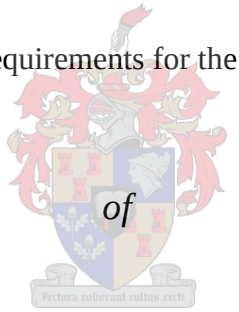
Evaluation of a flat sheet woven fabric membrane for use in water treatment processes suited to rural and peri-urban economies

by

Joshua Steven Asquith

Thesis presented in partial fulfilment

of the requirements for the Degree



MASTER OF ENGINEERING

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Declaration/ Verklaring

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Abstract

The present study was undertaken with the view of evaluating a woven fabric membrane as an alternative membrane technology for water treatment purposes for rural and peri-urban economies. Current membranes are not economically feasible or suited to the requirements of most rural and peri-urban economies. As a result, a woven fabric membrane material was developed to produce a membrane that was capable of overcoming these shortcomings.

The use of the woven fabric membrane as an alternative membrane for water treatment purposes was evaluated by (i) characterising the inherent membrane features, (ii) evaluating the membrane performance in terms of the product quality and fouling effects and (iii) investigating the effects of flux enhancement and cleaning methods. A literature review was performed to specify the separation requirements of the woven fabric.

(i) During the membrane characterisation the membrane's effective pore size range and morphology were evaluated. The bubble gas transport method was used to estimate the effective pore size range of unused and fouled membrane samples which were found to be 10-24 µm and 7-18 µm respectively. SEM imagine evaluation indicated that the complex multilayer membrane material was formed due the Plain Weave structure and the multiple strands used to produce the respective threads. The effects of pore narrowing due to fouling were concluded to be a result of the highly tortuous membrane pores that formed.

(ii) During the membrane performance evaluation the effect that the feed suspension's physical and chemical make-up had on the resultant product quality and fouling characteristics was indicated. From the performance evaluation it was concluded that the woven fabric membrane was able achieve the required 1 NTU product quality for sub-pore size (< 3 µm) feed suspensions with varying physical and chemical properties, through the formation of a porous fouling layer. This concluded the importance of dynamic membrane operation and how the estimated pore size was not an indication of the separation efficiency of the woven fabric.

(iii) The effects of continuous coarse air scouring (7.5 L/min and 2.5 L/min) and fine air scouring (5 L/min and 2.5 L/min) as a flux enhancement method resulted in higher fouling layer resistances compared to those of a dead end operation test. This was a result of the defouling potential of this method effectively defouling the membrane surface. This

compromised the dynamic operation of the membrane, as higher degrees of pore blocking occurred due to the absence of the surface fouling layer.

A cleaning evaluation was performed to investigate chemically-aided (25 PPM sodium hypochlorite) backflush methods for backflush fluxes of 50, 100 and 250 LMH in order to completely reverse the effects of fouling. Minimal recovery was exhibited by all these defouling methods, due to the high degree of irreversible fouling occurring as indicated by SEM evaluation of the membrane samples. The irreversible fouling was the result of the sub-pore size feed suspension. The only effective cleaning method was a physical scrubbing of the membrane surfaces. This was concluded from the results of a test that was operated with an initial permeate flux of 100 LMH, for 60-minutes, between physical cleaning, over a period of 10-hours, where no apparent effects of irreversible or irrecoverable fouling occurred. Due to its durability and robustness, the woven fabric was able to withstand these physical cleaning methods, which are not applicable to most available industrial membranes.

The findings of this study have clearly shown that the woven fabric membrane was capable of achieving the required separation efficiency for water treatment purposes. The membrane was however limited to physical cleaning methods due to the formation of high degrees of irreversible fouling within the membrane pores. Cleaning these membranes using a physical means was indicated as being a reliable means of defouling the woven fabric membrane. The ease of cleaning these membranes using a physical means, is of far more benefit to rural and peri-urban areas than a potentially hazardous chemically aided cleaning solution.

Opsomming

Die huidige studie is onderneem met die doel om 'n geweefde tekstielmembraan as 'n alternatiewe membraantegnologie vir waterbehandeling in landelike en buitestedelike ekonomieë, te evalueer. Huidige membrane is òf nie ekonomies lewensvatbaar nie, òf dit is nie geskik vir die vereistes van die meeste landelike en buitestedelike ekonomieë nie. Gevolglik is 'n geweefde tekstielmembraanmateriaal ontwikkel om 'n membraan op te lewer wat in staat is om hierdie tekortkominge die hoof te bied.

Die aanwending van die geweefde tekstielmembraan as 'n alternatiewe membraan vir die doeleindes van waterbehandeling is geëvalueer deur (i) die inherente membraaneienskappe te karakteriseer, (ii) die membraan se prestasie in terme van produkgehalte en bekorstingseffekte te evalueer, en (iii) die gevolge van vloeibevordering en skoonmaakmetodes te ondersoek. 'n Literatuuroorsig is uitgevoer om die skeidingsvereistes van die geweefde tekstiel te spesifiseer.

(i) Die karakterisering van die membraan het die effektiewe bestek in die grootte van die membraan se porieë, en die morfologie, geëvalueer. Die borrelgasvervoermetode het die effektiewe speling van die grootte van porieë van ongebruikte en vuil membraanmonsters as 10-24 µm en 7-18 µm onderskeidelik geskat. SEM skattingsevaluering het aangedui dat die komplekse membraanmateriaal met sy veelvuldige lae gevorm is deur die Plain Weave struktuur, en die veelvoud stringe gebruik is om die onderskeie drade te skep. Die gevolgtrekking was dat die vernouing van porieë weens bekorsting die gevolg was van die uiters gewronge membraanporieë wat gevorm is.

(ii) Evaluering van membraanprestasie het die effek wat die toevoersuspensie se fisiese en chemiese samestelling op die gevolglike produkgehalte en bekorstingseienskappe gehad het, bepaal. Die prestasie-evaluering het tot die slotsom gekom dat die geweefde tekstielmembraan in staat was om die nodige 1 NTU produkgehalte van toevoersuspensies met sub-poriegrootte (<3 µm) met wisselende fisiese en chemiese eienskappe te bereik deur die vorming van 'n poreuse bekorstingslaag. Dit het die vereiste dinamiese werking van die membraan afgesluit.

(iii) Die gevolge van voortdurende ruwe lugspoeling (7.5 L/min. en 2.5 L/min.) en fyn (5 L/min. and 2.5 L/min.) lugspoeling as metode om vloei te bevorder, het aanleiding gegee tot hoër bekorstingslaagweerstande in vergelyking met 'n doodloopbedryfstoets. Dit was die

gevolg van die ontkorstingspotensiaal van hierdie metode, wat die membraan se oppervlak doeltreffend ontkors het. Dit het die dinamiese werking van die membraan gekompromitteer, aangesien meer porieblokkering plaasgevind het weens die afwesigheid van die bekorstingslaag op die oppervlak.

'n Skoonmaakevaluasie is uitgevoer om terugspoelmetodes wat chemies aangehelp is (25 DPM natrium hipochloriet) vir terugspoelvloeiing van 20, 100 en 250 LMH om die gevolge van bekorsting heeltemal om te keer. Minimale herstel is deur al hierdie ontkorstingsmetodes getoon, weens die voorkoms van uiterste grade van onherstelbare bekorsting, soos getoon deur die SEM evaluering van die membraanmonsters. Die onherstelbare bekorsting was die gevolg van die toevoersuspensie wat kleiner was as die poriegrootte. Die enigste doeltreffende skoonmaakmetode was om die membraanoppervlak fisies te skrop. Dit is afgesluit deur die resultate van 'n toets wat uitgevoer is met 'n aanvanklike deurlaatvloeiing van 100 LMH, vir 60 minute, tussen fisiese skoonmaak, oor 'n tydperk van 10 uur, met geen ooglopende gevolge van onherstelbare of onherkrygbare bekorsting wat plaasgevind het nie. Weens die duursaamheid en stewige aard van die geweefde tekstiel kon dit hierdie fisiese skoonmaakmetodes, wat nie op die meeste beskikbare nywerheidstekstiele toegepas word nie, weerstaan.

Ter opsomming het hierdie studie duidelik getoon dat die gevolgtrekking bereik kon word dat die geweefde tekstielmembraan in staat is om doeltreffend die skeidingseffektiwiteit wat vir waterbehandelingsdoeleindes vereis word, te bereik. Net fisiese skoonmaakmetodes kon egter vir die membraan gebruik word, weens die vorming, binne die membraanporieë, van uiterste grade van onherstelbare bekorsting. Die gemak waarmee hierdie membrane fisies skoongemaak kan word is van veel groter waarde vir landelike en buitestedelike gebiede as wat potensieel skadelike chemiese skoonmaakoplossings sou wees.

Acknowledgements

I would like to thank my supervisor Professor V.L. Pillay of the Process Engineering Department at the University of Stellenbosch. The door to Prof. Pillay's office was always open whenever I ran into a problem with my research. He consistently allowed this work to be my own, but steered me in the right direction whenever he thought I needed it.

Finally, I must express my very profound gratitude to my parents and to my girlfriend for providing me with unfailing support and encouragement through these last two years of study. This accomplishment would not have been possible without them. Thank you.

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Nomenclature

Symbol or abbreviation	Interpretation
$\mathbf{A_{cross}}$	Membrane cross sectional area, m^2.
$\mathbf{A_d}$	Downcomer cross-sectional area in immersed membrane process, m^2.
$\mathbf{A_r}$	Riser cross-sectional area in immersed membrane, m^2.
BOD	Biochemical oxygen demand, mgO_2/L.
C	Concentration, g/L.
COD	Chemical oxygen demand, mgO_2/L.
DO	Dissolved oxygen, mg/L.
DOTM	Direct observation through the membrane.
F	Flow rate, kg/h.
FTN	Flavour threshold number.
$\boldsymbol{\gamma}$	Surface tension, N/m.
IMBR	Immersed membrane bioreactor.
J	Flux, LMH.
MF	Micro-filtration.
MWCO	Molar Weight Cut-off.
NF	Nano-filtration.
NTU	Nephelometric Turbidity Units.
P	Permeate flux, LMH.
POU	Point of Use device.

Q	Flow rate, L/h.
R	Membrane retention flux, LMH.
r	Rejection, %.
$\mathbf{r_{pore}}$	Membrane pore radius, µm.
RO	Reverse Osmosis.
SEM	Scanning Electron Microscope.
SMBR	Side Stream Membrane Bioreactor.
$\boldsymbol{\theta}$	Contact angle.
TMP	Trans-Membrane Pressure, Pa.
TOC	Total Organic Carbon, PPM.
TON	Threshold odour number.
UF	Ultra-filtration.
$\boldsymbol{v}$	Volume, L.
$\mathbf{W_R}$	Water recovery measured as a ratio.

Subscripts

F	Feed
f	Fouling
P	Permeate
R	Retentate

Chapter 1: Introduction

Overview

The chapter is divided into five sections, namely: 1.1 Background and motivation, 1.2 Objectives, 1.3 Approach, 1.4 Dissertation structure and 1.5 References. This chapter is used to introduce the reader to the woven fabric membrane material and to the requirements of the conducted study.

1.1 Background and motivation

The lack of access to safe, clean drinking water is a worldwide problem, this being especially true for developing economies due to a general lack of infrastructure. With large portions of South Africa falling in the category of rural[1] and peri-urban areas[2], the required infrastructure is simply not in place for large water treatment facilities to be constructed. For this reason, water treatment processes that incorporate membrane technology are being researched as possible water treatment alternatives to the conventional methods, due to their lower energy and infrastructure requirements [1-3].

The field of membrane technology is a rapidly advancing field of study. These advances have been fuelled by membrane processes being more and more suited to processes that until now have not been seen as membrane applicable [1,4]. This is demonstrably true for the water treatment industry where membrane processes are replacing the larger conventional wastewater and potable water production facilities [5]. The move towards membrane orientated processes lies in the benefit of achieving the same or improved water quality as the current water treatment processes, whilst on average, requiring less infrastructure and energy [6,7]. A relatively important characteristic of a membrane is that it is a physical separation barrier. Therefore, should a membrane be left unmonitored for extended periods, the product quality achieved will not decline but rather the yield will decrease with time to a point at which productivity ceases altogether, due to the effects of membrane fouling. Conventional water treatment processes rely on continual flocculation and disinfection dosing, that if left unmonitored will result in below standard water quality being produced. Whereas a

[1] Rural: sparsely populated area outside of the limits of a city or town or designated commercial, industrial, or residential centre [5].
[2] Peri-urban: categorises areas that fall between rural and urban, where an interaction of both influences is apparent [5].

membrane will ether produce a high quality product or will cease to produce a product altogether. This membrane characteristic is important for environments where highly skilled labourers are not available to continuously monitor the water treatment processes, such as in rural and peri-urban areas.

A short fall of all membrane processes is that they are affected by fouling[3], that overtime decreases the productivity and functioning of the membrane. The majority of a membrane's operating costs are as a result of the defouling processes that are incorporated into the membrane's operation [6]. Membrane defouling is a complex area of study, as a defouling strategy applicable to one membrane process, may not be as beneficial to another. This is largely due to the fouling characteristics being affected by the particular membrane configuration, the operation strategy and the nature of the feed suspension requiring processing.

Companies such as Kubota and Zenon have produced capillary and flat sheet membrane processes for potable water production and wastewater treatment. These membrane processes successfully meet the requirements of reduced energy and infrastructure needs. They are however unobtainable for rural and peri-urban areas due to the relatively high costs associated with these modern membranes. Additionally, the membranes utilised are not suited to the harsh nature of the environments for which they are required. This is due to the delicate make-up of the membranes incorporated into these membrane processes. The limitations of these processes therefore lie in the membrane technology incorporated in them. An improved membrane material is therefore required that can be incorporated in these modern membrane process that is economic viability and sufficiently robust to handle placement within the required harsh environments.

A new membrane technology has emerged that utilises a woven fabric microfiltration membrane material [1]. This membrane material is relatively less expensive than the industrially available competitive membrane materials whilst being more robust, making it easier to clean and maintain. This woven fabric has already been incorporated for use as a flat sheet membrane material for both wastewater treatment, as an immersed membrane bioreactor, and for potable water production, as point-of-use gravity fed membrane [1]. The flat sheet orientation promotes the use of physical cleaning methods that are cost effective

[3] Fouling: deposition of matter on or within the membrane pores [6].

and simple to perform. These woven fabric membrane processes have been successful in achieving positive results of permeate turbidity of lower than 1 Nephelometric Turbidity Units (NTU) and Chemical Oxygen Demand (COD) removal of up to 99% [1]. The woven fabric has therefore been proven capable of competing with some of the industrial membrane materials, thus making it an ideal candidate for water treatment applications as required in rural and peri-urban environments. However, comprehensive studies into the woven fabric membrane characterisation, performance effects, applicable flux enhancement and cleaning strategies have not been extensively covered and reported on. This has resulted in the stagnation of these woven fabric water treatment processes to further development.

This study was therefore proposed to develop a better understanding of the functioning of the woven fabric for use in water treatment processes. The study was performed by investigating the inherent membrane characteristics of the fabric, the resultant membrane performance in terms of the product quality and fouling effects exhibited and the effects flux enhancement and cleaning strategies have on the woven fabric membrane. The results will provide a basis on which further woven fabric membrane design and development can be based.

1.2 Objectives

The main goal with this investigation is to evaluate the woven fabric for use in water treatment processes suited to developing economies. The three objectives of this study are as follows:

i) Characterisation of the woven fabric membrane:

- Determine an effective membrane pore size range for the woven fabric material.
- Evaluate the woven fabric material morphology and the resultant properties.

ii) Performance evaluation of different aspects of woven fabric membrane:

- Investigate the effects different feed suspension physical properties and operating regimes have on the resultant membrane product quality achieved.
- Investigate the effects different feed suspension component properties have on the resultant fouling layer characteristics.

iii) Flux enhancement and cleaning methods evaluation to woven fabric membrane:

- Evaluate flux enhancement methods for managing the formation of the fouling layer.

- Evaluate cleaning methods for regaining the membrane functioning lost as a result of the effects of fouling.

1.3 Approach

The approach followed to meet the study objectives was subdivided into three sections.

1.3.1 Woven fabric material characterisation

The woven fabric material characterisation was evaluated by performing a bubble point test on woven fabric material samples to determine the effective membrane pore size range. The woven fabric morphology was evaluated using Scanning Electron Microscope (SEM) imaging of the fabric at various magnifications.

1.3.2 Woven fabric flat sheet membrane performance

The woven fabric flat sheet membrane performance was investigated by evaluating the effects different operating parameters and feed suspension compositions have on the product quality and the fouling characteristics exhibited.

1.3.3 Flux enhancement and cleaning methods

A combination of both flux enhancement and cleaning methods were investigated to evaluate their effectiveness to the woven fabric membrane. A preliminary evaluation of various flux enhancement and cleaning methods were performed to determine their applicability to the woven fabric membrane. A detailed investigation was then performed to further investigate the selected methods.

1.4 Dissertation structure

This dissertation is subdivided into 7 chapters. These chapters are used to guide the reader through the process routes followed to gain a better understanding of the functioning of the woven fabric membrane material.

In Chapter 1 the importance of the study, the objectives and the approach followed to meet these objectives, are provided.

In Chapter 2 the advancing field of membrane science and its use in water treatment applications is introduced. This is achieved with a review of the literature of various membrane processes and their functioning.

In Chapter 3 the woven fabric flat sheet membrane and the data processing methods followed during the subsequent investigations are described.

In Chapter 4 the characterisation of the woven fabric material is performed by evaluating empirical pore size range calculations and morphology related membrane phenomenon.

In Chapter 5 the woven fabric membrane performance evaluation is covered as well as the effects different feed suspensions and operating parameters have on the woven fabric membrane performance, in terms of the product quality achieved and fouling characteristics exhibited.

In Chapter 6 the evaluation of different flux enhancement and cleaning methods are investigated and the revision of the woven fabric flat sheet membrane design is covered.

Chapter 7 is a summary of the study as a whole with the final concluding remarks and recommendations is given.

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Chapter 2: Literature review

Overview

This chapter is divided into six sections, namely: 2.1 Membrane process overview, 2.2 Woven fabric membranes, 2.3 Membrane characterisation, 2.4 Water quality monitoring and standards, 2.5 Weaving terminology and 2.6 References. In this chapter a broad literature review of different membrane processes and their uses is given. Factors of importance to the investigation are also listed.

2.1 Membrane process overview

A membrane is a selective barrier, generally used to achieve some form of physical separation. Standalone membrane processes are generally non-invasive separation techniques that do not require any physical or chemical manipulation of the species being separated. Separation processes such as distillation, crystallisation and flash distillation require one or more of the species phases to be altered to achieve the desired separation [1]. It is this phase manipulation of the species that results in the aforementioned separation processes being highly energy intensive processes that are sensitive to variations in the operating parameters. These processes require strict control to be maintained over the operating conditions so as to keep them within the desired process specifications to insure stable product production. This is not the case for most membrane processes, as a membrane acts as selective barrier that will produce a product of a specific quality based on a physical seclusion forming due to the membranes pore size [2].

2.1.1 Membrane classification

Membranes are typically classified according to a nominal pore size. The nominal pore size is used to refer to the average equivalent perfect cylindrical pore size for a particular membrane. This is due to a membrane's pores, generally, not being perfectly cylindrical and often varying slightly in size over the membrane sample. The nominal pore size of a membrane gives an indication of the application to which the membrane would be suited, based solely on a size exclusion. The application ranges for the different membranes, namely sand filtration, microfiltration, ultrafiltration, nanofiltration and reverse osmosis are summarised in Figure 1:

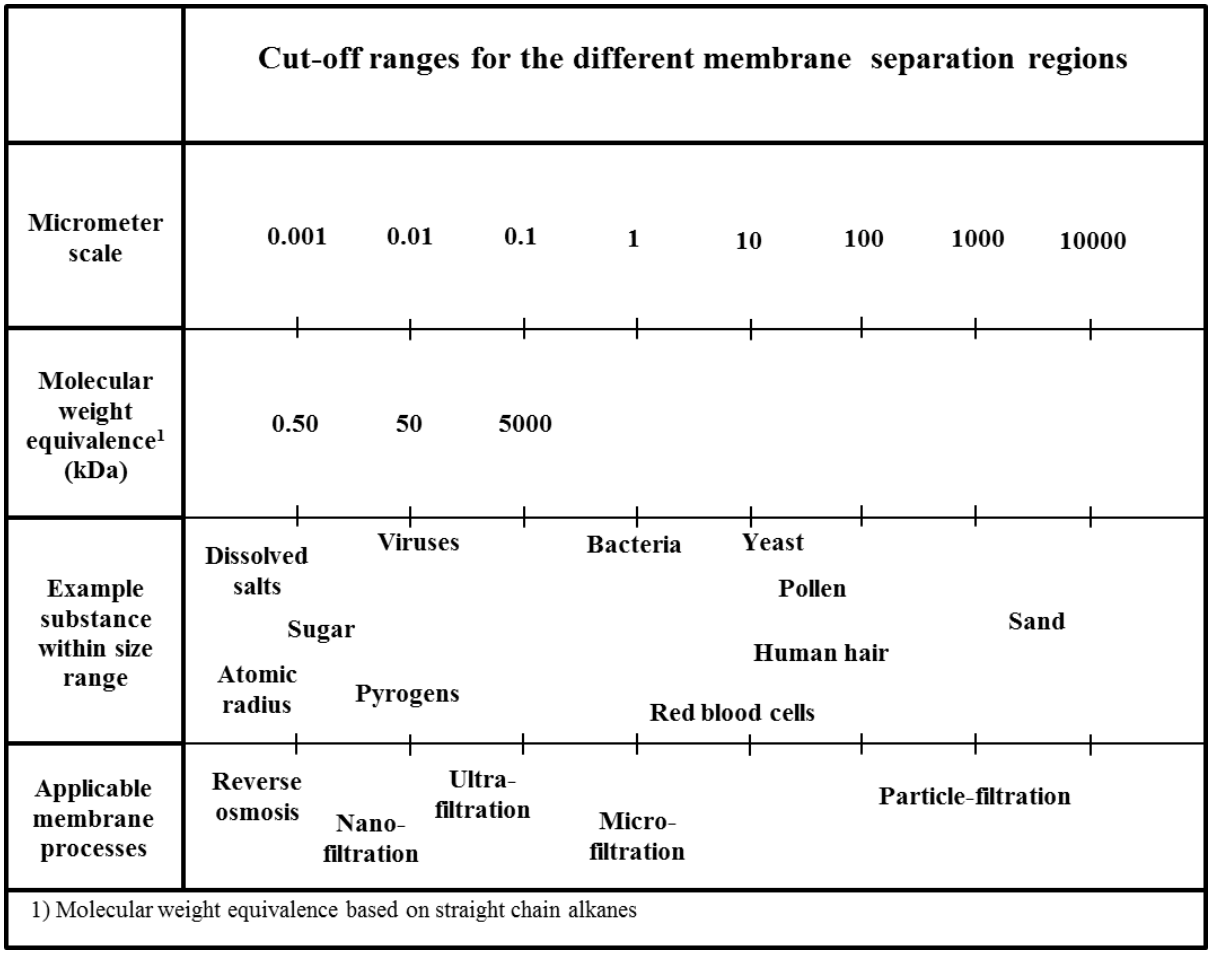

Figure 1: Membrane characterisation and process application, adapted from [3].

The figure indicates a generalised membrane pore size cut-off range used for classifying membrane types based on pore size. The figure also indicates different substances that fall within these pore size ranges.

Membrane manufacturers often give an indication of the molar weight cut-off (MWCO) of a membrane. The MWCO gives an indication of the largest straight chain alkane that will pass through that membrane based on either a 90 or 95% confidence interval, depending on the manufacturer [4]. It is often the case that two or more membrane materials can be applicable for use in the same membrane process. The selection is then made based on the desired final product quality.

The selection of a membrane separation process, up until recently, was solely based on size exclusion forming through the membrane pores. However, a new age of membrane technology has immerged that alters the membrane selectivity based on the chemical interactions that occur between the substance being separated and the membrane material [1].

A membrane can be engineered to either aid or hinder a particular species from passing through the membrane pores by altering the chemical selectivity of the membrane surface. Membrane processes can therefore incorporate both physical and chemical separation selectivity. These chemical interactions of the membrane surface with the species being separated, are as a result of, but not limited to, the construction material of the membrane and the type of membrane pre-treatment step utilised [4].

2.1.2 Membrane modes of operation

There are two general membrane operation modes, namely dead end operation and cross-flow operation. These two modes are represented in Figure 2:

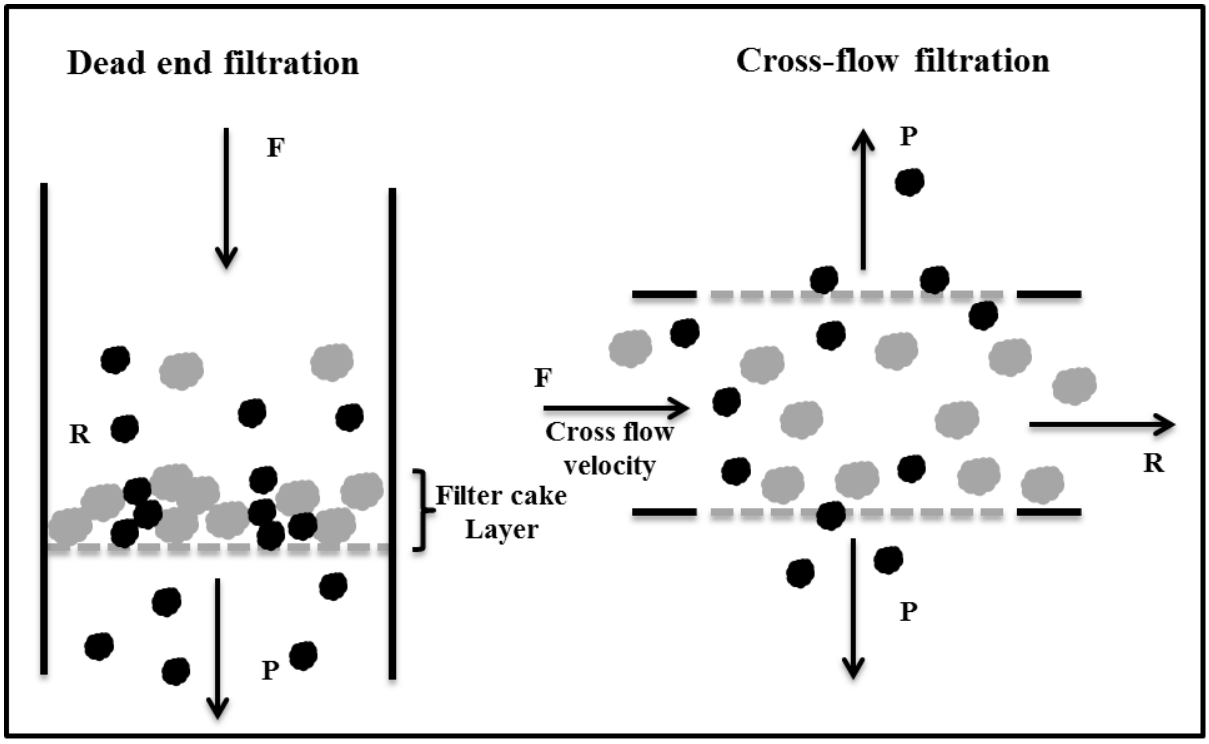

Figure 2: Membrane modes of operation, adapted from [5].

The difference between these two modes lies in the manner in which the feed stream is introduced to the membrane. Dead end operation is the mode that most people are familiar with, in that the feed is fed to the top of the membrane, such as when filtering sand with a sieve. The entire feed is emptied onto the membranes surface allowing the smaller particles to permeate through, leaving the concentrated larger particles on the membrane surface. This mode is relatively simple to implement, however results in a build-up of the retained substances on the surface of the membrane. The cross-flow operation mode is different in that the feed flow direction is tangential to the membrane's surface. This allows the permeate to

pass through the membrane while the remaining retentate is carried away from the membrane due to the tangential fluid flow.

Most industrial membrane processes are some or other adaption of these two operation modes. For example, an immersed membrane may resemble dead end operation mode, however with the addition of air scouring, it may function as a cross-flow operation mode process.

The variations of the filter cake layers formed as a result of the two different operation modes can be seen in Figure 3:

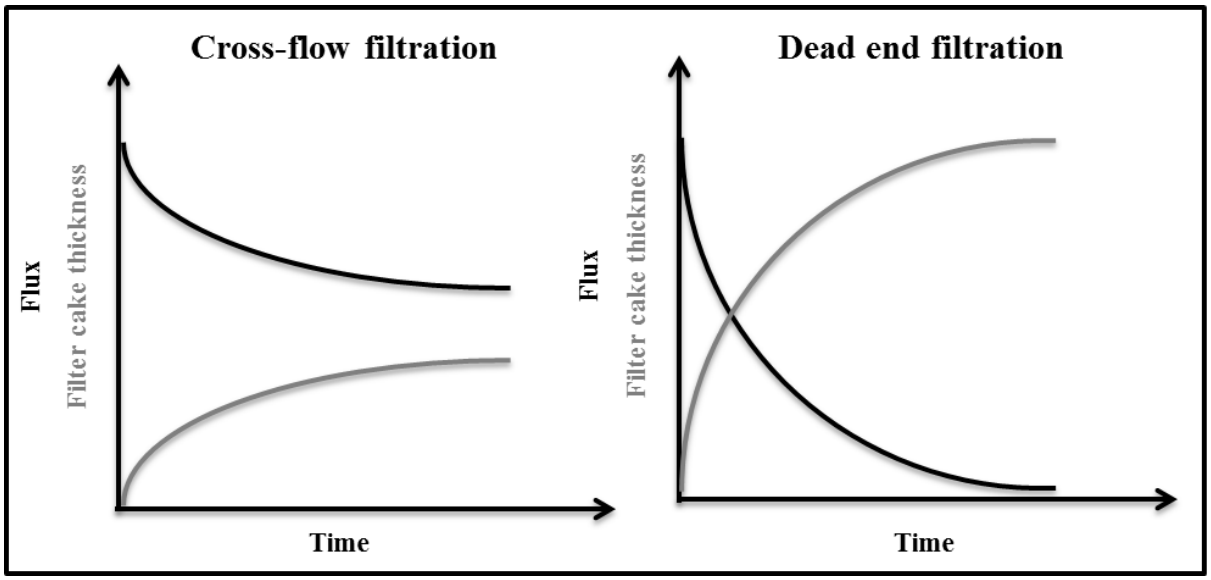

Figure 3: Permeate flux versus filter cake thickness relationships for cross-flow and dead end filtration modes [5].

The different operation modes result in varying flux and fouling characteristics. During dead end operation, a filter cake layer begins to form as a result of the retentate not being drawn away from the membrane. As the process continues, the filter cake increases in thickness until the permeate flux ceases due to the increased flow hindrance of the filter cake layer. During the cross-flow filtration mode, the propagation of the filter cake layer is potentially limited as a result of the cross-flow velocity carrying the retentate away from the membrane. The filter cake layer reaches a quasi-equilibrium thickness over time. In theory the higher the cross-flow velocity, the thinner the filter cake layer that is able to form [5].

2.1.3 Membrane operation configurations

When one thinks of a membrane, one generally envisions some form of sieve that is used to filter solid particle, such as sugar and flour. However, there are a wide range of different

membrane configurations available, each designed to meet certain requirements. The following are examples of some of the different membrane configurations available, namely: flat sheet membranes, tubular membranes, capillary membranes and spiral wrap membranes. A graphical representation of the flat sheet, capillary and tubular membranes can be seen in Figure 4:

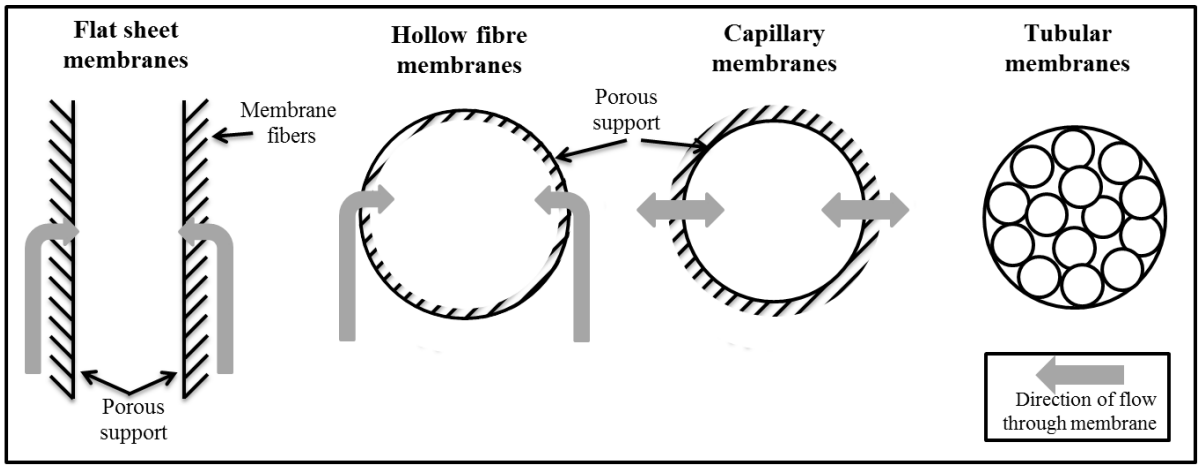

Figure 4: Variations in membrane flow orientations for different membrane configurations, adapted from [6].

The figure indicates the common flow orientations for these membrane configurations. It illustrates how hollow fibre membranes are designed in such a way that they do not collapse from the force of the liquid being exerted on the outside of the membrane by the inward flow direction, whereas the capillary membranes are constructed not to rupture from the force being exerted from either the inside or the outside of the membrane, depending on the flow orientation selected. Tubular membranes consist of an outer non-porous tube that contains multiple, capillary or hollow fibre membranes. The membranes used within tubular membranes generally do not have their own porous support layer and therefore require the additional rigidity from the outer tube to prevent them from deforming under pressure. This indicates how design measures are required to prevent the deformation of membranes due to the forces exerted during their intended operation configuration.

The investigation carried out as part of this report focusses on the use of flat sheet woven fabric membrane modules. There are both advantages and disadvantages to using a flat sheet style of membrane configuration. A graphical representation of a generalised flat sheet membrane module, as utilised for this investigation, can be seen in Figure 5:

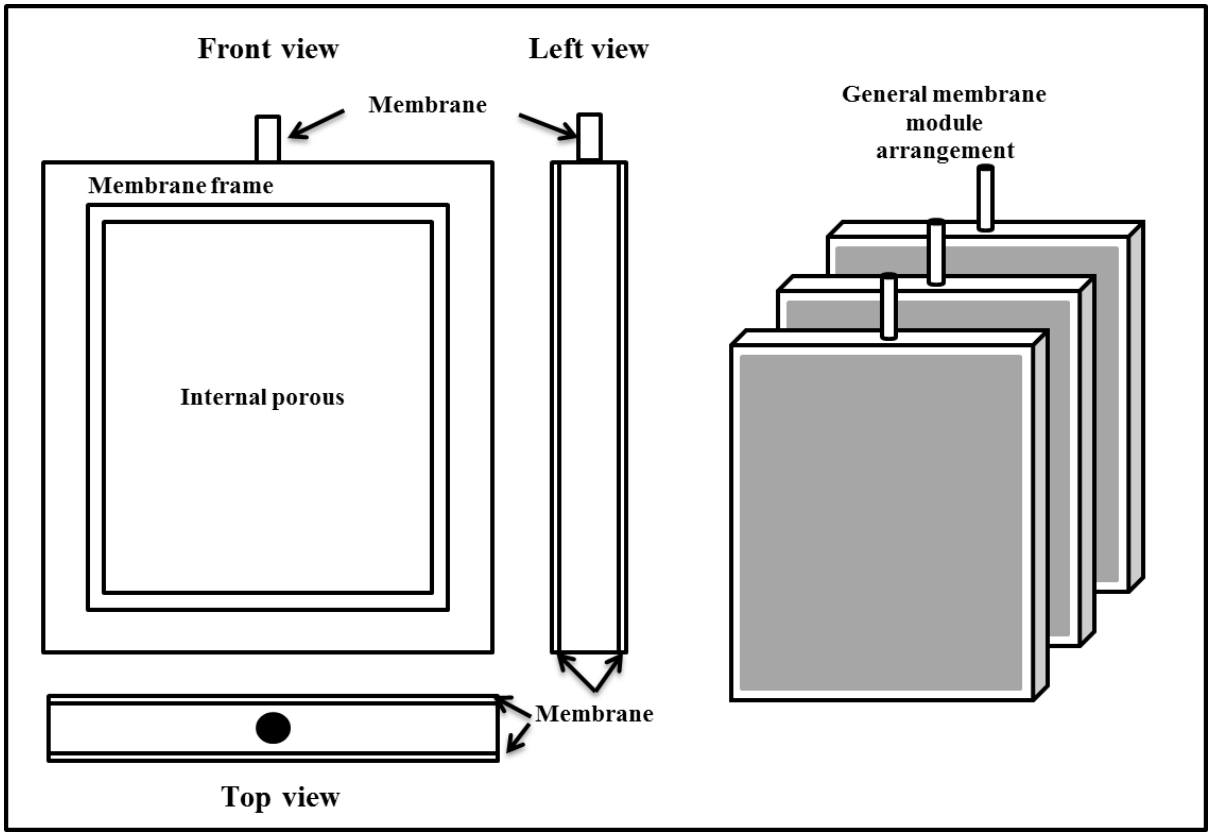

Figure 5: Flat sheet membrane design and layout.

The figure indicates the picture style frame used to secure the membrane layer and the use of a porous support layer between the two membrane layers. The membrane frame provides a surface area onto which the membrane layers can be fastened. The porous support layer is required to stop the membrane from collapsing when a suction force is applied through the membrane connection point. The porous support additionally channels the permeate fluid flow within the membrane.

Flat sheet membrane modules are used extensively in the field of membrane bioreactors due to the high suspended solids loading capability of the flat sheet orientation, as required by these treatment processes [2,3,5-10]. For this reason, further investigation into the operations and functioning of membrane bioreactors has been carried out to further evaluate the functioning of flat sheet membrane processes. The aim is to identify possible characteristics of the membrane bioreactors that can be utilised and carried across to a flat sheet woven fabric membrane, for use in water treatment processes.

2.1.4 Combined membrane processes

Membrane bioreactors (MBR) are a combination of a membrane separation process and a suspended growth bioreactor [7]. There are two general types of MBRs, namely immersed membrane bioreactors (IMBR) and side stream membrane bioreactors (SMBR) [5,7]. The difference between the two lies in the placement of the membrane within the process. An IMBR is a membrane process where the membrane is placed within the actual bioreactor vessel and the SMBR has the membrane placed in a separate unit to the bioreactor.

