | 9.13 | 3.24 | 8.51 |
| | | | | | Inlet $rQ_{MBR}$ (L/day) | 89,600 | 70,930 | 54,760 | 77,180 | 73,430 |
| **VARIABLE (0.3 - 0.5m/s)** | | | | | Outlet $V_{CROSSFLOW}$ (m/s) | 0.50 | 0.40 | 0.30 | 0.40 | 0.40 |

**Table D.1 (continued):** Operating parameters for modes 1 to 5 of operation

| **Pump Tag** | **Min. m$^3$/d** | **Max. m$^3$/d** | **Min. L/h** | **Max. L/h** | **Parameter** | **Mode 1** | **Mode 2** | **Mode 3** | **Mode 4** | **Mode 5** |
|---|---|---|---|---|---|---|---|---|---|---|
| **P6** | **59.76** | **99.60** | **2,490** | **4,150** | **Outlet $Q_{CROSSFLOW}$ (8.3 x V) = m$^3$/h** | **4.15** | **3.32** | **2.49** | **3.32** | **3.32** |
| | | | | | Outlet $Q_{CROSSFLOW}$ (L/d) | 99,600 | 79,680 | 59,760 | 79,680 | 79,680 |
| | | | | | Outlet $Q_{CROSSFLOW}$ (L/h) | 4,150 | 3,320 | 2,490 | 3,320 | 3,320 |
| | | | | | Outlet $rQ_{MBR/ANOX}$ (% x Vol.$_{ANOXIC}$) | 400 | 350 | 200 | 100 | 250 |
| | | | | | Outlet $rQ_{MBR/ANOX}$ (L/day) | 10,000 | 8,750 | 5,000 | 2,500 | 6,250 |
| **P7** | **4.40** | **9.80** | **183** | **408** | **Outlet $Q_{TOTAL}$ (L/day)** | **9,672** | **6,912** | **3,216** | **1,344** | **4,488** |
| | | | | | $HRT_{AEROBIC}$ | 6.20 | 8.68 | 18.66 | 44.64 | 13.37 |
| | | | | | **HRT (h) (total)** | **11.31** | **15.36** | **31.65** | **70.49** | **23.33** |
| **P1** | **SUBMERSIBLE (RECIRCULATION)** | | | | | | | | | |
| **P2** | **SUBMERSIBLE (RECIRCULATION)** | | | | | | | | | |
| **P5** | **SUBMERSIBLE (RECIRCULATION)** | | | | | | | | | |
| **P8** | **DOSING PUMP - UREA** | | | | | | | | | |
| **P9** | **DOSING PUMP - PHOSPHORIC ACID** | | | | | | | | | |

**Table D.2:** Operating parameters for modes 6 to 9 of operation

| **Pump Tag** | **Min. m$^3$/d** | **Max. m$^3$/d** | **Min. L/h** | **Max. L/h** | **Parameter** | **Mode 6** | **Mode 7** | **Mode 8** | **Mode 9** |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Membrane area (m$^2$) | 10.2 | 10.2 | 10.2 | 10.2 |
| **VARIABLE (5 - 40L/m$^2$/h)** | | | | | J (flux (L/m$^2$/h) | 21.27 | 9.80 | 8.53 | 9.80 |
| | | | | | Volumetric flux (L/d) | 5,208 | 2,400 | 2,088 | 2,400 |
| | | | | | Influent flow rate Q (L/d) | 5,208 | 2,400 | 2,088 | 2,400 |
| | | | | | COD mg/L (average) | 801 | 1680 | 2420 | 1950 |
| | | | | | COD mg/L (minimum) | 440 | 351 | 921 | 995 |
| | | | | | COD mg/L (maximum) | 1,280 | 3890 | 5155 | 3073 |
| | | | | | OLR$_{Avg}$ (kg COD/m$^3$/day) | 0.57 | 1.57 | 2.57 | 3.57 |
| | | | | | OLR$_{Min}$ (kg COD/m$^3$/day) | 0.44 | 0.35 | 0.92 | 1.00 |
| | | | | | OLR$_{Max}$ (kg COD/m$^3$/day) | 1.28 | 3.89 | 5.16 | 3.07 |
| | | | | | Loading rate (kg COD/day) avg. | 2.97 | 3.77 | 5.37 | 8.57 |
| | | | | | Loading rate (kg COD/day) min. | 2.29 | 0.84 | 1.92 | 2.39 |
| | | | | | Loading rate (kg COD/day) max. | 6.67 | 9.34 | 10.76 | 7.38 |
| | | | | | BOD mg/L (average) | 192.50 | 193.50 | 194.50 | 195.50 |
| | | | | | NH$_4$ mg/L (average) | 3.9 | 6.3 | 13.5 | 12.4 |
| | | | | | Loading rate (kg NH$_4$/day) | 0.020 | 0.015 | 0.028 | 0.030 |
| | | | | | PO$_4$ mg/L (average) | 0.4 | 1.7 | 1.1 | 1.2 |
| | | | | | Loading rate (kg PO$_4$/day) | 0.002 | 0.004 | 0.002 | 0.003 |
| | | | | | C:N:P (additional N required) | 100:0.05:0.05 | 100:0.04:0.10 | 100:0.06:0.05 | 100:0.06:0.06 |
| | | | | | Vol.$_{EQUALISATION}$ (L) | 10,000 | 10,000 | 10,000 | 10,000 |
| | | | | | Inlet Q$_{SUMP}$ (L/day) | 6,208 | 3,400 | 3,088 | 3,400 |
| | | | | | Outlet rQ$_{SUMP}$ (L/day) | 1,000 | 1,000 | 1,000 | 1,000 |
| **GRAVITY** | **9.67** | **5.21** | **403** | **217** | **Outlet Q$_{TOTAL}$ (L/day)** | **5,208** | **2,400** | **2,088** | **2,400** |
| | | | | | HRT$_{EQUALISATION}$ (h) | 46.08 | 100.00 | 114.94 | 100.00 |
| **ANAEROBIC TANK** | | | | | | | | | |
| | | | | | Vol.$_{ANAEROBIC}$ (L) | 1,000 | 1,000 | 1,000 | 1,000 |

**Table D.2 (continued):** Operating parameters for modes 6 to 9 of operation

| Pump Tag | Min. m$^3$/d | Max. m$^3$/d | Min. L/h | Max. L/h | Parameter | Mode 6 | Mode 7 | Mode 8 | Mode 9 |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Inlet $Q_{EQUAL}$ (L/day) | 5,208 | 2,400 | 2,088 | 2,400 |
| **VARIABLE (100-200%)** | | | | | Inlet $rQ_{ANOXIC}$ (% x Vol.$_{ANAEROBIC}$) | 100 | 100 | 100 | 100 |
| | | | | | Inlet $rQ_{ANOXIC}$ (L/day) | 1,000 | 1,000 | 1,000 | 1,000 |
| | | | | | Inlet $rQ_{ANOXIC}$ (L/h) | 42 | 42 | 42 | 42 |
| | | | | | Inlet $Q_{TOTAL}$ (L/day) | 6,208 | 3,400 | 3,088 | 3,400 |
| **P3** | **5.40** | **10.80** | **225** | **450** | **Outlet $Q_{TOTAL}$ (L/day)** | **6,208** | **3,400** | **3,088** | **3,400** |
| | | | | | $HRT_{ANAEROBIC}$ (h) | 3.87 | 7.06 | 7.77 | 7.06 |
| **ANOXIC TANK** | | | | | | | | | |
| | | | | | Vol.$_{ANOXIC}$ (L) | 2,500 | 2,500 | 2,500 | 2,500 |
| | | | | | Inlet $Q_{ANAEROBIC}$ (L/day) | 6,208 | 3,400 | 3,088 | 3,400 |
| **VARIABLE (100-400%)** | | | | | Inlet $rQ_{MBR/ANOX}$ (% x Vol.$_{ANOXIC}$) | 300 | 150 | 150 | 150 |
| | | | | | Inlet $rQ_{MBR/ANOX}$ (L/day) | 7,500 | 3,750 | 3,750 | 3,750 |
| | | | | | Inlet $rQ_{MBR/ANOX}$ (L/h) | 313 | 156 | 156 | 156 |
| | | | | | Inlet $Q_{TOTAL}$ (L/day) | 13,708 | 7,150 | 6,838 | 7,150 |
| | | | | | Outlet $rQ_{ANOXIC}$ (% x Vol.$_{ANAEROBIC}$) | 100 | 100 | 100 | 100 |
| | | | | | Outlet $rQ_{ANOXIC}$ (L/day) | 1,000 | 1,000 | 1,000 | 1,000 |
| **P4** | **6.90** | **20.00** | **288** | **833** | **Outlet $Q_{TOTAL}$ (L/day)** | **12,708** | **6,150** | **5,838** | **6,150** |
| | | | | | $HRT_{ANOXIC}$ (h) | 4.72 | 9.76 | 10.28 | 9.76 |
| **AEROBIC TANK** | | | | | | | | | |
| | | | | | Vol.$_{AEROBIC}$ (L) | 2,500 | 2,500 | 2,500 | 2,500 |
| | | | | | Inlet $Q_{ANOXIC}$ (L/day) | 12,708 | 6,150 | 5,838 | 6,150 |
| | | | | | Inlet $rQ_{MBR}$ (% of total recycle) | 10.39 | 6.70 | 6.70 | 6.70 |
| | | | | | Inlet $rQ_{MBR}$ (L/day) | 72,180 | 56,010 | 56,010 | 56,010 |
| **VARIABLE (0.3 - 0.5m/s)** | | | | | Outlet $V_{CROSSFLOW}$ (m/s) | 0.40 | 0.30 | 0.30 | 0.30 |