Membrane bioreactors became popular in the 1960s, as a result of polymeric ultrafiltration and microfiltration membranes becoming widely available [7]. These processes were used in the water industry as a means for treating wastewater as an alternative to the continuous activated sludge (CAS) process. The first MBR used in wastewater treatment made use of a SMBR design, where the activated sludge was pumped to the separate cross-flow membrane setup. This process proved to be effective for wastewater treatment, however high operating costs were incurred to partially overcome the rapid fouling rates [7]. To reduce these high operating costs, the membranes were placed directly in the bioreactor vessel. This resulted in the IMBR being produced. A graphical representation of an IMBR and SMBR can be seen in Figure 6:

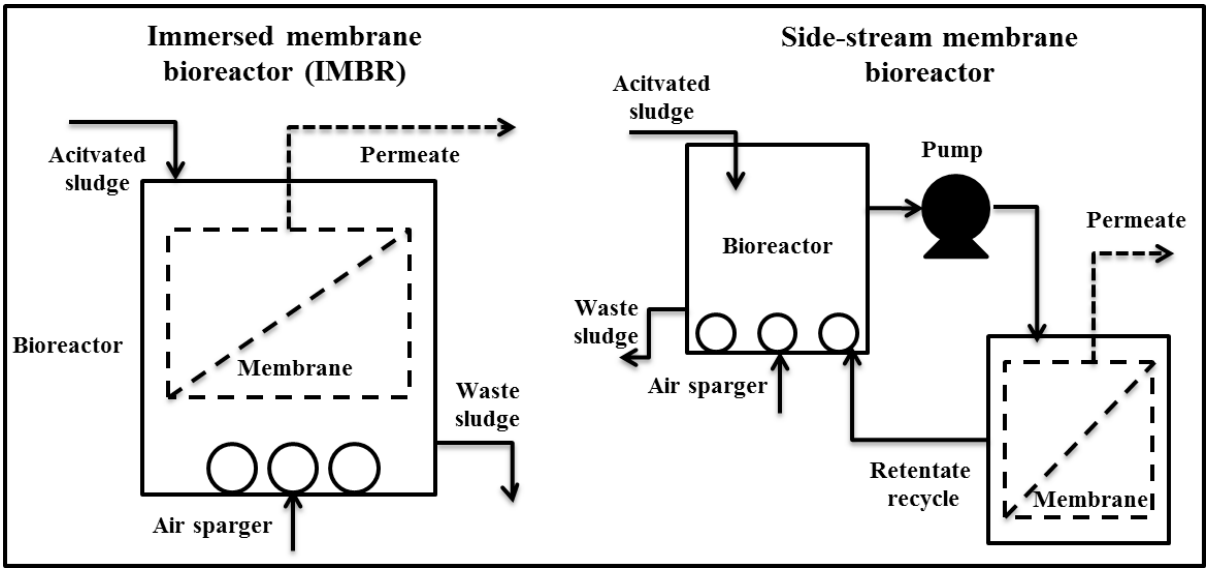

Figure 6: Comparison of an IMBR and a SMBR, adapted from [5].

The figure indicates the different positioning of the membrane within the membrane processes as well as the additional pumping requirements of the SMBR in order to pump the sludge to the external membrane unit. Pump selection is an important aspect of a SMBR

design, as a pump that creates too high of a sheer force on the biomass, would result in high biomass losses. The IMBR on the other hand is a simpler design, with less design constraints than the SMBR and therefore it is generally the more utilised membrane design.

Both the IMBR and the SMBR are currently used in industry. The SMBR may require additional operating costs, however due to the membrane being separate from the bioreactor, membrane maintenance can be performed in-situ or ex-situ without disturbing the bioprocess. The IMBR however requires that the membranes be removed before any chemical cleaners can be used. Therefore, both membrane processes have their own merits.

2.1.4.1 Immersed membrane bioreactors

As with all membrane processes, IMBR performance is greatly hampered by the effects of fouling. The addition of the biomass to the IMBR results in limited in-situ chemical cleaning options being possible without damaging the bioprocess aspect of the process [6]. Fouling in most cases has a negative impact on membranes, however it was noted by Wang *et al.* that the formation of thin bio-fouling layers, on the membrane surfaces, resulted in improved membrane functioning. This bio-fouling layer acted as an additional membrane separation layer [6]. However, as this bio-fouling layer increases with time, its positive impact rapidly diminishes as it restricts flow through the membrane. Air scouring is a measure used to control the formation of the bio-film layer and is therefore an important design consideration in an IMBR.

The air sparger for an IMBR is also an important design consideration based on the role it plays with regards to both the biological and membrane functioning aspects of the process. The air sparger provides dissolved oxygen to the biomass in order to sustain growth within the process. The air column additionally maintains the biomass in suspension. Another function of the air sparger is to provide a means of removing foulant from the surface of the membrane in order to control the propagation of the biofilm layer. The air is introduced using a series of diffuser units. Air scouring creates a shear over the surface of the membranes that results in the removal of foulant from the membrane surface. However, there is an upper limit above which an increase in air flow rate will no longer have an effect on improving the scouring efficiency [22,23].

2.1.5 Membrane performance measures

Membrane performance is a broad term that encompasses varying flow and recovery aspects of a membrane. Some of the more frequently utilised performance measures are as a follows: the permeate flux, the total resistance and the membrane rejection. For membranes used for water recovery, such as wastewater treatment, the membrane water recovery measure is an additional means of evaluating the membrane's performance.

The symbols and annotations used in the subsequent equations are based on the symbols represented in Figure 7:

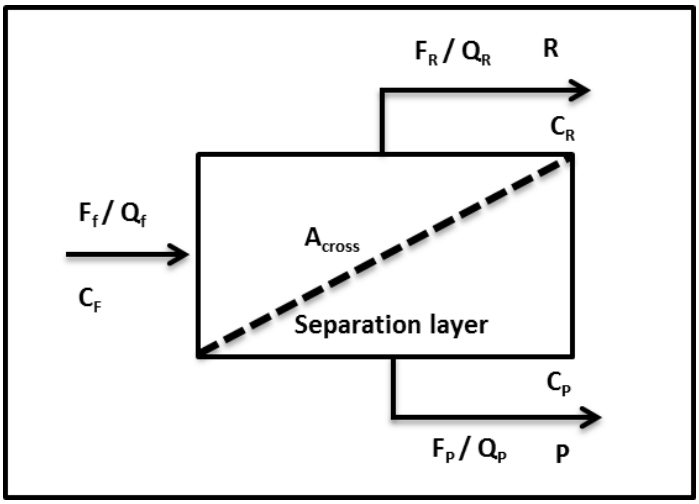

Figure 7: Membrane stream designations.

The figure is a block flow diagram used to illustrate the basic designation of the streams that enter and exit a membrane process. These stream designations are utilised by the different performance measures, as discussed below:

The permeate flux is used to indicate the mass or volume flow rate per square metre of active membrane surface area [4]. The membrane flux provides a means for comparing various membrane processes. The flux through a membrane is greatly affected by the following: the membrane nominal pore size, the membrane material and the operating pressure. The membrane operating pressure is also commonly known as the trans-membrane pressure drop (TMP). The permeate flux can be calculated for both continuous and batch membrane operation. The equations used to calculate the permeate flux for these are represented in Eq. (2.1) and (2.2):

$$J_m = \frac{F_P}{A} = \frac{m}{A\Delta t} = kg/m^2h \qquad (2.1)$$

$$J_v = \frac{Q_P}{A} = \frac{v}{A\Delta t} = l/m^2h \quad (2.2)$$

where J is the permeate flux, F_P the mass flow of the permeate, m the mass of the sample collected, Q the volumetric flow rate of the permeate, v the volume of the sample collected, Δt the time taken to collect the particular sample and A the area of the membrane exposed to the fluid being separated. The volume flux is often abbreviated as LMH (Litres per metre squared hour).

The total resistance is an important measure for membrane processes where both the flux and TMP of the membrane vary with time. This is due to the non-linear relationship that exists between these two measures as a result of the formation of a fouling layer [9]. For these membrane processes it is common practise to calculate the total resistance (R_T), using Darcy's Law [4], expressed in Eq. (2.3):

$$R_T = \frac{TMP}{J_p\mu} \quad (2.3)$$

where R_T is the total resistance, TMP the trans-membrane pressure drop, J_p the permeate flux and μ the fluid viscosity. It is important to note that the TMP, as referenced here, comprises the pressure differential created by the flow through the membrane and the fouling layer formed.

The membrane rejection is used to determine the percentage retention of a particular species by the membrane [4]. The rejection of a membrane is based on the selectivity of the membrane to allow certain species to pass through the membrane's selective barrier. The equation used to calculate the rejection can be seen in Eq. (2.4):

$$r = \left(1 - \frac{C_{Pi}}{C_{Fi}}\right).100 \quad (2.4)$$

where C_{Pi} is the concentration of species i in the permeate stream and C_{Fi} the initial feed concentration of species i in the feed solution.

The water recovery indicates the ratio of water that is retrieved through the permeate of the membrane. The closer the value is to one, the higher the portion of water recovered. The equation used to calculate the water recovery can be seen in Eq. (2.6):

$$W_R = \frac{Q_P}{Q_F} \tag{2.6}$$

where Q_P is the permeate flow rate and Q_F the feed flow rate.

2.1.6 Membrane fouling

Membrane fouling is an inevitable occurrence, in any membrane operation, that degrades the functioning of the membrane overtime. The nature of the fouling is dependent on the fluid being separated as well as the operating mode, operating pressure and any of the counter fouling measures used to control the fouling layer formation. The characteristics of a fouling layer are generally classified according to the chemical make-up or the physical adhesion of the foulant to the membrane. The chemical make-up of the fouling layer classifies it as either bio-fouling, organic fouling or inorganic fouling, whereas the physical adhesion classifies it as ether reversible fouling, irreversible fouling, residual fouling or irrecoverable fouling [6].

Bio-fouling takes place as a result of the growth of microorganisms on the membrane's surface. This type of fouling is only a problem with membranes that are operated on high organic matter concentrations, such as membrane bioreactors. The bio-fouling layer, if maintained correctly, acts as an additional membrane layer often improving the water quality achieved in an immersed membrane bioreactor [6,16,17]. The difficulties with bio-fouling layers lie in monitoring the thickness of this layer as it affects the membrane flow capability over time.

Organic fouling takes place as a result of the deposition of ether colloidal or soluble organic matter on the surface of the membrane. The organic matter is introduced by the feed to the membrane or as a result of microbial secretion [6,18,19]. The organic fouling forms a gel layer on the surface of the membrane and is known as gel layer fouling.

Inorganic fouling takes place as a result of metal ions and/or other crystalline precipitates present in the feed water. Inorganic matter often lodges itself within the membrane pores, creating large blockages [6,18,19].

Reversible fouling takes place as a result of fouling that lies loosely on the membrane surface that is easily removable using flux enhancement and or cleaning methods. Reversible fouling is seen as a more idealistic form of fouling. However, if it is left for long periods it can begin to harden on the membrane surface making it difficult to remove [6,18,20].

Irreversible fouling occurs when a fouling gel layer forms or as a result of reversible fouling that is left for long periods. Irreversible fouling results in pore narrowing due to the deposition of soluble matter on the walls of the membrane pores. This form of fouling cannot be removed without some form of chemical cleaning method that is able to dissolve the precipitated from the membrane pores [6,14].

Residual fouling is a new concept that is used to classify fouling that is not removed during the routine defouling of a membrane, but requires more intense cleaning strategy [7,10]. For example: maintenance cleaning may occur once a month, but once a year the membranes will require a more intense cleaning to remove any residual fouling that remains [6,14].

Irrecoverable fouling is used to describe the portion of the membrane's performance that is never recovered after the membrane is used for the first time, irrespective of the residual fouling cleaning methods utilised [6].

A graphical representation of reversible, irreversible and irrecoverable types of fouling can be seen in Figure 8:

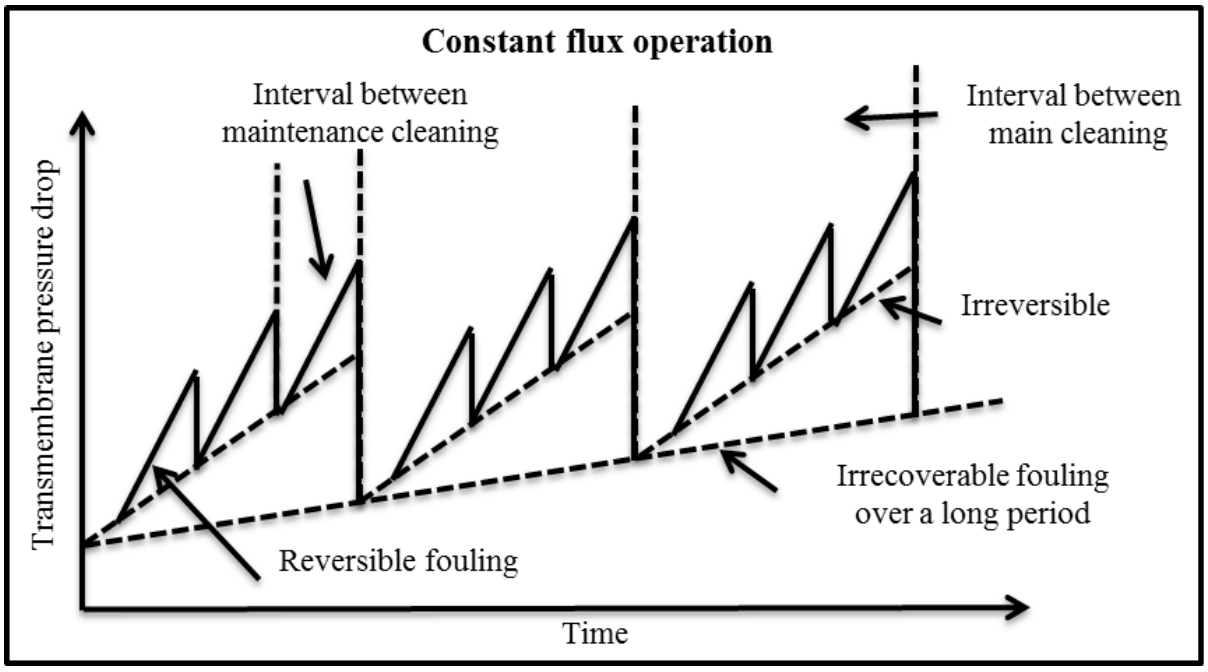

Figure 8: Membrane fouling characterisation using membrane TMP data, adapted from [8,10,14].

The figure indicates how TMP data from a constant flux membrane process can be used to give an indication of the types of fouling that is occurring. The figure indicates the difference between the maintenance and main cleaning intervals. The maintenance cleaning strategies are indicated as being ineffective at removing the irreversible portion of the fouling, only the

reversible fouling. The maintenance cleaning strategies are therefore applied in an effort to increase the operation period between applying the higher intensity, higher cost, main cleans.

The maintenance cleaning strategies are collectively termed flux enhancement methods. The flux enhancement methods can be applied as a periodic strategy or a continuous strategy parallel to the operation of the membrane. The maintenance cleaning strategies are aimed at managing the effects of fouling whereas the main cleaning strategies are aimed at completely reversing the effects of fouling. This is in an effort to regain the initial membrane functioning reduced as a result of fouling. The complete removal of a fouling layer, in general, is a far more intensive process in comparison to the managing the fouling layer and is therefore performed less frequently. The main cleaning strategies are collectively termed cleaning methods. The portion of the TMP that is never fully recovered once the membrane has been used, irrespective of the maintenance and/or main cleaning strategies used is the portion of irrecoverable fouling that forms.

2.1.7 Membrane defouling strategies

Membrane defouling strategies are categorized into two broad categories, namely: flux enhancement and cleaning. The intensity and the frequency of the applied defouling strategies are generally used to define whether it is classed as a flux enhancement method or a cleaning method. For this reason, a particular defouling strategy could be classified as both flux enhancement or cleaning depending on how it is applied.

2.1.7.1 Flux enhancement methods

Flux enhancement methods are often performed more frequently and or parallel to the operation of the membrane. The focus of this type of defouling is to manage the propagation of the fouling layer on the membrane surfaces. These methods are generally not able to completely remove or prevent the effects of fouling and therefore a higher intensity cleaning method is still required. The flux enhancement methods allow the membrane to operate for longer periods of time before needing to be taken off-line and rigorously cleaned. Examples of commonly applied flux enhancement methods are as follows: membrane backflush, membrane relaxation, air scouring, particle aided scouring, ultrasonification, vibration and back pulsing [6,15,32].

Membrane backflush is a process whereby the membrane flow is reversed in an attempt to dislodge the particles entrained within the membrane pores. The flow reversal is created by using a portion of the permeate to pass back through the membrane.

Membrane relaxation is a process whereby the flow through the membrane is stopped to allow the loosely packed particles on the membrane surface to dislodge. This method is effective for low TMP membrane processes. The relaxation method is often not sufficient as a cleaning measure due to lack of force or deactivation of the fouling layer.

Air scouring is a method commonly used in membrane bioreactors for the removal of the bio-film layer on the membrane surface [21-24]. An airstream is used to create a two-phase fluid flow over the membrane surface [7]. The two-phase fluid flow is a result of the upward airstream entraining fluid as it moves upward within the membrane vessel. The two-phase fluid stream applies a shear force to the fouling layer formed.

Particle aided scouring refers to the use of solid particles to scour the membrane surface whilst in-situ. The particles are maintained in suspension using an air stream and vary from solid bead-like particles to soft foam balls. The particles selected are dependent on the membrane material being utilised, as the particles can have an abrasive effect on the membrane surface structure if paired incorrectly [32].

Ultra-sonication refers to the use of an ultrasound generator to deactivate the constituents within the fouling layer using acoustic cavitation [32].

Vibration refers to the use of a vibration generator attached to the membrane module. The vibration energy instilled on the membrane module dislodges portions of the fouling material from the membrane surface [32].

Back pulsing is similar to a membrane backflush. The difference is that the reversed fluid flow through the membrane is pulsed to aid with the removal of the foulant constituents from the membrane surface. This method has benefits over the standard backflush method, however the back pulsing technique is often not applicable due to the strain applied to the membrane as a result of the pulsing motion.

2.1.7.2 Cleaning methods

Cleaning methods are generally more rigorous than flux enhancement methods as they are performed off-line with the focus on regaining the performance lost as a result of fouling.

Often a combination of physical and chemical cleaning is used for this [6,15]. Most of the above mentioned flux enhancement methods can be performed with the addition of a chemical disinfectant. However, these cannot generally be performed in-situ as chemical disinfectants affect the biological feed suspensions. Therefore, the addition of the chemical disinfectants are generally only utilised within cleaning methods, as opposed to flux enhancement methods.

2.1.8 Critical flux operation

Critical flux has been defined by Wang *et al* as the permeate flux below which no fouling is observed to occur on the surface of the membrane [8]. Therefore, if a membrane is operated below the critical flux, there will not be an increase in TMP or a decrease in the permeate flux with time. There are different methods for calculating the critical flux, namely flux stepping, pressure stepping, particle mass balances or by direct observations through the membrane (DOTM) [9]. The calculated critical flux is specific to the membrane process and fouling solution used and therefore cannot be extrapolated to other membrane processes due to the complex nature of the fouling characteristics, which differ for each process. For both the pressure and flux stepping methods, the critical flux is the flux after which the relationship between flux and TMP is no longer linear. The flux-stepping and pressure-stepping methods are illustrated in Figure 9:

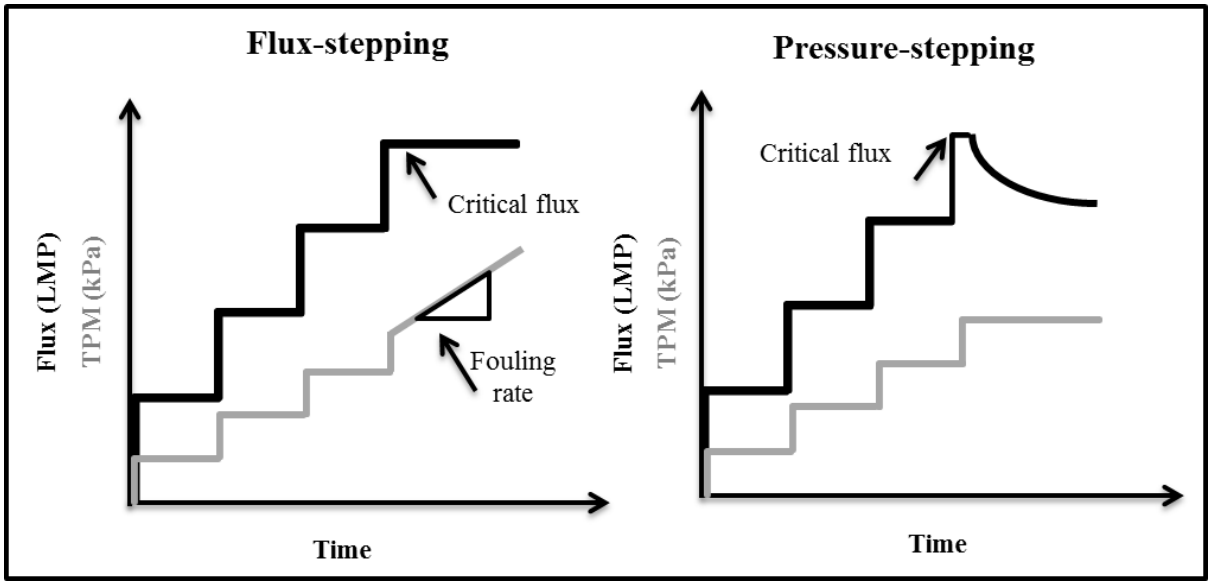

Figure 9: Flux-stepping and pressure stepping illustration, adapted from [9].

Flux-stepping is performed by selecting a desired operating flux and measuring the resultant TMP. This is repeated for increasing increments in the flux. It is important that the initial flux

selected is below that of the critical flux [9]. Once the critical flux is achieved, the rate of increase in TMP thereafter is the apparent rate of fouling. The fouling rate determines the increases in resistance through the membrane with time for the particular flux and solution as a result of the fouling layer formation [9]. The point at which the TMP increases linearly, and is no longer constant, is the critical flux pressure, with the corresponding flux at this point being the critical flux. It is important that relatively small increments in flux are made, as a too large increase will result in overshooting the actual critical flux.

Pressure–stepping is performed by selecting a desired operating pressure and recording the resultant flux. The pressure is then increased and the new flux recorded. It is important that the initial TMP selected is below the resultant critical flux. The pressure-stepping method indicates the steady-state flux for each of the operating TMPs selected below the critical flux. Once the critical flux is achieved, the flux will no longer be constant as a result of the fouling layer increasing with time.

The particle mass balance method uses a mass balance to determine the difference between the inlet and outlet concentrations of the feed suspension. A discrepancy between the amounts indicates entrainment of particles within the membrane as a result of fouling occurring on the membrane.

The DOTM method makes use of microscopic imagining of the membrane to determine if any fouling has occurred on the membrane surface. This method has limited accuracy as one cannot always see directly though the pores due to their potential tortuosity.

2.2 Woven fabric membranes

The woven fabric membrane is a relatively new membrane technology. The membrane is produced from a tightly woven polyester fabric. The woven nature of the membrane and the use of the polyester material have resulted in a membrane that is robust and economically viable in comparison with the majority of the other membrane technologies currently available [2]. Current flat sheet membrane technologies are made from polyimide-based materials. These membranes require a chemical pre-treatment step and once used, they cannot be left to dry out. The woven fabric membranes do not require any form of chemical pre-treatment step before they can be used, and once used, can be left to dry out with no adverse effects on the fabric. The polyimide membranes are in general sensitive to physical cleaning methods, as the membrane fibres are easily damaged. This limits the defouling methods to

chemically-orientated cleaning methods [6]. Chemical cleaning in general is relatively expensive, not readily accessible for all and a potential health risk, thus putting these membrane technologies out of reach for most rural environments [2].

The woven fabric is therefore well suited to rural environments due to its robustness which makes it cleanable using physical methods without the need of additional chemical cleaning steps. The woven fabric membranes have already been tested for use in a series of point-of-use (POU) potable water treatment units [2]. These units utilised additional purification steps, for example Mecha and Pillay incorporated an additional ultra-filtration membrane unit in one such POU potable water treatment unit. Another design by Mecha and Pillay impregnated the woven fabric membranes with silver nanoparticles to act as an additional purification medium for the water that passed through the membrane [2]. The impregnated woven fabric was able to improve E. coli removal from 91% with a standard woven fabric membrane, to 100% using the silver impregnated woven fabric membrane [2].

The woven fabric has been claimed to have a pore size range of 1-3 µm [2, 33]. This classifies the woven fabric as a microfilter membrane material. Tests conducted by Pillay and Buckely [33], indicated that the product quality achieved with the woven fabric is a function of the fouling layer that forms on the membrane surface. Therefore, as the fouling layer increases in thickness, the product quality achieved improves due to the smaller particles being entrained within the propagating fouling layer as opposed to passing through the membrane pores. This type of membrane process is referred to as a dynamic membrane process [33]. Fouling layer control for these membranes is crucial, as the fouling layer is required to achieve a particular product quality. However, if left to increase excessively the productivity of the membranes will be severely hampered. Cleaning methods are therefore required that are able to decrease the fouling layer propagation to a point that the membrane productivity increases, however the remaining fouling layer is adequate to achieve the required product quality. For feed solutions with low solids concentrations, Pillay and Buckley proposed the addition of limestone particles to the membrane as a pre-treatment step. This was to promote the formation of a fouling layer to improve product quality [33]. Membrane pre-treatment is common in dynamic membrane processes to aid the fouling layer formation.

The defouling methods as described in Chapter 2.1.7 Membrane defouling strategies, are not all applicable or feasible to this study due to the nature of the woven fabric being different to

that of other industrially available flat sheet membrane materials. The selected applicable defouling methods are explained in detail in the section that follows.

2.2.1 Backflush

Backflush is a process whereby the membrane flow is reversed in an attempt to dislodge the particles entrained within the membrane pores. The flow reversal is created by using a portion of the permeate to pass back through the membrane, by ether reversing the pump flow direction or by using a separate gravity fed line. The reason for using a gravity-fed line is to decrease the pumping costs of the process and to either increase or decrease the TMP applied to the membrane in reverse [10-20]. A chemical cleaning solution is often used as an alternative to using a portion of the permeate to backflush the membrane.

The woven fabric does not have the same rigidity as other industrial flat sheet membranes due to the woven fabric material not being impregnated with a porous support layer. Measures are therefore required to prevent adjacent membrane modules from coming into contact with one another when performing a backflush. The important design aspects of a membrane backflush for use in this investigation, are as follows:

- Backflush duration.
- Backflush flow intensity, pulsed or continuous.
- Backflush frequency, seldom or frequent.
- Backflush duration.
- The point within the operation cycle that it is initiated.
- Backflush driven by pump flow reversal or a gravity-fed backflush.

2.2.2 Membrane relaxation

Membrane relaxation is a process whereby the flow through the membrane is stopped to allow the loosely packed particles on the membrane surface to be dislodged. The efficiency of membrane relaxation may be increased if coupled with other flux enhancement cleaning methods. The important aspects of relaxation are:

- Relaxation duration
- The point within the operation cycle that it is initiated.

2.2.3 Air scouring

Air scouring is a common method used in immersed membrane bioreactors for the removal of the biofilm fouling layer that forms on the membrane surface [21-24]. An airstream is used to create a two-phase flow over the membrane surface [7]. The bubble size, flow rate and duration are important factors to take into consideration when designing the air sparger for a particular membrane process. There are two fields of thought as to whether fine bubble or coarse bubble air sparging is more beneficial for membrane defouling [7]. There is however no conclusive evidence as to which has a higher defouling potential, as is apparent from the fact that both methods are still being utilised in membrane processes. For this reason, both methods will be investigated.

To promote the air scouring effects, the membrane process geometry design is relatively important as to enhance fluid flow within the membrane process. The relatively important geometries that require consideration when designing a membrane apparatus can be seen in Figure 10:

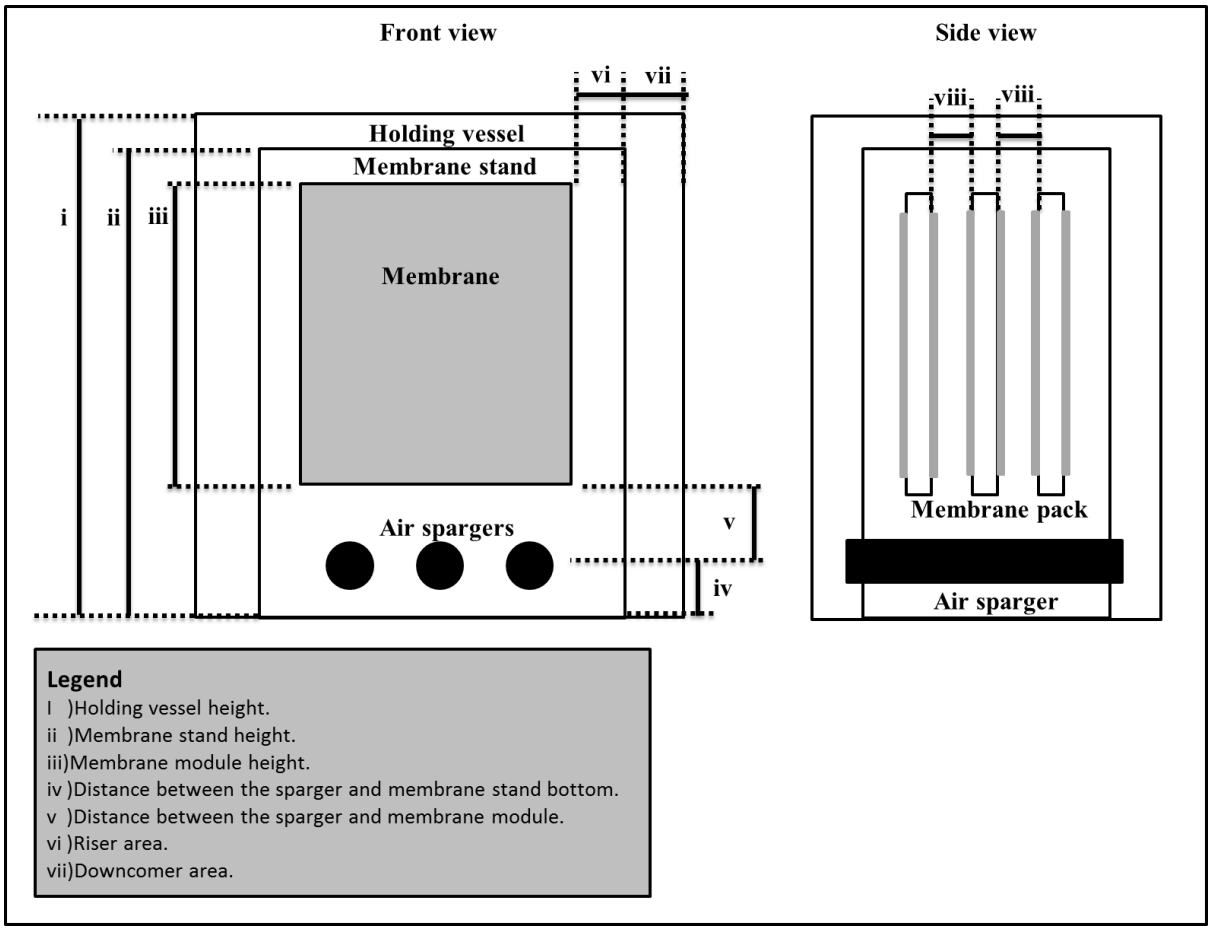

Figure 10: Air sparger design consideration, adapted from [11,22,24].

A brief explanation of the illustrated design geometries and their importance are given below:

i) The holding vessel height is an important geometric consideration due to the apparent increase in the bubble size with a decrease in the exerted pressure head. This affects the shear force impacted on the fouling layer. This may not be as important for laboratory scale membrane processes.

ii) The membrane stand height is dependent on the size of the holding vessel and the desired hydrodynamics. The membrane stand height will limit the membrane module dimensions and constrain the sparger diffuser design elements.

iii) The membrane module height will often be fixed by the manufacturer. However, for membrane processes with flexible membrane geometries, the height should be designed around the space required between the membrane and the sparger, and the sparger and the bottom of the vessel as these have an effect on the bubble formation and the induced two-phase fluid flow.

iv) The distance between the sparger and the bottom of the vessel has an effect on the two-phase fluid flow, as it acts as a reservoir for the fluid available for entrainment within the rising bubble column.

v) The distance between the air sparger and the membrane pack is important, primarily due to the effects it has on the bubbles size and distribution [34,35].

vi) The riser cross sectional area is the area available for fluid to pass between the adjacent membrane modules. The air sparger is situated within the riser section of the membrane process.

vii) The downcomer area is the area between the membrane stand and the holding vessel. No aeration occurs in this section of the apparatus. The downcomer area is required to promote recirculation of the fluid between the riser and downcomer sections.

The downcomer and riser areas are often referred to as the ratio of the downcomer area to the riser area (A_d/A_r). In a study conducted by Shim *et al.* into the effects of membrane geometry on air scouring efficiency in an IMBR, it was concluded that the ratio between the downcomer area and the riser cross sectional area was an important design consideration. Where a larger area ratio was indicated as aiding the air scouring efficiency [24]. A study conducted by M.L. Hamann *et al*. concluded that the higher A_d/A_r ratio configurations

resulted in relatively higher cross-flow velocities in the riser section of airlift reactors, which resulted in a proportionally thinner fouling layer forming on the surface of the membranes [22]. Literature indicates that A_d/A_r ratios of 2 and larger enhance the defouling effects of air scouring in an immersed membrane process [20-22,24].

viii) The inter-membrane spacing is an important geometric constraint as it has an effect on the fluid flow pattern between the membrane modules. A spacing that is too narrow will result in large coalescences of the air bubbles passing over the membranes. For an IMBR the most common inter-membrane spacing range utilised is 5-12 mm, however spacing of up to 32 mm are still utilised [11].

2.3 Membrane characterisation

The membrane characterisation procedure is used to develop an understanding of the functioning of a membrane material, in order to determine the applications to which the membrane is potentially suited. This is achieved by investigating the different membrane properties. Three of the key factors for determining a membrane's properties are the membrane pore size, the membrane morphology and the membrane hydrophobicity. The membrane pore size is no longer the only important aspect to be considered when selecting a membrane due to the current advancements that have been made in the field of membrane science.

2.3.1 Membrane pore size

Membrane pore size is an important membrane characterisation tool as this is often the main contributing factor to the separation efficiency of a membrane that is based on size exclusion alone. Even membrane processes that rely on chemical interactions between the membrane material and the feed suspension to form an effective separation, rely, to a degree, on the membrane pore size for an effective separation to occur. The membrane pore size is often expressed as either a nominal pore size or an absolute pore size. The difference between the two lies in the confidence level to which the pore size rating applies. For example, a nominal pore size implies that there is a 60-90 percentage certainty that a particle of the stated nominal pore size will be retained by the membrane, whereas an absolute particle size implies a certainty of 99% that the same size particle will be retained [27]. An important factor to note when characterising a membrane pore size is the that the pores are often not perfectly cylindrical or uniform in geometry throughout [27].

An additional pore size defining term, used extensively for membrane material with no real defined pore geometry, is an effective pore size. The effective pore size refers to the largest diameter round particle that would pass through the membrane pores. Effective pore size and nominal pore size are generally used interchangeably. Below follows a number of potential methods for characterising a membrane pore size.

2.3.1.1 The Bubble gas transport method

The bubble gas transport method determines the minimum pressure and maximum pressure required for a fluid to pass through a water filled porous membrane pore [27, 28]. This pressure is then correlated to an effective membrane pore size. This method of membrane pore size estimation is commonly used for membrane integrity tests and is commonly known as the bubble point test. For integrity test purposes, the pressure required for air to pass through the wetted membrane pore of a new, unused, membrane sample is compared to the pressure required for a used membrane sample. This way a discrepancy between the two readings is used to indicate potential deformation or the presence of residual fouling of the membrane pores [27]. A schematic representation of the bubble point mechanism can be seen in Figure 11:

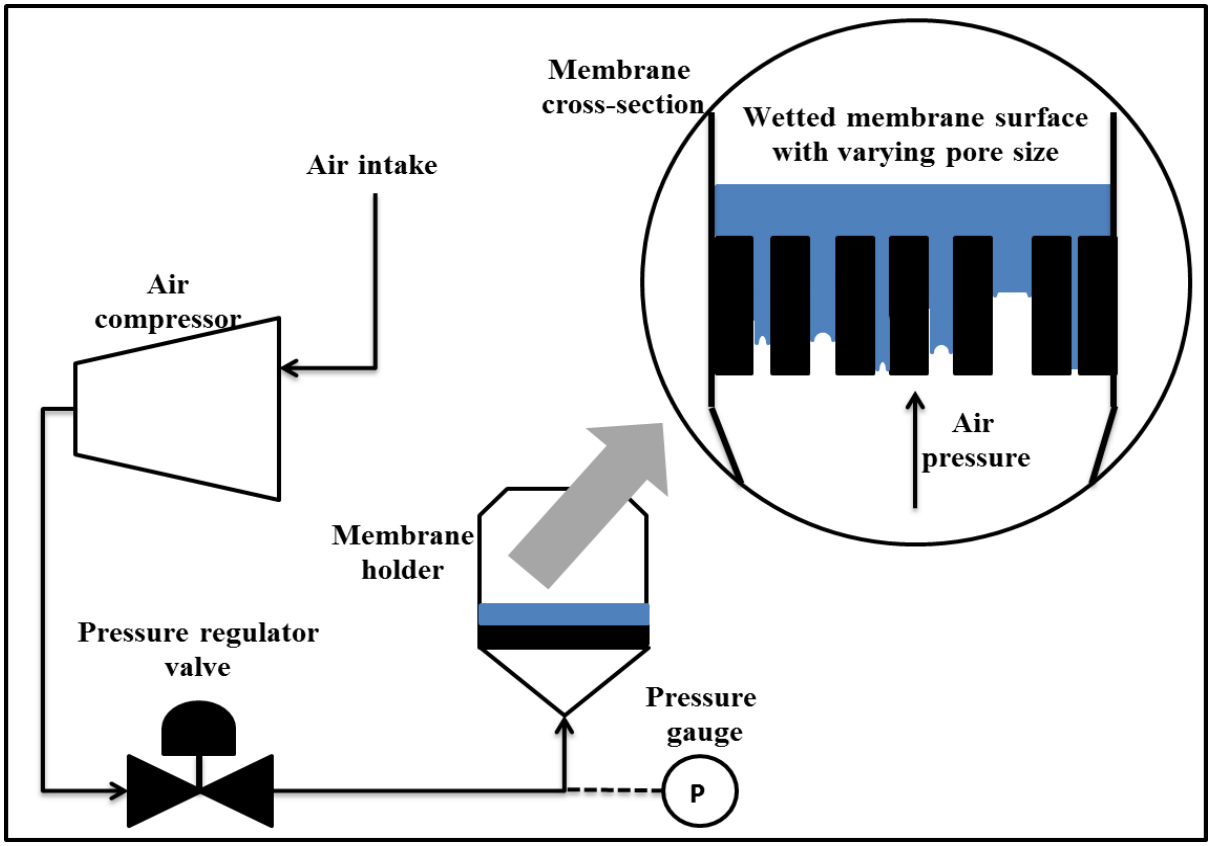

Figure 11: Bubble point test apparatus and mechanism, adapted from [27, 28].

The figure indicates how the first air bubbles that appear are as a result of the air stream pushing through the larger of the wetted membrane pores, with a relatively higher pressure required to push the air through the smaller wetted pore sizes.

It has been proposed that the bubble point test can be used to estimate the maximum and minimum pore size of a membrane sample [27]. The largest pore size is estimated using the pressure at which air just begins to pass through the membrane, and the smaller pore size is estimated using the pressure at which air begins to pass through the entire membrane surface. In terms of accuracy, the test is dependent on the operator's discretion with regard to when to distinguish between these two pressures. The equation used to correlate the air pressures to an effective pore size is based on the following Laplace equation, as represented in Eq. (2.6) [28]:

$$r_{pore} = \frac{2\gamma \cos\theta}{\Delta P} \tag{2.6}$$

where r_{pore} is the effective pore size, γ the surface tension of the membrane wetting fluid used, θ the contact angle of the wetting fluid with the membrane surface and ΔP the trans-membrane pressure differential required to pass air through the wetted membrane pore. A limitation of Eq. (2.6) with regard to its applicability to a woven fabric membrane lies in the assumption of perfectly cylindrical pores.

2.3.1.2 Membrane comparisons tests

The membrane comparisons test compares different product quality measures of an unknown membrane against a range of different known membrane pore size samples. A product quality measure, such as fluid turbidity, can be used as the comparative measure.

2.3.1.3 Particle size distribution of the feed and permeate streams

An evaluation of the particle size distribution of both the feed and the permeate streams is a means for determining a membrane pore size. The size distribution comparison gives a clear indication of the size of particles that remain entrained within the membrane pores. For this a single uniform particle distribution is required. The size distribution can be performed using any Scanning Electron Microscope image software or refractive index based methods. The refractive index methods are however limited to large solids concentration samples, which may not be achievable for low solids concentration processes. This method may not be applicable to dynamic membrane processes due to the product quality, of these membrane

processes, varying as a function of the fouling layer thickness. This could result in an over estimating the membrane pore size.

2.3.2 Membrane morphology

The membrane morphology defines the membrane material structure. The structure of the membrane is determined by the chemical makeup of the material as well as the process used to produce the membrane material. The morphology can be used to determine the membrane pore size, pore density, tortuosity and chemical selectivity.

Examples of the membrane morphology of a polyimide nanofibre membrane and an acid etch nucleopore membrane, captured using a Scanning Electron Microscope (SEM), can be seen in Figure 12:

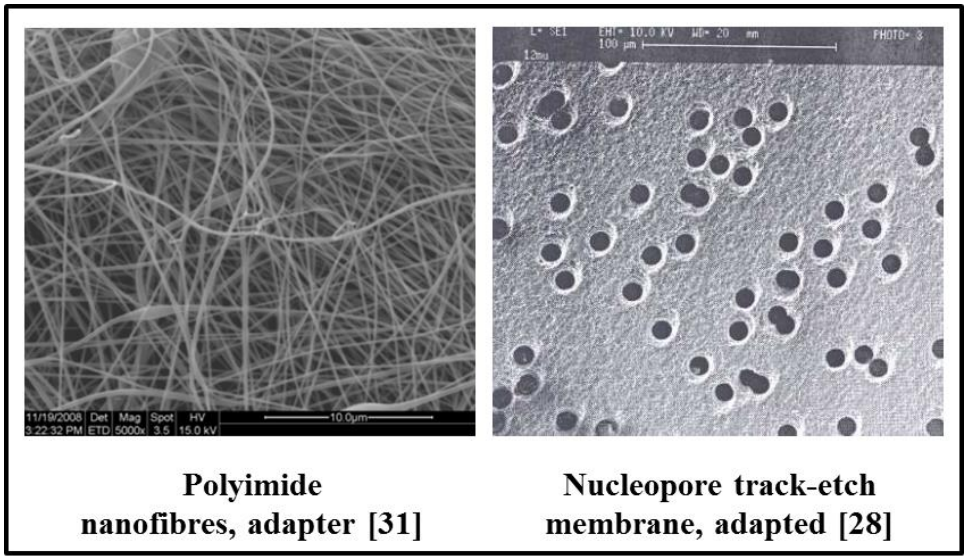

Figure 12: Illustrations of various membrane sample morphologies, adapted from [28,31].

The morphology of a membrane is dictated by the membrane manufacturer; however, it can be altered by the operator to some degree. The morphology can be altered by impregnation of the membrane material with different chemical compounds [29]. This chemical impregnation step is often used in polyimide membrane modules where the chemical used to pre-treat the membranes is selected so as to achieve a particular membrane chemical selectivity [4]. A SEM is used as a method for evaluating a membrane's morphology. The SEM produces high resolution microscopic images using an electron beam that can be used to determine the sample's topology and composition.

2.3.3.1 Membrane hydrophobicity

Membrane manufacturers often state if a membrane is hydrophobic or hydrophilic. This refers to a type of chemical interaction with the membrane and polar compounds, such as water. Hydrophobic membrane materials have a low affinity to polar molecules, whereas hydrophilic membrane materials have a high affinity to polar molecules. The hydrophobicity of a membrane material is a result of the membrane's morphology. The hydrophilic and hydrophobic nature of a membrane material can be determined by calculating the contact angle that water makes with the membrane surface. A contact angle (θ) of greater than 90° indicates a more hydrophobic membrane material whereas a contact angle of less than 90° indicates a hydrophilic membrane material. This is known as the Sessile Drop Method for measuring the contact angle that water makes with a solid surface [29,30]. An illustration of the key differences between a hydrophilic and hydrophobic interaction and how the angle is measured for both types can be seen in Figure 13:

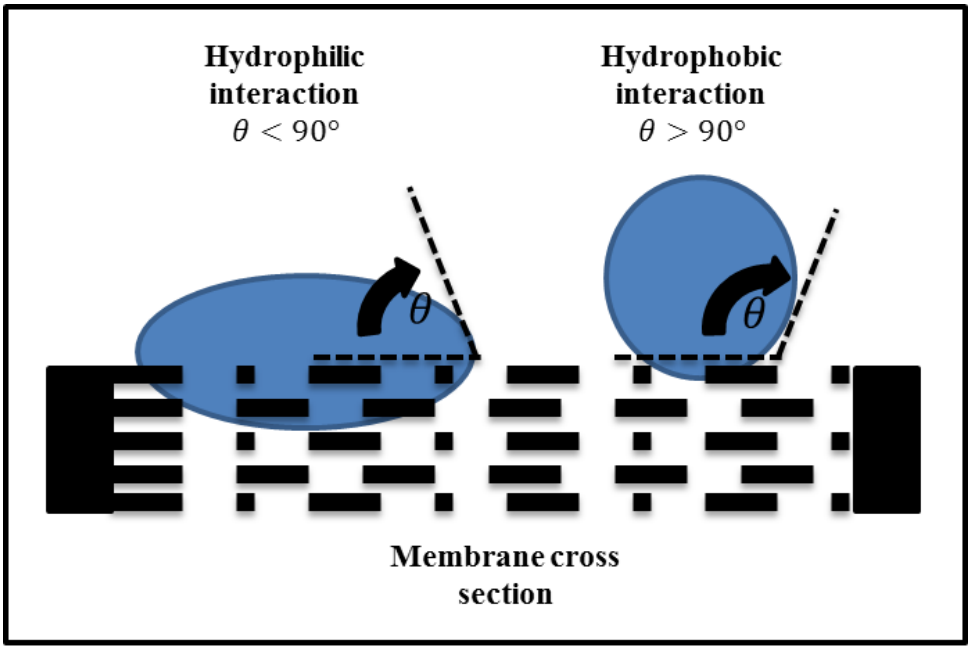

Figure 13: Sessile Drop Test Method, adapted from [29,30].

The hydrophilic or hydrophobic nature of a membrane can have an effect on the formation of a fouling layer on a membranes surface should the membrane have a low affinity to the particular foulant that is being removed.

2.4 Water quality monitoring and standards

2.4.1 Turbidity

Turbidity is the degree of 'cloudiness' or the loss of transparency of a water sample as a result of suspended solids [5]. Turbidity is measured in terms of Nephelometric Turbidity Units (NTU). A high NTU value indicates a high turbidity and therefore a large concentration of suspended solids within the sample. Due to turbidity relying on the refraction of light through the sample, the magnitude of the turbidity is affected greatly by the particular constituents and the particle geometry.

2.4.2 Classification of solid particles

The classification of a particle as a solid particle is based on the particle size. Solid particles are often larger than molecules but small enough to be undistinguishable with the naked eye [5]. In the water treatment industry, particles larger than 1 µm are termed suspended solids, particles between 0.001 and 1 µm are classified as colloidal particles. And particles smaller than 0.001 µm are classified as dissolved particles [5].

2.4.3 Dissolved solids

The term dissolved solids refers to any particle that dissolves in water, such as minerals, salts, metals, cations and anions [5]. The total dissolved solids are a combination of the inorganic salts and organic substances within water that are either in a molecular, colloidal, solid or ionized form [5].

2.4.4 Coliform bacteria and pathogens

Coliforms are bacteria that are present in the digestive tract of animals and humans, and are therefore present in their waste, as well as being present in plant and soil matter [5]. These bacteria are unlikely to cause health risks, however the presence of these bacteria is often used as an indicator for the presence of other disease-causing organisms. These disease-causing organisms are collectively termed pathogens [5]. The reason that coliforms are able to indicate the presence of pathogens is due to the majority of pathogens that can contaminate water being the result of faecal pollution from humans and animals into a water source. Testing water samples for the presence of all possible pathogens is time-consuming and expensive; therefore, instead of testing water samples for the presence of pathogens, the presence of the indicator coliforms is tested. Coliforms are generally present in larger

concentrations than pathogens making them easier to identify [5]. The most common method for testing for the presence of pathogens is to perform a total coliforms test. The total coliforms test indicates the presence of coliforms from human and animal waste as well as the coliform bacteria found in plant and soil matter. The total coliforms test therefore uses an indirect test to determine the possible presence of pathogens in a water sample.

2.4.5 Organic matter

Organic matter is introduced into water by plant and animal life. It is important to monitor organic concentrations within wastewater effluent, as high organic concentrations can result in esterification of the lakes and dams into which the effluent water is discharged. There are different methods used in industry to determine the organic matter concentrations within water samples, such as: BOD, COD and TOC.

The biochemical oxygen demand (BOD) test is a method used to determine the amount of oxygen required by aerobic biomass to oxidise all the organic matter present in the water sample. The BOD test is used in the wastewater treatment industry as an indication of the effectiveness of the bioprocesses in removing organics from the wastewater [5]. The BOD test indicates the amount of oxygen required to oxidise the organic matter only.

The chemical oxygen demand (COD) test is an indirect method used to determine the concentration of organic matter within a water sample. It is used in the wastewater and drinking water industries to give an indication of the concentration of organic pollutants within a sample. The COD method indicates the amount of oxygen consumed per litre of water. The test is based on the principle that all organic matter can be oxidised to form carbon dioxide, ammonia and water. The organic matter is oxidised using a strong oxidising agent, such as acidified potassium dichromate, rather than bacteria such as in the BOD test [5]. The COD is calculated based on the concentration of potassium dichromate that remains once the sample has been completely oxidised. A disadvantage of the COD method is that some of the inorganic material present may also be oxidised.

The total organic carbon (TOC) test is an analytical method for determining the amount of carbon present in the dissolved and suspended organic matter in a water sample. The TOC test is also used to monitor the total organic discharge to the environment from effluent plants [5].

2.4.6 Raw water

Raw water refers to any natural occurring water source found in the environment. Raw water therefore embodies a large collection of different water sources, such as ground water, surface water and rain water. Raw water samples can therefore comprise of relatively vast variations of organic and non-organic matter as a result of the geological position and any upstream occurrences. Additionally, the raw water sources are generally inconsistent in composition due to seasonal changes, dilution from rain and any variations in the upstream occurrences [37]. An example of such fluctuations in raw water sources can be seen in Figure 14:

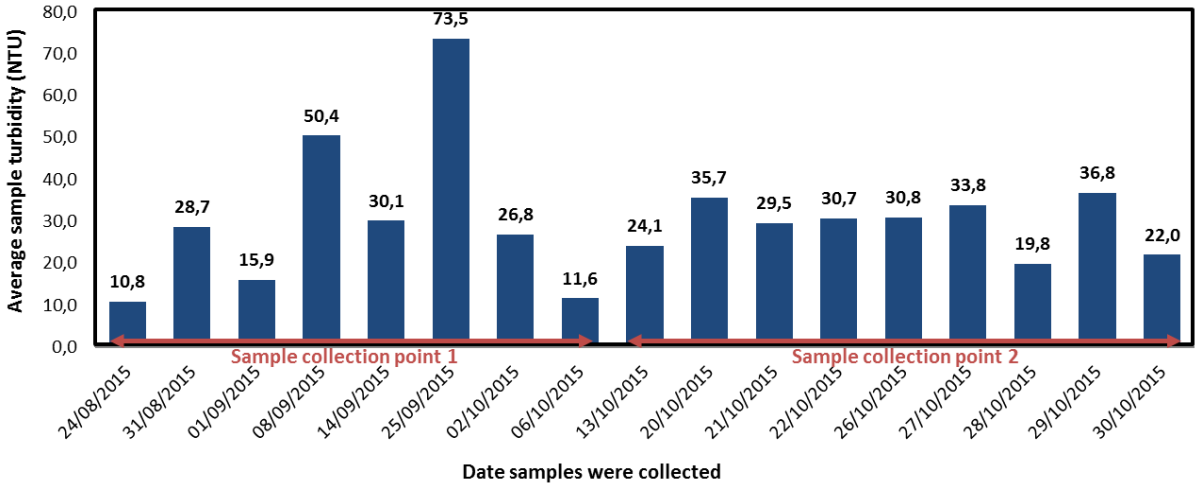

Figure 14: Raw water sample turbidity variation for two sample collection points located in the same river.

The figure indicates the variations in the raw water turbidity for two different raw water collection points, along the same river system. The red arrows are used to indicate which data is linked to which raw water collection point. The ‘Sample collection point 1’ was located directly after an informal settlement and the ‘Sample collection point 2’ was taken 5km downstream of the informal settlement and the ‘Sample collection point 1’. The figure indicates how the upstream occurrences affect the degree by which the turbidity can vary between two points that are a relatively short distance apart, over short periods of time. It is for this reason that most research studies utilise some or other synthetic feed suspension to increase the repeatability of the research performed.

A globalised standard for surface raw water was proposed by the Environmental Action Programme (EAP) in 2007 [38]. This was used to categorise different raw water sources. The

purpose of classifying the raw water sources was to indicate the applicability of the water source to use for drinking and food production, recreation or agriculture [38].

From the above text it is clear that for a membrane to be effective, the membrane has to be capable of handling variations in the feed suspension composition. Or alternatively strong control over the feed source constituents, prior to their entering the membrane would be required in order to maintain a relatively constant feed make-up to the membrane. In the case of the woven fabric membrane, where the focus is on a membrane suited to rural environments, tight control over the feed make-up to the membrane is not plausible. Therefore, the woven fabric membrane is required to handle variations in the feed make-up in order to be best suited to placement within the rural environment.

2.4.7 Water quality standards for potable water production

For a membrane to be deemed applicable to producing water that is safe for human consumption, there are a number of water production standards that need to be met. A summary of the potable water production standards required for safe human consumption, as set by the European Union (EU), Republic of South Africa (RSA) and World Health Organisation (WHO) are listed in Table 1:

Table 1: Potable water production standards, as set by the EU, RSA and WHO [12,25,26].

Parameters and constituents	EU set standard [25]	RSA set standard, SANS 241 [26]	WHO set standard [12]
Physical requirements			
Turbidity	≤1 NTU	≤1 NTU	≤5 NTU
Colour	Inoffensive	≤15 mg/L Pt-Co	≤15 mg/L Pt-Co
Odour	Inoffensive	<5 TON	<5 TON
Taste		<5 FTN	<5 FTN
pH (25°C)	6.5-8.5	5-9.7	6.5-8.5
Dissolved solids		<1200	<1000
Conductivity (20°C)	2500 µS/cm		

Microbiological requirements			
E. Coli	0 count/250ml	0 count/100ml	0 count/250ml
Total coliforms	0 count/100ml	≤10 count/100ml	0 count/ 100ml
Protozoan parasites	0 count/10L	0 count/10L	0 count/10L
TOC	No abnormal change	≤10 mg/L	No abnormal change

The potable water standards indicated in the table differ amongst the organisations. The EU, which is made up of developing countries, has higher water purification standards due to the infrastructure being in place to meet the higher standards. The RSA, which is considered a developing nation, has standards that are lower as the infrastructure is still lacking within certain regions. The World Health Organization (WHO) sets global standards and thus takes into consideration rural and slow developing nations who have limited resources for achieving the same standards as developed nations. For this reason, the WHO sets lower standards than both the EU and the RSA. For the purpose of this investigation, it would be ideal to aim for potable water production that meets the EU standards. However, as the membrane is aimed at rural and peri-urban environments, water that meets the WHO standards is classified as acceptable.

The table does not indicate the ionic constituent concentration standards. Ionic constituents will not be removed by a microfilter or ultrafilter membrane, due to the membrane nominal pore sizes being too large for ionic constituents, thus indicating the importance of proper water source selection prior to testing.

2.5 Weaving characteristics

In order to sufficiently understand the functioning of the woven fabric as membrane material, it is important to gain knowledge of weaving characteristics. This provides a means of characterising some of the woven fabric properties according to particular weave phenomena.

In weaving there are different fibre configurations, however in general, a woven material is made up of a weft and a warp thread. The warp is the lengthwise fibre that is initially loaded into the loom, whereas the weft is the fibre that is woven around the loaded warp fibres. The fibres used are either single fibres or a combination of spun fibres. Spun refers to the process

where multiple strands are wound to produce a single thread. A single pass of the weft thread is referred to as a pick. The weave tightness is then defined by the number of picks per inch (P.P.I.). An illustration of the warp and weft designations and P.P.I can be seen in Figure 15:

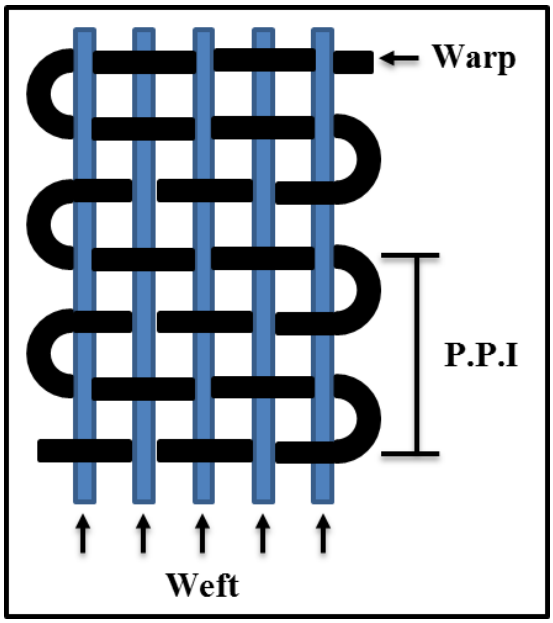

Figure 15: Weft and warp designation, adapted from [38].

The figure indicates a Plain Weave structure. The manner in which the weft is woven around the warp determines the type of weave produced, which has a direct effect on the strength and visual appearance of the resultant fabric [38].

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Chapter 3: Research apparatus and data processing

Overview

This chapter is divided into three sections, namely: 3.1 Research apparatus design, 3.2 Data processing and 3.3 References. This chapter covers an overview of the woven fabric flat sheet membrane apparatus design and the data processing procedures followed in the subsequent chapters.

3.1 Research apparatus design

The purpose of the study is to evaluate the woven fabric material for use as an immersed flat sheet membrane for water treatment purposes. For this study, the woven fabric material is fixed, with no potential to vary the physical make up or chemical composition of the fabric. The flat sheet membrane frame design, orientation, operating protocol and other parameters are however, to be designed as part of this study. A generalised flat sheet immersed membrane processes is made up of a membrane module, a membrane pack, a holding vessel, a membrane stand, an air sparger unit and other operational components. These are discussed in the sections that follow.

3.1.1 Membrane module

The generalised membrane module design utilised for the woven fabric flat sheet membrane modules can be seen in Figure 16:

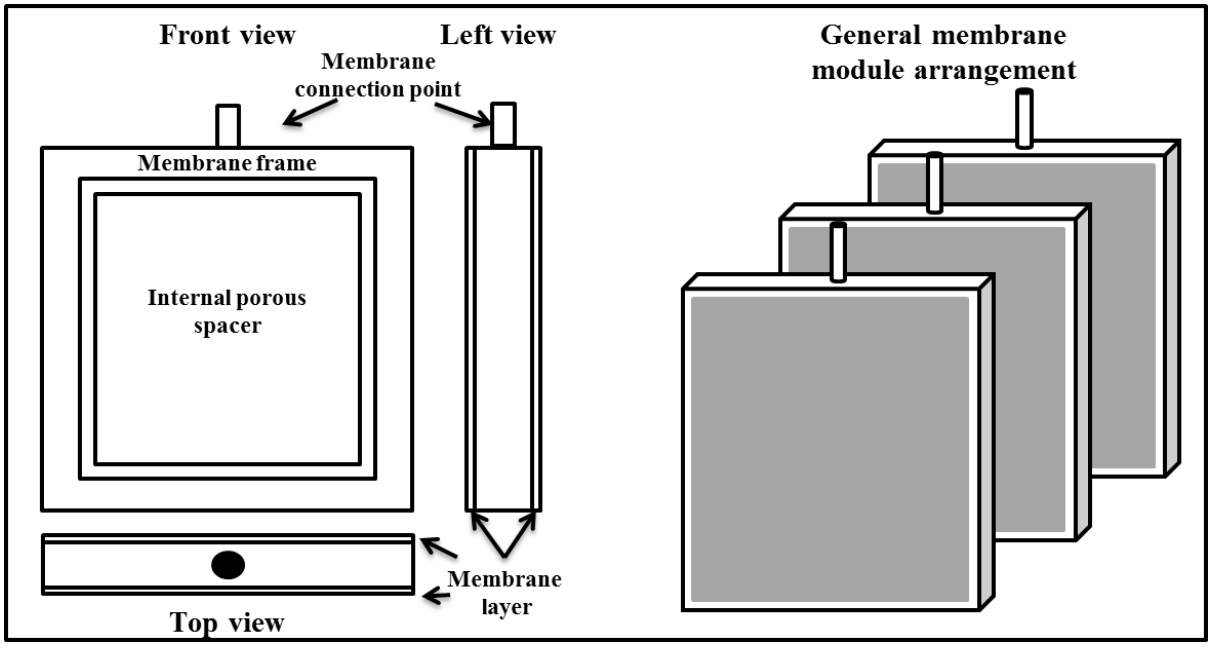

Figure 16: Woven fabric flat sheet membrane module design.

The woven fabric flat sheet membrane modules are made up of four parts, namely: a PVC frame, an internal porous spacer, woven fabric sheets and a membrane connection point. The PVC membrane frame is required to provide a surface onto which the membrane material could be glued in order to increase the membrane module rigidity. The thickness of the module was dictated by the internal porous membrane spacer unit positioned within the membrane module. The internal porous spacer was required to counter the expansion of the fabric when operating under suction. The membrane connection point was used to connect each of the membrane modules to a central manifold to which the pump could be connected.

The procedure followed for fabricating the woven fabric flat sheet membrane module is discussed in Appendix C: Flat sheet woven fabric membrane fabrication.

3.1.2 Membrane pack

The term membrane pack is used to describe multiple membrane modules that are fastened together. The spacing between the membrane modules is important when designing the membrane pack as it promotes the flow of the feed suspension between the modules. For membrane processes that use cross-flow operation mode techniques as a fouling control measure, the spacing has an effect on the magnitude of the shear force induced on the fouling layer adjacent to the membrane surface. The membrane spacing has been discussed in more detail in Chapter 2.1.7 Membrane defouling strategies and is investigated as part of Appendix E: Air scour design revision

3.1.3 Membrane holding vessel

The membrane holding vessel is required to house the membrane modules and the feed suspension. The holding vessel dictates the largest available membrane surface area that can be housed by the apparatus. The membrane holding vessel additionally determines the downcomer area, which has an effect on the membrane hydrodynamics. The membrane hydrodynamics is an important design consideration as has been discussed in Chapter 2.1.7 Membrane defouling strategies and is investigated further as part of Appendix E: Air scour design revision.

3.1.4 Membrane stand

An illustration of a membrane stand within a holding vessel can be seen in Figure 17:

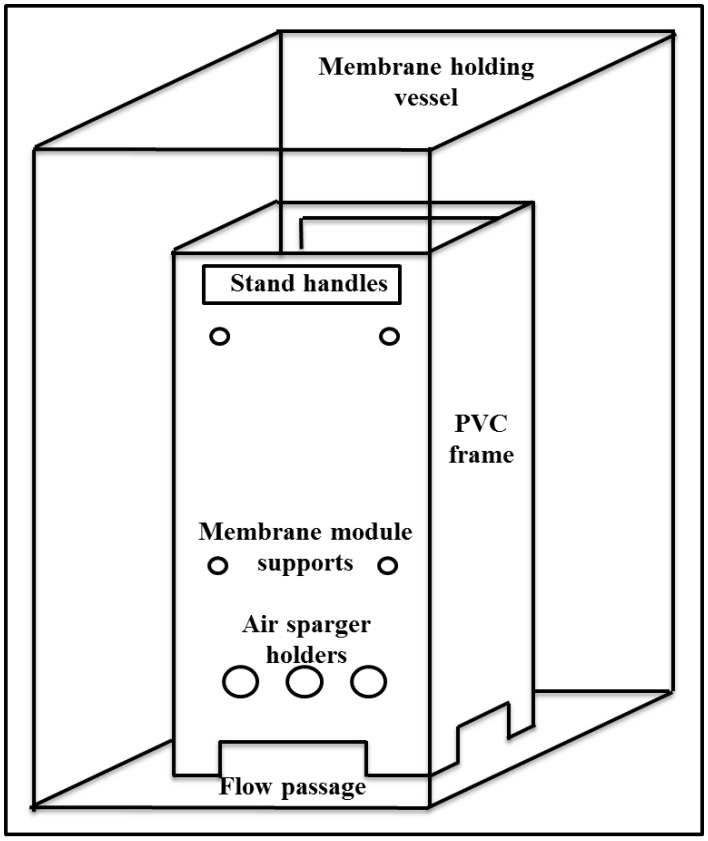

Figure 17: Membrane stand and holding vessel.

The membrane stand is an integral part of the immersed membrane apparatus. It is used to support and increase the rigidity of the membrane modules, provide an interface between the downcomer and riser sections to promote fluid circulation, and to house the air sparger diffuser units. The membrane stand is therefore an important design consideration for improved operation efficiency of the membrane. The membrane stand dimensions of the apparatuses used as part of this study were constrained such that the A_d/A_r ratio was equal to or greater than 2. The importance of the A_d/A_r ratio was made apparent in literature, where the outcome of the evaluated studies indicates the benefits of utilising an A_d/A_r ratio of 2 or greater [2,3,5]. This has been discussed in Chapter 2.1.7 Membrane defouling strategies.

The membrane stands were made from 8 mm thick PVC sheeting and produced in-house. PVC was selected due to its robust and durable properties. A membrane stand was not required for the tests that comprised of single membrane modules only.

3.1.5 Air sparger

The air sparger is used to create the bubble column required for the air scouring. The air sparger units utilised were produced using 10 mm PVC solid tubing, machined to size. A

combination of coarse and fine air scouring was tested depending on the requirements of the particular investigation. The air sparger's distribution hole pattern was determined experimentally using a purpose built Perspex tank, where the selection was based on bubble column formation requirements of the particular investigation.

3.1.6 Operational components

The operational components required are as follows:

- An air blower was required for the operation of the air sparger units.
- A positive displacement pump was used to draw the permeate fluid through the membrane modules.
- A pressure gauge was used to monitor the operational suction pressure drawn by the pump.
- A collection vessel was used to collect permeate samples.
- A turbidity meter was used for in-line monitoring of the permeate samples
- A stop watch was used to determine the fluid flow rate through the membrane at a given point during operation.

3.2 Data processing

The methodology followed for each of the investigations performed varied based on the requirements of the investigation. However, the testing procedure and data processing for each of these investigations remained relatively unchanged and are therefore discussed below.

3.2.1 Flow rate and permeate flux calculations

In order to calculate the permeate flux for a given experimental run, the permeate fluid flow rate and membrane surface area are required. The fluid flow rate was calculated using a measuring cylinder and a stop watch. The stop watch was started as the permeate filled the measuring cylinder. The time taken to fill a certain volume of fluid in the measuring cylinder was then recorded. The membrane surface area was calculated by multiplying the length and width of the membrane surface and then multiplying by a factor of 2, as there are two flat sheet membranes per membrane module. Eq. (3.1) was used to calculate the permeate flux:

$$J_P = \frac{v}{A\Delta t} = l/m^2h \qquad (3.1)$$

where J_P is the permeate flux, v the volume of the permeate sample collected, Δt the time taken to collect the particular sample and A the active area of the membrane exposed to the fluid being separated.

3.2.2 Resistance calculations

For membrane processes where both the flux and TMP vary with time, it is common practice to calculate the total resistance using Darcy's Law [4], expressed in Eq. (3.2):

$$R_T = \frac{TMP}{J_p \mu} \tag{3.2}$$

where R_T is the total resistance, TMP the trans-membrane pressure drop, J_p the permeate flux and μ the fluid viscosity. It is important to note that the TMP, as referenced here, comprised the pressure losses of both the fluid flow through the membrane and the fouling layer formed, as expressed in Eq. (3.3):

$$TMP = TMP_{membrane} + TMP_{fouling} \tag{3.3}$$

$$TMP_{fouling} = TMP - TMP_{membrane} \tag{3.4}$$

Similar to the TMP comprising of the pressure losses of both the membrane and fouling layer, the total resistance term, R_T, comprises the membrane resistance, R_m, and the resistance caused by the formation of the fouling layer, R_f, represented in Eq. (3.5):

$$R_T = R_m + R_f \tag{3.5}$$

The resistance of the fouling layer, R_f, is of more importance than the total resistance, R_T. However, in order to calculate the fouling layer resistance, the membrane resistance is required. The membrane resistance is the result of the fluid flow hindrance through the membrane and connectors and is specific to a membrane process due to the influence the connectors and the membrane module dimensions have on the resistance to fluid flow. The fouling layer resistance provides a means of comparing fouling layer formations, amongst different woven fabric membrane processes, without the influence of the varying membrane resistance. Combining Eq. (3.2), Eq. (3.3) and Eq. (3.5) the total resistance can be separated into the separate resistance terms and calculated as follows in Eq. (3.6), Eq. (3.7) and Eq. (3.8):

$$R_T = R_m + R_f = \frac{TMP}{J_p \mu} = \frac{1}{J_p \mu}\left(TMP_{membrane} + TMP_{fouling}\right) \quad (3.6)$$

$$R_f = R_T - R_m$$

$$R_m = \frac{1}{J_p \mu} TMP_{membrane} \quad (3.7)$$

$$R_f = \frac{1}{J_p \mu} TMP_{fouling} \quad (3.8)$$

The $TMP_{membrane}$ and the $TMP_{fouling}$ are both functions of the fluid flow magnitude through the membrane. Therefore, it was not a valid assumption to assume that the $TMP_{membrane}$ would remain constant for the duration of the investigation. This was due to the fouling layer formation decreasing the fluid flow through the membrane over time. For this reason, the $TMP_{membrane}$ was calculated, for each of the woven fabric membrane processes utilised, as a function of the fluid flow rate through the membrane. Linear regression was applied to determine the $TMP_{membrane}$ as a function of the flow rate, as indicated in Eq. (3.9):

$$TMP_{membrane} = K.Q_p + C \quad (3.9)$$

Eq. (3.9) allowed the $TMP_{membrane}$ to be calculated and subtracted from the TMP in order to determine the pressure loss as a result of the fouling layer only, for varying flow magnitudes. Eq. (3.8) was used to calculate the fouling layer resistance only.

3.3 References

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Chapter 4: Characterisation

Overview

This chapter is divided into three sections, namely:4.1 Membrane characterisation procedure, 4.2 Overall conclusion of woven fabric characterisation and 4.3 References. This chapter covers the characterisation of the woven fabric material by evaluating empirical pore size range calculations and morphology related membrane phenomena.

4.1 Membrane characterisation procedure

The woven fabric has been utilised in water treatment processes as an IMBR for wastewater treatment and as a gravity-fed point of use device (POU), for raw water treatment [1,4,7]. The woven fabric has however undergone a series of revisions since its initial conception, due to the woven fabric material initially not being intended for use as a membrane material. For this reason, the currently utilised woven fabric required characterisation in order to further develop these membrane processes. The characterisation procedure evaluated the woven fabric material's effective pore size and morphology. The characterisation was focussed on evaluating the woven fabric in its supplied form.

The characterisation procedure was sub-divided into two parts, namely membrane pore size estimation and membrane morphology.

4.1.1 Membrane pore size estimation

The membrane pore size was an important aspect of characterising the woven fabric for use as a membrane material. The effective pore size of a membrane gives an indication of the physical separation potential of the membrane, based on a size exclusion formed through the membrane pores. The bubble gas transport method was selected for the membrane pore size estimation.

4.1.1.1 The bubble gas transport method

The bubble gas transport method was used to determine an effective pore size range for the woven fabric material. The pore size was expected to vary between a maximum and a minimum pore size as a result of the woven fabric pores being produced due to the irregular spacing forming between the intersections of the weft and warp threads of the fabric. The bubble gas transport method determined the pressure required to pass fluid through a fluid

filled, wetted, membrane pore. The equation used to correlate the air pressures to a pore size was based on the following Laplace equation, represented in Eq. (4.1):

$$r_{pore} = \frac{2\gamma \cos\theta}{\Delta P} \tag{4.1}$$

where r_{pore} is the effective pore size, γ the surface tension of the membrane wetting fluid used, θ the contact angle of the wetting fluid with the membrane surface and ΔP the trans-membrane pressure differential required to pass the fluid through the wetted membrane pore. A limitation of applying Eq. (4.1) to the woven fabric membrane was in the assumption of perfectly cylindrical pores.

The contact angle required for use in Eq. (4.1) was determined to be $76.3^{o} \pm 18^{o}$ using the Sessile Drop method. This was calculated as part of an investigation by Cleophas [1] evaluating the effectiveness of impregnating the woven fabric with silver nanoparticles for disinfection purposes.

4.1.1.2 Apparatus

A bubble point apparatus was purpose-built to determine the pressure at which water ($\gamma_{water} = 0.07197\ n/m$) would pass through the largest and smallest pores of a woven fabric membrane sample. The apparatus included a compressor to pressurise the membrane sample and a mercury manometer to monitor the pressure exerted. An illustration of the complete setup can be seen in Figure 18:

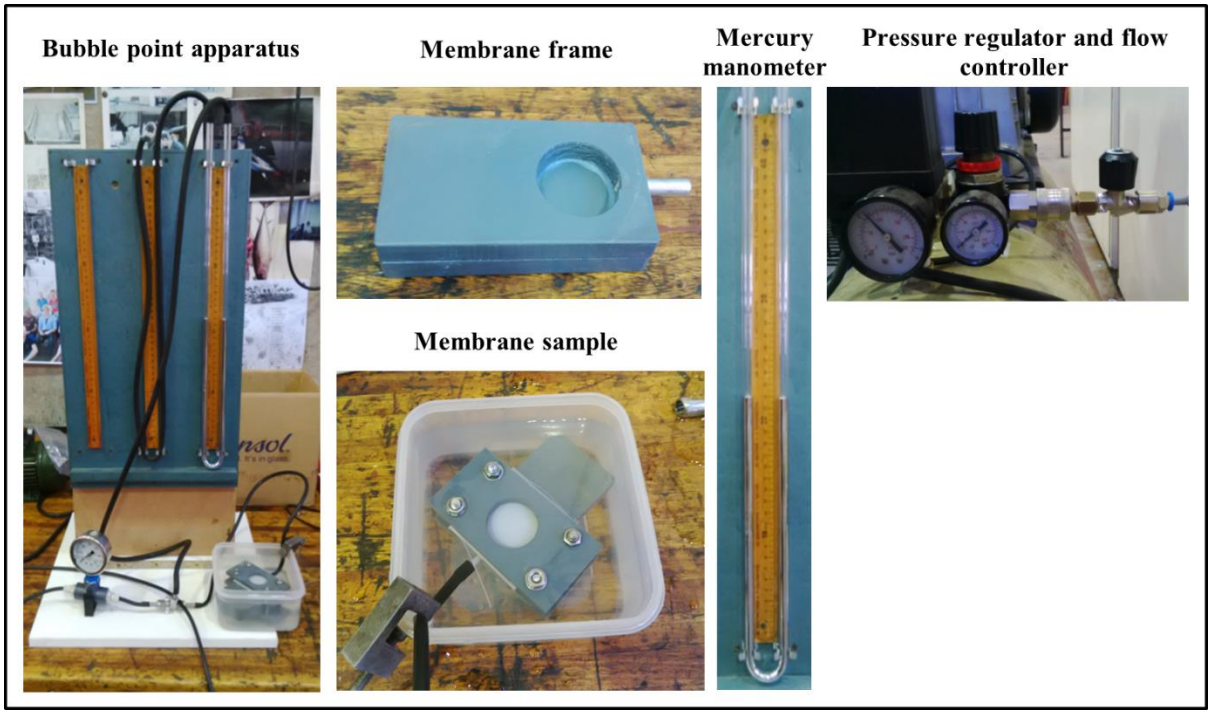

Figure 18: Bubble transport experimentation apparatus.

The mercury manometer had an effective pressure range of 0-0.6 bar (430 mmHg) with an uncertainty of +-0.001 bar (1 mmHg). A secondary pressure gauge was used to indicate any pressure leaks in the apparatus.

The apparatus utilised a woven fabric sample size of 50x60 mm, which had an effective active surface area of $0.8x10^{-3}$ m^2. The sample was secured to the membrane frame using a general purpose hot melt glue. An additional 8 mm PVC securing plate was used to reinforce the membrane sample to the frame. This prevented leaks occurring between the woven fabric sample and the PVC frame as a result of the pressure applied. This was illustrated in Figure 18, 'membrane sample'.

The bubble point setup utilised both a pressure regulator and a flow controller. This was to allow the pressure to be set using the pressure regulator, and the flow controller used to control the rate of increase to the set pressure. This resulted in a relatively low increase in the exerted pressure on the membrane sample over time. This was used to pin point the pressure at which the first bubble stream was witnessed passing through the membrane sample and the point at which the entire membrane sample surface became fluidised. This method was proposed rather than the use of set pressure increments, which would have resulted in over estimation of the applied pressure required, illustrated in Figure 18, 'Pressure regulator and flow control'.