**Table D.2 (continued):** Operating parameters for modes 6 to 9 of operation

| **Pump Tag** | **Min. m$^3$/d** | **Max. m$^3$/d** | **Min. L/h** | **Max. L/h** | **Parameter** | **Mode 6** | **Mode 7** | **Mode 8** | **Mode 9** |
|---|---|---|---|---|---|---|---|---|---|
| **P6** | **59.76** | **99.60** | **2,490** | **4,150** | **Outlet $Q_{CROSSFLOW}$ (8.3 x V) = m$^3$/h** | **3.32** | **2.49** | **2.49** | **2.49** |
| | | | | | Outlet $Q_{CROSSFLOW}$ (L/d) | 79,680 | 59,760 | 59,760 | 59,760 |
| | | | | | Outlet $Q_{CROSSFLOW}$ (L/h) | 3,320 | 2,490 | 2,490 | 2,490 |
| | | | | | Outlet $rQ_{MBR/ANOX}$ (% x Vol.$_{ANOXIC}$) | 300 | 150 | 150 | 150 |
| | | | | | Outlet $rQ_{MBR/ANOX}$ (L/day) | 7,500 | 3,750 | 3,750 | 3,750 |
| **P7** | **4.40** | **9.80** | **183** | **408** | **Outlet $Q_{TOTAL}$ (L/day)** | **5,208** | **2,400** | **2,088** | **2,400** |
| | | | | | $HRT_{AEROBIC}$ | 11.52 | 25.00 | 28.74 | 25.00 |
| | | | | | **HRT (h) (total)** | **20.11** | **41.81** | **46.79** | **41.81** |

| | |
|---|---|
| **P1** | **SUBMERSIBLE (RECIRCULATION)** |
| **P2** | **SUBMERSIBLE (RECIRCULATION)** |
| **P5** | **SUBMERSIBLE (RECIRCULATION)** |
| **P8** | **DOSING PUMP - UREA** |
| **P9** | **DOSING PUMP - PHOSPHORIC ACID** |

# APPENDIX E: Ammonium ($NH_4^+$) determination

Ammonium has a molecular weight of 18.05 and is formed by the protonation of ammonia ($NH_3$). Ammonium is therefore also known as ionised ammonia ($NH_3$) and due its positive electrical charge is a cation with the chemical formula $NH_4^+$. Ammonia nitrogen ($NH_3$-N) is conventionally removed by the biological process of nitrification-denitrification in wastewater treatment.

## Method for determining ammonium (2 – 75 mg/L range):

Using a Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001).

1. Pipette 5 ml of the $NH_4$-1 solution into a test tube using a P5000 pipette.
2. Add 0.2 ml of the sample to the test tube with a P200 pipette.
3. Add 1 level blue microspoon of the $NH_4$-2 powder to the test tube. The microspoon is located in the cap of the $NH_4$-2 bottle.
4. Place the cap on the test tube and mix vigorously with the vortex mixer until the $NH_4$-2 reagent has completely dissolved.
5. Leave the test tube in a test tube rack for 15min.
6. Pipette 1 ml of the mixture from the test tube into a 10 mm cuvette using a P1000 pipette.
7. Place the Autoselector tube for the 2 – 75 mg/L ammonium range into the Nova 60 Spectroquant.
8. Place the 10 mm cuvette in the 10 mm slot of the Nova 60 Spectroquant and record the measurement displayed on the display screen.

## Method for determining ammonium (5 – 150 mg/L range):

Using a Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001).

1. Pipette 5 ml of the $NH_4$-1 solution into a test tube using a P5000 pipette.
2. Add 0.1 ml of the sample to the test tube with a P200 pipette.
3. Add 1 level blue microspoon of the $NH_4$-2 powder to the test tube. The microspoon is located in the cap of the $NH_4$-2 bottle.
4. Place the cap on the test tube and mix vigorously with the vortex mixer until the $NH_4$-2 reagent has completely dissolved.
5. Leave the test tube in a test tube rack for 15 minutes.
6. Pipette 1 ml of the mixture from the test tube into a 10 mm cuvette using a P1000 pipette.
7. Place the Autoselector tube for the 5 – 150 mg/L ammonium range into the Nova 60 Spectroquant.

8. Place the 10 mm cuvette in the 10 mm slot of the Nova 60 Spectroquant and record the measurement displayed on the display screen.

*Notes:*

- Make sure all test tubes are clean and dry.
- All samples must be tested in duplicate. If samples differ with more than 1 unit, the sample must be re-analysed.
- Turbid samples should be filtered before analysis.
- Due to the strong temperature dependence of the colour reaction, the temperature of the reagents should be between 20 and 30°C.
- Do not allow the samples to stand for longer than 15 minutes after all the reagents have been mixed. Decolourisation will start and the readings will be incorrect.
- The colour of the measurement solution remains stable for at least 60 minutes after the end of the reaction time stated above.
- Ammonium determination should be done as soon as the samples have been taken in order to obtain accurate results.
- The measurements obtained from the Nova 60 Spectroquant are $NH_4$-N and therefore these needs to be converted to $NH_4^+$ using Equation E1.

$$NH_4^+(mg/L) = 1.2877(NH_4 - N/mg/L) + 0.0247 \qquad \text{Eq. E1}$$

## References

Merck Spectroquant $NH_4^+$ test kit, Cat. No. 1.00683.0001).

## APPENDIX F: Chemical oxygen demand (COD) determination

Chemical oxygen demand (COD) is a measure of the oxygen required to oxidise all compounds, both organic and inorganic, present in wastewater.

### Method for determining total COD (tCOD):

1. All samples must be tested in duplicate.
2. All apparatus should first be inspected to ensure all is in working order and not damaged.
3. The test cells should be inspected to ensure they are clean and not scratched.
4. Switch on the Spectroquant thermoreactor TR 420 to the preset setting of 148˚C for two hours and allow the thermoreactor to heat up to the desired temperature. This will take approximately 10 minutes.
5. Place approximately 100 ml of distilled water in a 250 ml beaker.
6. When using COD solutions A and B for the 500 to 10 000 mg/L range:

   Using 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.
   - 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.
   - Tightly attach the screw caps to the test cells.
   - Vigorously mix the contents of the test cells with a vortex mixer.
   - 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. Do not cool with cold water.
   - 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.
   - Wipe the outside of the test cells clean and place in the Nova 60 Spectroquant.
   - 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.
7. When using COD solutions A and B for the 100 to 1500 mg/L range:

   Using 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:
   - Pipette 0.30 ml of COD solution A into the test cell.
   - Pipette 2.85 ml of COD solution B into the test cell.

- Pipette 3 ml of sample into the test cell.

### Method for determining soluble COD (sCOD):

- A Büchner funnel is attached to 500 ml suction flask.
- The suction flask is either connected to a water pump or a vacuum pump.
- Glass microfibre filter discs, 5.5 cm in diameter with a 0.45 µm pore size, without organic binder (i.e. Whatman type GF/F (0.7 Fm)) is placed inside the Büchner funnel.
- 200 ml of the raw sample is filtered.
- The COD of the filtered sample is then determined, using the same procedure as for total COD determination.
- The insoluble COD (iCOD) is the difference between the total COD (tCOD) measurement and the soluble COD (sCOD) measurement, calculated using Equation F1.

$$tCOD \quad (mg/L) \; - \; sCOD \quad (mg/L) \; = \; iCOD(mg/L) \qquad \text{Eq. F1}$$

### 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 G: Nitrate ($NO_3$) determination

In wastewater nitrate is a form of organic nitrogen in its highest oxidised form, it is a univalent compound with a formula of $NO_3$. Nitrification is the process by which ammonia is first converted to nitrite and then to nitrate (Judd, 2006).