4.1.1.3 Methodology

The tests were performed by positioning the membrane sample in a clear dish, in such a way that there was 20 mm of water above the sample. The sample was allowed to soak for 5-minutes, to allow the membrane pores to be sufficiently wetted before any pressure was applied to the sample. The pressure regulator was set to 0.13 bar, with the flow controller closed. The flow controller was then set to allow a gradual pressure increase. The manometer differential height was recorded at two distinct points. The first point was when the first bubble stream was noted, indicating the largest pore size of the membrane sample. The second point was when the entire membrane surface became fluidised by the bubble stream, indicating fluidisation of the majority of the membrane pores and thus indicating the smallest pore size of the membrane sample.

The results were represented as a membrane pore size range as opposed to an average pore size between the two pore size limits calculated. An average between the largest and smallest determined pore sizes would not be a true representation of the effective pore size of the membrane samples as this would have based the average pore size on the assumption of an equal pore size distribution between the calculated ranges. The non-uniform distribution of the membrane pore sizes is evaluated in Chapter 4.1.2 Membrane morphology.

Validation of the experimental apparatus and procedure was required. This was performed using an ANOVA single factor evaluation. To limit additional variations based on the membrane sample, 8 new membrane samples were utilised where each sample was tested three times, resulting in 24 pore size range calculations to base the ANOVA evaluation on.

The experimentation procedure utilised for the pore size range comparison, utilised 3 new unused membrane samples, 3 samples of the original woven fabric material prior to being reworked, and 3 samples of a fouled membrane sample that had been utilised in a membrane test. Each of the membrane samples were tested three times. The final size range was calculated by taking the average of each of the sample repetitions performed. Each test comprised of 3 individual samples per membrane type, each with 3 repetitions performed per sample. This resulted in 9 calculated pressures per pore size estimate (3x3). These were utilised to calculate the final average pore size estimate value, for both the largest and smallest pore sizes.

An indication of the point at which the pressures were recorded, for the largest and smallest pore size calculations, can be seen in Figure 19:

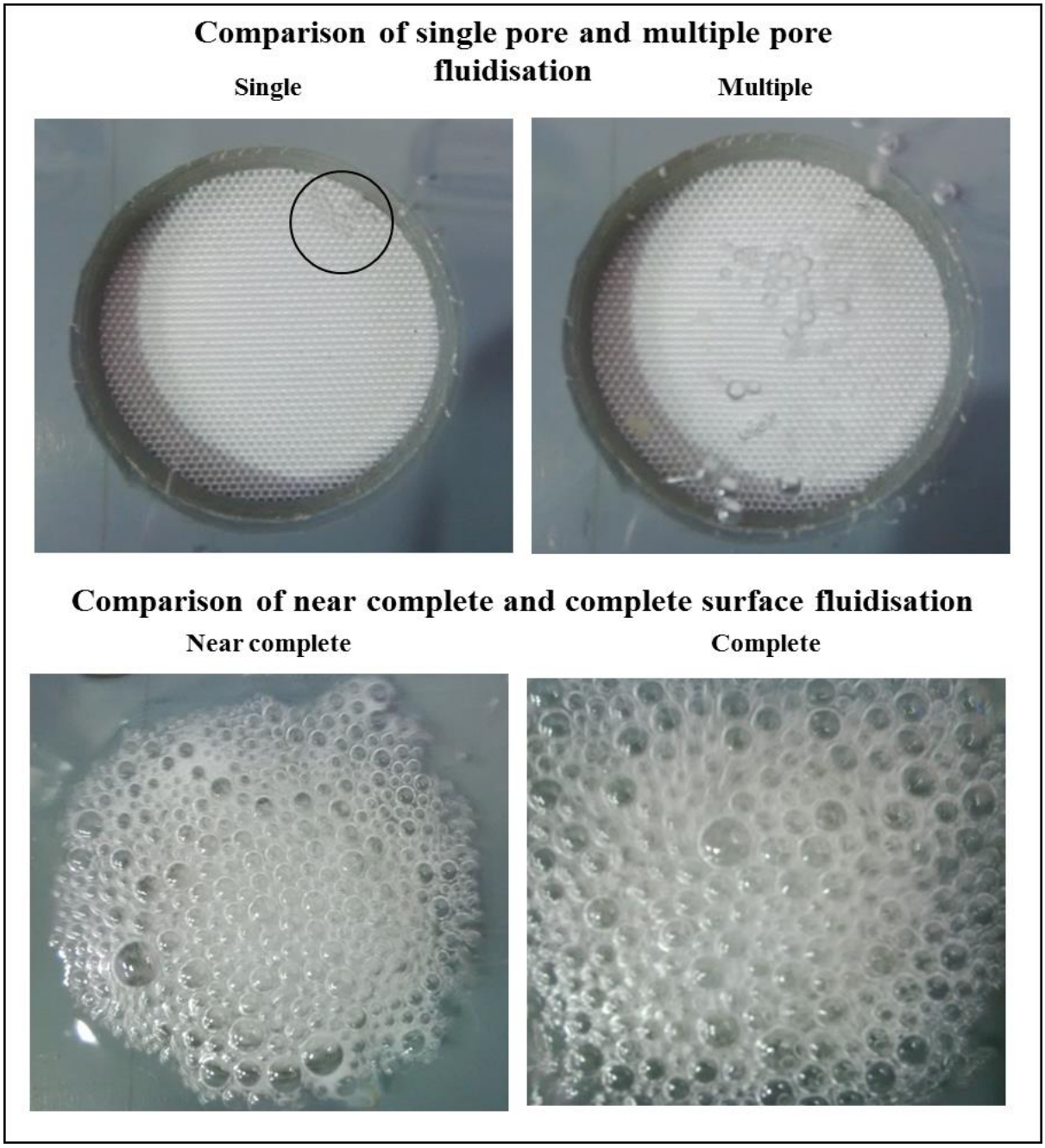

Figure 19: Bubble transport pressure comparison of single pore and multiple pore fluidisation and near complete and complete surface fluidisation.

The figure indicates the difference between single and multiple pore fluidisations and the difference between near complete and complete surface fluidisation.

4.1.1.4 Results and discussion

4.1.1.4.1 Bubble transport experimental procedure validation

The full ANOVA evaluation is indicated in Appendix A: Validation of bubble point experimentation with a summary of the results following below:

The ANOVA single factor evaluation was performed at a 95% confidence level ($\alpha = 0.05$), with the following null and alternative hypotheses:

Null Hypothesis (H_0): $\mu_1 = \mu_2 = \cdots = \mu_n$

Alternative Hypothesis (H_a): $\mu_1 \neq \mu_2 \neq \cdots \neq \mu_n$

The Null Hypothesis was to be rejected based on the criteria of $F_0 > F_{critical}$. The results of the respective F_0, $F_{critical}$ and P-values are illustrated in Table 2:

Table 2: ANOVA single factor analysis of the bubble transport experimentation procedure.

Anova analysis	F_0	P-value	$F_{critical}$
Largest pore size estimate	0.654	0.707	2.657
Smallest pore size estimate	1.127	0.394	2.657

The Null Hypothesis could not be rejected based on the results of the ANOVA evaluation indicated in the table above. This was based on the $F_0 > F_{critical}$ criteria not being met for both pore size estimates. The implication of not being able to reject the Null Hypothesis as stated above, was that the variation of the average pore size between the samples and the sample replications was not significant. Therefore, the procedure followed and the apparatus used to evaluate the membrane pore size range calculated was sufficiently repeatable based on a 95% confidence level. The results of the ANOVA evaluation did not indicate whether the bubble transport method was applicable to the woven fabric material, it only indicated the repeatability of the results based on the apparatus and methodology followed.

4.1.1.4.2 Bubble transport comparisons

The pore size range estimates for the three types of membrane samples are summarised in Table 3:

Table 3: Pore size range estimation.

WF material sample	Estimated size range (µm)	Difference (µm)
Currently available WF material	10 - 24	14
Original WF material	11 - 26	15
Fouled WF material	7 - 18	11

The table indicates the pore size range and the difference between the largest and smallest pore sizes for; the currently available woven fabric material, the original woven fabric material prior to being reworked and fouled samples of the current woven fabric material. The results indicated that the currently available membrane samples had a smaller average pore size range, apparent by the smaller deviation between the larger and smaller pore sizes, than the original material samples. The difference was relatively small, with the current fabric samples having a 1 µm smaller smallest pore size and a 2 µm smaller largest pore than the older membrane sample. This was an indication that the current woven fabric material had been reworked to produce a smaller effective pore range.

The estimated pore size of the unused woven fabric, 10-24 µm, indicates that should the membrane process be based solely on a size exclusion forming between the membrane pores and the feed suspension, the woven fabric would not be applicable for use in water treatment processes. This is due to the expected feed water constituents being in the range of 1-3 µm. A means to overcome this, would be to operate the woven fabric as a dynamic membrane process [7].

The bubble transport experimentation indicated that the fouled samples of the woven fabric material produced a relatively significant, smaller pore size range and smaller effective pore size than the unused samples. The fouled samples had an effective pore size range of 7-18 µm compared to the 10-24 µm of the unused samples. The results of the pore size reduction are

an indication of narrowing of the membrane pores by the effects of irreversible fouling. Literature defines irreversible fouling as a pore narrowing occurring when a precipitate or solid particle becomes lodged within the membrane pores, resulting in a partial or complete pore becoming blocked or restricted for fluid flow [8,9]. The definition for irreversible fouling correlates with the pore narrowing phenomenon indicated by the bubble transport test. The magnitude of the pore narrowing of the fouled samples was not important due to it being dependent on the characteristics of the fouling medium. Additional evaluation is required to conclude if the pore narrowing was indeed a result of irreversible fouling. This will indicate the potential for operating the woven fabric as a dynamic membrane process.

The pore size estimate determined was expected to vary slightly from the actual pore size of the woven fabric material. Literature indicated that the woven fabric had a claimed effective pore size of 1-3 µm [1,7]. This measurement deviates by an approximate factor of 10 from the range estimated using the bubble transport method, 10-26 µm. The reason for the variation between the two pore size estimates was linked to the assumption of perfect cylindrical pores. This assumption resulted in an underestimation of the pore range size using the bubble transport method. However, it must be noted that the previous method utilised in the literature studies, calculated the pore size based on the evaluation of the separation results of a woven fabric membrane using limestone particles of a known size distribution. There are two inherent issues with that method. The first is the limestone particles were not perfectly uniform, therefore potentially resulting in an overestimation of the separation efficiency of the woven fabric material [1,7]. And the second issue arises from literature indicating the effects of fouling on improving the product quality achieved through the woven fabric membrane. Therefore, the use of a fouling feed suspension would have overestimated the membrane pore size based on the formation of the fouling layer during the test.

It must be noted that the bubble transport method indicates a pore size range, and not a pore size distribution. Thus indicating how the largest pore size estimates is an extreme within the actual average membrane pore size.

4.1.1.5 Summary

The bubble transport method tests indicted:

- The currently available woven fabric has been reworked, producing a smaller effective pore size than that of the originally produced woven fabric material.

- This indicated the requirement to re-evaluate the woven fabric membrane performance.
- The estimated pore size range of the new and original fabric were estimated as 10-24 µm and 11-26 µm respectively.
- A size exclusion alone would not be sufficient for the required water treatment processes.
- The effective pore size range of the fouled samples was estimated as 7-18 µm.
- The comparison between the unused and the fouled membrane samples indicated the potential presence of pore narrowing.
- The pore narrowing was considered to be due to irreversible fouling.
- Further evaluation is required to confirm the presence of the irreversible fouling.
- Pore narrowing indicates a potential for dynamic membrane operation.
- The assumption of perfectly cylindrical pores by the bubble point transport method resulted in a underestimation of the pore size.
- The bubble transport method indicated the pore size range and not the pore size distribution.

4.1.2 Membrane morphology

An evaluation of a membrane's morphology focusses on the structure and surface properties of the membrane material. The woven fabric's morphology was evaluated using SEM imaging taken at various magnifications, for a set of woven fabric material samples. The evaluation focussed on the weave pattern utilised for the fabric and the pore designations, if apparent. The woven fabric samples selected were a combination of new unused membrane samples and fouled membrane samples. The fouled samples were selected as to further investigate the effects of pore narrowing.

The surface chemistry of the fabric was not investigated as a result of the woven fabric threads being made from unenhanced polyester fibres. The SEM images were taken at progressively greater magnifications, namely: 79X, 109X, 1000X and 3500X magnifications. This allowed for evaluation of the required aspects of the woven fabric.

4.1.2.1 Results and discussion

The membrane morphology was investigated using SEM imaging, as presented in Figure 20:

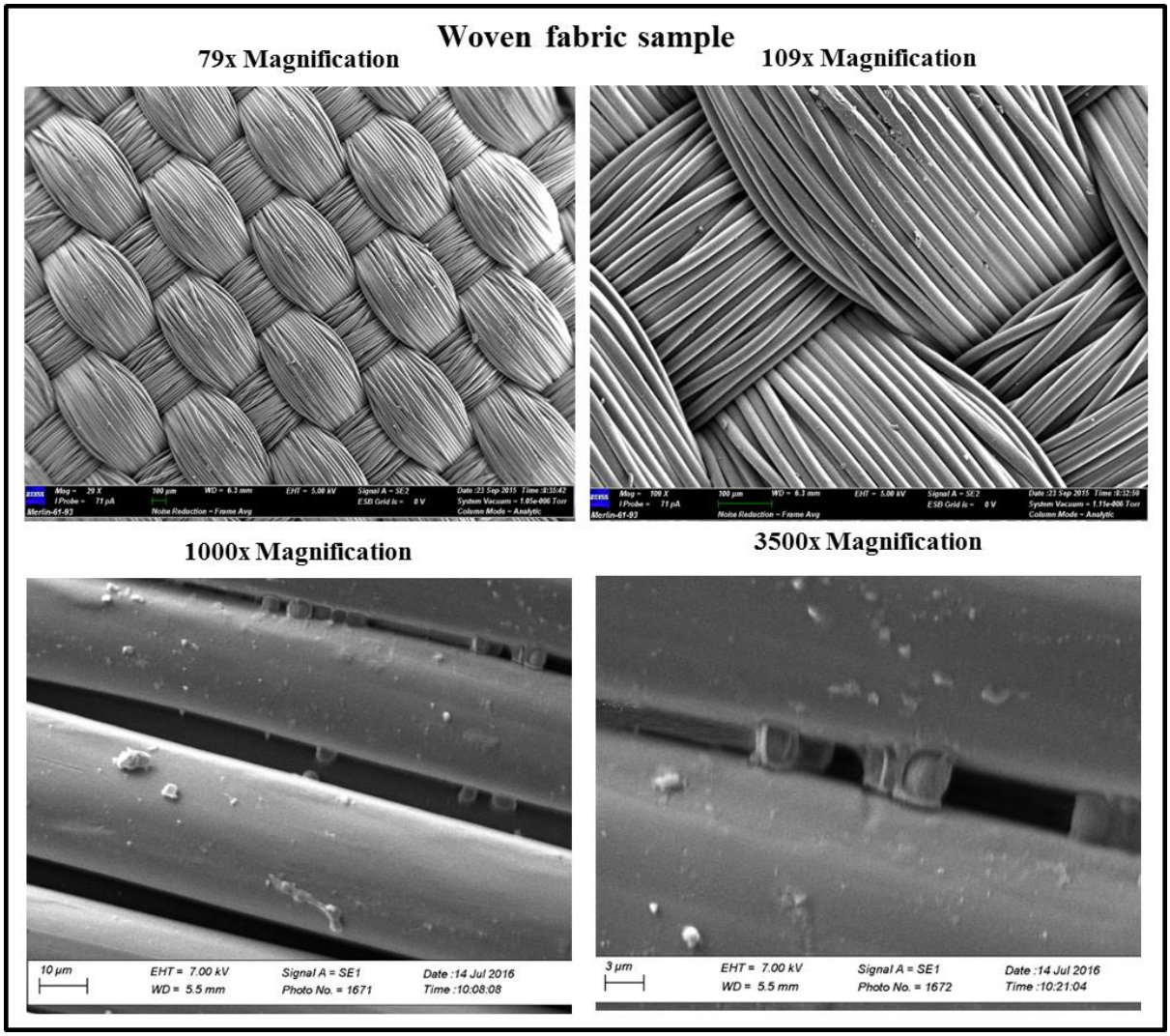

Figure 20: Woven fabric SEM image evaluation of both clean and fouled membrane samples, ZEISS EVO MA15VP, CAF Stellenbosch University.

The figure indicates four SEM images of woven fabric samples at different magnifications. From the images it is apparent how the woven fabric utilised a Plain Weave structure, as indicated by the alternating weft over and under the warp thread. Evaluating the 79X magnification image only, one would conclude that the membrane pore designations were a result of spaces forming between the intersections of the weft and warp layers only. However, on further investigation of the 109X magnification image, it was apparent how the pore designation was not as clear cut as initially believed. The pores were illustrated as being formed due to the overlapping of the various strands within the weft and warp threads. These overlapping strands formed passages for fluid to pass through. The 1000 and 3500X magnification images validated the pore designation as initially indicated by the 109X magnification image.

The pore designations indicated by the 1000 and 3500X magnification images indicated passages that pass between the strands within the weft and warp threads, and not directly through the material. The woven fabric comprises two perpendicular weft and warp thread layers, both comprising of multiple strands. This therefore results in the formation of a complex multilayer membrane material with resultant highly tortuous membrane pores formed.

This discovery was important, as this double layer effect of the overlapping weft and warp threads, and the weft and warp threads being made up of multiple strands, are the reason for the woven fabric's membrane properties. The overlapping threads indicated a potential difficulty when defouling the woven fabric utilising standardised flux enhancement and cleaning methods. This is the result of the potential for the particulate matter being entrained within these numerous layers of the fabric due to the tortuous nature of the membrane pores. Additionally, the double layer effect has validated why SEM imaging was not an applicable method for pore size estimation. This is based on the tortuous pores that formed hindering any form of visual inspection.

On closer inspection of the 79X magnification image, it can be seen how the 'overlapping nature' of the strands within the warp and weft threads are random, and follow no defined pattern. This random overlapping of the strands within the threads create the relatively large pore size range of the woven fabric material, as initially indicated in the bubble point evaluation performed, as described in Chapter 4.1.1 Membrane pore size.

The 1000 and 3500X magnification images were taken of the fouled woven fabric samples. The 3500X image clearly indicates the effects of pore narrowing as a result of entrained particulate matter between adjacent threads. This pore narrowing has a significant impact on increasing the hindrance of fluid flow through the membrane pores. This was initially indicated by the effective pore size reduction of the fouled membrane samples indicated in Chapter 4.1.1 Membrane pore size. This form of fouling is known as irreversible fouling, as the foulant matter is entrained within the membrane's pores, as opposed to forming on the membrane surface only [8,9]. This indicates how the bubble transport method is effective for indicating deviations between the membrane samples. The effects of pore narrowing, due to fouling, resulting in an improved separation ability of the woven fabric correlates with literature [1,2,7].

4.1.2.2 Summary

The membrane morphology was investigated by evaluating SEM imaging at 79X, 109X, 1000X and 3500X magnification of the woven fabric material. This indicated that:

- The woven fabric had a Plain Weave structure.
- The 109X magnification SEM image indicated the membrane pores forming as a result of overlapping strands within the warp and weft threads.
- The 'overlapping nature' of the strands was random as no defined pattern was noted when inspecting the 79X magnification SEM image.
- The overlapping strands and perpendicular weft and warp layers resulted in a complex multilayer membrane material.
- Pore narrowing was indicated in the fouled 1000X and 3500X magnification SEM images by the presence of residual fouling within the membrane pores.
- The bubble transport method was concluded to be capable of indicating deviations between the membrane samples.

4.1.3 Conclusions

Effective membrane pore size estimation:

It was concluded from the bubble transport method tests that the currently available woven fabric had been reworked resulting in a smaller effective pore size. This was indicated by the effective pore sizes of the current and the original fabric samples being 10-24 µm and 11-26 µm respectively. The pore size range estimate classified the woven fabric as a microfilter membrane.

The comparison between the unused and the fouled membrane samples indicated the presence of irreversible fouling material reducing the effective pore size range of the fouled membrane samples. The effective pore size range of the fouled samples was estimated as 7-18 µm, in comparison to the 10-24 µm of the unused membrane samples. However, it could not be concluded if this pore size reduction was due to irreversible fouling without additional investigation.

Membrane morphology:

From the membrane morphology it was concluded that the woven fabric material had a Plain Weave structure. The 109X magnification SEM image indicated the presence of the

membrane pores being the result of overlapping strands within the warp and weft treads. The 'overlapping nature' of the strands was random as no defined pattern was noted in the 79X magnification SEM image. It was concluded that this gave the woven fabric the relatively larger pore size range.

Pore narrowing was concluded to be present by the fouled 1000X and 3500X magnification SEM images. This validated the bubble transport method for indicating the presence of pore narrowing as a result of irreversible fouling matter within the membrane pores. The pore narrowing concluded how the formation of a fouling layer resulted in a pore size reduction.

An important discovery of the characterisation procedure was the complex multilayer property of the woven fabric membrane material. The complex layering nature of the fabric was as a result of the multiple strands utilised to produce the weft and warp threads, as well as the perpendicular overlapping of these weft and warp threads. These layers resulted in highly tortuous membrane pores being formed.

4.2 Overall conclusion of the membrane characterisation evaluation

The characterisation procedure led to the woven fabric membrane being classified as a microfilter membrane. The characterisation evaluation made it possible to conclude how the effects of fouling were able to result in a pore size reduction of the fabric. This indicated how the formation of fouling layer could improve the separation ability of the woven fabric membrane. From the characterisation procedure it was apparent how the woven fabric would require operation as a dynamic membrane process to successfully meet the requirements of a water treatment process.

The membrane morphology indicated that the woven nature of the woven fabric membrane resulted in a complex multilayer membrane material being formed. It was concluded that this multi-layering effect of the fabric promoted the use of the woven fabric for dynamic operation as a result of the highly tortuous membrane pores formed.

4.3 References

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Chapter 5: Performance evaluation

Overview

This chapter is divided into four sections, namely:5.1 Product quality evaluation, 5.2 Evaluation of fouling characteristics, 5.3 Overall conclusion of the performance evaluation, 5.4 References. This chapter covers the performance evaluation of various operating parameters and feed suspensions physical and chemical properties on the woven membrane product quality and fouling characteristics.

5.1 Product quality evaluation

The product quality evaluation is subdivided into two separate investigations, namely the effects of permeate flux and solid size distribution on permeate quality and the effects of feed solids concentration on permeate quality. Prior to these investigations being performed an evaluation of the synthetic feed suspensions utilised is covered.

For the woven fabric to meet the requirements of a membrane suited to water treatment applications, the membrane has to achieve a product quality of 1 NTU or lower for feed suspensions containing particles as small as 1-3 µm. This has been stipulated by the SANS 241 guidelines for potable water production. It is therefore important to determine the limitations of the woven fabric to produce the required product quality.

5.1.1 The development of synthetic feed suspensions

The product quality evaluation requires the use of a repeatable feed suspension that will adequately indicate the effects of varying feed suspension properties, such as concentration and particle size range. For this, a synthetic feed suspension was selected to allow the feed suspension properties to be manipulated as desired.

Limestone particles were selected as the feed suspension constituent for this phase of the product quality evaluation. Limestone was selected due to its low surface force interaction properties, resulting in testing that would indicate the physical separation aspects of the woven fabric. The limestone feed suspensions utilised had the following particle size D_{50}s, namely 5, 2 and 1 µm. This allowed adequate evaluation of the effects of varying particle size distributions on the woven fabric product quality. The particle size distributions of the limestone distributions utilised, supplied by the manufacture, can be seen in Figure 21:

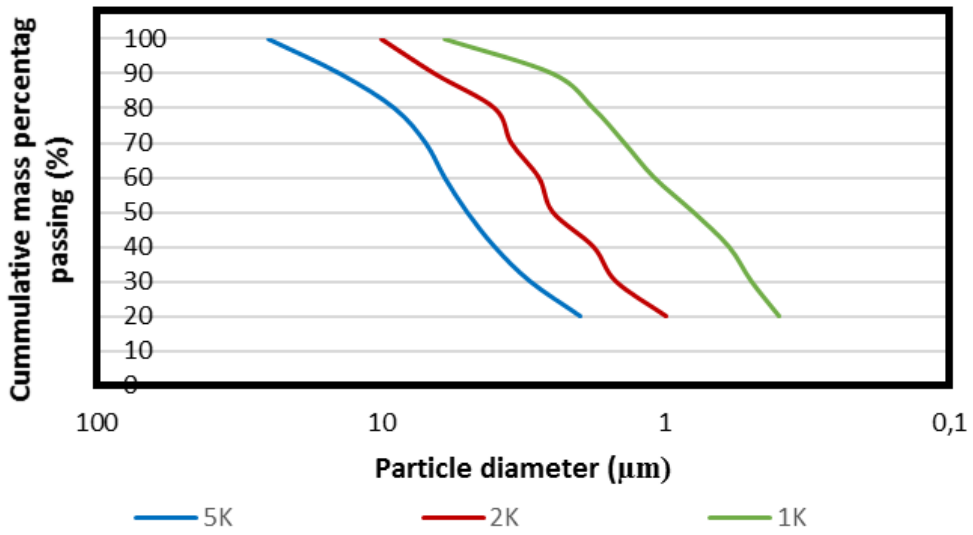

Figure 21: Limestone particle size distributions utilised, 1K, 2K, 5K.

The limestone particle size distributions are referred to as 5K, 2K and 1K, which correspond to the particle size distribution D_{50}s of 5, 2 and 1 µm respectively. The figure indicates how the 5K, 2K and 1K size distributions have varying portions of particles larger and smaller than the effective pore size range of the woven fabric, as is determined in Chapter 4.1.1 Membrane pore size estimation. The 2K and 1K particle size distributions are predominantly below the estimated pore size. This was desired to determine the limitations of the membrane in removing sub-pore sized particulate matter.

A means for measuring the permeate solids concentration was required due to this phase of the performance evaluation focussing on the product quality achieved for varying feed suspension physical properties. Turbidity was selected as this measure, due to the relatively low solids concentration expected in the permeate stream for the different tests. Alternatively, evaporation of the permeate samples, followed by SEM size distribution evaluation of the remaining solids, could have been utilised. This method was however not selected due to the intense resource requirements of such a process.

5.1.2 The effects of permeate flux magnitude and solid size distribution on permeate quality

5.1.2.1 Investigation

This investigation was performed to determine the effects the permeate flux magnitude and feed suspension particle size distribution had on the product quality achieved. The operating flux magnitudes selected were 50, 100 and 200 LMH, in combination with the three

limestone D_{50} particle size distributions (5K, 2K and 1K). The concentration of the feed was monitored and maintained at 200 NTU for the duration of each of the tests. An aeration stone was used to maintain the limestone particles in suspension, due to its relatively high settling rate. The TMP, permeate flux and permeate stream turbidity were recorded for the duration of the 2-hour tests for comparison between the tests performed.

5.1.2.2 Apparatus

The apparatus utilised for this phase of the performance evaluation can be seen in Figure 22, with the various components that make up the membrane process discussed below the figure:

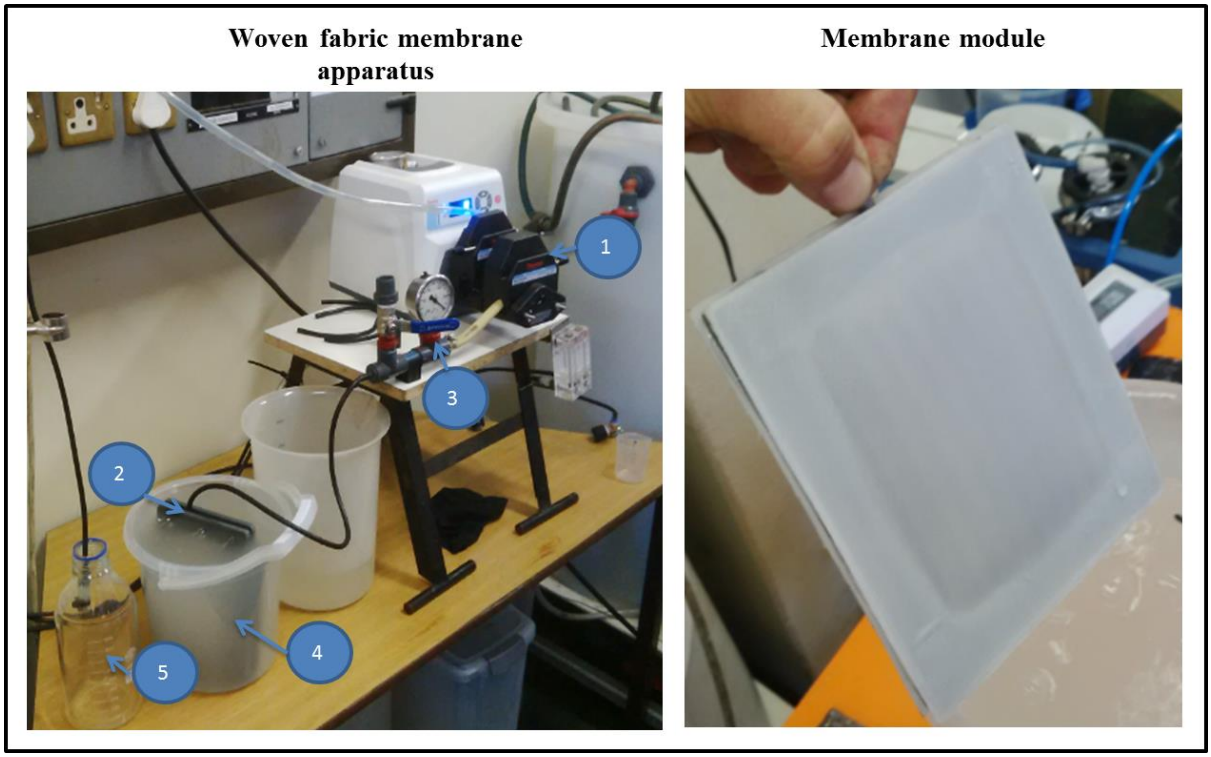

Figure 22: Woven fabric flat sheet membrane apparatus for product quality evaluation.

1: The permeate driving force was provided by a MasterFlex 6-600rpm peristaltic pump.

2: The membrane modules utilised had dimensions of 180x130x8 mm.

3: The TMP was monitored using a non-stepped vacuum gauge with a pressure range of -100-0 kPa.

4: A 5 L holding vessel was utilised.

5: The permeate was collected in a separate vessel and not recycled back to the membrane.

5.1.2.3 Methodology

The product quality evaluation procedure consisted of two parts, namely: an initiation procedure and an operation procedure.

Step 1: Initiation procedure:

1) The membrane module was tested for leaks prior to being utilised.
2) The holding vessel was filled with RO water.
3) The membrane module was placed in the holding vessel and left for 10-minutes to allow the pressure within the membrane module to equalise.
4) The pump speed was set to the desired flow and left to stabilise for 10-minutes before the first permeate flux was recorded.
5) If the pure water flux, RO water, recorded was within ±2 LMH of the desired permeate flux, the pump was turned off and the holding vessel drained. If it was lower or greater than ±2 LMH of the desired flux, the pump speed was altered, either increased or decreased, and the flow left to stabilise as before, before the new permeate flux was recorded. This was repeated until the desired permeate flow was achieved.

Step 2: Operation procedure:

1) The holding vessel was filled with the desired feed suspension.
2) The membrane module was placed in the holding vessel and left for 10-minutes to allow the pressure within the membrane module to equalise.
3) The pump flow was not altered.
4) The permeate flux, TMP, feed and permeate turbidity were recorded after 5-minutes of operation and thereafter in 5-minute intervals.
5) The tests were performed for the desired duration or until a desired TMP, permeate flux or permeate turbidity was achieved.
6) The feed suspension composition was monitored and approximately maintained.

5.1.2.4 Results and discussion

This phase of the woven fabric flat sheet membrane performance testing was used to investigate the effects the feed suspension particle size distribution and permeate flux magnitude had on the permeate product quality achieved. The results of the change in the

product turbidity as a function of the operation time, for the tests performed, can be seen in Figure 23:

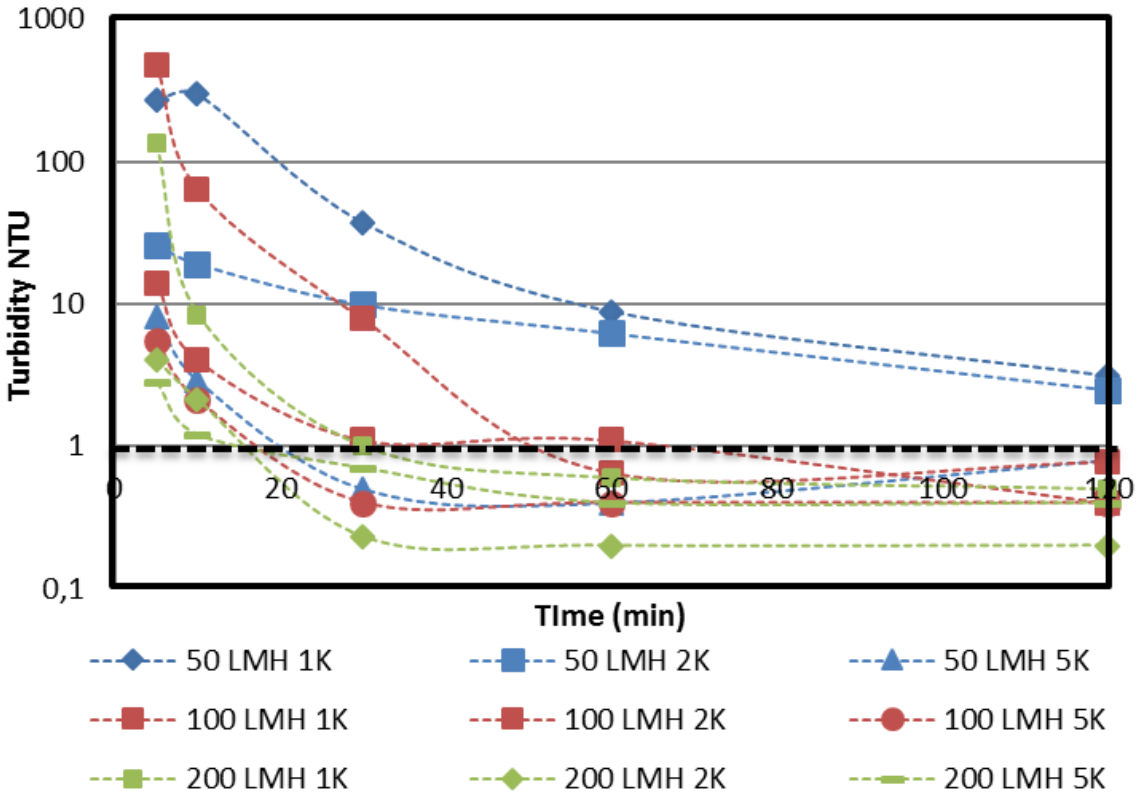

Figure 23: The effects of permeate flux and feed solid particle size distribution on permeate product quality.

The blue markers indicate the tests performed at 50 LMH, for all three particles size distributions, the red markers indicate the tests performed at 100 LMH, for all three particle size distributions and the green markers indicate the tests performed at 200 LMH, for all three particle size distributions. From the figure it was apparent how the permeate turbidity decreased as a function of the operation time for all three particle size distributions and permeate flux magnitudes tested. The rate of decrease in the product quality varied between the particle size distributions and permeate flux magnitude tests.

The figure indicates that of the 5K limestone feed suspensions tested, all achieved a permeate product quality of 1 NTU and lower within the first 15-minutes of operation, for the three fluxes tested (50, 100 and 200LMH). The initial, larger than 1 NTU turbidity, indicated that a portion of the limestone particles within the suspension were small enough to initially pass through the membrane pores. However, after a period of operation the majority of these particles were being entrained by the woven fabric, as indicated by the decrease in the product turbidity to below 1 NTU. This was the result of the portion of the 5K particle size

distribution that was larger than the membrane pore size, forming a fouling layer that was able to reduce the effective membrane pore size. The pore narrowing increased the separation ability of the fabric, as the sub-pore size particles were no longer able to pass through the membrane pores. This correlates with literature, where the particles larger than the membrane pore size result in the formation of a fouling layer that is capable of entraining the sub-pore sized particles [1,2]. Additionally, the effects of fouling resulting in a pore size reduction, specifically with regards to the woven fabric pores, was concluded in Chapter 4.1 Membrane characterisation procedure. Therefore, it could be deduced that the claimed 1-3 µm woven fabric pore size from literature is not a valid claim based on the formation of a fouling layer being required to separate the 5K particle size distribution. This is due to the entire 5K limestone particle size distribution being larger than 1-3 µm, indicating that the formation of the fouling layer should not have been required to achieve the lower than 1 NTU product quality.

The effects of the permeate flux magnitude on product quality were more prevalent for the 2K and 1K feed suspension tests. From the figure it was apparent how the 1K feed suspension, operated at a permeate flux of 50 LMH, was unable to achieve a product quality of lower than 1 NTU within the 120-minute operation period. However, during the 100 LMH and 200 LMH tests it was possible to achieve a product quality of less than 1 NTU after a period of approximately 50-minutes and 25-minutes respectively. The same trend was witnessed for the 2K feed suspensions, where at 50 LMH the membrane was unable to achieve a product quality of lower than 1 NTU within the 120-minute operation period, whereas during the 100 LMH and 200 LMH tests, the permeate turbidity achieved was below 1 NTU within 30 and 15-minutes respectively.

These tests indicated how the rate of achieving a product quality of lower than 1 NTU was affected by both the particle size distribution and the permeate flux magnitude. The greater the permeate flux magnitude, the higher the rate of decrease in permeate turbidity was. And the larger the particle size distribution, the higher the rate of decrease in permeate turbidity was. These two phenomena correlate with literature on the separation effects of a MF and UF membrane [2,3]. However, what did not correlate, was the fact that the entire 2K and 1K particle size distributions were below the pore size range estimated in Chapter 4.1.1 Membrane pore size estimation. The woven fabric membrane was therefore indicated as being able to effectively remove sub-pore size particulate matter.

It was expected that the method utilised to estimate the pore size range of the membrane was not perfectly suited to the woven fabric, due to the method used being based on perfectly cylindrical membrane pores. However, this was not the sole reason for the unique separation ability of the woven fabric, as indicated in the SEM image evaluation. The separation ability of the woven fabric was due to the complex multilayer membrane material that formed during the weaving process. The multilayer membrane material resulted in the formation of relatively tortuous membrane pores. It was the tortuous nature of the membrane pores that promoted the deposition of the sub-pore size particulate matter within the membrane pores. The deposition of these particles, over-time, resulted in the formation of a porous fouling layer, that was able to effectively entrain the remaining sub-pore sized limestone particles. The permeability of the fouling layer arose from the overlapping of the limestone particles not completely blocking flow. The variation in the time taken to achieve a product quality of 1 NTU between the 2K and 1K tests indicated how the smaller the relative particle size distribution, the higher the quantity of particles requiring deposition within the membrane pores to achieve the required separation efficiency.

The effects the permeate flux magnitude had on the formation of the fouling layer were not apparent from the figure above. It was not clear if the rate of formation of a fouling layer was as a result of the increased force of entrainment on the particulate matter within the pores or as a result of the increased mass transfer rate occurring at the relatively higher permeate flux magnitudes. To deduce the mechanism for the increased rate, the permeate turbidity was plotted as a function of the cumulative permeate volume collected for the different flux magnitudes tested. The results of these tests can be seen in Figure 24:

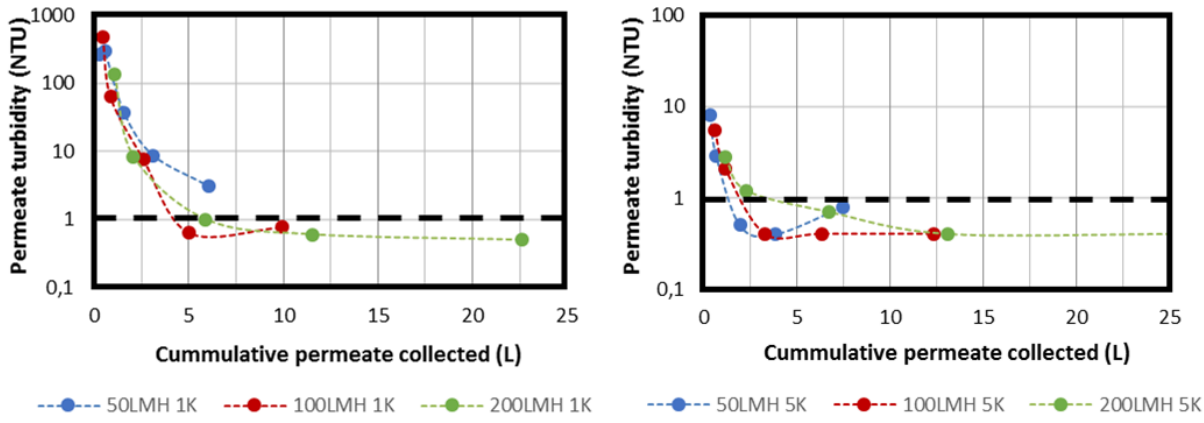

Figure 24:The effects of permeate flux and feed solid particle size distribution on permeate product quality, cumulative permeate volume evaluation.

The figure indicates how the permeate turbidity decreased with an increase in the cumulative permeate volume collected for both the 1K and 5K series of tests. From the figures it was apparent that both the 1K and 5K tests, achieved a product turbidity of less than 1 NTU after approximately similar respective amounts of cumulative permeate volume were collected for the permeate flux magnitudes tested.

For the 1K test, both the 100 and 200 LMH tests achieved a product turbidity of less than 1 NTU, after approximately 6 L of permeate was collected. The 50 LMH test can be seen to tend towards 1 NTU, however the volume of fluid was insufficient to achieve the desired product turbidity for the test duration. The same trend was exhibited by the 5K test, where the 50, 100 and 200 LMH tests permeate turbidity tended to be less than 1 NTU after approximately 2.5 L of permeate was collected.

The trend for both particle size distribution tests tending towards a permeate turbidity of less than 1 NTU, after a certain quantity of permeate was collected, indicates how the product quality was not affected by the force entraining the particles within the membrane pores. The product quality was rather affected by the quantity of suspended particulate matter that passed through the membrane pores. The importance of this phenomenon was that it indicated that the performance of a 50 LMH and 200 LMH test, for instance, would result in similar membrane product quality being achieved should the cumulative permeate volume collected be similar. Additionally, the test indicated how the larger particle size distribution feed suspensions required relatively less cumulative permeate volume to pass through the membrane pores as opposed to the smaller size distribution. This indicated how the rate of formation of the fouling layer was proportional to the feed suspension particle size.

5.1.2.5 Summary

This phase of the product quality evaluation investigated the effects of the permeate flux (50, 100 and 200 LMH) and feed solids size distribution (1K, 2K and 5K) on the permeate quality. The investigation indicated:

- The membrane pore size was concluded to be larger than what was claimed in literature, namely 1-3 µm.
- A relatively higher permeate flux resulted in a lower time period required to achieve a product quality of lower than 1 NTU, for all three particle size distributions tested.

- The large distribution required incrementally less operation period than the smaller particle size distributions, to achieve a particular permeate turbidity.
- The woven fabric was able to achieve a product quality of lower than 1 NTU for the 1K feed suspension, even though the entire particle size distribution was smaller than the estimated pore size range.
- This was a relatively important discovery, as it indicated how the woven fabric was able to remove particulate matter that was smaller than the effective pore size range of the material.
- The woven fabric effectively removed particles in the size range required by water treatment processes.

5.1.3 The effects of feed solids concentration on permeate quality

5.1.3.1 Investigation

The effects the feed solid concentrations had on the product quality achieved using the woven fabric was of importance. This was as a result of the woven fabric being indicated as capable of achieving adequate product quality when utilising feed suspensions that contained particulate matter smaller than the woven fabric's effective pore size. The required product quality relied on the entrainment of particulate matter within the membrane pores, to result in a pore narrowing effect. It was therefore important to investigate how the feed suspension concentrations would affect this pore narrowing effect.

The tests consisted of testing three D_{50} Limestone particle size distributions (5K, 2K, and 1K), at a feed turbidity of 10, 50, 100, 200, 500 and >1000 NTU. The purpose of these tests was to determine whether an increase in the solid concentrations would have an effect on the product quality achieved and whether the same trend would be observed for all three limestone particle size distributions. A single operating flux of 100 LMH was selected for the tests. The concentration of the feed was monitored and maintained at the desired initial feed turbidity. An aeration stone was used to maintain the limestone particles in suspension for the duration of the investigations, due to its relatively high settling rate. The TMP, permeate flux and permeate stream turbidity were recorded for the duration of the tests, in order to compare the variation of each over the 40-minute operation period.

5.1.3.2 Apparatus

The apparatus utilised for this phase of the performance evaluation was the same as the apparatus designed for use in Chapter 5.1.2 The effects of permeate flux magnitude and solid size distribution on permeate quality.

5.1.3.3 Methodology

The methodology followed for this phase of the performance evaluation was the same as the methodology outlined in Chapter 5.1.2 The effects of permeate flux magnitude and solid size distribution on permeate quality.

5.1.3.4 Results and discussion

The results of the varying solid feed suspension concentrations tests, using the 5K particle feed suspension can be seen in Figure 25:

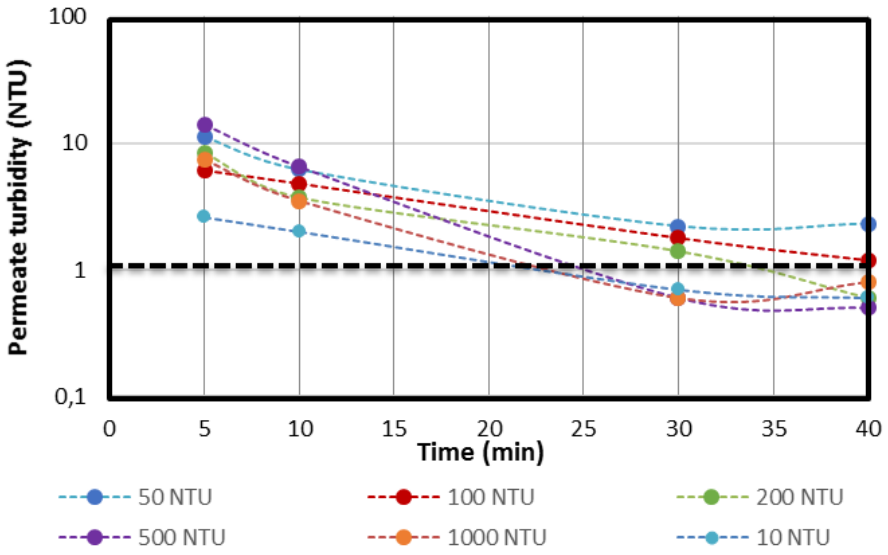

Figure 25: Permeate turbidity evaluation for varying feed solids concentration, 100 LMH 40-minutes, 5K.

The figure indicates that a decrease in the permeate turbidity was achieved over the 40-minute operation period, for each of the 5K particle distribution concentrations tested. The rate of decrease varied between concentrations tested. The general trend followed was that an increase in the solid concentrations increased the rate of decrease in permeate turbidity over the time period tested. The 50 NTU test was unable to achieve a permeate turbidity of 1 NTU over the 40-minute operation period, whereas the 10, 100, 200, 500 and 1000 NTU tests resulted in permeate turbidity of lower than 1 NTU within the 40-minute operation period tested. The rate of decrease in permeate turbidity can be seen to decrease in the following sequence: 10, 1000, 500, 200, 100 and 50 NTU. The 10 NTU test was an outlier from this

trend. The results correlate with literature, where an increased solid concentration had a positive effect on the product quality achieved through a MF and UF membrane, due to the increased rate of formation of a fouling layer [2,3]. The positive effect here, is with regard to the product quality only as these tests did not indicate the effects the increased concentration had on the permeate flux decline or TMP increase.

The results of the varying solid concentrations test for the 2K particle feed suspension can be seen in Figure 26:

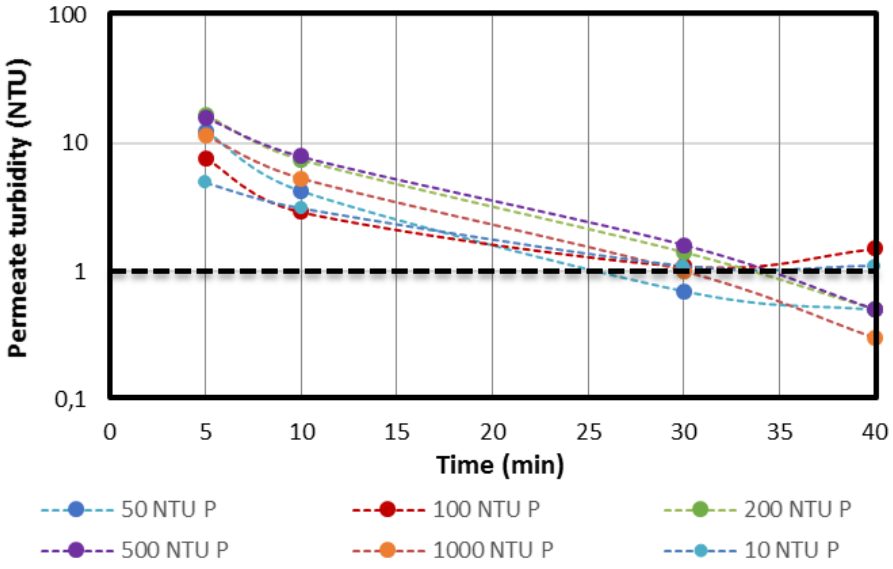

Figure 26: Permeate turbidity evaluation for varying feed solids concentration, 100 LMH, 40-minutes, 2K.

The figure indicates that a decrease in the permeate turbidity was achieved over the 40-minute operation period, for each of the concentrations tested. The rate of decrease varied between the concentrations tested. The general trend followed was that an increase in solid concentrations increased the rate of decrease of the permeate turbidity over the 40-minute operation period tested. The 10 NTU test was an outlier from this trend. The 10 NTU test turbidity can be seen to plateau with a lower rate of decrease in turbidity than in the other tests.

The results of the varying solid concentrations test for the 1K particle feed suspension can be seen in Figure 27:

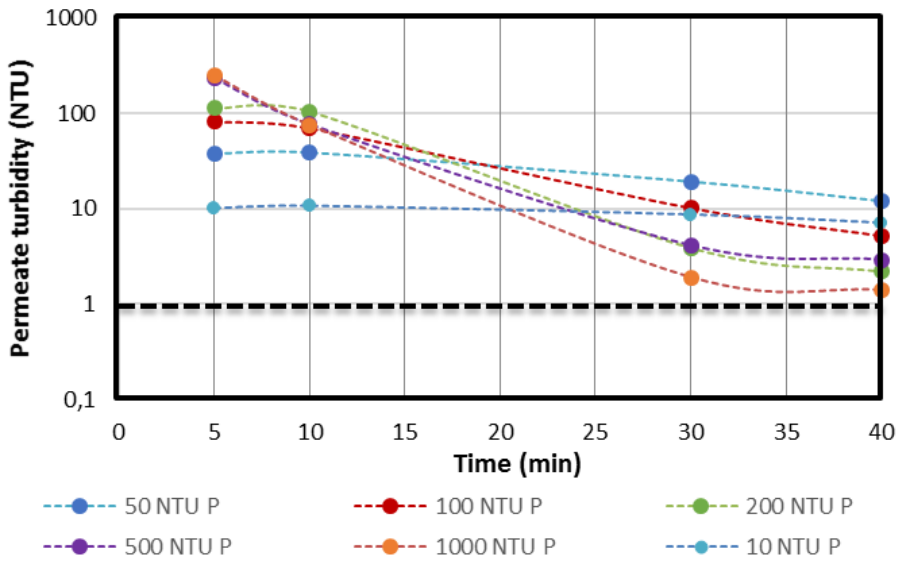

Figure 27:Permeate turbidity evaluation for varying feed solids concentration, 100 LMH, 40-minutes, 1K.

The figure indicates that a decrease in the permeate turbidity was achieved over the 40-minute operation period, for each of the concentrations tested. The results of the 1K test at the varying solid concentrations indicate the effects of the solid concentrations on permeate quality more clearly than the 5K and 2K tests. This is as a result of the entire 1K particle distribution falling below the estimated effective pore size of the woven fabric membrane. Therefore, to entrain these particles, a sufficient fouling layer is required for an effective separation to take place as a result of pore narrowing. The permeate turbidity of the tests after the 40-minute operation period, in decreasing order, was as follows: 50, 10, 100, 500, 200 and 1000 NTU respectively. The rate of decrease in the permeate turbidity increased following a similar trend as the final permeate turbidity decreased, except in the 10 NTU test. The 10 NTU test can be seen to show only a minor decrease in permeate turbidity over the 40-minute period in comparison to the other tests. This is a result of the relatively low solid concentrations not being sufficient to form an effective fouling layer. The 1K tests indicated the necessity for fouling layer formation in order to remove the sub-pore sized particles, and how the feed suspension concentration has an effect on the rate of decrease in the permeate turbidity.

To better understand the reason for the 10 NTU feed suspension results varying for all three limestone suspensions tested, the three 10 NTU feed suspension turbidity tests, were plotted on the same axis as a function of operation period, as illustrated in Figure 28:

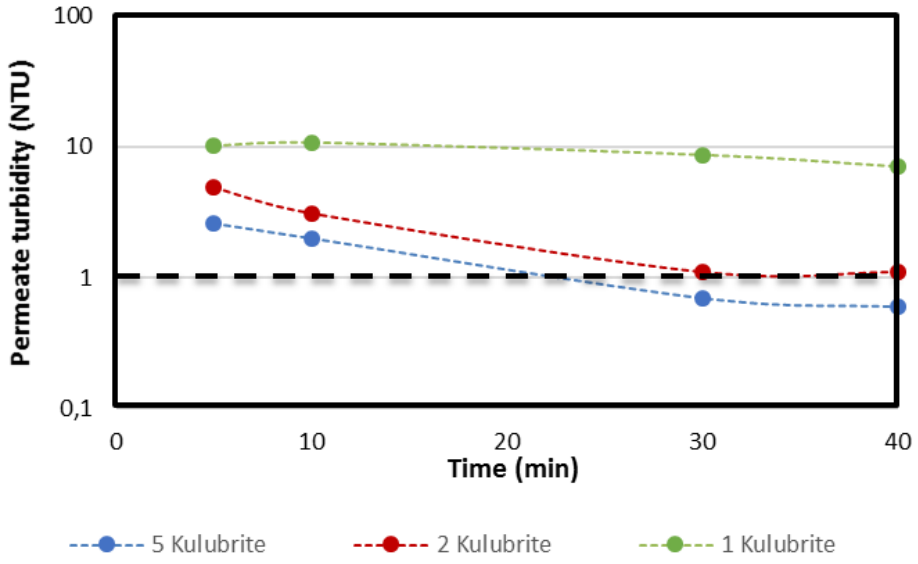

Figure 28: Permeate turbidity evaluation for 5K, 2K and 1K feed suspension concentration of 10 NTU, 100 LMH, 40-minutes.

The figure indicates that a decrease in the permeate turbidity was achieved over the 40-minute operation period, in each of the 5K, 2K and 1K 10 NTU concentration tests. From the figure it was apparent how the larger feed suspension particle size distributions resulted in lower permeate turbidity over the 40-minute operation period. The smaller the feed suspension particle sizes, the more important the formation of a fouling layer became as was indicated in the investigation above. However, at the relatively low solids concentration required to produce a feed suspension of 10 NTU, the number of particles available to form the fouling layer were limited.

The 5K feed suspension had a relatively large portion of particles that were greater than those of the woven fabric membrane pore size range. Therefore, the separation of the 5K suspension was formed based on a size exclusion of the larger particles through the membrane pores as well as the formation of a fouling layer capable of removing the smaller particle sizes. However, for the 2K and 1K particle distributions, there were limited particles in the distribution larger than those of the woven fabric pore size. Therefore, the separation ability of the membrane relied heavily on the formation of the fouling layer. The 2K particle distribution was able to achieve a product quality of lower than 1 NTU, but required additional operation time to compensate for the smaller particle sizes. Yet for the 1K distribution the particle concentration was too low at 10 NTU, for the degree to which the 1K size distribution was sub-pore sized. Therefore, the fouling layer formed was not sufficient to achieve a product quality of lower than 1 NTU.

This is important to note, as it validates that the 1K particle size distribution was smaller than the effective pore size of the membrane due to the formation of a fouling layer being required for a separation to occur. Additionally, the woven fabric has a minimum threshold feed suspension concentration, below which the membrane would not be able to remove sub-pore sized suspended matter. This indicated the necessity for membrane pre-coating when utilising low concentration sub-pore size feed suspensions with the woven fabric membranes [18].

5.1.3.5 Summary

In this phase of the laboratory scale characterisation procedure the effects of the feed solid concentrations (10, 50, 100, 200, 500, >1000 NTU), of the feed suspensions tested, on the product quality achieved were investigated. The investigation indicated that:

- The relatively higher feed suspension concentrations, for all three limestone particle size distributions tested, resulted in relatively higher rates of decrease in the permeate turbidity to below the required 1 NTU.
- The relatively higher solid concentrations increased the rate at which the fouling layer formed on the membranes surface.
- The 1K particle size distribution was below the pore size range of the woven fabric membrane.
- The woven fabric membrane had a minimum threshold particle concentration, below which the membrane was unable to remove sub-pore sized particulate matter.

5.1.4 Conclusions

The effects of permeate flux magnitude and solid size distribution on permeate quality:

In this phase of the performance evaluation investigated the effects of varying the permeate flux magnitude and feed solids size distribution on the permeate quality achieved. The investigation indicated how the product quality achieved through the membrane increased with an increase in the operation period, for all three limestone particle size distributions and permeate flux magnitudes tested. The varying rate of increase was concluded to be a function of both the feed suspension particle size distribution and the permeate flux magnitude. Where the relatively larger particle size distribution was concluded to result in a higher rate of decrease in the permeate turbidity.

The woven fabric was able to achieve a product quality of lower than 1 NTU for the 1K feed suspension, where the particle size distribution was smaller than the pore size range estimate

of the fabric. This was a relatively important discovery, as it showed how the woven fabric was able to remove particulate matter that was smaller than the effective pore size range of the material. This phenomenon was concluded to be as a result of the woven fabric pores being highly tortuous, due to the complex multi-layering of the woven fabric material. The tortuous nature of the pores promoted the entrainment of the sub-pore size particles within these pores. The effects of pore narrowing being as result of the deposition of the particulate matter within the woven fabric pores was validated using SEM imaging. The pore narrowing effect was concluded to increase the effectiveness of the membrane's physical separation barrier to removing sub-pore sized particulate matter. This indicated how the separation efficiency of the woven fabric was not directly related to the physical pore size of the fabric.

The effects of the increase in flux magnitude increasing the rate of achieving the lower than 1 NTU product quality was concluded to be a result of the higher cumulative permeate volume being collected with time. This was indicated by the 5K and 1K suspension tests which resulted in a permeate turbidity of lower than 1 NTU after 2.5 L and 6 L of cumulative permeate volume was collected respectively for the range of flux magnitudes tested. This indicated how the operation of a membrane at higher flux magnitudes could be used to decrease the operation period required to obtain the same product quality phenomenon, as a respective lower flux test. This concluded how the permeate turbidity was a function of the cumulative permeate volume collected.

The effects of feed solids concentration on permeate quality:

In this phase of the performance evaluation the effects of varying the feed suspension solid concentrations on the product quality achieved over a period of operation, was investigated. The findings of the investigation indicated that the relatively higher feed suspension concentrations, of all three feed limestone particle size distributions tested, resulted in an increased rate of decrease in the permeate turbidity to below 1 NTU. This was concluded to be as a result of the relatively higher solid concentrations increasing the rate of pore narrowing. The fouling layer formed due to the propagation of the entrained particulate matter layer within the pores, which increased with an increase in solids concentration. This result correlated with the trend of increasing solid concentrations increasing the rate of fouling in the UF and MF membrane process [3,5].

It was concluded that for the sub-pore sized feed suspensions the more noticeable the effects of varying the feed suspension concentration became on the product quality achieved. This

was indicated by the role the concentration had on the effects of pore narrowing for the formation of the required fouling layer. This was indicated by the 1K particle distribution not being able to achieve the required product quality for a feed suspension concentration of 10 NTU. This was due to the relatively low suspension particle concentration not being sufficient to form the degree of pore narrowing required to form an effective separation. This is important, as it implies that the woven fabric has a minimum threshold feed suspension concentration below which the membrane will not be able to remove suspended matter below the pore size range of the fabric. Additionally, this validated that the entire 1K feed suspension particle size distribution was below the pore size of the woven fabric. This was due to a negligible decrease in permeate turbidity being achieved by the 10 NTU 1K feed suspension, indicating the how the fouling layer was required to separate the 1K feed suspension.

5.2 Evaluation of fouling characteristics

A series of experiments were performed to evaluate the effects different feed suspension physical properties had on the fouling characteristics of the woven fabric membrane. The fouling layer formation was indicated as being required for the separation of sub-pore sized particulate matter by the woven fabric, in order to achieve the required product quality. However, the implications of the fouling layer formation on the functioning of the woven fabric membrane were not known.

Raw water sources are known to vary vastly in composition from day to day, as was discussed in Chapter 2.4 Water quality monitoring and standards. For this reason, an understanding of how these variations in the composition of feed suspensions could affect the woven fabric flat sheet membrane functioning was required. This would indicate the potential sensitivity of the woven fabric membrane to changes in the composition and concentrations of feed suspension. The functioning of the membrane was monitored by calculating the fouling layer resistance formed as a function of the operation time. A relatively high fouling layer resistance would indicate a degradation of the membrane functioning through hampering the fluid flow through the membrane.

In order to adequately determine the effects of varying the physical feed suspension properties on the woven fabric, the selection of the feed suspension was important. Water sources in general comprise of both organic matter and inorganic particulate matter, each in

varying concentrations. Therefore, organic and inorganic feed suspension properties were evaluated to adequately replicate a potential water source that the membrane material could be utilised for. The following synthetic feed suspensions were evaluated: bentonite, kaolin, limestone and rehydrated yeast cells.

Bentonite is a clay type substance which was selected due to its low settling rate as a result of its strong colloidal properties. The strong colloidal properties arise from the anisomeric (varying, no two alike) properties of the clay. These properties are the result of the different crystalline faces of the particles varying greatly in charge and magnitude due to the bentonite clay particles varying chemical make-up [6].

Kaolin is a clay type substance which was selected due to its extensive use as a filler medium in coagulation and flocculation investigations. This is as the result of its ability to mimic other inorganic elements generally found in raw water sources [7-9].

Limestone is a sedimentary rock which is in abundance as a result of it**s** being present in the majority of raw water sources. This is due to it constituting 10% of all sedimentary rocks.

Yeast is a single cell fungus which was selected in order to investigate the effects organic suspensions had on the woven fabric membrane. For health and safety reasons the use of yeast was selected as opposed to utilising bacteria as the organic source.

Subcritical flux operation was not considered as part of the investigation. The reasoning for this was due to the woven fabric microfilter being indicated as requiring the formation of a fouling layer to remove sub-pore sized particulate matter. Therefore, any subcritical flux operation will result in relatively poor product quality for feed suspensions comprising of sub-pore sized particulate matter.

This investigation comprised of three parts, namely the development of synthetic feed suspensions, a membrane fouling layer resistance evaluation and an evaluation of the fouling effects of varying organic suspension compositions.

5.2.1 The development of synthetic feed suspensions

5.2.1.1 Investigation

The development of synthetic feed suspensions was required to determine an applicable concentration range for which the synthetic feed suspension components would remain

relatively stable in suspension. A feed suspension with a relatively high settling rate, was unlikely to be utilised in a membrane process, as the separation of the suspended matter could be achieved using settling alone. Therefore, these suspensions would not mimic real water sources and were not investigated further as part of the fouling layer resistance investigation.

The feed suspensions selected for the fouling layer investigation all fell within the range of fine suspended solids and colloidal particles. This resulted in the surface forces of these particles becoming the predominant acting forces, rather than the body forces, on the particles when within suspension. This was an important consideration as it is the surface forces that resulted in the particles forming varying degrees of stability in suspension. For this reason, the concentration limits of the selected feed suspension components were required, before the different characteristics of the feed suspensions fouling layer could be evaluated. The concentration limits were selected based on the range in which the components exhibited a low settling rate.

The investigation monitored the change in the suspension turbidity over a period of time. The turbidity quantified the cloudiness of the water sample and was monitored by the dispersion of a light source passing through a sample of the suspension. Therefore, as the suspended particles settle out of the solution, the concentration of these suspended particles within the solution decreased, resulting in a decrease in the solution turbidity. It was this change in turbidity that was used to determine the settling nature of the suspension. The investigation monitored the change in turbidity over a 6-hour period. The change in turbidity was correlated to the percentage by which the suspension concentration decreased. The percentage by which the concentration decreased was calculated using Eq. (5.1):

$$Percentage\ decrease = \left(\frac{C_0 - C_t}{C_0}\right).100 \qquad (5.1)$$

where C_0 is the suspension concentration at time zero and C_t the concentration at time t. The relationship of the feed suspension concentration to the resultant suspension turbidity was required before the concentration of the settling tests could be determined. This was required for each of the components over the range of concentrations to be tested, namely: 1, 0.5, 0.4, 0.3, 0.2, 0.1 and 0.05 g/L. For the purpose of this investigation, a stable suspension refers to a suspension where a 25 ±3% or lower decrease in the suspension concentration, over the desired time period, was witnessed.

5.2.1.2 Apparatus

A photograph of the apparatus used to determine the settling characteristics of the feed suspensions can be seen in Figure 29:

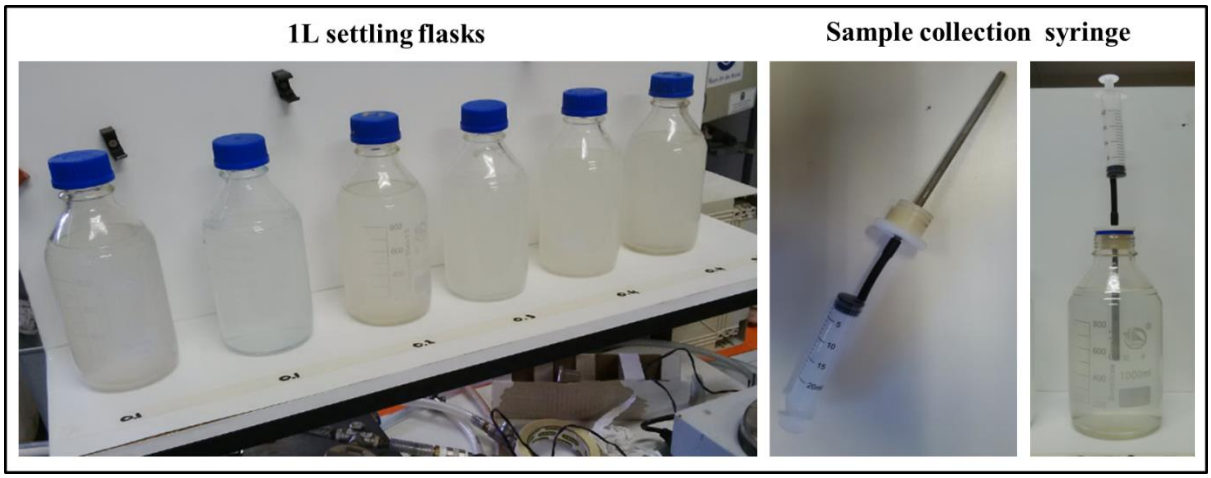

Figure 29: Settling characteristics evaluation apparatus.

The apparatus utilised 1 L glass flasks to contain the settling suspensions and a sample collection syringe that was designed with a plastic stopper to ensure that the samples were drawn from the same point within the flask, to ensure repeatability.

5.2.1.3 Methodology

The settling tests were performed using the stepwise procedure indicated below:

1) The feed suspensions were prepared using a bench scale, with an uncertainty of 0.001 g, to prepare the 1 L feed suspensions.
2) The samples were vigorously shaken and left for 1-minute to stabilise, before the first turbidity samples (t_0) were recorded, using the syringe with adapter.
3) Samples were taken in 2-hour increments, with the first sample taken at t_0 and the last sample taken after 6 hours.
4) Each suspension was replicated for reproducibility purposes.

The results were calculated based on the average turbidity recorded of the two repeat settling tests performed. The relationship of the resultant suspension turbidity of the particular feed suspension concentration was determined by plotting the concentration of the feed suspension versus the resultant turbidity (recorded at t_0) on an x-y axis. Linear regression was used to model the relationship between the two parameters.

5.2.1.4 Results and discussion

The results of the suspension concentration versus turbidity regression can be seen in Figure 30:

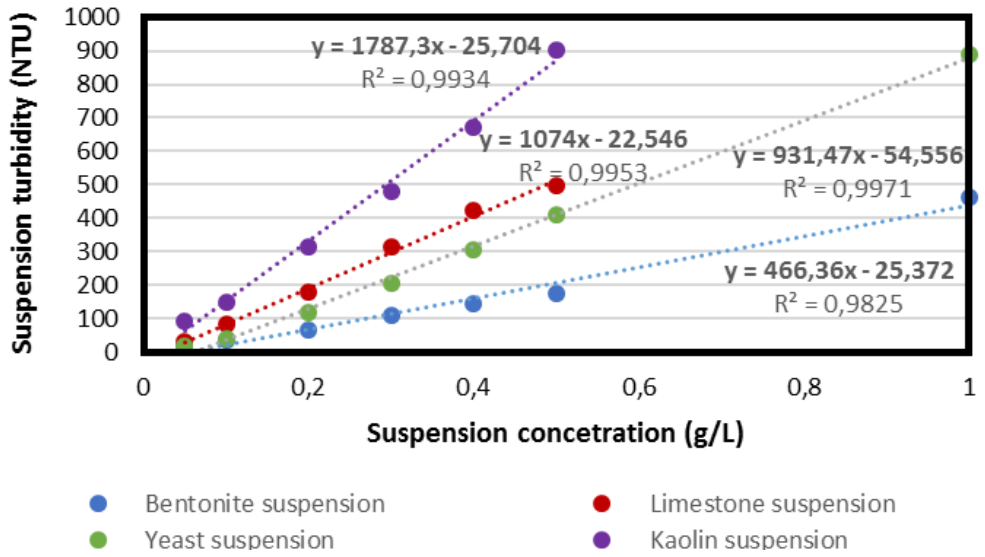

Figure 30: Linear relationship between suspension turbidity and suspension concentration validation.

The figure indicates how an increase in the suspension concentration, for all the suspensions tested, resulted in a relative increase in the resultant turbidity. The degree by which the turbidity increased was dictated by the composition of the suspension. The figure indicates a linear relationship between the suspension composition and resultant turbidity, for the concentration range tested, therefore, justifying the use of linear regression to model the relationship between these two parameters. Literature validates both the varying turbidity of the different components and the linear relationship between the component concentrations and the resultant turbidity [10].

The results of the settling investigation are summarised in Table 4:

Table 4: Settling characteristics after 2 and 6 hour durations, bentonite, kaolin, 5K limestone, yeast.

Settling characteristics after 2 and 6 hour intervals														
Feed concetration (g/L)	1.0 g/L		0.5 g/L		0.4 g/L		0.3 g/L		0.2 g/L		0.1 g/L		0.05 g/L	
Duration	2 hours	6 hours	2 hours	6 hours	2 hours	6 hours	2 hours	6 hours	2 hours	6 hours	2 hours	6 hours	2 hours	6 hours
Feed suspension	**Percentage concetration decrease (%)**													
Bentonite (7.5µm)	10	16	24	28	24	28	14	16	18	20	0	0	0	0
Limestone (5µm)	na	na	80	90	68	85	62	80	59	68	51	65	35	50
Kaolin (6µm)	na	na	14	27	18	29	20	32	24	37	18	27	0	0
Yeast (5,7µm)	4	21	5	29	10	36	21	45	13	34	2	15	0	0

The table indicates the percentage by which the suspension concentration decreased, for the components tested, over periods of 2 and 6 hours, as a result of settling. The feed suspension particles D_{50}s are indicated in the first column to the left of the table. The D_{50} for the yeast cannot be considered a true D_{50} but rather an average cell size based on the method used to calculate it.

The results indicate how the bentonite suspensions tested were all able to remain stable in suspension (< 25% ±3%), over the 6-hour duration for the range of concentrations tested. The limestone suspensions tested were unable to remain stable in suspension, which was expected as a result of limestone being a sedimentary rock. It was therefore proposed that limestone would not be an adequate synthetic water constituent for this phase of the performance evaluation. Both the kaolin and the yeast were able to remain stable in suspension (< 25% ±3%) within the first 2-hours of the settling tests, thereafter both settled out beyond the 25% upper settling bound, for the range of concentrations tested. Both suspensions were indicated as being applicable synthetic raw water components for investigations performed for 2 hours or less. The varying settling characteristics of the suspensions achieved above was validated in literature to be as a result of, but not limited to, the varying electrokinetic potential (ζ) of the suspensions tested [13].

5.2.1.5 Summary

From the above investigation it was indicated:

- The bentonite was capable of forming a stable suspension for the concentration range of 1.0-0.025 g/L for a period of 6 hours.
- The kaolin and yeast were unable to form stable suspensions for periods longer than 2-hours for a concentration range of 1.0-0.025 g/L.
- The limestone would not be an adequate synthetic feed suspension component due to its high settling rate.

5.2.2 Fouling layer resistance evaluation

5.2.2.1 Investigation

The membrane fouling layer resistance tests evaluated the effects various concentrations and compositions of bentonite, kaolin and yeast had on the woven fabric flat sheet membrane functioning. The fouling layer resistance was derived using Darcy's law equation, to

formulate an expression for calculating the resistance to flow through the membrane due to the formation of the fouling layer, expressed in Eq. (5.2):

$$R_f = \frac{1}{J_p \mu} TMP_{fouling} \quad (5.2)$$

The derivation required to arrive at Eq. (5.2) is discussed in Chapter 3.2.2 Resistance calculations.

The fouling layer resistance tests were used to determine the effects varying the feed suspension component's concentrations had on the resultant fouling layer resistance through the woven fabric membrane. The D_{50} of the various feed suspensions selected were all within 5±2.5 µm of each other, as indicated in Table 4 above. This was selected so that the variations in the fouling formed would be predominately due to the varying particle properties and not solely as a result of varying feed suspensions particle D_{50}s. An increase in the fouling layer resistance, during the membrane operation, would be an indication of an increase in the hindrance of the fluid flow to passing through the membrane pores. The hindrance would be a result of the formation of a fouling layer causing partial or complete pore blocking.

The bentonite, kaolin and yeast feed suspensions were investigated at concentrations of 0.5, 0.3, 0.1, 0.05 and 0.025 g/L. An additional test was performed to evaluate the effects of combining yeast and bentonite, to form a 0.3 g/L yeast and 0.025 g/L bentonite suspension (0.325 g/L suspension). This was done to indicate whether a cumulative effect on the fouling layer resistance would occur.

5.2.2.2 Apparatus

The apparatus utilised for this phase of the performance evaluation was designed in such a way as to allow the parallel operation of two separate membrane modules. The apparatus utilised can be seen in Figure 31:

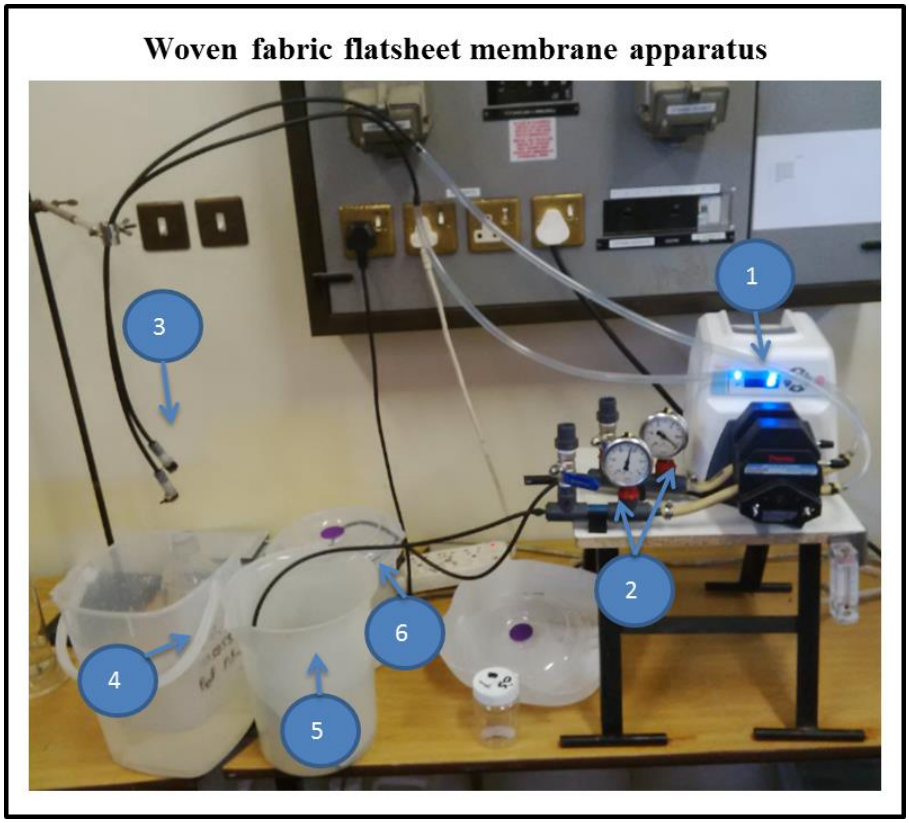

Figure 31: Woven fabric flat sheet membrane apparatus.