### Method for nitrate determination (0.5 – 20.0 mg/L $NO_3$-N (2.2 – 88.5 mg/L $NO_3$)):

Using a Merck Spectroquant Nitrate cell test, Cat. No. 1.14773.0001.

1. Place 1 level blue microspoon $NO_3$-1 powder into a test tube. The microspoon is located in the cap of the $NO_3$-1 bottle.
2. Add 5.0 ml of the $NO_3$-2 solution to the test tube using a P5000 pipette.
3. Place the cap on the test tube and mix vigorously with a vortex mixer until the reagent has completely dissolved.
4. Slowly add 1.5 ml of the sample to the test tube using a P5000 pipette.
5. Place the cap on the test tube and mix vigorously with a vortex mixer. *CAUTION*: the test tube will become very hot!
6. Place the test tube in a test tube rack and leave for 10 minutes.
7. Add 1 ml of the mixture from the test tube to a 10 mm cuvette using a P1000 pipette.
8. Place the Autoselector tube for 0.5 – 20.0 mg/L nitrate range in Nova 60 Spectroquant.
9. Place the 10 mm cuvette in the 10 mm slot of the Nova 60 Spectroquant and take measurement.

*Notes*

- The reagents should be stored sealed at a temperature range of 20 to 30°C.
- Nitrate determination should be done as soon as the samples have been taken in order to obtain accurate results.
- Make sure all test tubes are clean and dry.
- All samples must be tested in duplicate.
- Turbid samples should be filtered.

### References

Judd, S. 2006. *The MBR Book: Principles and Applications of Membrane Bioreactors in Water and Wastewater Treatment.* Elsevier: Amsterdam.

Merck Spectroquant Nitrate cell test, Cat. No. 1.14773.0001.

## APPENDIX H: Phosphate ($PO_4$) determination

Total phosphorous is the sum of orthophosphate ($PO_4$-P), polyphosphate and organophosphate in the water sample.

### Method for total phosphate determination (0.2 – 15.3 mg/L $PO_4$ (0.11 – 11.46 mg/L $P_2O_5$))

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

1. Check the pH of the sample; it should be in the specified range of pH 0 – 10. If needed, add dilute sulphuric acid ($H_2SO_4$) drop-by-drop to the sample to adjust the pH. If the sample pH is more than 10, reduce the pH by adding 1+1 HCl before dilution. In order to notice the difference, add 0.05 ml phenolphthalein to 50 ml of the sample, upon addition of the acid the red colour will decrease and disappear.
2. Add 1 ml of the sample to a barcoded test cell with a P1000 pipette.
3. Place a cap on the barcoded test cell and mix vigorously with a vortex mixer.
4. Add 1 dose of P-1K to the barcoded test cell using the green dose-metering cap.
5. Place the cap on the barcoded test cell and mix vigorously with the vortex mixer.
6. Heat the test cells in the Spectroquant thermoreactor TR 420 at 120˚C for 30 minutes.
7. Carefully remove the test cells after 30 minutes and place in a test tube rack to cool down to room temperature.
8. Add 5 drops of P-2K to the barcoded test cell.
9. Place the cap on the barcoded test cell and mix vigorously with the vortex mixer.
10. Add 1 dose of P-3K to the barcoded test cell using the blue dose-metering cap.
11. Place the cap on the barcoded test cell and mix vigorously with the vortex mixer.
12. Wait 5 minutes for the reaction to occur.
13. Place the test cells into the Nova 60 Spectroquant to measure for $PO_4$.

*<u>Notes:</u>*

- Total phosphorus determination should be done as soon as the samples have been taken in order to obtain accurate results.
- Turbid samples should be filtered.
- Make sure all barcoded test cells are clean and free of marks/scratches on the outside, as this could affect the measurement.
- All samples must be tested in duplicate.
- When using the Nova 60 Spectroquant, make sure the indicator line on the test cell, lines up with the indicator line on the Nova 60 Spectroquant.

## Method for orthophosphate determination:

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

1. Check the pH of the sample; it should be in the specified range of pH 0 – 10. If needed, add dilute sulphuric acid ($H_2SO_4$) drop-by-drop to the sample to adjust the pH. If the sample pH is more than 10, reduce the pH by adding 1+1 HCl before dilution. In order to notice the difference, add 0.05 ml phenolphthalein to 50 ml of the sample, upon addition of the acid the red colour will decrease and disappear.
2. Add 1.0 ml of the sample to a barcoded test cell with a P1000 pipette.
3. Place a cap on the barcoded test cell and mix vigorously with a vortex mixer.
4. Add 5 drops of P-2K to the barcoded test cell.
5. Add 1 dose of P-3K to the barcoded test cell using the blue dose-metering cap.
6. Place the cap on the barcoded test cell and mix vigorously with the vortex mixer.
7. Wait 5 minutes for the reaction to occur.
8. 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 I: Total suspended solids (TSS) determination

Total suspended solids (TSS) are the total number of particles that is un-filterable and in suspension in the wastewater (ESS Method 340.2: Total Suspended Solids, Mass Balance (Dried at 103 - 105°C).

## Method for determining TSS:

1. Preparation of the glass fibre filter disk:
    - Insert the glass fibre filter disk into a Büchner funnel attached to a collection flask. While 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 vacuum after the Milli-Q water has passed through.
    - Remove the Büchner funnel from the collection flask and place the filter in an aluminum dish and ignite in a 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 the aluminum dish from the oven, desiccate, and weigh the glass fibre filter.
2. Select a sample volume, a maximum of 200 ml that will yield no more than 200 mg of TSS.
3. Place the filter in the Büchner funnel attached to a collection flask and apply vacuum. Wet the filter with a small volume of Milli-Q water to seal the filter against the Büchner funnel.
4. Shake the sample vigorously and quantitatively transfer the sample to the glass fibre filter using a large orifice, volumetric pipette. Remove all traces of water by continuing to apply vacuum after the sample has passed through.
5. Rinse the pipette and Büchner funnel onto the glass fibre filter with a small volume of Milli-Q water. Remove all traces of water by continuing to apply vacuum after the Milli-Q water has passed through the filter.
6. Carefully remove the glass fibre filter from the Büchner funnel. Dry the filter at 103 - 105°C for at least one hour. Cool the filter in a desiccator and weigh.
7. TSS is calculated using Equation I1, where A is the weight of the glass fibre filter and sample residue as well as the aluminium dish (mg); B is the weight of the glass fibre filter and the aluminium dish (mg); and C is the volume of sample filtered (ml).

$$\text{TSS (mg/L)} = \frac{(\text{A} - \text{B}) \times 1000}{\text{C}} \qquad \text{Eq. I1}$$

## References

Greenberg, A.E., Trussell, R.R. & Clesceri, L.S. 1985. *Standard methods for examination of water and wastewater.* 16th edition. Washington: American Public Health Association, American Water Works Association and Water Pollution Control Federation - ESS Method 340.2: Total Suspended Solids, Mass Balance (Dried at 103 - 105°C).

## APPENDIX J: Turbidity determination

Turbidity is the cloudiness or haziness of a fluid caused by individual particles such as suspended solids that are generally invisible to the naked eye. The measurement of turbidity is a key test of water quality. Turbidity is the amount of solid particles that are suspended in water and that cause light rays shining through the water to scatter. Therefore, turbidity is the measure of relative sample clarity, not colour. Turbidity measured this way uses an instrument called a nephelometer. The units of turbidity from a calibrated nephelometer are called nephelometric turbidity units (NTU).

### Calibration Procedure:

1. Place TN-100 turbidimeter on a flat level surface.
2. Insert the CAL 1 (800 NTU) calibration standard into the sampling well, aligning the mark on vial with the mark on the meter.
3. Press the vial down until it snaps into the instrument.
4. Cover the vial with the light shield cap.
5. Press the ON/OFF key to switch on the meter. The meter will go into measuring mode after the start up sequence.
6. Press the CAL key to switch to calibration mode. The meter will prompt for the CAL 1 standard to be inserted.
7. Press the READ/ENTER key.
8. The annunciator will blink for 12 seconds and then prompt for the CAL 2 (200 NTU) calibration standard to be inserted.
9. Repeat steps 2, 3, 4 7 and 8 for CAL 2, CAL 3 (100 NTU) and CAL 4 (0.02 NTU) calibration standards.
10. After CAL 4 (0.02 NTU) calibration standard is calibrated, the display will show STbY.
11. The meter is now ready for measurement.

### Turbidity Measuring Procedure:

Preparation of Sample Vial:

1. Obtain a clean dry sample vial.
2. Take care to handle the sample vial by the lid.
3. Rinse the sample vial with approximately 10 ml of the sample, capping the vial with a black screw cap and inverting gently several times. Discard the used sample and repeat the rinsing procedure twice.
4. Fill the sample vial with approximately 10 ml of sample (i.e. up to the mark indicated on the sample vial). Cap the vial with a black screw cap.

5. Wipe the sample vial with a soft, lint-free cloth. Ensure that the outside of the vial is dry, clean and free from smudges.

*Notes*

- Apply a thin film of silicone oil (i.e. 1 drop) onto the glass surface of the sample vial should any scratches be visible on the glass.
- Wipe with a soft cloth to obtain an even distribution over the entire vial surface
- The purpose of oiling the glass surface is to fill small scratches and mask the imperfections in the glass.
- Do not apply a large quantity of oil as this may result in the collection of dirt and dust.
- The sample vial is now ready to be inserted into the sample well of the meter for measurement.

### Measurement Procedure:

1. Place the sample vial inside the sample well of the TN-100 turbidimeter and align the vial's index mark with the meter's index mark.
2. Push the sample vial until it is fully snapped in.
3. Cover the vial with the light shield cap.
4. Turn the meter on by pushing the ON/OFF key.
5. Following the power up sequence, the meter will go into measuring mode and the display will blink "Rd" approximately 10 times.
6. The measured reading will appear on the display.
7. If necessary, place a second sample vial into the sample well, push down till it is snapped into place and press the READ/ENTER key. Wait for the measured reading to appear on the display screen.
8. Repeat steps 1 to 8 for all samples.

Dilutions:

- The dilution procedure is necessary only when measuring above 1,000 NTU.
- Dilute the sample with turbidity free water (i.e. water obtained from a Milli-Q).
- Measure the volume of the sample before dilution and record the value ( $V_S$ ), in millilitres (ml).
- Add the measured volume ( $V_D$ ) of dilution water to the sample.
- Pour 10 ml of the diluted sample into the measuring vial and measure the turbidity of the diluted sample. Record the value ( $T_D$ ) in NTU.
- Calculate the true turbidity ( $T_T$ ) of the sample using Equation J1.

$$T_T = \frac{T_D \quad x \quad V_S \quad + \quad V_D}{V_S}$$ Eq. J1

## References

ISO 7027 compliant nephelometric method

## APPENDIX K: True colour determination using the colour Hazen method

Colour is not a toxic characteristic but is listed as a secondary parameter affecting the appearance and platability of the water (Hongve & Åkesson, 1996; Platinum-cobalt standard method analogous to APHA 2120B, DIN53409).

### Method for true colour determination:

For the measuring range: 0 – 1000 Pt/Co (Hz)

1. Allow any samples stored in the refrigerator for preservation purposes to return to room temperature before testing for colour.
2. Filter 5 ml of the water sample through a membrane filter with a 0.45 µm pore size, using a Luer-lock syringe.
3. Transfer about 2 ml of the filtered sample into a 50 mm glass cuvette, using a P5000 Gilson pipette.
4. Place the 50 mm glass cuvette into the cell compartment of the NOVA 60 Spectroquant, and select the method (code no. **179**) on the menu.

For the measuring range: 0 – 500 Pt/Co (Hz)/0 – 250 Pt/Co (Hz)/0 – 100 Pt/Co (Hz)

1. Allow any samples stored in the refrigerator for preservation purposes to return to room temperature before testing for colour.
2. Filter 5 ml of the water sample through a membrane filter with a 0.45 µm pore size, using a Luer-lock syringe.
3. Transfer about 2 ml of the filtered sample into a 10 mm glass cuvette for the 0 – 500 Pt/Co (Hz) measuring range, a 20 mm glass cuvette for the 0 – 250 Pt/Co (Hz) measuring range and a 50 mm glass cuvette for the 0 – 100 Pt/Co (Hz) measuring range, using a P5000 Gilson pipette.
4. Place the corresponding glass cuvette into the cell compartment of the NOVA 60 Spectroquant, and select the method (code no. **032**) in the menu.

*Notes:*

- Unfiltered sample = Apparent colour
- Filtered sample = True colour
- Ensure the glass cuvette is free of scratches and marks. Wipe the cuvette surface with a soft tissue before inserting into the NOVA 60 Spectroquant.
- All samples are read in triplicate.
- For the measuring range: 0 – 1000 Pt/Co (Hz) the code number 179 must be selected on the NOVA 60 Spectroquant menu.

For the measuring ranges: 0 – 500 Pt/Co (Hz)/0 – 250 Pt/Co (Hz)/0 – 100 Pt/Co (Hz) the code number 032 must be selected on the NOVA 60 Spectroquant menu.

## References

Hongve, D. & Åkesson, G. 1996. Spectrophotometric determination of water colour in Hazen units. *Water Research,* 30(11):2771-2775.

Greenberg, A.E., Trussell, R.R. & Clesceri, L.S. 1985. *Standard methods for examination of water and wastewater.* 16th edition. Washington: American Public Health Association, American Water Works Association and Water Pollution Control Federation - Platinum-cobalt standard method analogous to APHA 2120B, DIN53409.

## APPENDIX L: True colour determination using the American dye manufacturing index (ADMI) method

1. Samples on which ADMI must be performed should be stored in the refrigerator at 4°C for preservation reasons.
2. Allow two 50 ml samples to reach room temperature.
3. One sample will be used at its original pH.
4. If necessary, the pH of the second sample will be adjusted to 7.6 using sulphuric acid ($H_2SO_4$) and sodium hydroxide (NaOH) prepared at concentrations that the resulting volume change does not exceed 3%. (NOTE: A standard pH is needed, because of the variation in colour with pH).
5. For this study no pH adjustment was necessary.
6. The ten ordinate method (Greenberg *et al.,* 1985) was used for determination of ADMI true colour values.
7. Filter the sample in order to remove excess quantities of suspended solids using a glass filtering crucible, fitted to a flask connected to a vacuum pump. (Note: Use glass fibre filter paper to filter the samples since polymer membranes absorbed the dye).
8. Using a P5000 Gilson pipette, transfer 3 ml of the filtered sample to a plastic 10mm cuvette, free of scratches.
9. The samples were analysed using a Cary 300 Bio UV-VIS spectrophotometer.
10. Each sample will be read in duplicate with a third blank cuvette containing distilled water. The blank will be used during measurements in order to obtain a base reading where the absorbance is equal to zero.
11. The photometric scan determined the absorbance of each sample at ten different predetermined wavelengths (indicated in Table L.1) for the columns X, Y and Z.
12. From the absorbance values obtained the % transmittance is calculated using Equation L1:

$$\%\ \text{Transmittance} = \frac{10^{2\text{-Absorbance}}}{100} \qquad \text{Eq. L1}$$

13. The tristimulus values (Equation L2) are determined for X, Y and Z by multiplying the sum of the % transmittance for X, Y and Z, respectively, for each of the ten ordinates by the multiplication factors for X (0.09806), Y (0.1) and Z (0.11814) and multiplying the total by 100.

$$\text{Tristimulus value}_{X,Y,Z} = \text{Sum of \% Transmittance} \times \text{Multiplication factor}_{X,Y,Z} \times 100 \qquad \text{Eq. L2}$$

14. The calculated tristimulus values are used to read the Munsell values (i.e. $V_X$ , $V_Y$ , $V_Z$ ) from Tables located in Greenberg *et al.,* 1985.
15. Using the Adams Nickerson colour difference (DE) the ADMI was calculated using Equation L3.

$$\text{ADMI value}=\frac{\text{Calibration factor (F)}\times\text{DE}}{\text{Cell path length (b)}} \qquad \text{Eq. L3}$$

Where the calibration factor ( $F$ ) was 1400, the cell path length ( $b$ ) 1 cm and the Adams Nickerson colour difference (DE) was calculated using Equation L4.

$$DE=\sqrt{0.23\ \ V_{Yblank}-V_{Ysample}\ {}^{2}+\ \Delta\ \ V_X-V_Y\ {}^{2}+\ 0.4\Delta\ \ V_Y-V_Z\ {}^{2}} \qquad \text{Eq. L4}$$

**Table L.1:** Selected ordinates for spectrophotometric colour determinations (Greenberg *et al.,* 1985)

| Wavelength No. | X | Y | Z |
|---|---|---|---|
| **1** | 435.5 | 498.5 | 422.2 |
| **2** | 461.2 | 515.2 | 432.0 |
| **3** | 544.3 | 529.8 | 438.6 |
| **4** | 564.1 | 541.4 | 444.4 |
| **5** | 577.4 | 551.8 | 450.1 |
| **6** | 588.7 | 561.9 | 455.9 |
| **7** | 599.6 | 572.5 | 462.0 |
| **8** | 610.9 | 584.8 | 468.7 |
| **9** | 624.2 | 600.8 | 477.7 |
| **10** | 645.9 | 627.3 | 495.2 |

## References

Greenberg, A.E., Trussell, R.R. & Clesceri, L.S. 1985. *Standard methods for examination of water and wastewater.* 16th edition. Washington: American Public Health Association, American Water Works Association and Water Pollution Control Federation.