1: The permeate driving force was provided by a MasterFlex 6-600rpm, dual head, peristaltic pump.

2: The TMP, for both of the membrane apparatuses, A and B, were monitored using two non-stepped vacuum gauges with a pressure range of -100-0 kPa.

3: Indicates the permeate return lines.

4: Permeate for both membrane processes were collected in separate vessels and not recycled back to the membranes.

5: A 5 L holding vessel was utilised for process A.

6: A 5 L holding vessel was utilised for process B.

Membrane pressure curves were required for both membranes. The curves can be seen in Figure 32, with the calculated membrane pressure linear regression model, represented alongside the respective membrane pressure curve.

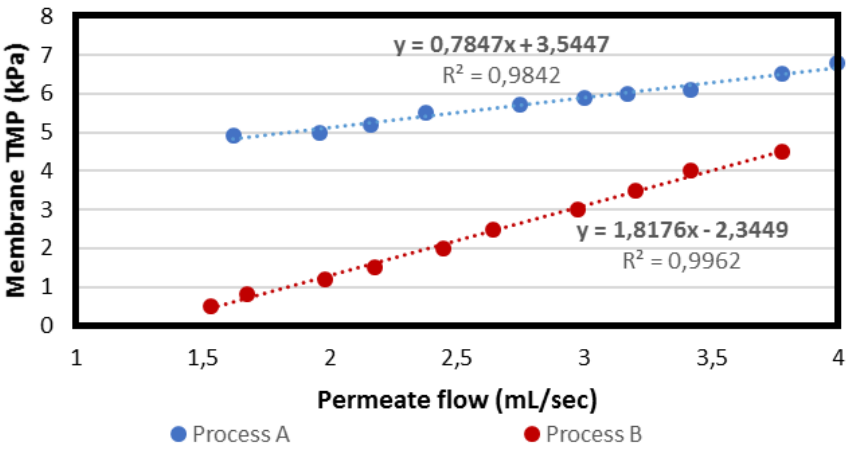

Figure 32: Membrane TMP linear regression model.

The membrane TMP curves were produced following the methodology described in Chapter 3.2.2 Resistance calculations. The two separate apparatus were designed to be as close to unity as possible. It was apparent from the regression models how the membrane pressure varied between the apparatuses. This indicates the importance of comparing the fouling layer resistance data only as opposed to the total resistance. The apparatus had an upper operation limit TMP of 45 kPa to prevent failure of the PVC connectors utilised.

5.2.2.3 Methodology

The fouling layer resistance evaluation procedure consisted of two steps namely: the initiation procedure and the operation procedure.

Step 1: Initiation procedure:

1) The membrane modules were tested for leaks prior to being utilised.
2) The holding vessels were filled with RO water.
3) The membrane modules were lowered into the respective holding vessels and left for 10 minutes to allow the pressure within the membrane modules to equalise.
4) The pump speed was set to the desired flow and left to stabilise for 10 minutes before the first permeate flux was recorded.
5) If the pure water flux recorded was 50±2 LMH, the pump was turned off and the holding vessels drained, if it was lower or greater than 50±2 LMH, the pump flow was altered and the flow left to stabilise as before, before the new permeate fluxes were recorded. This was repeated until the desired flow was achieved for both apparatuses.

Step 2: Operation procedure:

1) The holding vessels were filled with the desired feed suspensions.
2) The membrane modules were lowered into the respective holding vessels and left for 10 minutes to allow the pressure within the membrane modules to equalise. This was done to prevent initial cavitation of the pump, to prevent premature wear of the peristaltic tubing.
3) The pump flow was not altered.
4) The permeate flux and turbidity were recorded after 5-minutes of operation and thereafter in 5-minute intervals.
5) The tests were performed for 2 hours or until a TMP of 45 kPa was achieved.
6) The feed suspension composition was monitored and approximately maintained.

The membranes utilised were single membrane modules with dimensions of 50x105x8 mm. New membrane modules were utilised for each of the tests, due to the potential for irreversible and irrecoverable fouling occurring within the membrane pores. Both Steps 1 and 2 were repeated for each of the feed suspensions investigated.

5.2.2.4 Results and discussion

The fouling layer resistance for the bentonite suspensions tests can be seen in Figure 33:

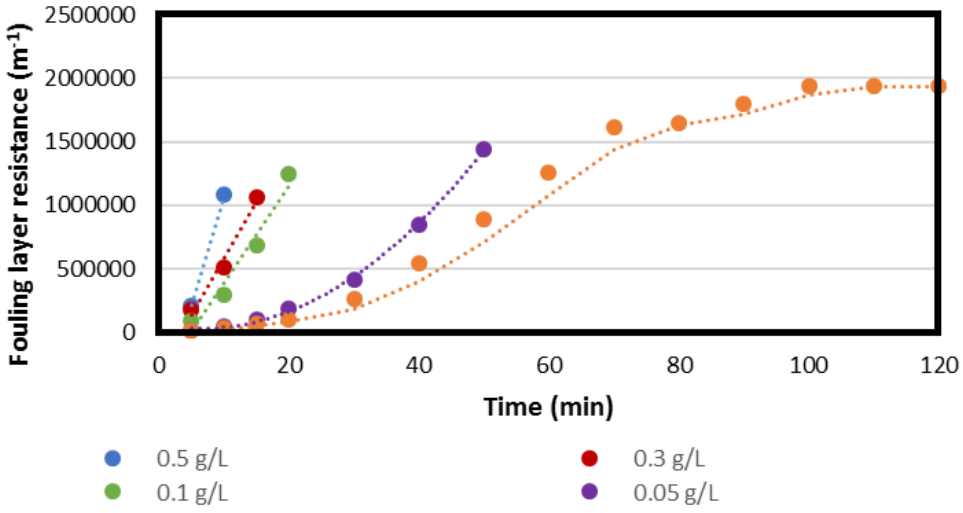

Figure 33: Bentonite fouling layer resistance evaluation for various concentrations, 0.5, 0.3, 0.1, 0.05, 0.025 g/L bentonite, 50 LMH.

The figure indicates the change in fouling layer resistance as a function of operation period for the bentonite suspensions tested. The results indicated that an increase in the fouling resistance rate was achieved for an increased bentonite concentration. The 0.5, 0.3, 0.1 and

0.05 g/L tests were all stopped prematurely as a result of the TMP achieved exceeding the cut-off value of approximately 45 kPa. It was apparent from the figure how sensitive the fouling layer resistance was to additions in concentrations of bentonite. The trend of an increase in solid concentrations increasing the fouling layer resistance correlates with literature where an increase in the solid concentrations, increased the fouling rate achieved for both UF and MF membranes [3,5].

The fouling layer resistance for the kaolin suspensions tests can be seen in Figure 34:

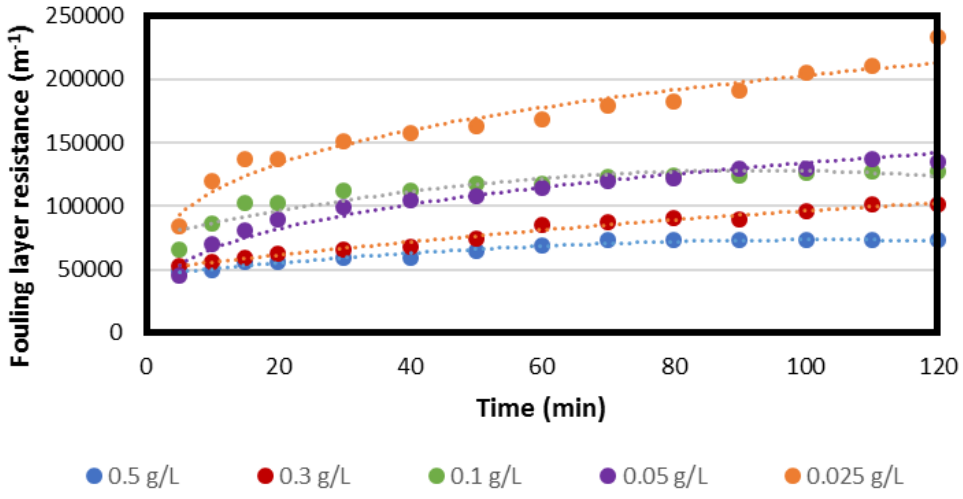

Figure 34: Kaolin fouling layer resistance evaluation for various concentrations, 0.5, 0.3, 0.1, 0.05, 0.025 g/L kaolin, 50 LMH.

The figure indicates the change in fouling layer resistance as a function of operation period for the kaolin suspensions tested. The results indicated that an increase in the fouling layer resistance rate was achieved by a decrease in the suspension concentration. This trend was the opposite to what was witnessed for the bentonite suspensions tested as well as contradictory to what was indicated in literature where an increase in the solids concentration is expected to result in an increase in the fouling layer resistance, in the case of MF and UF membranes [3,5].

The figure indicates how the fouling layer resistance was not a sole function of the fouling layer thickness that formed, as the lower kaolin suspension concentration resulted in a higher fouling layer resistance being achieved than the comparative higher concentration test. This indicated how the fouling layer formation can be of both detriment and benefit to the functioning of a membrane, when operated on sub-pore sized feed suspensions. The kaolin suspension tests were important as they indicate that the complex multi-layering of the woven

fabric results in fouling characteristics that are not always relatable to other nonwoven MF and UF membrane materials.

The fouling layer resistance for the yeast suspensions tests can be seen in Figure 35:

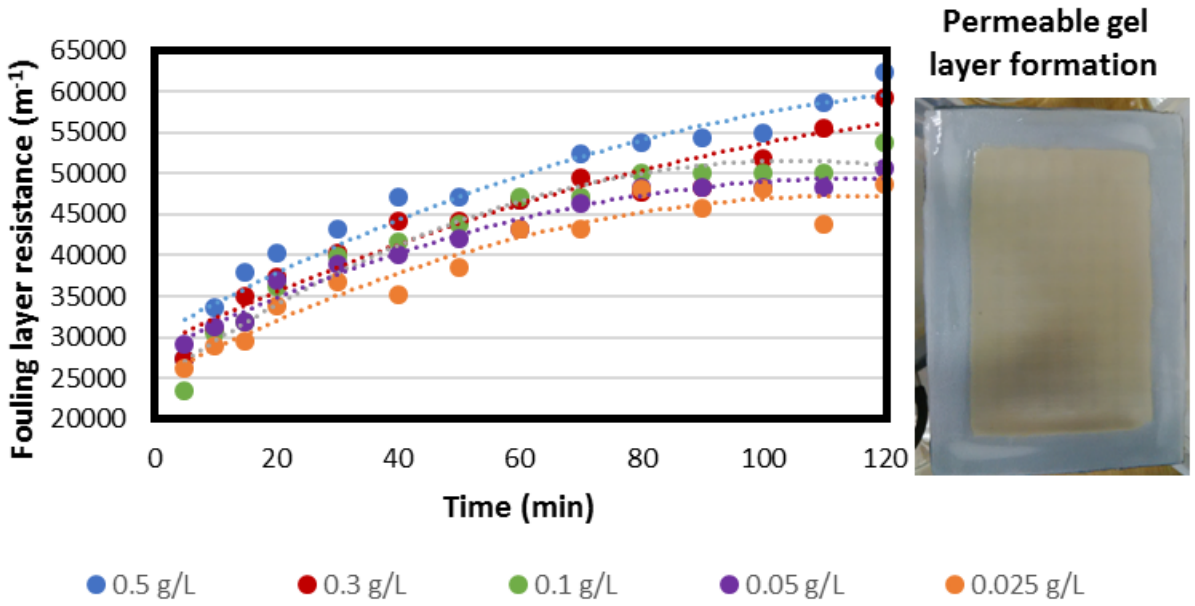

Figure 35: Yeast fouling layer resistance evaluation for various feed suspension concentrations, 0.5, 0.3, 0.1, 0.05, 0.025 g/L yeast, 50 LMH.

The figure indicates the change in fouling layer resistance as a function of operation period for the yeast suspensions tested. The results indicated that an increase in the fouling resistance rate was achieved for an increased suspension yeast concentration. The fouling rate for the concentrations tested are seen to be very similar in magnitude, indicating that the membrane was relatively insensitive to changes in the yeast suspension concentrations in comparison to the sensitivity of the membrane to the bentonite concentration.

The yeast suspensions comprised of living single cell fungal organisms which were protected by a rigid cell wall. The cell wall resulted in the yeast cells having a uniformly oval shape. The similar resistance magnitude and resistance rates for the range of concentrations tested was a result of the uniformly oval shape of the yeast cell walls. This prevented large flow hindrance from occurring when multiple cells were packed on one another. The yeast cells resulted in the formation of a permeable gel layer, as can be seen on the right hand side of the figure above. Although the fouling layer thickness increased with an increase in suspension concentration, the fouling resistance rate was not greatly affected due to the permeability of this fouling layer. This type of fouling layer is classed as a gel layer due to its compressible and permeable nature [16].

The fouling layer resistance tests for evaluating the combined yeast and bentonite suspension can be seen in Figure 36:

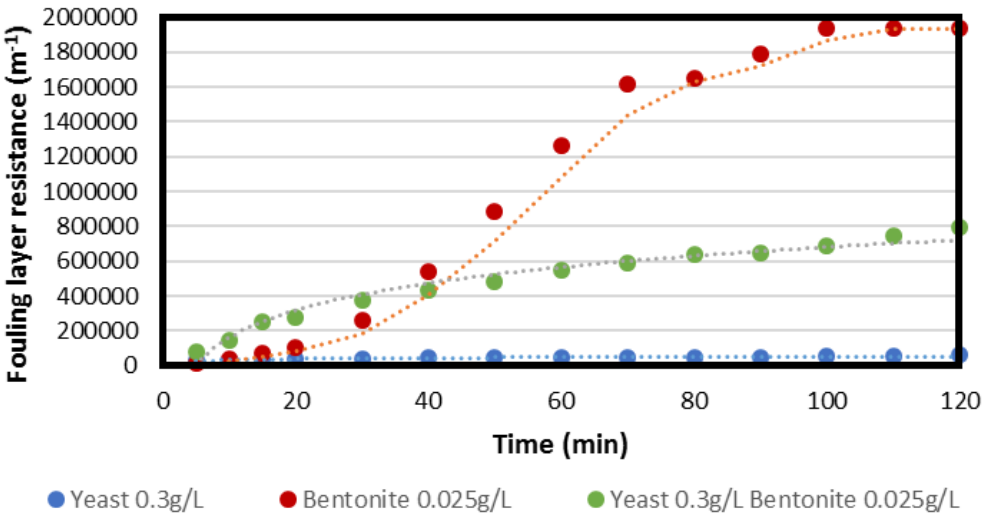

Figure 36: Fouling layer resistance comparison.

The figure indicates the change in fouling layer resistance as a function of operation period for the three suspensions tested. It can be seen in the figure that the fouling rate decreased in the following order: the bentonite 0.025 g/L suspension, the combination of yeast 0.3 g/L and bentonite 0.025 g/L suspension (0.325 g/L) and the yeast 0.3 g/L suspension.

The comparison of the suspension fouling layer resistances, indicates the effect the fouling layer composition has on the resultant resistance magnitude. The bentonite suspension formed a non-compressible, low porosity fouling layer. The bentonite particles are jagged in shape due to their anisomeric surface properties which promoted the complex surface plane geometries of the particles [6]. These jagged shapes led to the formation of a tightly interconnected particle fouling layer. This resulted in the relatively high fluid flow hindrance of the bentonite fouling layer as indicated by the relatively high resistance magnitude in comparison to the yeast porous gel layer [15-17]. The high permeability of the yeast layer was due to the oval yeast cell shape preventing high fluid flow hindrance occurring when the cells overlapped within the fouling layer [15-17]. When these two suspensions were combined, the resultant fouling layer was a combination of both suspension types where the porous yeast layer reduced the formation of the low porosity bentonite layer. The yeast was unable to completely prevent the formation of the bentonite layer and therefore the resultant fouling layer resistance was greater than that of the yeast only but less than the bentonite only

suspension. This indicates the functioning of membrane pre-coating, where a low fouling substance is used to reduce the fouling effects of a higher fouling substance [18].

The effect of the higher suspension concentration, of the combined suspension (0.325 g/L compared to 0.3 g/L and 0.025 g/L), was noticeable during the first 30 minutes of the membrane operation. Where the initial fouling layer resistance of this test was greater than that of both the yeast and bentonite only suspension tests. However, thereafter the formation of the yeast gel layer hindered the formation of the higher resistance bentonite fouling layer.

5.2.2.5 Summary

The comparison of the various feed suspension fouling layer resistances indicated:

- The fouling resistance rate was affected by the size, shape and chemical properties of the feed suspension.
- The woven fabric interacted with the different suspension components differently.
- The feed suspension components physical properties resulted in varying degrees of permeability of the fouling layer formed.
- A low fouling feed suspension component could be used to reduce the fouling effects of a higher fouling feed suspension component.
- Membrane pre-coating was indicated as being effect for use with the woven fabric membrane, where a lower fouling layer resistance feed suspension component is use to reduce the fouling effects of a highly fouling feed suspension.

5.2.3 Fouling effects of organic feed suspensions

5.2.3.1 Investigation

A series of investigations were performed to evaluate the effects of organic rich feed suspensions on the woven fabric membrane. An investigation was performed to determine the sensitivity of the woven fabric membrane to changes in the yeast feed suspension composition. The yeast composition was altered by preparing a 25 L suspension of 0.3 g/L yeast and performing a test at the beginning of three consecutive days, using the same initial prepared yeast suspension. The aging process was to indicate how living feed suspensions compositions vary over a period of time, and how these changes affect the fouling exhibited on the woven fabric.

A second series of tests were performed to evaluate the effects varying the permeate flux had on fouling layer resistance. This was to determine whether an increase in flux by a factor of X would result in an increase in the fouling layer resistance rate by the same X factor. This was required to correlate the effects of an increased permeate flux magnitude on the product quality achieved due to the resultant fouling layer formation. The effects of permeate flux on the fouling layer resistance was investigated for magnitudes of 50, 100 and 200 LMH

An additional investigation was performed to indicate the potential need for a membrane defouling process to be applied between membrane operations on a yeast feed suspension. A membrane module was operated for three 120-minute intervals, at an initial flux of 50 LMH. After 120 minutes of operation, the operation was stopped for 10 minutes, before continuing the operation for another 120-minute period. This was repeated three times to validate that the permeate flux and TMP would not return to the initial values of the first 120-minute operation due to the absence of a defouling measure applied between operations.

5.2.3.2 Apparatus

The apparatus utilised for this phase of the performance evaluation is the same as the apparatus designed for use in Chapter 5.2.2 Fouling layer resistance evaluation.

5.2.3.3 Methodology

The methodology followed for this phase of the performance evaluation is the same as the methodology outlined in Chapter 5.2.2 Fouling layer resistance evaluation.

5.2.3.4 Results and discussion

The fouling layer resistance tests for evaluating the sensitivity of the woven fabric to variations in the living cell composition of a yeast suspension can be seen in Figure 37:

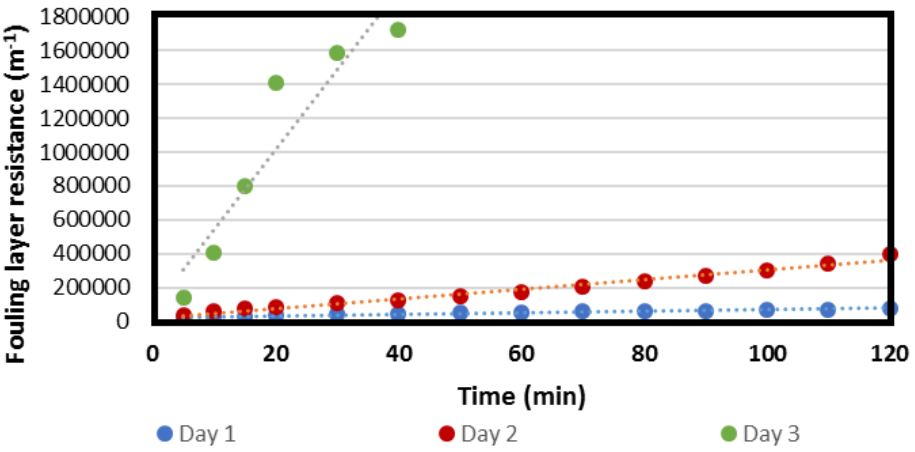

Figure 37: Fouling layer resistance sensitivity evaluation for varying yeast suspension compositions, 0.3 g/L yeast, 50 LMH.

The figure indicates the change in fouling layer resistance as a function of the membrane operation period for the different yeast suspension compositions tested. It is apparent from the figure that there was an increase in the rate of the fouling layer resistance as the yeast suspension was aged in the absence of a substrate. The resistance difference over the 3-day period was significant, with an increase in resistance by a factor of 20 between the first and third day tests. The results indicate the sensitivity of the woven fabric membrane to changes in yeast suspension compositions. The results indicated how processes containing living cells, such as raw water and wastewater sources, can change within a short period of time and how these changes affect the resultant membrane functioning.

The results of the fouling layer resistance test for initial fluxes of 200, 100 and 50 LMH can be seen in Figure 38:

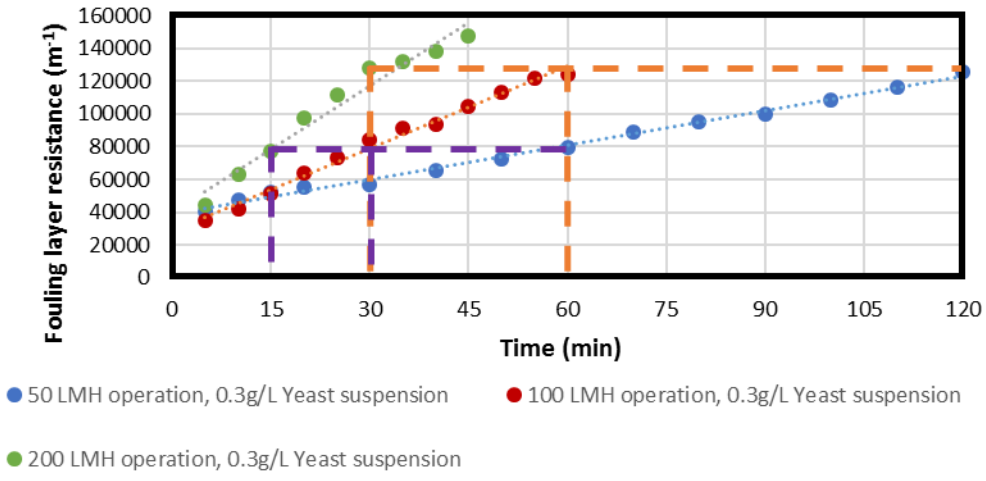

Figure 38: Effects of permeate flux magnitude on fouling layer resistance rate: 200, 100 and 50 LMH, 0.3 g/L yeast suspension.

The figure indicates the fouling layer resistance versus operation time for the three initial permeate flux values tested. From the results it is apparent that an increase in the initial flux by factors of 2 and 4 for the initial permeate flux of 50 LMH, resulted in an increase in the fouling layer resistance rate by the same factor. This was apparent from the 100 LMH and 200 LMH flux tests, as it took approximately 60 minutes and 30 minutes respectively to achieve the same approximate fouling layer resistance value as the 50LMH test did after 120 minutes of operation. This is indicated by the orange dashed line in the figure above. The same trend is apparent when comparing the 50 LMH test after 60 minutes, as indicated by the purple dashed line in the figure above. This correlates with the results indicated in Chapter 5.1.2 The effects of permeate flux magnitude and solid size distribution on permeate quality, where the product quality was indicated as being a function of the cumulative permeate volume collected and not as a result of the force of entrainment applied to form the fouling layer. This is reiterated in this section as the resultant fouling layer resistance is indicated as not being a strong function of the force of entrainment but rather the cumulative permeate volume collected.

The effects of permeate flux and TMP as a function of operation period for three consecutive tests where no defouling measure was applied to the membrane between operations, can be seen in Figure 39:

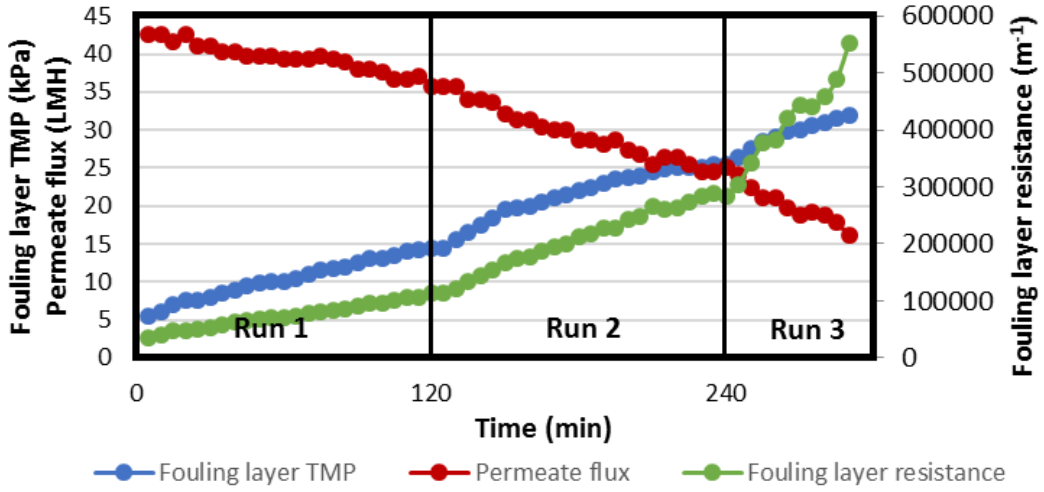

Figure 39: The effects of halting membrane operation and restarting without applying any form of defouling strategy.

The figure indicates the change in the Fouling layer TMP, permeate flux and the fouling layer resistance as a function of the subsequent operation cycles. The figure indicates that the membrane did not regain the initial permeate flux and TMP between the 120-minute operation periods, as a result of a defouling measure not being applied. This indicates the

importance of investigating potential defouling strategies to regain the membrane functioning lost to the effects of fouling. This correlates with literature, where the effects of fouling on the membrane functioning are described as being a major shortcoming of every membrane process due to the high operating costs associated with controlling it [2-5].

5.2.3.5 Summary

The organic rich feed suspension fouling effects evaluation indicated the following:

- The freeze dried yeast cells did not all rehydrate with the sole addition of water.
- The presence of both dead and living yeast cell matter promoted the formation of an EPS layer between the cells.
- The fouling layer resistance of the EPS containing suspension was significantly larger (20-times larger) than that of the EPS free suspensions.
- This indicated how processes containing cell matter could change in short periods of time with relatively vast variations in the resultant fouling characteristics occurring.
- It was indicated that defouling measures were required to reverse the effects of fouling between subsequent membrane operation cycles.

5.2.4 Conclusions

Fouling layer resistance evaluation:

The fouling layer resistance evaluation determined the effects of different feed suspension properties on the fouling layer resistance of a woven fabric membrane module. The investigation concluded that the fouling layer resistance was affected by the size, shape and chemical properties of the feed suspensions tested. The bentonite and yeast suspensions resulted in an increased fouling layer resistance with an increase in the suspension concentration, whereas the kaolin resulted in a decrease in the fouling layer resistance with an increase in the suspension concentration. This was concluded to be as a result of the complex multi-layering of the woven fabric membrane material affecting the interaction of the suspension particles differently based on the various particle shapes and chemical makeup.

From the comparison of the fouling layer resistance magnitudes of the different bentonite and yeast suspensions, it was concluded that the fouling layer resistance magnitude was a strong function of the fouling layer composition. The bentonite particles formed a non-compressible, low porosity fouling layer that resulted in a relatively high fouling layer resistance being

achieved. This was attributed to the bentonite particles' chemical make-up forming complex particle shapes, which promoted the low permeability of the overlapping particle fouling layer that formed. The shape and properties of yeast cells results in the formation of a compressible, porous fouling layer that results in a relatively high porosity fouling layer. It was concluded that a lower fouling substance could be used to decrease the fouling nature of a higher fouling substance. This indicated the applicability of utilising membrane pre-coating to improve the functioning of the woven fabric membrane utilising high fouling feed suspensions.

Evaluation of the fouling effects of organic feed suspensions:

The effects that varying organic feed suspensions compositions have on the fouling layer resistance of a woven fabric membrane module were investigated. The results indicated how processes containing living cells such as raw water and wastewater sources change within a short period of time and how these changes affect the resultant membrane functioning.

5.3 Overall conclusion of the performance evaluation

The complex multi-layering of the woven fabric membrane was concluded to result in the different trends exhibited in the product quality and the fouling layer resistance evaluations performed. A 1 µm particle should not be capable of being entrained within a larger pore. However, the complex multi-layering of the woven fabric resulted in the formation of the membrane pores being highly tortuous. The tortuous pores promoted the occurrence of pore narrowing with sub-pore sized particulate matter as indicated by the woven fabric producing a product quality of lower than 1 NTU as well as an increase in the fouling layer resistance. This indicated that an entrainment of these sub-pore size particles was occurring within the woven fabric membrane pores.

It was therefore this unique complex multi-layering effect of the woven fabric that resulted in the woven fabric not correlating with the functioning of other MF and UF membranes. It was for this reason that the microfilter categorised woven fabric membrane was capable of achieving UF degrees of separation ability. The property of the woven fabric membrane to remove sub-pore size suspended matter indicated the applicability of the membrane's separation ability to water treatment processes and how a pore size estimate of the fabric could not be used to pre-empt the separations capability of the fabric.

5.4 References

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Chapter 6: Flux enhancement and cleaning evaluation

Overview

This chapter is divided into six sections, namely:6.1 Preliminary evaluation of flux enhancement methods, 6.2 Woven fabric membrane design, 6.3 Flux enhancement evaluation, 6.4 Cleaning evaluation 6.5 Overall conclusion of the flux enhancement and cleaning evaluations and 6.6 References. In this chapter the investigations into the flux enhancement and cleaning methods evaluated are covered.

6.1 Preliminary evaluation of flux enhancement methods

6.1.1 Investigation

The preliminary investigation was performed to determine the effects different flux enhancement methods had on the woven fabric membrane. The investigation evaluated the different flux enhancement methods at two operation levels, as summarised in Table 5:

Table 5: Flux enhancement methods upper and lower testing levels.

Method	Levels tested	
Backflush duration (continuous)	5 minute	10 minute
Backflush duration (pulsed)	5 minute	10 minute
Air scour intensity (continuous)	5 L/min	10 L/min
Relaxation duration	5 minute	10 minute
Backflush + periodic air scour	5 minute 5 L/min	10 minute 5 L/min
Feed solution	50/50 D_{50} 5µm lime stone D_{50} 75µm bentonite	

The purpose of utilising two levels of testing was to better evaluate the defouling phenomena that were occurring. The flux enhancement methods evaluated were as follows: gravity backflush, air scouring and membrane relaxation. The results of the flux enhancement methods were compared based on the percentage of the initial flux value remaining after the 2-hour operation period. The flux enhancement measures were compared to a dead-end operation test where no defouling measure was applied to the membrane. Additionally, the product quality trends achieved by the different methods investigated were evaluated. This was to determine how the dynamic operation of the membrane was affected by the flux enhancement measures.

The full investigation performed is covered in Appendix D: Preliminary investigation of flux enhancement methods.

6.1.2 Results and discussion summary

A summary the flux enhancement results can be seen in Table 6:

Table 6: Preliminary flux enhancement methods results comparison.

Increasing energy input →										
Flux enhacement method	**Dead end**	**Relaxation**		**Gravity backflush (continuous)**		**Gravity backflush (pulsed 2min on 1min off)**		**Air scour coupled periodic backflush**	**Continuous air scouring**	
Level tested		5 min	10 min	5 min	10 min	5 min	10 min	5 min + 5 L/min	5L/min air scour	10L/min air scour
Percentage initial flux remaining	31%	37%	40%	43%	45%	44%	47%	46%	58%	53%

The table indicates the percentage of initial flux remaining after the 2-hour operation period for the flux enhancement methods tested. A higher percentage remaining is an indication of a higher defouling potential achieved by the flux enhancement method.

The table was ordered in such a way that the flux enhancement methods are listed from left to right in order of the increasing power utility required by the methods tested. The first entry, ‘Dead end’, describes the test performed where no flux enhancement method was applied. The table indicates how the flux enhancement ability varied between the flux enhancement methods tested where the relatively higher power utility methods resulted in a higher defouling efficiency of the membrane.

The effects of a membrane backflush and continuous air scouring on the product quality achieved can be seen in Figure 40:

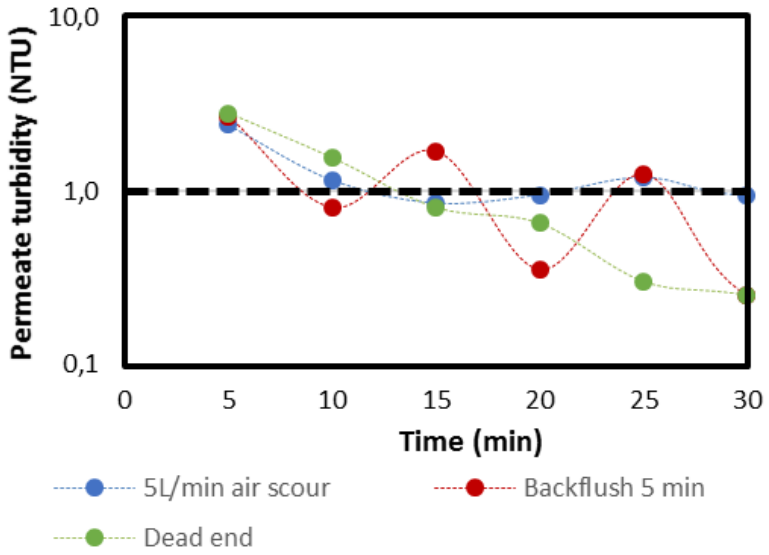

Figure 40: Comparison of the effects of air scouring and backflush on permeate turbidity.

The figure indicates the permeate turbidity for the first 30 minutes of the 5 L/min continuous air scouring test, the 5-minute backflush test and the dead end operation test. The figure indicates how all three tests started off with a turbidity in the range of 3 and 4 NTU. Thereafter the trends varied between the tests performed. The product quality of the 5 L/min continuous air scouring test can be seen to oscillate slightly around 1 NTU as a result of the continuous air scouring reducing the propagation of the fouling layer. The backflush test product quality can be seen to oscillate largely. This was as a result of the periodic nature of the backflush removing a portion of the fouling layer that was allowed to form between the backflush cleaning cycles. The product quality of the dead end test decreased with no oscillation or periodic increase over the operation period. This was as a result of the fouling layer being left to propagate with no flux enhancement controlling measures. This indicated the sensitivity of the product quality to variations in the fouling layer formation.

The investigation indicated the varying feasibility of the flux enhancement methods tested to the woven fabric membrane. The membrane relaxation method resulted in relatively low flux recovery in comparison to the other methods tested and was indicated as having low potential for improvement; it was therefore not investigated further. The membrane backflush and air scouring orientated flux enhancement methods tested indicated the potential benefits these methods have on woven fabric membrane operation. It was however apparent from the

product quality evaluation of these methods that additional control was required to better regulate the propagation of the fouling layer to maintain the desired product quality. For this reason, refinement of the membrane design was required to better suit these methods to the dynamic operation of the woven fabric membrane.

6.1.3 Summary

The preliminary flux enhancement evaluation indicated:

- Varying degrees of flux recovery were achievable utilising the different flux enhancement measures.
- Further revision of the membrane design was required to better suit these methods to the dynamic operation of the woven fabric membrane.

6.1.4 Conclusions

From the flux enhancement study as a whole it was concluded that varying degrees of flux recovery were achievable using the different flux enhancement methods. Both the continuous and periodic type of flux enhancement methods evaluated resulted in oscillation of the permeate turbidity above and below the required product quality. Therefore, both methods affected the dynamic functioning of the membrane. A revision of the membrane process was required to determine whether these flux enhancement methods could be better suited to the required dynamic operation of the woven fabric membrane.

6.2 Flux enhancement evaluation

The focus of this investigation was to evaluate the effects of different flux enhancement methods on the fouling layer resistance and product quality achieved for a woven fabric membrane. A synthetic feed suspension of 0.3 g/L yeast was selected for these investigations. The selection was based on the composition and the effective particle size distribution of the yeast suspensions that formed. The particle size range of the yeast suspension was determined to be approximately 3-13 µm. The yeast feed suspension had both varying concentrations of living and dead cell matter and was in a size range that mimicked potential bacteria. This met the requirements of both raw water and wastewater suspensions.

This investigation comprised of two parts, namely the development of the dead end operation protocol and the continuous air scouring flux enhancement evaluation.

6.2.1 The development of the dead end operation protocol

6.2.1.1 Investigation

A means of comparing the effects of the flux enhancement methods was required to effectively evaluate the different methods tested. A dead end test was selected as it allowed both the fouling layer resistance and the product quality of a test where no defouling measure had been applied as a comparison means. It was important to first determine whether the dead end test resulted in repeatable fouling layer formation and product quality, in order to provide a consistent means of comparison against.

To test the replicability of the fouling layer resistance and the product quality of the yeast suspension, three dead end operation tests were performed. These tests were performed a week apart to indicate any potential variations in the results due to changes in ambient conditions, such as the air temperature. The ambient air temperature was expected to affect the activation of the freeze dried yeast cells. A 25% deviation between the results of dead end tests was considered plausible for a membrane process with biological matter in a non-temperature controlled environment.

Additionally, it was important to indicate whether it was possible to completely defoul a woven fabric membrane module once it had been operated on a yeast feed suspension before evaluating the flux enhancement methods. It was expected that a large percentage of the fouling that occurred would result in the formation of irreversible pore blocking, based on the woven fabric being categorised as a microfilter and it investigated for use in an ultrafiltration applicable water treatment process. It was therefore important to indicate to what degree irreversible fouling could be expected, by utilising a cleaning method that had already been proven to be effective on a woven fabric membrane. The investigation utilised a single membrane module, which was physically cleaned using a scrubbing brush and rinsing after 60-minutes of dead end operation at an initial permeate flux of 100 LMH. This was to be repeated 10 times.

The yeast suspension would not have been utilised if the physical cleaning method resulted in premature failure of the woven fabric material due to the magnitude of the force required to reverse the effects of fouling. An SEM evaluation of the membrane utilised was evaluated at 90X and 3000X magnification to indicate any degradation of the membrane material.