# APPENDIX M: Total organic carbon (TOC) and dissolved organic carbon (DOC) determination

Total organic carbon (TOC) describes the total content of organically bound carbon present in wastewater. Dissolved organic carbon (DOC) is the DOC is the fraction of TOC that passes through a 0.45 µm filter (Greenberg *et al.,* 1985).

## Method for TOC determination (5.0 - 80.0 mg/L TOC):

Using a Merck Spectroquant TOC cell test, Cat. No. 1.14878.0001

1. Transfer 25 ml of the pretreated sample, with a temperature of 10 – 30°C, into a 40 ml glass beaker.
2. Add 3 drops of TOC-1K reagent to the glass beaker.
3. Stir the mixture for 10 minutes at a medium speed, using a magnetic stirrer.
4. Check the pH of the mixture after stirring. The pH must be below 2.5. Adjust the pH with sulphuric acid if necessary.
5. Add 3 ml of the stirred sample to the barcoded test cell with a P5000 pipette.
6. Add 1 level grey microspoon of TOC-2K reagent to the barcoded test cell. The microspoon is located in the lid of the TOC-2K reagent bottle.
7. Immediately close the barcoded test cell tightly with an aluminium cap.
8. Heat the barcoded test cell, standing on its head, at 120°C in a preheated Spectroquant thermoreactor TR 420 for 120 minutes.
9. Remove the barcoded test cell from the thermoreactor and allow the closed test cell to cool in a test tube rack, standing on its head, for 60 minutes. Do not cool with cold water.
10. After cooling, turn the test cell upright and measure in a Nova 60 Spectroquant within 10 minutes.

<u>Notes:</u>

- Ensure the TOC-1K reagent bottle is held vertically while adding the reagent to the test cell in step 2.
- Turbid solutions may yield false-low readings.
- Make sure all barcoded test cells are clean and free of marks/scratches on the outside, as this could affect the measurement.
- All samples must be tested in duplicate.
- When using the Nova 60 Spectroquant, make sure the indicator line on the test cell, lines up with the indicator line on the Nova 60 Spectroquant.

## Method for DOC determination (5.0 - 80.0 mg/L DOC):

Using a Merck Spectroquant TOC cell test, Cat. No. 1.14878.0001

1. Attach a Büchner funnel to 500 ml suction flask.
2. The suction flask is either connected to a water pump or a vacuum pump.
3. Glass microfibre filter discs, 5.5 cm in diameter with a 0.45 µm pore size, without organic binder (i.e. Whatman type GF/F (0.7 Fm)) is placed inside the Büchner funnel.
4. 200 ml of the raw sample is filtered.
5. The DOC of the filtered sample is then determined, using the same procedure as for TOC determination.
6. Transfer 25 ml of the filtered sample, with a temperature of 10 – 30°C, into a 40 ml glass beaker.
7. Add 3 drops of TOC-1K reagent to the glass beaker.
8. Stir the mixture for 10 minutes at a medium speed, using a magnetic stirrer.
9. Check the pH of the mixture after stirring. The pH must be below 2.5. Adjust the pH with sulphuric acid if necessary.
10. Add 3 ml of the stirred sample to the barcoded test cell with a P5000 pipette.
11. Add 1 level grey microspoon of TOC-2K reagent to the barcoded test cell. The microspoon is located in the lid of the TOC-2K reagent bottle.
12. Immediately close the barcoded test cell tightly with an aluminium cap.
13. Heat the barcoded test cell, standing on its head, at 120°C in a preheated Spectroquant thermoreactor TR 420 for 120 minutes.
14. Remove the barcoded test cell from the thermoreactor and allow the closed test cell to cool in a test tube rack, standing on its head, for 60 minutes. Do not cool with cold water.
15. After cooling, turn the test cell upright and measure in a Nova 60 Spectroquant within 10 minutes.

## References

Greenberg, A.E., Trussell, R.R. & Clesceri, L.S. 1985. *Standard methods for examination of water and wastewater.* 16th edition. Washington: American Public Health Association, American Water Works Association and Water Pollution Control Federation.

Merck Spectroquant TOC cell tests, Cat. No. 1.14878.0001.

## APPENDIX N: Sludge volume index (SVI) determination

Sludge Volume Index (SVI) is an indication of the sludge settleability. It is a useful test that indicates changes in the sludge settling characteristics and quality. The SVI is the volume of settled sludge in millilitres occupied by 1 gram of dry sludge solids after 30 minutes of settling in a 1000 ml graduated cylinder or a settleometer (Operation and control, n.d.).

### Method for determining the SVI of the aerobic tank:

1. Collect 1 litre of sample at or near the outlet of the aerobic tank.
2. Allow the sample to settle for 30 minutes in a 1 litre graduated cylinder.
3. Measure the volume occupied by the sludge in millilitres.
4. Calculate the SVI of the sample using Equation N1.

$$\text{SVI (ml/g)} = \frac{\text{Settled sludge volume after 30 minutes (ml/L) x 1000}}{\text{Mixed liquor suspended solids in the aerobic tank (mg/L)}} \qquad \text{Eq. N1}$$

### References

Operation and control, n.d. Sludge volume index. http://water.me.vccs.edu/courses/ENV149/operation3.htm [10 May 2010].

# APPENDIX O: Minimum, maximum and average values for the experimental timeline

**Table O.1:** Composition of the wastewater in the sump fed to Phase 1 over the 220 days pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 12.3 | 29.6 | 22.8 |
| **pH** | - | 5.3 | 12.2 | 9.8 |
| **Conductivity** | µS/cm | 495 | 8,500 | 2,716 |
| **TDS** | ppm | 169 | 6,260 | 1,973 |
| **COD** | mg/L | 45 | 2,820 | 763 |
| **Ammonium** | mg/L | 1.1 | 28.5 | 9.0 |
| **TSS** | mg/L | 3.0 | 430 | 54 |
| **Turbidity** | NTU | 14.6 | 575 | 45 |
| **Phosphate** | mg/L | 0.2 | 4.0 | 1.5 |
| **Nitrate** | mg/L | 0.7 | 6.8 | 3.0 |
| **True colour** | mg Pt/L | 31 | 427 | 131 |
| **ADMI** | ADMI units | 195 | 2070 | 659 |

**Table O.2:** Composition of the wastewater in the anaerobic tank over the 220 days of pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 13.1 | 28.9 | 21.7 |
| **pH** | - | 4.7 | 10.9 | 7.9 |
| **Conductivity** | µS/cm | 588 | 9,945 | 3,504 |
| **TDS** | ppm | 458 | 8,381 | 3,597 |
| **COD** | mg/L | 205 | 1759 | 767 |
| **Ammonium** | mg/L | 1.4 | 114 | 21.5 |
| **TSS** | mg/L | 23.0 | 398 | 80.7 |
| **SVI** | ml/g | 0.0 | 10.1 | 6.4 |
| **Turbidity** | NTU | 18.6 | 499 | 87.8 |
| **Phosphate** | mg/L | 0.35 | 4.1 | 1.8 |
| **Nitrate** | mg/L | 1.0 | 6.1 | 3.0 |
| **True colour** | mg Pt/L | 50.3 | 336 | 163 |
| **ADMI** | ADMI units | 372 | 1279 | 799 |

**Table O.3:** Composition of the wastewater in the anoxic tank over the 220 days of pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 13.3 | 28.8 | 21.3 |
| **pH** | - | 6.7 | 10.2 | 8.0 |
| **Conductivity** | µS/cm | 663 | 9,378 | 4,521 |
| **TDS** | ppm | 678 | 8,105 | 3,625.8 |
| **COD** | mg/L | 235 | 4,160 | 792.8 |
| **Ammonium** | mg/L | 0.9 | 208 | 28.0 |
| **TSS** | mg/L | 34.0 | 2,150 | 249 |
| **SVI** | ml/g | 1.6 | 50.4 | 13.1 |
| **Turbidity** | NTU | 30.8 | 784 | 169 |
| **Phosphate** | mg/L | 0.40 | 4.8 | 1.6 |
| **Nitrate** | mg/L | 0.80 | 11.1 | 3.9 |
| **True colour** | mg Pt/L | 55.0 | 367 | 153 |
| **ADMI** | ADMI units | 406 | 649 | 539 |

**Table O.4:** Composition of the wastewater in the aerobic tank over the 220 days of pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 13.8 | 28.9 | 21.5 |
| **pH** | - | 7.0 | 16.2 | 8.2 |
| **Conductivity** | µS/cm | 528 | 9,091 | 4,787 |
| **TDS** | ppm | 654 | 8,220 | 3,612 |
| **COD** | mg/L | 177 | 3,860 | 901 |
| **Ammonium** | mg/L | 3.0 | 205 | 31.0 |
| **TSS = MLSS** | mg/L | 6.0 | 6,460 | 1,329 |
| **SVI** | ml/g | 1.6 | 27.3 | 15.8 |
| **Turbidity** | NTU | 36.3 | 948 | 334 |
| **Phosphate** | mg/L | 0.32 | 4.8 | 1.4 |
| **Nitrate** | mg/L | 1.5 | 10.2 | 3.9 |
| **True colour** | mg Pt/L | 41.3 | 458 | 145 |
| **ADMI** | ADMI units | 387 | 920 | 553.5 |

**Table O.5:** Composition of the UF permeate over the 220 days of pilot plant operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 14.5 | 27.6 | 22.2 |
| **pH** | - | 7.2 | 10.6 | 8.4 |
| **Conductivity** | µS/cm | 517 | 9,088 | 4,302 |
| **TDS** | ppm | 771 | 7,186 | 3,657 |
| **COD** | mg/L | 0.0 | 1460 | 191 |
| **Ammonium** | mg/L | 0.67 | 139 | 25.3 |
| **TSS** | mg/L | 0.0 | 471 | 43.1 |
| **Turbidity** | NTU | 0.0 | 32.6 | 2.7 |
| **Phosphate** | mg/L | 0.10 | 4.0 | 1.3 |
| **Nitrate** | mg/L | 1.2 | 10.3 | 3.2 |
| **True colour** | mg Pt/L | 41.7 | 243 | 129 |
| **ADMI** | ADMI units | 224 | 613 | 471 |

**Table O.6:** Composition of the wastewater in the sump during the 4 days of NF membrane operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 24.9 | 29.6 | 27.2 |
| **pH** | - | 10.1 | 11.5 | 10.8 |
| **Conductivity** | µS/cm | 1,601 | 2,040 | 1,820 |
| **TDS** | ppm | 1,014 | 1,721 | 1,367 |
| **COD** | mg/L | 742 | 848 | 795 |
| **Ammonium** | mg/L | 8.4 | 9.7 | 9.0 |
| **TSS** | mg/L | 18.0 | 30.0 | 24.0 |
| **Turbidity** | NTU | 16.3 | 26.9 | 21.6 |
| **Phosphate** | mg/L | 0.83 | 1.1 | 1.0 |
| **Nitrate** | mg/L | 1.6 | 2.4 | 2.0 |
| **True colour** | mg Pt/L | 63.7 | 232 | 144 |
| **ADMI** | ADMI units | 195 | 1,271 | 935 |

**Table O.7:** Composition of the UF permeate during the 4 days of NF membrane operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 22.4 | 27.5 | 24.9 |
| **pH** | - | 9.2 | 9.5 | 9.4 |
| **Conductivity** | µS/cm | 8,401 | 9,067 | 8,734 |
| **TDS** | ppm | 6,003 | 6,085 | 6,044 |
| **COD** | mg/L | 32.0 | 41.5 | 36.8 |
| **Ammonium** | mg/L | 11.0 | 13.5 | 12.3 |
| **TSS** | mg/L | 13.0 | 14.0 | 13.5 |
| **Turbidity** | NTU | 0.40 | 1.9 | 1.1 |
| **Phosphate** | mg/L | 0.29 | 0.81 | 0.55 |
| **Nitrate** | mg/L | 1.4 | 1.9 | 1.6 |
| **True colour** | mg Pt/L | 143 | 153 | 148 |
| **ADMI** | ADMI units | 463 | 463 | 463 |

**Table O.8:** Composition of the NF permeate

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 22.6 | 22.7 | 22.6 |
| **pH** | - | 9.1 | 9.2 | 9.2 |
| **Conductivity** | µS/cm | 6,071 | 6,084 | 6,077 |
| **TDS** | ppm | 4,072 | 4,082 | 4,077 |
| **COD** | mg/L | 99.5 | 127 | 113 |
| **Ammonium** | mg/L | 6.5 | 9.0 | 7.8 |
| **TSS** | mg/L | 3.0 | 6.0 | 4.5 |
| **Turbidity** | NTU | 0.0 | 0.19 | 0.12 |
| **Phosphate** | mg/L | 0.40 | 0.41 | 0.40 |
| **Nitrate** | mg/L | 2.4 | 2.5 | 2.4 |
| **True colour** | mg Pt/L | 13.0 | 13.0 | 13.0 |
| **ADMI** | ADMI units | 8.1 | 16.0 | 11.9 |

**Table O.9:** Composition of the wastewater in the sump during the 13 days of RO membrane operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 24.6 | 25.9 | 25.3 |
| **pH** | - | 7.8 | 10.4 | 9.4 |
| **Conductivity** | µS/cm | 995 | 1,659 | 1,340 |
| **TDS** | ppm | 963 | 1,016 | 983 |
| **COD** | mg/L | 728 | 1,033 | 840 |
| **Ammonium** | mg/L | 7.8 | 25.1 | 13.5 |
| **TSS** | mg/L | 16.0 | 46.0 | 33.3 |
| **Turbidity** | NTU | 16.4 | 26.3 | 20.8 |
| **Phosphate** | mg/L | 0.83 | 1.3 | 1.0 |
| **Nitrate** | mg/L | 1.2 | 2.3 | 1.8 |
| **True colour** | mg Pt/L | 79.0 | 288 | 153 |
| **ADMI** | ADMI units | 222 | 2,070 | 870 |

**Table O.10:** Composition of the UF permeate during the 13 days of RO membrane operation

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 23.7 | 25.5 | 24.8 |
| **pH** | - | 8.9 | 10.8 | 9.5 |
| **Conductivity** | µS/cm | 6,040 | 9,080 | 8,215 |
| **TDS** | ppm | 4,050 | 7,041 | 5,688 |
| **COD** | mg/L | 50.0 | 171 | 77.0 |
| **Ammonium** | mg/L | 9.0 | 20.6 | 12.0 |
| **TSS** | mg/L | 8.0 | 40.0 | 16.4 |
| **Turbidity** | NTU | 0.06 | 0.77 | 0.41 |
| **Phosphate** | mg/L | 0.43 | 1.5 | 1.1 |
| **Nitrate** | mg/L | 1.3 | 4.0 | 2.0 |
| **True colour** | mg Pt/L | 102 | 182 | 153 |
| **ADMI** | ADMI units | 432 | 590 | 503 |

**Table O.11:** Composition of the RO permeate

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 23.5 | 25.3 | 24.7 |
| **pH** | - | 8.1 | 10.5 | 9.3 |
| **Conductivity** | µS/cm | 92.5 | 348 | 204 |
| **TDS** | ppm | 67.2 | 237 | 146 |
| **COD** | mg/L | 69.0 | 94.5 | 81.6 |
| **Ammonium** | mg/L | 6.5 | 18.7 | 11.0 |
| **TSS** | mg/L | 0.0 | 14.0 | 1.8 |
| **Turbidity** | NTU | 0.0 | 0.73 | 0.39 |
| **Phosphate** | mg/L | 0.68 | 4.2 | 1.6 |
| **Nitrate** | mg/L | 1.2 | 2.3 | 1.7 |
| **True colour** | mg Pt/L | 13.0 | 16.0 | 14.1 |
| **ADMI** | ADMI units | 6.4 | 38.5 | 19.9 |