6.2.1.2 Apparatus

A photograph of the revised woven fabric flat sheet membrane apparatus can be seen in Figure 41:

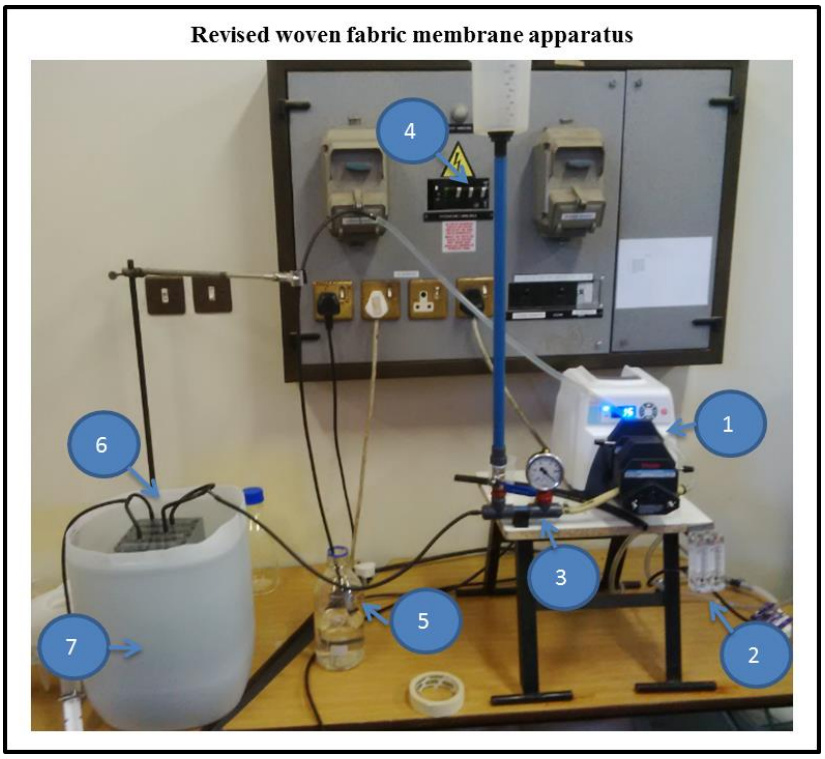

Figure 41: Revised laboratory scale woven fabric membrane apparatus.

The components that make up the complete membrane apparatus are numbered and illustrated in the figure, with the description of the components discussed below:

1: The permeate driving force was provided by a MasterFlex 6-600rpm, easy load, peristaltic pump.

2: The air scouring air flow rate was monitored using a rotary meter with a flow range of 0-10 L/min.

3: The TMP was monitored using a non-stepped vacuum gauge with a pressure range of -100-0 kPa.

4: A height variable backflush reservoir with a backflush pressure range of 3-10 kPa was used to vary the gravity backflush intensity.

5: The permeate was collected in a separate vessel and not recycled back to the membrane.

6: Indicates the membrane stand within the holding vessel.

7: A 25 L holding vessel was utilised.

The method used to determine the membrane TMP is outlined in Chapter 3.2.2 Resistance calculations. However, the increased surface area of the membrane modules utilised resulted in a negligible membrane TMP for the flow range utilised in the subsequent investigations.

6.2.1.3 Methodology

The dead end operation procedure consisted of two parts, namely the initiation procedure and the operation procedure:

Step 1: Initiation procedure:

1) The membrane modules were tested for leaks prior to being utilised.
2) The holding vessel was filled with RO water.
3) The membrane stand was lowered into the holding vessel and left for 10-minutes to allow the pressure within the membrane modules to equalise.
4) The pump speed was set to the desired flow, and left to stabilise for 10-minutes before the first permeate flux was recorded.
5) If the pure water flux recorded was 50±2 LMH, the pump was turned off and the holding vessel drained, if it was lower or greater than 50±2 LMH, the pump speed was altered and the flow left to stabilise as before, before the new permeate flux was recorded. This was repeated until the desired flow was achieved.

Step 2: Operation procedure:

1) The holding vessel was filled with the prepared 0.3 g/L yeast feed suspension.
2) The membrane stand was lowered into the holding vessel and left for 10 minutes to allow the pressure within the membrane modules to equalise.
3) The pump flow was not altered.
4) The permeate flux was recorded after 5 minutes of operation and thereafter in 5-minute intervals, the permeate turbidity was recorded in 15-minute intervals.
5) The tests were performed for the desired duration or till a desired TMP or permeate flux was achieved.
6) The feed suspension composition was monitored and approximately maintained.

To reduce the potential of irrecoverable fouling affecting the results of the subsequent tests performed, a new membrane module was used for each of the series of tests performed.

6.2.1.4 Results and discussion

The average fouling layer resistance and the permeate turbidity of the three repeat tests performed can be seen in Figure 42:

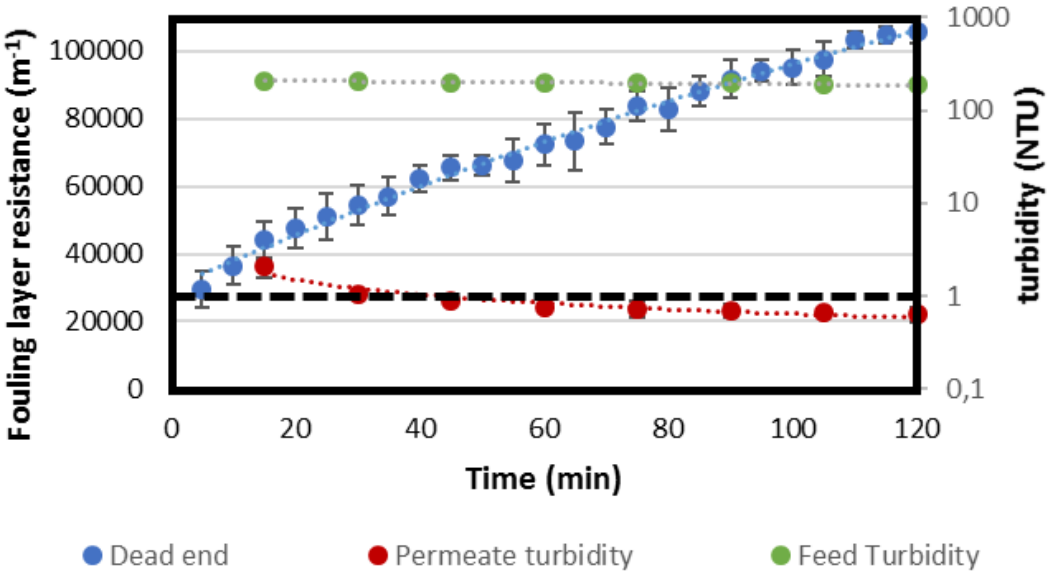

Figure 42: Dead end operation fouling layer resistance and permeate turbidity evaluation, 120-minute operation, 50 LMH permeate flux.

The figures indicate the average change in fouling layer resistance and the average change in the product and feed suspension turbidity as a function of time of three dead end operation tests. With the standard deviation indicated using error bars. All three tests achieved a product quality of below 1 NTU within approximately 30 minutes of operation with the cumulative permeate turbidity being 1 NTU for all three tests after the 120 minute operation period.

A visual comparison of a 1 L sample of the feed suspension and the total permeate collected, over the 2-hour operation period, can be seen in Figure 43:

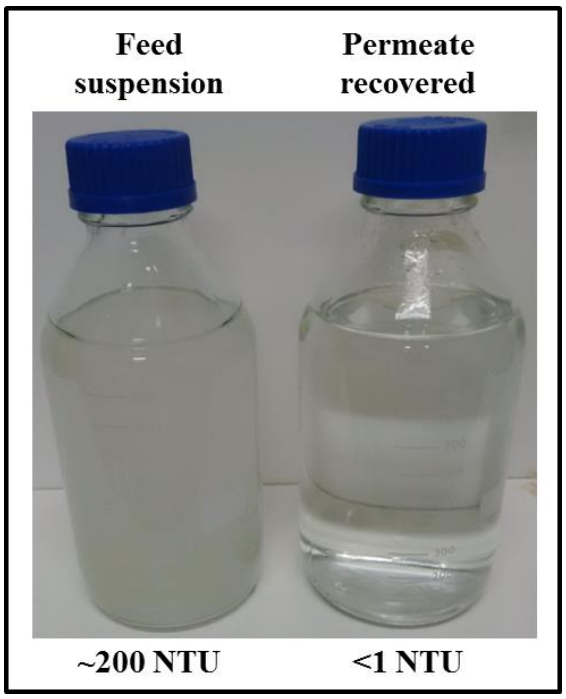

Figure 43: Visual comparison of the feed suspension and the permeate collected after the duration of the Dead end 3 test.

The figure indicates the visual variation of the 200 NTU feed suspension and the lower than 1 NTU permeate turbidity produced.

The results of the physical cleaning measure applied for reversing the effects of fouling can be seen in Figure 44:

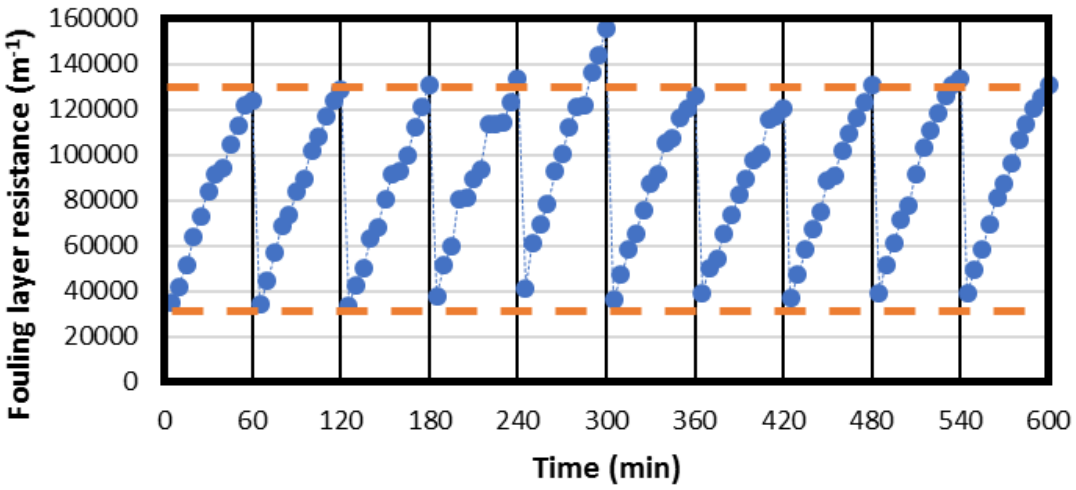

Figure 44: Physical defouling evaluation for prolonged operation, 600-minutes, 100 LMH, 10 defouling cycles.

The figure indicates the change in the fouling layer resistance as a function of the operation period. The orange dotted lines in the figure indicate the lower and upper resistance bounds of the first 60-minute dead end test. The subsequent dead end operation tests can be seen to

keep approximately within these bounds. This indicated how the cleaning method was able to completely reverse the effects of irreversible, with negligible amounts of irrecoverable fouling forming, over the 600-minute operation period. A flux of 100 LMH was selected for this investigation to indicate that at an elevated flux, complete recovery of the membrane performance could be achieved utilising a physical cleaning method.

The SEM image evaluation of two samples of the physically cleaned membrane material can be seen in Figure 45:

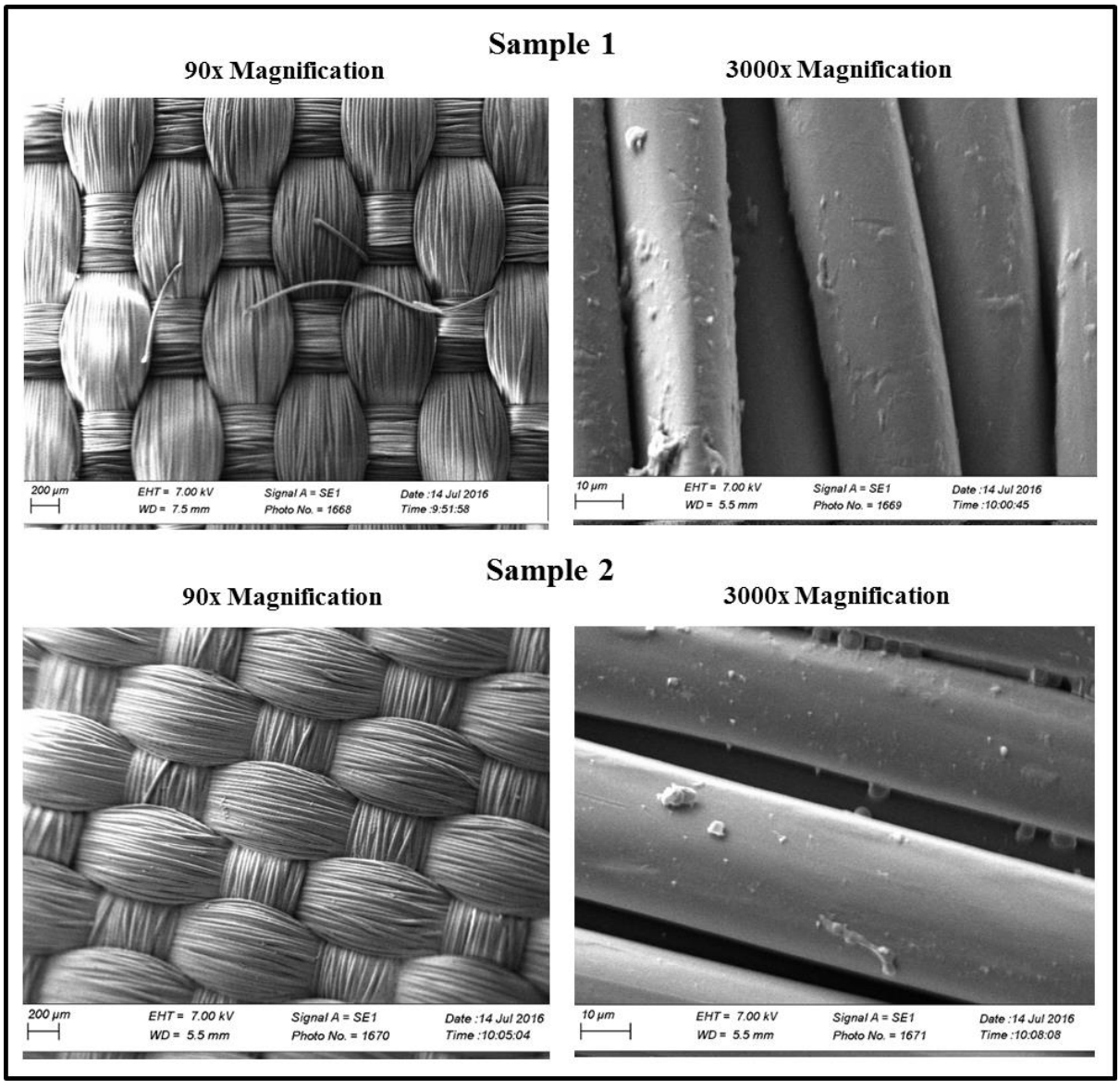

Figure 45: Membrane integrity, post physical cleaning evaluation, SEM imaging at 90X and 3000X magnification, ZEISS EVO MA15VP, CAF Stellenbosch University.

The figure indicates the SEM imaging of two samples of the membrane material utilised in the physical cleaning trial, at 90X and 3000X magnification. The ‘Sample 1 90X

magnification' image indicates the presence of loose membrane strands. This phenomenon was however not exhibited in the 'Sample 2 90X magnification' image, and was therefore concluded to be as a result of a manufacturing fault. It was expected that fraying of the weft and warp strands within the thread was likely to occur due to the scrubbing action applied to the membrane surfaces. In the 3000X magnification images, there was no apparent fraying of the strands within the material threads for both membrane samples. From this it was apparent that the physical cleaning method applied did not have an adverse effect on the integrity of the woven fabric material.

It was the durability of the woven fabric that resulted in the inherent applicability of the fabric for use as a membrane that was suited to developing environments. It was therefore a significant finding that the durability of the fabric had not been lost, or misinterpreted in previous studies [1].

6.2.1.5 Summary

The dead end operation procedure indicated that:

- The yeast feed suspension resulted in a repeatable fouling layer formation as a result of the fouling layer resistance versus operation period relationship having minimal deviation amongst the repeat tests.
- The dead end operation tests resulted in an average permeate turbidity of lower than 1 NTU.
- The fouling layer formed as a result of the yeast suspension, was indicated as being fully reversible when defouled utilising a physical cleaning method.
- The reversibility of the yeast suspension fouling layer indicated the applicability of the suspension for further testing utilising flux enhancement and cleaning methods.

6.2.2 Continuous air scouring flux enhancement evaluation

The preliminary investigation indicated that continuous air scouring had a greater defouling potential than the other methods tested. For this reason, further evaluation of the effects of different continuous air scouring operating methods was performed before further flux enhancement methods were investigated.

6.2.2.1 Investigation

The investigation evaluated the effects of both coarse and fine air scouring at an upper and lower air scouring flow rate. The fouling layer resistance and permeate turbidity were recorded for each of the tests and compared to the dead end operation test. An ineffective flux enhancement method was considered when the fouling layer resistance was larger than that of the dead end operation test and/or there was an inability of the membrane to achieve a permeate turbidity of less than 1 NTU over the 2-hour operation period.

Continuous air scouring is different to periodic flux enhancement methods, such as backflushing, as it continually acts against the formation of a fouling layer. For this reason, the fouling layer resistance of the air scouring tests would have to remain below the comparative dead end operation test for the duration of the tests to be considered effective.

6.2.2.2 Apparatus

The apparatus utilised for this phase of the defouling evaluation was the same as the apparatus utilised in 6.2.1 The development of the dead end operation protocol.

6.2.2.3 Methodology

The continuous air scouring procedure consists of two parts, namely an initiation procedure and an operation procedure. The initiation procedure utilised was the same as the initiation procedure outlined in Chapter 6.2.1 The development of the dead end operation protocol.

Step 2: Operation procedure:

1) The holding vessel was filled with the prepared 0.3 g/L yeast feed suspension.
2) The membrane stand was lowered into the holding vessel and left for 10-minutes to allow the pressure within the membrane modules to equalise.
3) The pump flow was not altered.
4) The air pump was turned on and set to the desired air sparger flow rate.
5) The permeate flux was recorded after 5-minutes of operation and thereafter in 5-minute intervals, the permeate turbidity was recorded in 15-minute intervals.
6) The test was performed for a duration of 2 hours.

The coarse air scouring flow rates tested were 7.5 L/min and 2.5 L/min and the fine air scouring flow rates tested were 5 L/min and 2.5 L/min. Each of the air scouring flow rate tests were repeated, with the fouling layer resistance calculated as an average of the two tests.

To reduce the potential of irrecoverable fouling affecting the results of the tests performed, two new membrane modules were used for each of the series of tests performed.

6.2.2.4 Results and discussion

The variations in fouling layer resistance for the air scouring tests performed can be seen in Figure 46:

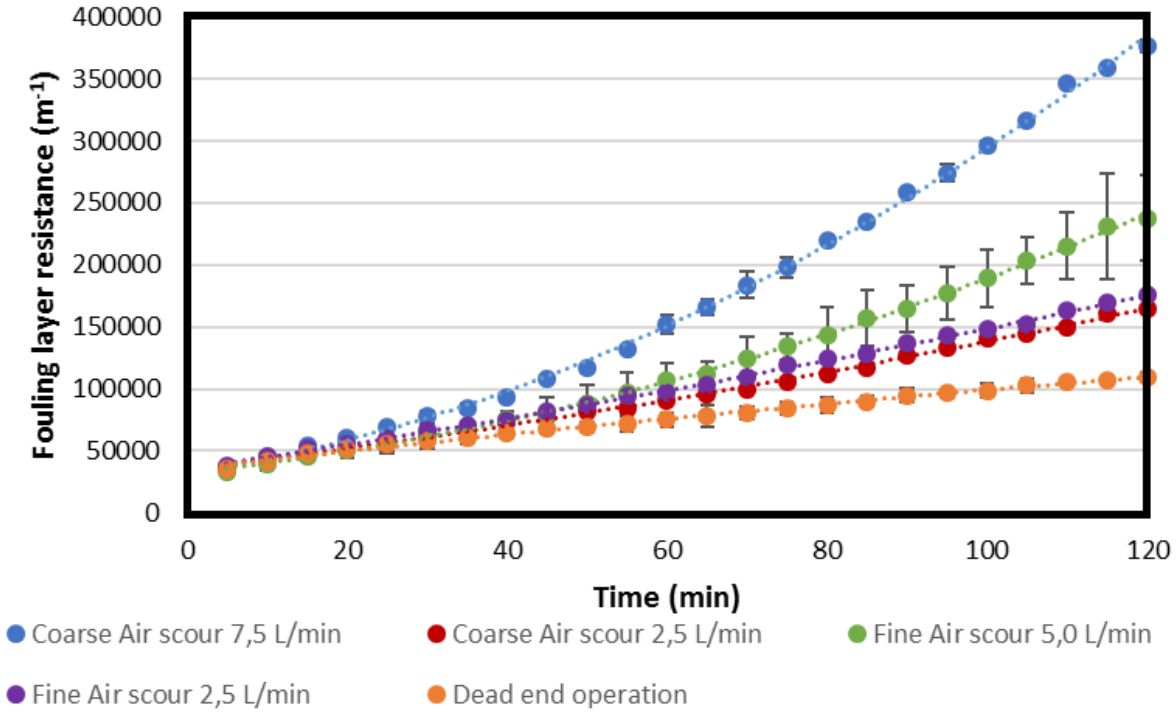

Figure 46: Continuous air scouring evaluation, for both coarse and fine air scouring, fouling layer resistance, 120-minute operation period, 50 LMH permeate flux.

The figure indicates the change in the fouling layer resistance as a function of the operation time for the coarse and fine air scouring tests performed. The standard deviation is indicated for the 'Coarse Air scour 7,5L/min', 'Fine Air scour 5L/min' and 'Dead end operation'. The figure indicates how the fouling layer resistance of all the air scouring flow rates tested increased at a relatively higher rate than that of the dead end operation test, over the entire 2-hour operation period.

It is apparent from the figure that a decrease in the permeate flux magnitude would not have resulted in an improved defouling potential for the air scouring tests performed. This was due to the fouling layer resistance of the air scouring tests being larger than the dead end test over the full duration of the testing period. The coarse and fine air scouring tests are discussed individually:

Coarse air scouring:

It is apparent from the figure that the 7.5 L/min tests resulted in a greater fouling layer resistance than the 2.5 L/min test. This indicates that an increase in the air scouring flow resulted in a greater fouling layer resistance being achieved. The effects of the increased air scouring rate having a negative effect on the membrane performance was initially indicated in Chapter 6.1 Preliminary evaluation of flux enhancement methods, Table 6, where a higher decrease in the permeate flux was achieved with an increase in the air scouring flow rate in comparison to the respectively lower flow rate test.

Fine air scouring:

The trend of an increase in air scouring flow rate increasing the fouling resistance was repeated in the fine air scouring tests where the higher 5 L/min flow resulted in a relatively greater fouling layer resistance than that of the 2.5 L/min air scouring test.

A visual inspection of the membrane modules, after the 2-hour operation, was performed to inspect the variations in the surface fouling layer that formed, as can be seen in Figure 47:

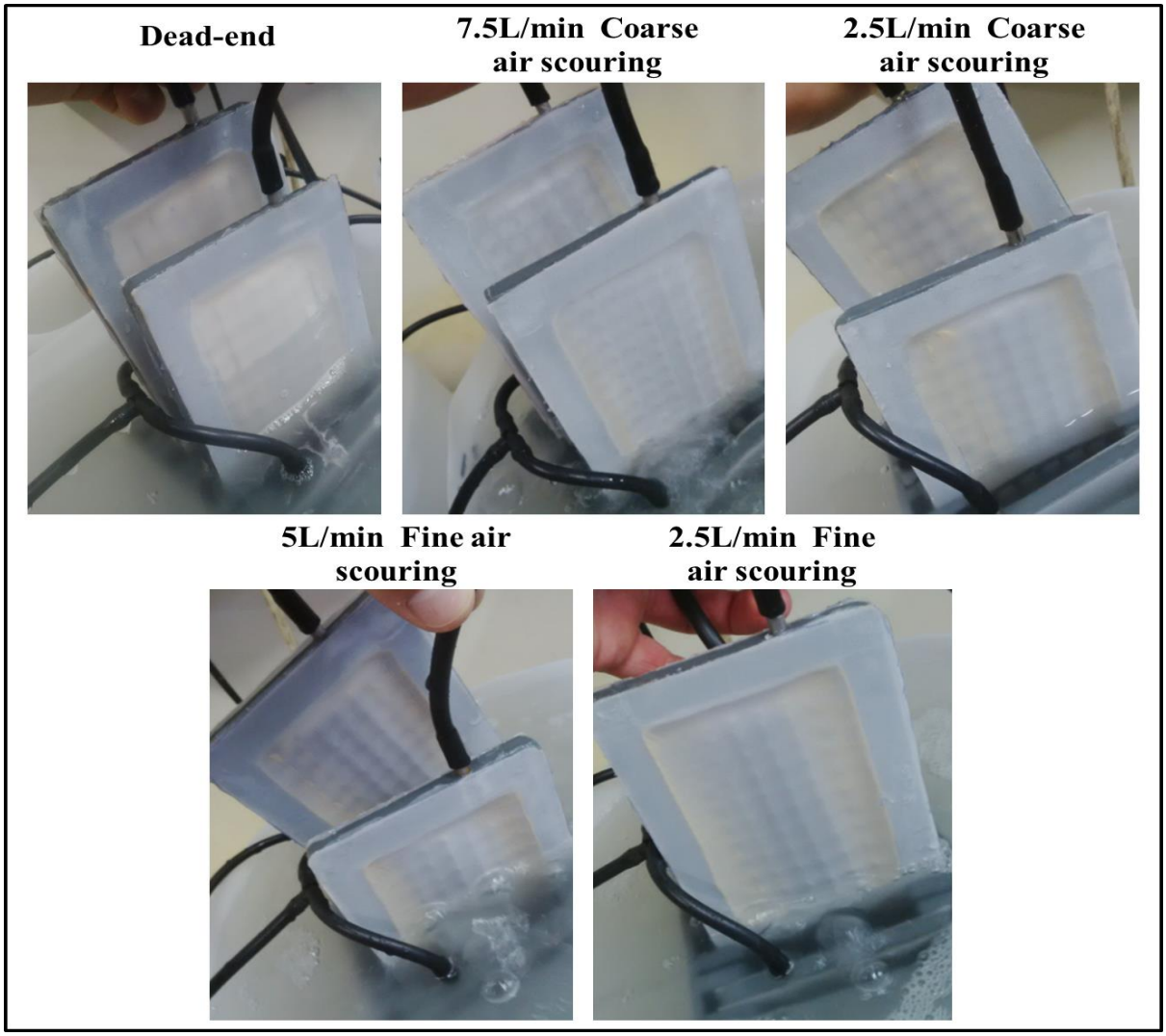

Figure 47: Visual fouling layer formation comparison.

The figure indicates the variations in the visual fouling layer present in the tests performed, where the fouling layer can be seen as the cream coloured layer on the membrane surfaces. It is apparent from the figure that the dead end test resulted in the membrane surface being evenly coated by the porous yeast fouling layer whereas the air scouring tests had varying amounts of the foulant on the opposing sides of the membrane. The remaining portions were as a result of air scouring dead-zones that formed. Both the 7.5 L/min coarse and the 5 L/min fine air scouring tests can be seen to have lower amounts of visual foulant, than both the lower 2.5 L/min air scouring tests performed. This was a result of the increased bubble column dispersion and higher defouling potential of the relatively higher air scouring flow rates. It is apparent from the visual inspection that the continuous air scouring tests resulted in a lower apparent visual surface fouling layer forming in comparison to the dead end test.

However, the lower apparent visual fouling of the air scouring tests resulted in a higher fouling layer resistance being achieved than the dead end test.

The second area of evaluation was the permeate quality achieved during the air scouring tests. The permeate quality of the coarse and fine continuous air scouring tests and the dead end operation test can be seen in Figure 48:

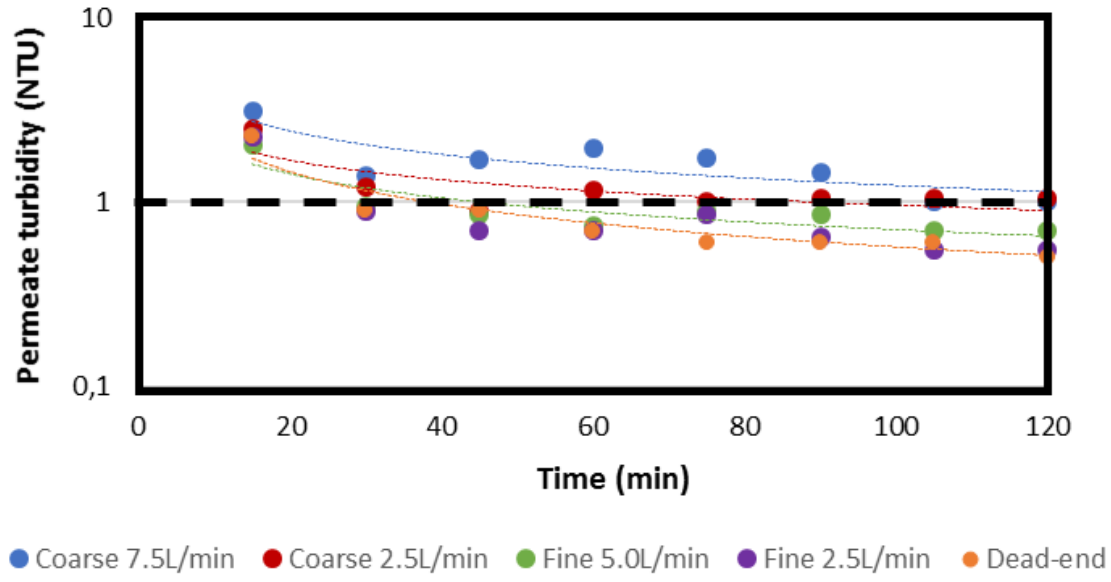

Figure 48: Continuous air scouring evaluation, for both coarse and fine air scouring, permeate turbidity, 120-minute operation period, 50 LMH permeate flux.

The figure indicates the change in the permeate turbidity as a function of the operation period for the tests performed. The figure indicates how the turbidity followed an overall decreasing trend during all the tests performed. The decrease in the permeate turbidity over time indicates that the formation of a fouling layer was occurring, despite the low visual surface fouling being present for the air scouring tests, as indicated in Figure 47. This indicates that the fouling layer was forming within the membrane pores, and not predominately on the membrane surface. This was a result of the air scouring preventing the formation of the surface fouling layer, by continually removing the larger yeast cells that would have been entrained on the membrane surface. The removal of the surface fouling layer promoted the smaller yeast cells to be entrained within the membrane pores, as opposed to being entrained within a porous surface fouling layer, resulting in partial or complete pore blocking. This was validated by the lower air scouring rates, resulting in a higher visual surface fouling layer forming whilst resulting in relatively lower permeate turbidity and lower comparative fouling layer resistance, than that of the higher flow rate tests.

From this investigation the importance of the formation of the porous fouling layer to the functioning of a woven fabric membrane, was apparent. The fouling layer played a role in decreasing the fouling layer resistance and improving the permeate turbidity. The purpose of investigating the effects of fine air scouring at a lower flow rate, 5.0 L/min as opposed to 7.5 L/min, was to lower the defouling potential of the air scouring whilst maintaining the high bubble column dispersion. This was performed in an effort manage the propagation of a slight fouling layer, as opposed to completely removing. This was to better control the dynamic operation of the membrane. However, both fine air scouring tests were unable to sufficiently manage the effects of the surface fouling layer. This was indicated by the higher fouling layer resistance than during the dead end test.

The trend of an increase in the air scouring rate increasing the defouling potential correlates with literature [2,3,7,8]. However, for the woven fabric it was determined that the increased defouling potential resulted in an increase in the fouling layer resistance. This was due to the dynamic operation of the membrane being compromised by the increased defouling potential, due to the continuous defouling measure applied [6]. Additionally, the sensitivity of the dynamic operation to flux enhancement methods was indicated, where tampering with the formation of the fouling layer using flux enhancement methods resulted in an increased fouling layer resistance and a decrease in the product quality.

6.2.2.5 Summary

The flux enhancement evaluation investigated the effects of continuous air scouring on a woven fabric membrane for reducing the effects of fouling. The results indicated the following:

- A higher fouling layer resistance was achieved for all the air scouring tests performed compared with the dead end operation tests.
- The higher air scouring flow rates resulted in an increased defouling potential, which resulted in an increased fouling layer resistance for the woven fabric.
- The continuous air scouring prevented the formation of the fouling layer by removing the large particles that were too large to pass through the membrane pores.
- This prevented the formation of the surface fouling layer which resulted in a higher resistance inter-pore fouling occurring.

- It was therefore apparent from the air scouring investigation that continuous defouling measures were not applicable to the woven fabric membrane due to the sensitivity of the dynamic operation.
- A move away from flux enhancement methods was required, due to the sensitivity of the dynamic operation to continuous and high frequency periodic flux enhancement methods.
- Low frequency cleaning methods was proposed as an alternative to the flux enhancement methods.

6.2.3 Conclusions

Dead end operation:

From the dead end operation investigation, it was concluded that the feed yeast suspension resulted in repeatable fouling layer formation as a result of the fouling layer resistance versus operation period relationship having minimal deviation amongst the replicate tests performed. The 2-hour dead end operation tests resulted in an average permeate of lower than 1 NTU.

The fouling layer formed as a result of the yeast feed suspension, was concluded to be fully reversible when defouled using a physical cleaning method. The test utilised an initial permeate flux of 100 LMH, which was operated dead end for 60-minutes, between physical cleans, over a 10-hour period. The approximate fouling layer resistance values of the subsequent dead end tests indicated the removal of both irreversible and irrecoverable fouling matter from within the woven fabric membrane pores. The fully reversible nature of the yeast suspension fouling layer, indicated the applicability of the suspension for use in the flux enhancement and cleaning methods evaluation.

Continuous air scouring evaluation:

An investigation was performed to evaluate the effects of using air scouring techniques on the woven fabric membrane as flux enhancement methods. From the results it was concluded that an increased fouling layer resistance was achieved by all the air scouring tests performed in comparison to the dead end tests.

The increased air scouring flow rate tests were concluded to result in an increased defouling potential of the membrane surface. This trend was validated by the visual evaluation of the membrane surfaces, indicating a lower degree of the surface fouling layer being present for

the relatively higher air flow rate tests. Additionally, a higher permeate turbidity was achieved for the higher flow rate air scouring tests concluding the removal of a higher degree of the porous fouling layer was achieved.

The increased defouling potential was concluded as being of detriment to the woven fabric membrane's functioning. The surface fouling layer was required to prevent the sub-pore size particles from passing through the membrane and/or becoming lodged within the membrane fibres. This resulted in an increased fouling layer resistance due to the increased occurrence of pore blocking. The fouling layer was formed as a result of the larger particles forming partial pore blocking of the membrane pores. This effectively decreased the effective pore size of the membrane. The continuous air scouring prevented the formation of this layer by removing the large particles that were too large to pass through the membrane pores.

The lower flow rate fine air scouring tests were unable to manage the propagation of the fouling layer better the respective higher flow coarse air scouring tests. This was concluded to be as a result of the sensitivity of the dynamic operation to changes in the fouling layer formation, due to the use of continuous air scouring. Flux enhancement methods were concluded not be applicable to the woven fabric membrane due to the sensitivity of the dynamic operation of the membrane to continuous or high frequency defouling measures.

For these reasons, it was concluded that a move away from flux enhancement was required, and that more emphasis should be placed on the complete reversal of the effects of fouling using different cleaning methods. The dead end operation tests were concluded to result in lower effective fouling layer resistance and higher product quality than any of the flux enhancement methods investigated, including the preliminary flux enhancement methods evaluated.

6.3 Cleaning evaluation

6.3.1 Evaluation of standard flat sheet membrane cleaning

An investigation was performed to evaluate the effects of different cleaning methods on the woven fabric membrane. The cleaning evaluation was proposed over further evaluation of flux enhancement methods due to the cleaning methods not affecting the dynamic functioning of the membrane. This was based on the results of the flux enhancement evaluation indicating the importance of the fouling layer formation for both reducing the fouling layer resistance

and ensuring a permeate turbidity of less than 1 NTU. The cleaning measure proposed was an ex-situ chemically aided gravity backflush coupled with continuous air scouring. The preliminary defouling evaluation performed, described in Chapter 6.1 Preliminary evaluation of flux enhancement methods, indicated the enhanced cleaning potential of combining periodic air scouring and gravity backflush methods. The benefits of performing the cleaning method ex-situ allowed for the addition of a chemical aid to the backflush fluid.

The inherent differences between the use of continuous air scouring as a flux enhancement method compared to its use as a cleaning method must be noted. For flux enhancement purposes, the air scouring was required to reduce the propagation of the fouling layer. This was required to maintain the dynamic operation of the membrane. For cleaning purposes, the air scouring was required to completely reverse the effects of fouling, as the dynamic operation was achieved using the dead end operation. Therefore, to maintain the dead end operation between cleaning cycles, complete reversal of the effects of fouling was required to achieve prolonged membrane operation.

The strategy for this investigation was to start with the most rigorous cleaning method first. If this method was successful, further evaluation of lower intensity alternatives would be investigated.

6.3.1.1 Investigation

The investigation evaluated the effects of a chemically aided (sodium hypochlorite) gravity backflush combined with continuous air scouring. The effectiveness of the cleaning method was evaluated by comparing the fouling layer resistance of a dead end operation test performed before and after the cleaning method. Complete reversal of the effects of fouling would be indicated by the fouling layer resistance of the subsequent dead end tests being approximate in rate and magnitude, for the duration of the operation period. An ineffective cleaning method was considered when the fouling layer resistance was significantly larger than that of the previous dead end operation test.

The effects of varying the permeate flux magnitude were also investigated to indicate whether an increased defouling potential was achieved by a lower permeate flux dead end test.

An experiment was performed to determine the upper limit of the gravity backflush intensity that the standard membrane modules could withstand, before the cleaning evaluation was

performed. The intensity was varied by increasing the differential height between the backflush reservoir and the top of the membrane module. The results of the experiment indicted that a maximum pressure of approximately 3 kPa (positive pressure operation) could be withstood by the membrane modules. This resulted in a height differential of 260 mm being selected for the gravity backflush reservoir. A higher backflush intensity resulted in failure of the woven fabric bond to the PVC frame.

6.3.1.2 Apparatus

The apparatus utilised for this phase of the defouling evaluation was the same as the apparatus utilised in 6.2.1 The development of the dead end operation protocol.

6.3.1.3 Methodology

The methodology followed for this phase of the investigation is split into two sections, namely the dead end operation and the cleaning operation procedure. The dead end operation follows the same procedure as outlined in Chapter 6.2.1 The development of the dead end operation protocol.

Cleaning operation procedure:

1) The cleaning holding vessel was filled with RO water.
2) The membrane stand was lowered into the holding vessel and left for 10 minutes to allow the pressure within the membrane modules to equalise.
3) The air pump was turned on and set to a flow of 7.5 L/min.
4) The chemical aided backflush solution was prepared using 1 ml/L of 3.5% m/vol sodium hypochlorite resulting in a ~25 PPM CLO^- solution.
5) The gravity backflush reservoir was filled with the prepared solution to the upper limit, 500 ml, and topped up as required to maintain the level within the vessel to maximise the backflush TMP.
6) The backflush flux was recorded in 5-minute intervals.
7) The cleaning method was performed for a duration of 2-hours.