**Table O.12:** Composition of the potable water of the industrial partner

| Parameters | Units | Minimum | Maximum | Average |
|---|---|---|---|---|
| **Temperature** | °C | 19.1 | 24.1 | 22.2 |
| **pH** | - | 6.5 | 10.2 | 8.7 |
| **Conductivity** | µS/cm | 71.1 | 148 | 92.6 |
| **TDS** | ppm | 50.4 | 107 | 66.3 |
| **COD** | mg/L | 28.0 | 99.0 | 58.9 |
| **Ammonium** | mg/L | 8.4 | 13.2 | 11.2 |
| **TSS** | mg/L | 0.0 | 3.0 | 1.5 |
| **Turbidity** | NTU | 0.62 | 2.8 | 1.5 |
| **Phosphate** | mg/L | 0.85 | 1.9 | 1.3 |
| **Nitrate** | mg/L | 2.0 | 2.6 | 2.3 |
| **True colour** | mg Pt/L | 14.7 | 88.7 | 22.7 |
| **ADMI** | ADMI units | 11.1 | 21.2 | 16.0 |

**Table O.13:** Composition of the anaerobic feed for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | 16.2 | 24.8 | 25.1 | 24.8 | 26.2 |
| **pH** | - | 11.1 | 10.9 | 10.7 | 11.7 | 7.4 |
| **Conductivity** | μS/cm | 10.79 | 1,179 | 9,007 | 1,359 | 3,012 |
| **TDS** | ppm | 759 | 8,024 | 6,044 | 9,062 | 2,020 |
| **COD** | mg/L | 738 | 548 | 623 | 561 | 715 |
| **Ammonium** | mg/L | 10.3 | 9.0 | 14.2 | 12.3 | 18.7 |
| **TSS** | mg/L | 51.0 | 66.0 | 30.0 | 24.0 | 34.0 |
| **Turbidity** | NTU | 43.1 | 30.0 | 20.5 | 22.5 | 30.5 |
| **Phosphate** | mg/L | 1.9 | 1.5 | 1.3 | 1.6 | 1.6 |
| **Nitrate** | mg/L | 2.0 | 2.3 | 1.6 | 1.7 | 1.7 |
| **TOC** | mg/L | 107 | 106 | 108 | 107 | 103 |
| **DOC** | mg/L | 78.4 | 75.8 | 100 | 102 | 62.7 |
| **True colour** | mg Pt/L | 278 | 393 | 291 | 511 | 68.0 |
| **ADMI** | ADMI units | 1,027 | 1,649 | 1,829 | 2,388 | 290 |

**Table O.14:** Composition of the anaerobic tank for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | 24.3 | 25.1 | 23.5 | 25.6 | 25.3 |
| **pH** | - | 7.2 | 6.8 | 10.6 | 9.2 | 9.3 |
| **Conductivity** | μS/cm | 1,131 | 1,027 | 9,095 | 1,320 | 1,327 |
| **TDS** | ppm | 7,434 | 7,026 | 6,094 | 6,037 | 6,044 |
| **COD** | mg/L | 775 | 580 | 594 | 737 | 748 |
| **Ammonium** | mg/L | 15.5 | 9.7 | 19.9 | 10.3 | 11.6 |
| **TSS** | mg/L | 94.0 | 84.0 | 60.0 | 62.0 | 28.0 |
| **Turbidity** | NTU | 160 | 158 | 37 | 66 | 71 |
| **Phosphate** | mg/L | 0.63 | 1.2 | 0.71 | 0.85 | 1.0 |
| **Nitrate** | mg/L | 2.5 | 1.9 | 1.9 | 1.4 | 1.9 |
| **TOC** | mg/L | 102 | 106 | 107 | 107 | 106 |
| **DOC** | mg/L | 65.4 | 81.3 | 58.3 | 100 | 87.8 |
| **True colour** | mg Pt/L | 200 | 330 | 240 | 274 | 99.3 |
| **ADMI** | ADMI units | 372 | 1279 | 797 | 966 | 581 |

**Table O.15:** Composition of the anoxic tank for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | 19.5 | 25.1 | 25.4 | 25.2 | 25.8 |
| **pH** | - | 8.1 | 7.7 | 9.5 | 8.3 | 8.1 |
| **Conductivity** | μS/cm | 8,176 | 1,018 | 1,062 | 1,152 | 1,110 |
| **TDS** | ppm | 6,751 | 7,025 | 7,063 | 8,105 | 8,105 |
| **COD** | mg/L | 750 | 676 | 633 | 647 | 893 |
| **Ammonium** | mg/L | 18.1 | 17.4 | 12.3 | 9.7 | 7.8 |
| **TSS** | mg/L | 270 | 210 | 130 | 225 | 154 |
| **Turbidity** | NTU | 306 | 188 | 149 | 215 | 203 |
| **Phosphate** | mg/L | 1.1 | 1.1 | 1.5 | 1.3 | 0.80 |
| **Nitrate** | mg/L | 1.9 | 2.9 | 2.0 | 2.2 | 1.9 |
| **TOC** | mg/L | 107 | 105 | 107 | 107 | 106 |
| **DOC** | mg/L | 92.5 | 71.2 | 97.9 | 72.9 | 67.6 |
| **True colour** | mg Pt/L | 197 | 368 | 216 | 234 | 129 |
| **ADMI** | ADMI units | 484 | 516 | 642 | 649 | 406 |

**Table O.16:** Composition of the aerobic tank for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | 20.1 | 25.1 | 25.2 | 25.4 | 24.5 |
| **pH** | - | 8.7 | 8.0 | 7.8 | 8.1 | 9.2 |
| **Conductivity** | μS/cm | 8,102 | 9,091 | 9,090 | 9,061 | 7,063 |
| **TDS** | ppm | 7,928 | 7,109 | 7,002 | 7,601 | 5,040 |
| **COD** | mg/L | 825 | 725 | 676 | 771 | 716 |
| **Ammonium** | mg/L | 29.0 | 12.3 | 8.4 | 8.4 | 12.9 |
| **TSS = MLSS** | mg/L | 4,710 | 4,160 | 5,240 | 4,700 | 1,730 |
| **Turbidity** | NTU | 267 | 510 | 408 | 509 | 948 |
| **Phosphate** | mg/L | 0.84 | 1.6 | 0.86 | 0.91 | 1.0 |
| **Nitrate** | mg/L | 1.7 | 2.8 | 1.8 | 1.6 | 1.5 |
| **TOC** | mg/L | 106 | 106 | 108 | 108 | 107 |
| **DOC** | mg/L | 98.9 | 94.1 | 92.3 | 105 | 67.7 |
| **True colour** | mg Pt/L | 178 | 458 | 108 | 206 | 153 |
| **ADMI** | ADMI units | 409 | 439 | 613 | 920 | 387 |

**Table O.17:** Composition of the UF permeate for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | 21.3 | 25.1 | 25.2 | 25.2 | 24.3 |
| **pH** | - | 8.7 | 8.9 | 8.9 | 9.0 | 10.5 |
| **Conductivity** | μS/cm | 6,805 | 9,088 | 973 | 981 | 8,008 |
| **TDS** | ppm | 6,730 | 6,097 | 6,085 | 6,092 | 5,061 |
| **COD** | mg/L | 80.0 | 74.5 | 59.0 | 83.5 | 258 |
| **Ammonium** | mg/L | 10.3 | 18.1 | 20.6 | 7.1 | 9.7 |
| **TSS** | mg/L | 15.0 | 16.0 | 12.0 | 11.0 | 15.0 |
| **Turbidity** | NTU | 0.74 | 0.61 | 0.64 | 1.0 | 0.01 |
| **Phosphate** | mg/L | 1.1 | 1.6 | 0.81 | 0.82 | 1.3 |
| **Nitrate** | mg/L | 1.8 | 2.0 | 2.2 | 2.9 | 3.1 |
| **TOC** | mg/L | 104 | 100 | 105 | 102 | 102 |
| **DOC** | mg/L | 57.0 | 61.0 | 48.4 | 41.9 | 45.6 |
| **True colour** | mg Pt/L | 171 | 171 | 168 | 180 | 129 |
| **ADMI** | ADMI units | 463 | 507 | 613 | 595 | 423 |

**Table O.18:** Composition of the RO permeate for the hydraulic retention time (HRT) sample sets

| Parameters | Units | HRT1 | HRT2 | HRT3 | HRT4 | HRT5 |
|---|---|---|---|---|---|---|
| **Temperature** | °C | - | 24.7 | 25.3 | 25.1 | 24.2 |
| **pH** | - | - | 8.1 | 8.7 | 9.5 | 9.5 |
| **Conductivity** | μS/cm | - | 312 | 348 | 256 | 175 |
| **TDS** | ppm | - | 226 | 237 | 182 | 125 |
| **COD** | mg/L | - | 89.0 | 86.5 | 69.5 | 87.0 |
| **Ammonium** | mg/L | - | 8.4 | 10.3 | 7.8 | 11.0 |
| **TSS** | mg/L | - | 1.0 | 0.0 | 0.0 | 0.0 |
| **Turbidity** | NTU | - | 0.58 | 0.57 | 0.59 | 0.0 |
| **Phosphate** | mg/L | - | 0.68 | 0.90 | 1.2 | 1.2 |
| **Nitrate** | mg/L | - | 2.3 | 1.8 | 1.6 | 1.3 |
| **TOC** | mg/L | - | 22.9 | 10.3 | 8.7 | 8.8 |
| **DOC** | mg/L | - | 13.6 | 11.0 | 9.4 | 11.5 |
| **True colour** | mg Pt/L | - | 15.7 | 16.0 | 14.7 | 13.0 |
| **ADMI** | ADMI units | - | 38.5 | 10.2 | 6.4 | 27.8 |

# APPENDIX P: Pump and blower specifications for the proposed full-scale wastewater treatment plant

**Table P.1:** Specifications of pump (P2)

| **Pump - P2:** | Tsurumi Model KTZ 32.2-50 Submersible Pump |
|---|---|
| **Closed valve head** | 20,5 m |
| **Actual pump head at 10 L/sec** | 10 m |
| **Pump Power** | 2,2 kW |
| **Pump speed** | 2860 rpm (2 Pole) |
| **Pump Voltage** | 380 Volt |
| **Starting method** | Direct on line |
| **Maximum solids passed** | 8,5 mm |
| **Discharge size** | 80 mm |

**Table P.2:** Specifications of pump (P3)

| **Pump - P3** | Tsurumi Model KTZ 21.5-50 Submersible Pump |
|---|---|
| **Closed valve head** | 22 m |
| **Actual pump head at 1,125 L/sec** | 20 m |
| **Pump Power** | 1,5 kW |
| **Pump speed** | 2850 rpm (2 Pole) |
| **Pump Voltage** | 380 Volt |
| **Starting method** | Direct on line |
| **Maximum solids passed** | 8,5 mm |
| **Discharge size** | 50 mm |

**Table P.3:** Specifications of pump (P5)

| **Pump - P5** | Tsurumi Model KTZ 21.5-50 Submersible Pump |
|---|---|
| **Closed valve head** | 22 m |
| **Actual pump head at 0,52 L/sec** | 21,5 m |
| **Pump Power** | 1,5 kW |
| **Pump speed** | 2850 rpm (2 Pole) |
| **Pump Voltage** | 380 Volt |
| **Starting method** | Direct on line |
| **Maximum solids passed** | 8,5 mm |
| **Discharge size** | 50 mm |

**Table P.4:** Specifications of pump (P6)

| **Pump - P6** | Curo Model DL/DB 125-250-32 End Suction Pump |
|---|---|
| **Closed valve head** | 13,9 m |
| **Impeller diameter required for duty** | 200 mm |
| **Power absorbed at duty point** | 7,9 kW |
| **Recommended motor power** | 11 kW |
| **Pump speed** | 1450 rpm (4 Pole) |
| **Pump Voltage** | 380 Volt |
| **Starting method** | Direct on line |
| **Suction branch size** | 150 mm |
| **Discharge branch size** | 125 mm |

**Table P.5:** Specifications of blower (B1)

| **Roots Blower Package Unit Design duty 130 m$^3$/h at 40 kPa** | |
|---|---|
| Bare-shaft air blower | URAI 33 |
| Motorelli or equal electric motor IP55, 380V, 50 Hz, 3ph | 4 kW |
| Motor slide rails | included |
| Vee-belt drive assembly | included |
| Driveguard assembly | included |
| Combined suction filter & silencer | 50 mm NB |
| Filter restriction indicator | included |
| Suction vacuum switch to shut down blower in event of blocked filter | included |
| Combined base-frame & discharge silencer | 50 mm NB |
| Pressure relief valve | 50 mm NB |
| Pressure gauge set | 0 - 100 kPa (g) |
| Non-return valve | 50 mm NB |
| Discharge flexible pipe connection | 50 mm NB |
| Anti-vibration mountings | included |
| Assembly of package unit | included |
| Sowerby standard paint specification | included |
| Arrangement drawing & instruction manual | included |

# APPENDIX Q: Actual oxygen required ($AOR$) calculation

All calculations were based on the average flow rate (i.e. 4.05 $m^3$/h) entering the proposed full-scale textile wastewater treatment plant design, with a flow rate of 97 $m^3$/d entering the aerobic tank from the anoxic tank.

**Table Q.1:** Values calculated and used in the actual oxygen required calculations

| Parameter | Units | Value |
|---|---|---|
| **Average volumetric flow rate entering the proposed wastewater treatment system** | $m^3$/d | 97 |
| **$K_{LA}$ factor for tapwater to wastewater on transfer coefficient ($\alpha$)** | dimensionless | 0.8 |
| **Arrhenius constant to correct the effect of temperature ($\theta$)** | dimensionless | 1.024 |
| **Saturation water vapour pressure (ρ)** | kg/$m^3$ | 23.69 |
| **Saturation water vapour pressure under STD conditions ($\rho_{std}$)** | kg/$m^3$ | 17.51 |
| **Saturation Concentration of water ($C_S$)** | kg/L | 9.02 |
| **Design dissolved oxygen concentration ($C_L$)** | kg $O_2$/L | 2.00 |
| **Constituents in wastewater impact the solubility of $O_2$ ($\beta$) [$C_S$ / $C_L$]** | dimensionless | 0.95 |
| **Temperature correction for $C_S$ ($\tau$)** | dimensionless | 0.91 |
| **Pressure correction for $C_S$ ($\omega$)** | dimensionless | 0.98 |
| **Ammonium-nitrogen present in the effluent ($N_{Effective}$)** | kg/L | 0.0054 |
| **Oxygen required for nitrification ($Con.N$)** | kg $O_2$/kg $NH_4$ | 4.57 |
| $OTR_{Nitrification}$ **for 60% nitrification** | kg/h | 839.1 |
| $AOR/SOR$ | dimensionless | 7.31 |
| **Actual oxygen required ($AOR$)** | kg $O_2$/h | 0.0087 |
| **Actual oxygen required ($AOR$)** | $m^3$/h | 0.0061 |
| **Density of Air ($\rho_{Air}$)** | kg Air/$m^3$ | 1.21 |
| **Oxygen % in air** | dimensionless | 0.23 |
| **Actual air required ($AAR$)** | $m^3$/h | 0.031 |
| **Actual air required ($AAR$)** | L/h | 31.43 |
| **Actual air required ($AAR$)** | L/min | 0.52 |
| **Density of oxygen at standard conditions** | g/L | 1.43 |

**Table Q.2 (continued):** Values calculated and used in the actual oxygen required calculations

| **Parameter** | **Units** | **Value** |
|---|---|---|
| **Volume of air the blower must put out** | $m^3/h$ | 0.031 |
| **Volume of air the blower must put out** | L/h | 31.43 |
| **23% Oxygen in the air put out by the blower** | $m^3/h$ | 0.0072 |
| **23% Oxygen in the air put out by the blower** | L/h | 7.23 |
| **Amount of oxygen provided by blower** | kg/d | 0.25 |
| **Amount of oxygen provided by blower** | kg/h | 0.01 |
| **Ratio of oxygen provided by the blower to the actual amount of oxygen required (i.e. correction factor)** | dimensionless | 1.187 |
| **Volume of air the blower must put out multiplied by the correction ratio** | $m^3/h$ | 0.037 |
| **Volume of air the blower must put out multiplied by the correction ratio** | L/h | 37.30 |
| **23% Oxygen in the air put out by the blower after accounting for the correction ratio** | $m^3/h$ | 0.0086 |