Once the cleaning cycle was complete, the membrane modules were returned to the feed suspension holding vessel and the dead end operation test procedure repeated. To reduce the potential of irrecoverable fouling affecting the results of the tests performed, two new membrane modules were used for each of the series of tests.

The cleaning methods were evaluated by comparing the fouling layer resistance of subsequent dead end operation tests. The format utilised for the cleaning methods evaluation was as follows: A dead end operation test 'Run 1' was performed followed by the particular cleaning method, this was then followed by the dead end operation test 'Run 2'. This test was then followed by the same cleaning method which was lastly followed by the dead end operation test 'Run 3'. Two orange lines were added to the fouling layer resistance curves. This was to indicate the time taken for the subsequent dead end operation test to achieve approximately the same fouling layer resistance magnitude as was achieved over the 2-hour operation period during the previous test. This gave an indication of the extent to which the fouling was reversed by the cleaning method applied.

6.3.1.4 Results and discussion

The dead end operation fouling layer resistance curves for two chemically-aided gravity backflush coupled with coarse air scouring tests can be seen in Figure 49:

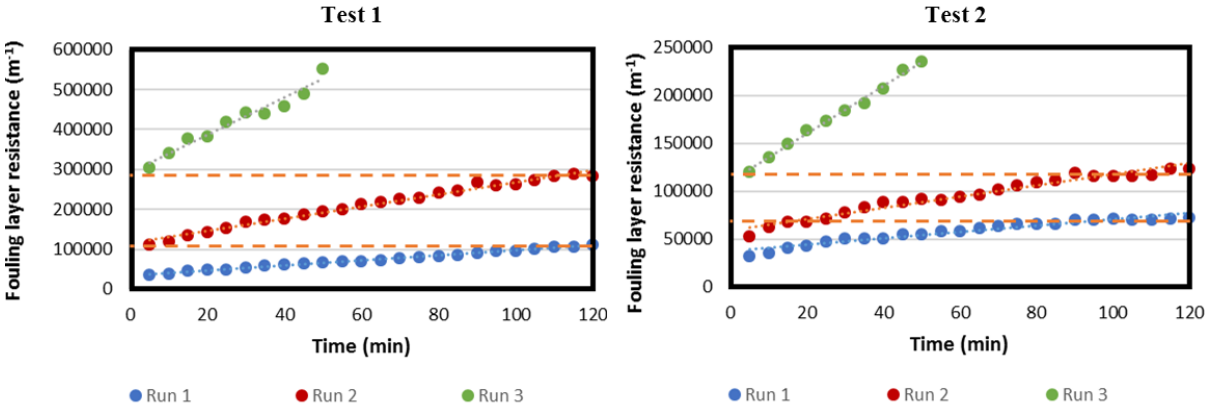

Figure 49: Chemical aided gravity backflush coupled with air scour cleaning evaluation, 2-hour duration.

The figure indicates the change in the fouling layer resistance as a function of the operation period for three subsequent dead end operation tests. The figure indicates two different tests: 'Test 1' and 'Test 2'. 'Test 1' utilised an initial flux of 50 LMH and 'Test 2' utilised an initial flux of 40 LMH. From the figure it is apparent that the subsequent dead end operation tests resulted in higher fouling layer resistance magnitudes over the 2-hour period for both 'Test 1' and 'Test 2'. This was an indication of incomplete reversal of the effects of fouling on the membrane using the selected cleaning method. 'Test 2' resulted in a slight recovery of the effect of fouling between 'Run 1'and 'Run 2'. This is indicated by the lower initial fouling layer resistance of 'Run 2' in comparison to the final fouling layer resistance of 'Run 1'. However, this recovery was negligible as the 'Run 2' fouling layer resistance surpassed the

'Run 1' fouling layer resistance within 30 minutes of operation. The comparison of the 'Run 2' and 'Run 3' of the 'Test 2', indicates no recovery of the effect of fouling. This correlates with literature where an ineffective removal of the effects of reversible fouling resulted in the formation of irreversible fouling [4].

To further investigate the increased defouling potential of the 'Test 2' compared to the 'Test 1' as a result of the lower permeate flux increasing the defouling potential, an investigation was performed where the duration of the dead end test was dictated by the time taken to achieve a TMP of 10 kPa. The results of two tests, 'Test 1'and 'Test 2', where the dead end operation tests were performed for the duration required to achieve a TMP of 10 kPa, each with an initial flux of 50 LMH, can be seen in Figure 50:

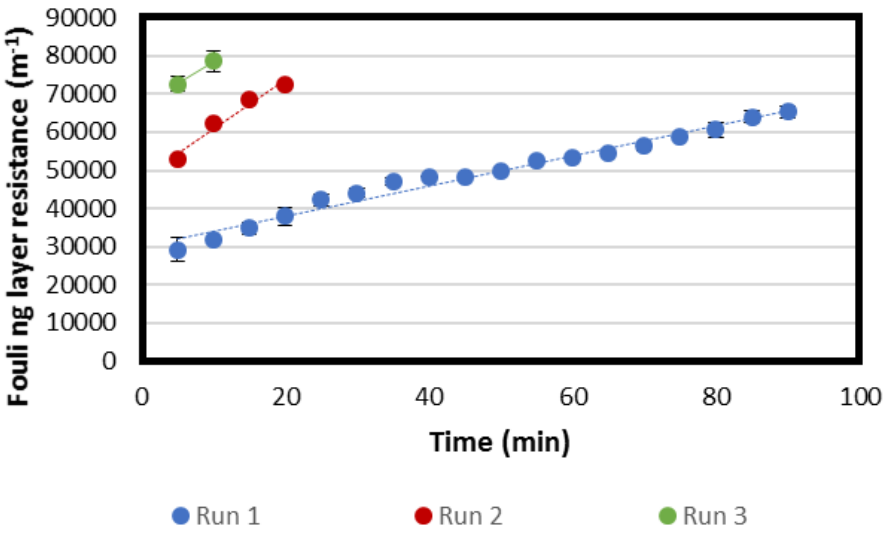

Figure 50: Ex-situ chemical aided gravity backflush and air scour, 10 kPa TMP operation limit between cleans.

The figure indicates the change in the fouling layer resistance as a function of the operation time for three subsequent dead end operation tests. Error bars have been utilised to indicate the standard deviation between the two repeat tests. From the figure it is apparent that the recovery between the subsequent dead end operation tests was minimal, with the 'Run 2' test taking approximately 15-minutes to achieve the same fouling layer resistance magnitude as the final fouling layer resistance of the 'Run 1' for both tests. There was relatively no recovery between the fouling layer resistance for the 'Run 3'and the 'Run 2' tests. This indicated the effects of a compounding reversible fouling layer as a result of the ineffective cleaning measure.

From the investigation it is apparent that a lower fouling layer resistance magnitude resulted in a marginally larger recovery of the effects of fouling between the subsequent dead end operation tests. This correlates with literature where the membrane backflush defouling potential was dependent on the membrane operating parameters, such as TMP and Flux, and the point in the operation cycle that the backflush was performed [8]. The intensity of the cleaning method investigated was insufficient for the fouling layer formed, therefore a higher intensity cleaning method was required.

6.3.1.5 Summary

The investigation was performed to evaluate the effects of cleaning subsequent dead end operation tests using an ex-situ chemically-aided gravity backflush combined with a coarse air scouring cleaning method. During the investigation it was found that:

- The force exerted by this cleaning method was unable to dislodge the entrained particulate matter from within the membrane pores.
- When a lower force was applied to entrain the particulate matter within the membrane pores a marginal increase in the defouling potential was achieved by the cleaning method.
- There is a need to investigate higher intensity cleaning methods.

6.3.2 Intensified cleaning evaluation

The results of the cleaning evaluation performed above indicated the limitations of the cleaning method for reversing the effects of fouling on the woven fabric flat sheet membrane. This was concluded to be a result of the low cleaning intensity applied by this method, in comparison to the force applied to entrain the particulate matter within the membrane pores.

A revised membrane module was required that would allow for a greater backflush intensity to be tested without rupturing the membrane modules. An alternative glue for bonding the woven fabric material to the PVC membrane frame had already been investigated, where the currently utilised glue was selected as described in Appendix B: Membrane to PVC frame glue strength evaluation. Therefore, a securing device was proposed to reinforce the bond between the woven material and the PVC frame. This would allow for an increased backflush flow to be tested in order to match the suction force used to entrain the particulate matter within the membrane fibres.

6.3.2.1 Investigation

The investigation evaluated the effects of a chemical-aided backflush, where the backflush flow was tested over a range of 1 to 5 times that of the initial 50 LMH dead end operation flux. The effectiveness of the intensified backflush methods was evaluated by comparing the fouling layer resistance of dead end operation tests performed before and after the cleaning method was applied. Complete reversal of the effects of fouling would be indicated by the fouling layer resistance of the subsequent dead end tests being approximate in rate and magnitude, for the duration of the operation period. An ineffective cleaning method was considered when the fouling layer resistance was significantly larger than that of the previous dead end operation and/or an inability of the subsequent dead end tests to achieve a permeate turbidity of less than 1 NTU. This would indicate potential deformation of the membrane material due to the intensified cleaning method applied.

Samples of the different tests were evaluated using SEM imagine at 3000X magnification. This evaluation was performed to rule out a potential degradation of the membrane material integrity being the resultant cause of the increased fouling layer resistance as opposed to an ineffective cleaning method.

6.3.2.2 Apparatus

A revised membrane module was required to prevent the membrane module from rupturing when operated at the elevated backflush flow. A membrane module reinforcing frame was developed for this purpose. The use of the reinforcing frame resulted in the membrane no longer working within a flatsheet membrane stand. For this reason, a single membrane apparatus was designed as can be seen in Figure 51:

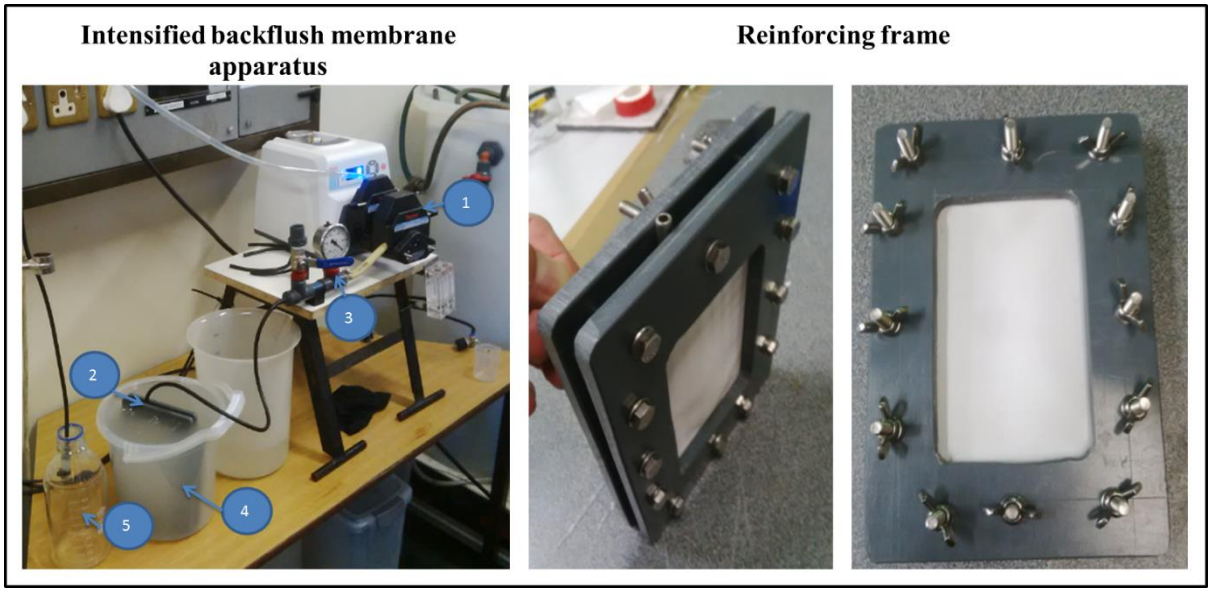

Figure 51: Photograph of the intensified backflush membrane apparatus and reinforced membrane frame.

A description of the various apparatus components is given below:

1: The permeate driving force was provided by a MasterFlex 6-600rpm peristaltic pump.

2: The reinforced frame.

3: The TMP was monitored using a non-stepped vacuum gauge with a pressure range of -100-0 kPa.

4: A 5L holding vessel was utilised.

5: The permeate was collected in a separated vessel and not recycled to the membranes.

The membrane pressure curve for the reinforced membrane apparatus can be seen in Figure 52, with the calculated membrane pressure linear regression model indicated:

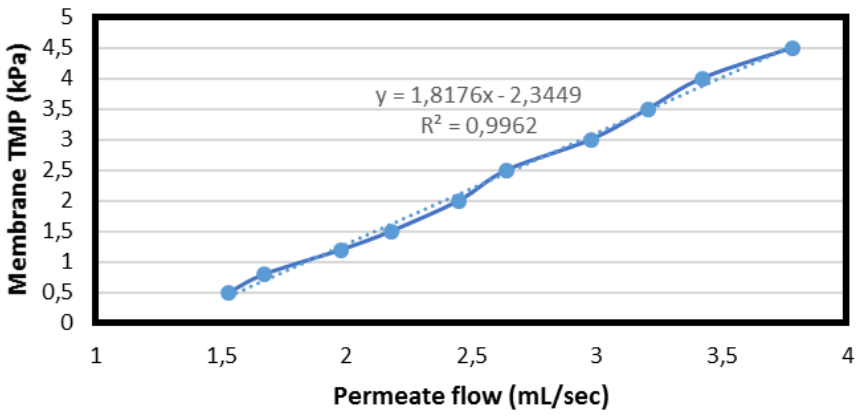

Figure 52: Membrane TMP linear regression

6.3.2.3 Methodology

The methodology followed during this phase of the investigation was split into two sections, namely the dead end operation and the intensified cleaning operation. The dead end operation followed the same procedure as outlined in Chapter 6.2.1 The development of the dead end operation protocol.

Intensified cleaning procedure:

1) The cleaning holding vessel was filled with RO water.
2) The reinforced membrane module was lowered into the holding vessel and left for 10 minutes to allow the pressure within the membrane to equalise.
3) The chemically aided backflush solution was prepared using 1 ml/L of 3.5% m/vol sodium hypochlorite resulting in a ~25 PPM ClO^- solution.
4) The backflush vessel was filled with 4 L of the prepared solution.
5) The pump flow direction was reversed and the desired backflush flow set.
6) The backflush flux was recorded in 5-minute intervals.
7) The test was performed for the duration required to backflush the prepared 4 L of solution.

Once complete, the membrane was returned to the feed suspension holding vessel and the dead end operation test procedure repeated.

The cleaning methods were evaluated by comparing the fouling layer resistance of subsequent dead end operation tests. The format utilised for the subsequent cleaning method was as follows: A dead end operation test ‘Run 1’ was performed followed by the particular cleaning method, this was then followed by the dead end operation test ‘Run 2’. This test was then followed by the same cleaning method which was followed by the dead end operation test ‘Run 3’. Two orange lines were added to the fouling layer resistance curves. This was to indicate the time taken for the subsequent dead end operation test to achieve approximately the same final fouling layer resistance magnitude as was achieved over the 2-hour operation period of the previous test. This gave an indication of the extent to which the fouling was reversed by the cleaning method applied.

6.3.2.4 Results and discussion

The dead end operation fouling layer resistance curves for a chemically aided backflush performed at 1X the forward initial pure water flux, 50 LMH backflush flux, can be seen in Figure 53:

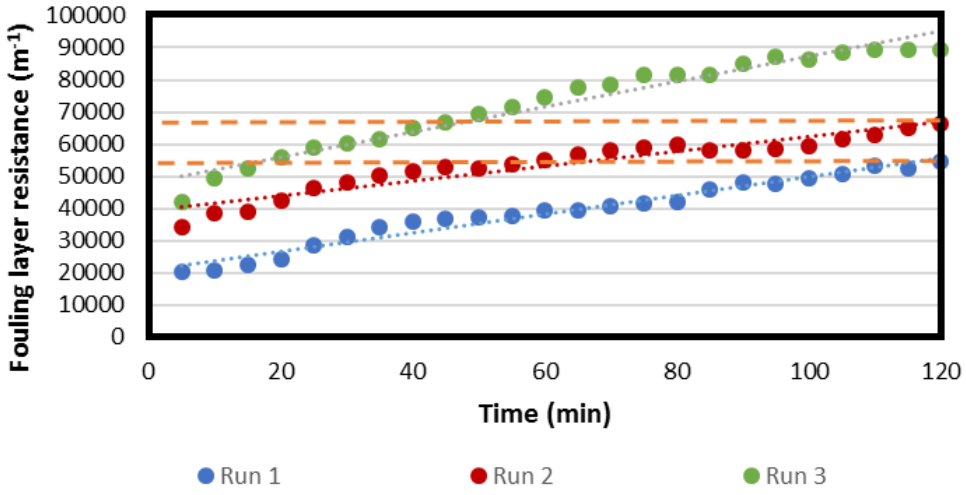

Figure 53: Ex-situ chemical aided backflush, 1 times forward flux intensity cleaning method.

The figure indicates the change in the fouling layer resistance as a function of the operation time for three subsequent dead end operation tests. From the figure it is apparent that the subsequent dead end operation tests resulted in higher fouling layer resistance magnitudes over the 2-hour operation period. This is an indication of incomplete defouling of the membrane module using the intensified cleaning method performed. However, it can be seen that it took the 'Run 2' dead end test approximately 60-minutes to achieve the same fouling layer resistance value as that of the final fouling layer resistance achieved by the 'Run 1' test, as indicated by the lower dashed orange line. The 'Run 3' dead end test took approximately 45-minutes to achieve the same fouling layer resistance value as that of the final fouling layer resistance achieved by the 'Run 2' test, as indicated by the upper orange dashed line.

These results indicate how an increased defouling potential is achieved by the intensified backflush cleaning method compared with the results achieved using the gravity fed backflush.

The dead end operation fouling layer resistance curves for a chemically aided backflush performed at 2X the forward initial flux, 100 LMH backflush flux, can be seen in Figure 54:

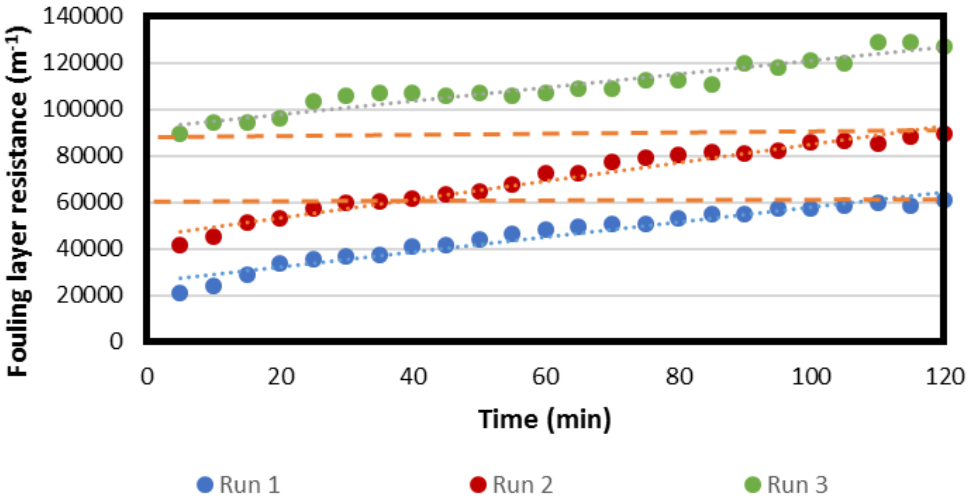

Figure 54: Ex-situ chemical aided backflush, 2 times forward flux intensity cleaning method.

The figure indicates the change in the fouling layer resistance as a function of the operation time for three subsequent dead end operation tests. From the figure it is apparent that the subsequent dead end operation tests resulted in higher fouling layer resistance magnitudes over the 2-hour operation period. This is an indication of incomplete defouling of the membrane module using the intensified cleaning method performed. It can be seen that it took the 'Run 2' dead end test approximately 30 minutes to achieve the same fouling layer resistance value as that of the final fouling layer resistance achieved by the 'Run 1' test, as indicated by the lower dashed orange line. And the 'Run 3' dead end had no potential defouling recovery, as the tests initial fouling layer resistance magnitude was approximately the same as the final fouling layer resistance values achieved at the end of the 'Run 2' dead end operation test, as indicated by the upper dashed orange line.

The performance of the 2X backflush cleaning method can be seen to result in a lower defouling effectiveness than that of the 1X backflush test. This is indicated by the decrease in the time taken to achieve the same fouling layer resistance value as the previous dead end operation tests. The 1X backflush and 2X backflush 'Run 2' dead end operation tests took 60-minutes and 30-minutes respectively to achieve the same fouling layer resistance as the respective 'Run 1' dead end test.

The dead end operation fouling layer resistance curves for a chemically aided backflush performed at 5X the forward initial flux, 250 LMH backflush flux, can be seen in Figure 55:

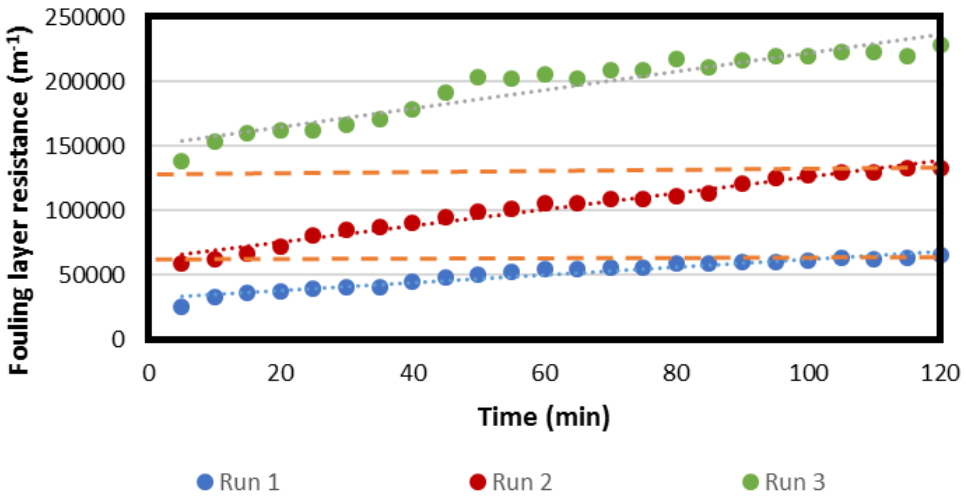

Figure 55: Ex-situ chemical aided backflush and air scour, 5 times forward flux intensity cleaning method.

The figure indicates the change in the fouling layer resistance as a function of the operation time for three subsequent dead end operation tests. From the figure it is apparent that the subsequent dead end operation tests resulted in higher fouling layer resistance magnitudes over the 2-hour operation period. This is an indication of incomplete defouling of the membrane module using the intensified cleaning method performed. It can be seen that there was no defouling recovery between the subsequent dead end tests performed. This is indicated by the tests initial fouling layer resistance values being approximately the same as the final fouling layer resistance values achieved at the end of the previous dead end operation test. This is indicated by the dashed orange lines.

The turbidity of the cumulative permeate volume collected for the 'Run 1', 'Run 2' and 'Run 3' dead end operation tests, were 0.9, 0,7 and 0.6 NTU respectively. This was sufficient to indicate that there was no apparent loss in the woven fabrics material integrity due to being operated at a backflush flux of 250 LMH. The gradual decrease in turbidity of the subsequent tests indicated an increased fouling layer formation between the tests. This was validated by the increased fouling layer resistance during the respective tests. Further evaluation of the membrane samples was required to conclude the results of the low defouling potential of the intensified defouling methods.

The results of the SEM evaluation of the intensified cleaning methods effects on the woven fabric membrane material can be seen in Figure 56:

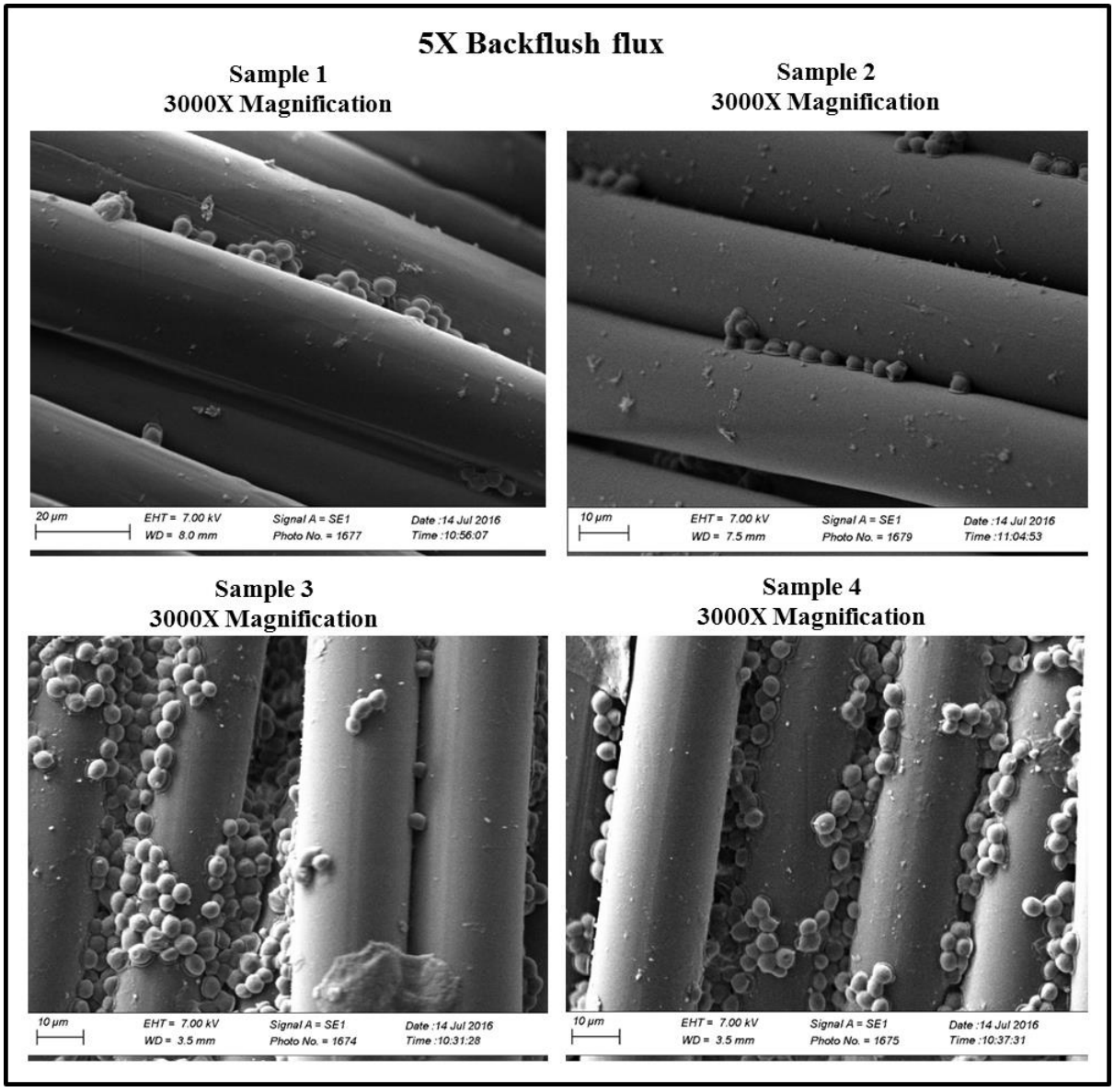

Figure 56: SEM evaluation of the intensified cleaning methods fouling phenomena, ZEISS EVO MA15VP, CAF Stellenbosch University.

The figure indicates SEM images of the 5X backflush. The round particulate matter present in SEM evaluated samples are yeast cells that remained entrained within the membrane threads. From the figure it is apparent that the surface fouling layer was effectively removed as the majority of the indicated fouling in the figure was within the membrane pores. The inter-pore foulant material is indicated by the yeast cells that are relatively out of focus in the figure. This indicates that the cells are present at varying planes to the membrane surface, as the membrane surface was set as the plane of focus for the SEM detector. The inter-pore fouling is referred to as irreversible fouling in the membrane industry as a result of the inherent difficulty to remove such fouling phenomena [4]. The tortuous nature of the woven fabric's pores promotes the difficulty associated with removing irreversible fouling.

Literature indicates the most effective means of removing irreversible fouling is to dissolve it using an acidic solution, such as nitric acid [8]. However, the objectives of this study was to develop a membrane suited to rural environments therefore making such cleaning methods unsuitable for the membrane's intended use. The tortuous pores of the woven fabric therefore result in the increased separation ability of the woven fabric, however at the inherent cost of high quantities of difficult to remove irreversible fouling forming.

The figure allows the conclusion to be made that both samples were not effectively defouled due to the presence of yeast cells within the membrane threads.

6.3.2.5 Summary

The intensified backflush investigations performed indicated the follow:

- The intensified cleaning methods were unable to effectively reverse the effects of fouling on the woven fabric membranes.
- The backflush fluxes tested were only able to recover relatively small portions of the flux and TMP lost to the effects of fouling.
- This was the result of the presence of inter-pore fouling.
- The inter-pore fouling is commonly known as irreversible fouling.
- The woven fabric pore size range resulted in the woven fabric being classed as a microfilter pore membrane, however, this investigation was aimed at achieving ultrafiltration level product quality.
- The woven fabric was able to achieve the required separation ability based on the lower than 1 NTU permeate quality.
- This resulted in irreversible fouling forming.

6.3.3 Conclusions

Evaluation of standard flat sheet membrane cleaning:

The investigation was performed to determine the effects of using a chemically aided (~25 PPM Sodium hypochlorite) gravity backflush combined with a coarse air scouring cleaning method on subsequent dead end operation tests. From the investigation it is apparent that the force exerted by this cleaning method was unable to dislodge the entrained particulate matter from within the membrane pores. From the investigation it was noted that when a lower force was applied to entrain the particulate matter within the membrane pores a marginally higher

defouling potential by the cleaning method was achieved. The relatively higher defouling potential was apparent from the larger decrease in fouling layer resistance between the initial and final fouling layer resistance magnitudes of two subsequent dead end operation tests.

From this investigation it was concluded that further evaluation of cleaning methods that resulted in a higher relative force being applied to dislodge the entrained particulate matter from within the membrane pores required evaluation. Therefore, a revision of the membrane modules was required to prevent the modules from rupturing due to a higher intensity cleaning method being applied.

Evaluation of intensified cleaning:

The intensified backflush investigations performed at backflush magnitudes of 1X, 2X and 5X the initial forward flux magnitude of 50 LMH, as a whole, were unable to effectively reverse the effects of fouling on the woven fabric membrane modules. It was expected that the increased backflush flow would effectively remove the particles entrained within the membrane pores. However, the backflush fluxes tested were only able to recover relatively small portions of the flux and TMP lost to the effects of fouling. This was indicated by an inability of the membrane to achieve the same initial fouling layer resistance as the previous dead end tests.

The low recovery of the effects of fouling were concluded not to warrant the extreme measures required to achieve them. The higher intensity backflush flow required the use of a securing frame and the same volume fluid recovered through the permeate to be passed back through the membrane pores. The low recovery of these methods was a result of a large portion of the fouling layer that formed, forming as irreversible fouling within the membrane pores. This form of fouling requires the use of physical cleaning or dissolving of the fouling matter out of the membrane pores.

The use of extreme measures to reverse the effects of fouling were not in line with meeting the objectives of this study as a membrane technology was required to meet the demands of rural and peri-urban economies.

6.4 Overall conclusion of the flux enhancement and cleaning evaluation

The dynamic operation of the membrane was concluded to be crucial to the operation of the woven fabric membrane, where any controlling measure applied to manage the propagation of the fouling layer resulted in a higher fouling layer resistance occurring. This indicated how any continuous or high frequency periodic flux enhancement method applied to the woven fabric resulted in both a decreased product quality and an increased fouling layer resistance being formed.

The dead end operation of the membrane was seen as the most idealist operating strategy due to the lower fouling layer resistance and higher product quality being achieved. The cleaning measures were applied only after a predetermined dead end operation to reverse the effects of fouling that occurred. However, the cleaning methods were unable to effectively reverse the effects of fouling on the woven fabric membrane modules. This was concluded to be a result of the feed suspension forming larger quantities of irreversible fouling within the membrane pores. The irreversible fouling formed as a result of the requirement of the woven fabric to separate sub-pore size particulate matter. It was concluded that a physical orientated cleaning method was capable of completely reversing the effects of irreversible fouling on the woven fabric membrane modules. The physical cleaning method was able to dislodge the particulate matter entrained within the woven fabric's highly tortuous membrane pores.

6.5 References

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Chapter 7: Overall conclusions and recommendations

Overview

This chapter is split into two sections, namely 7.1 Overall Conclusion and 7.2 Recommendations for further studies. In this chapter the main conclusions drawn from the investigations performed in line with fulfilling the objectives of this study are listed and potential areas that have potential for further investigations are mentioned.

7.1 Conclusions

The conclusions drawn from the investigations performed, in line with meeting the objectives of this study, are discussed below.

7.1.1 Characterisation evaluation

During the characterisation procedure the woven fabric membrane was classified as a microfilter membrane. It was concluded that the effects of fouling were able to result in a pore narrowing of the fabric. This indicated how the formation of fouling layer could improve the separation ability of the woven fabric membrane. From the characterisation procedure it was apparent that the woven fabric would require operation as a dynamic membrane process to successfully meet the separation requirements of a water treatment process.

The membrane morphology indicated the woven nature of the woven fabric membrane resulted in a complex multilayer membrane material being formed. It was concluded that this multi-layering effect of the fabric promoted the use of the woven fabric for dynamic operation as a result of the highly tortuous membrane pores formed.

7.1.2 Performance evaluation

The complex multi-layering of the woven fabric membrane was concluded to result in the different trends exhibited in the product quality and the fouling layer resistance evaluations performed. A 1 µm particle should not be capable of being entrained within a larger pore. However, the complex multi-layering of the woven fabric resulted in the formation of the membrane pores being highly tortuous. The tortuous pores promoted the occurrence of pore narrowing with sub-pore sized particulate matter as indicated by the woven fabric producing a product quality of lower than 1 NTU as well as an increase in the fouling layer resistance.

This indicated that an entrainment of these sub-pore size particles was occurring within the woven fabric membrane pores.

It was therefore this unique complex multi-layering effect of the woven fabric that resulted in the woven fabric not correlating with the functioning of other MF and UF membranes. It was for this reason that the microfilter categorised woven fabric membrane was capable of achieving UF degrees of separation ability. The property of the woven fabric membrane to remove sub-pore size suspended matter (<1µm) indicated the applicability of the membrane's separation ability to water treatment processes and how a pore size estimate of the fabric could not be used to pre-empt the separations capability of the fabric.

7.1.3 Flux enhancement and cleaning evaluation

The dynamic operation of the membrane was concluded to be crucial to the operation of the woven fabric membrane while any controlling measure applied to manage the propagation of the fouling layer resulted in a higher fouling layer resistance occurring. This indicated how any continuous or high frequency periodic flux enhancement method applied to the woven fabric resulted in both a decreased product quality and an increased fouling layer resistance being formed.

The dead end operation of the membrane was seen as the most ideal operating strategy due to the lower fouling layer resistance and higher product quality being achieved, while the cleaning measures were applied only after a predetermined dead end operation to reverse the effects of fouling that occurred. However, the cleaning methods were unable to effectively reverse the effects of fouling on the woven fabric membrane modules. This was concluded to be a result of the feed suspension forming larger quantities of irreversible fouling within the membrane pores. The irreversible fouling formed as a result of the requirement of the woven fabric to separate sub-pore size particulate matter. It was concluded that a physical orientated cleaning method was the only method, that was in-line with the requirements of this study, capable of completely reversing the effects of irreversible fouling on the woven fabric membrane modules.

7.1.4 Overall conclusion of the study

The overall goal of the study, of evaluating the woven fabric's suitability for use in water treatment applications within rural and peri-urban economies, was achieved.

The study indicated how the woven fabric successfully achieved the required separation efficiency for a range of different feed suspension properties, as would be required for a water treatment process.

It was concluded that the large pore size range of the membrane resulted in large quantities of irreversible fouling forming within the membrane pores due to the size range of the components requiring separation. This limited the effective cleaning methods to physical cleaning methods to reverse the effects of the irreversible fouling. The durability of the membrane which can withstand physical cleaning methods indicated its suitability to water treatment processes within rural and peri-urban economies. Additionally, a physical cleaning method is of more benefit in a rural setting in comparison to a potentially hazardous chemical aided cleaning method.

Therefore, the woven fabric was concluded to be a suitable membrane for water treatment processes that are required within rural and peri-urban environments.

7.2 Recommendations for further studies

There are two prominent areas requiring further investigation, namely the woven fabric material and the woven fabric flat sheet membrane design and operation.

7.2.1 Woven fabric flat sheet membrane design

The woven fabric operation and defouling investigations performed as part of the various investigations indicated the need for further design revisions in the following areas:

Improved glue or clamping mechanisms used to better bond the woven fabric material to the PVC frame. This is required to overcome some of the operation constraints and limitations that arose with the current membrane module configuration proposed.

Automation of the physical scrubbing mechanism used, which successfully defouled the woven fabric flat sheet membrane modules, would be of benefit to the rural and peri-urban economies. This could be achieved utilising a series of high pressure water jets. The method used to distribute the water jets uniformly over the membrane surface may prove problematic and highly energy intensive. An automated scrubbing brush could perhaps be utilised as a means of automating the physical process.

7.2.2 Woven fabric material further research

From the various investigations performed it was apparent that the woven fabric expanded under both suction and positive operation operating modes. The inherent issues with such behaviour was the effects it had on the hydrodynamic design, in order to compensate for the expansion of the woven fabric. The dissertation evaluated the characterisation of the woven fabric for membrane use, where the woven fabric was selected as a fixed design element that was not revised as part of the various investigations. However, it was made apparent how a revision to the fabric that evaluated impregnating the material with a porous support layer, would be of benefit to the membrane functioning. The increased rigidity, that the impregnated porous support layer would provide, would allow for performing a pulsed backflush. The pulsed backflush was indicated as resulting in relatively low defouling recovery as a result of the expansion of the woven fabric absorbing the pulsed backflush energy as opposed to it being instilled on the entrained particulate matter. Additionally, the packing density of the membrane modules would increase due to the reduced expansion of the woven fabric.

Recommendations such as revising the woven pattern utilised by investigating a tighter weave pattern, would require that the entire woven fabric material be revised. This would be at the potential cost of losing the inherent woven fabric membrane properties that resulted in its original conception as a potential membrane material. A tighter weave would require revision of the membrane strands within the threads to achieve a tighter packing of the threads, to effectively reduce the woven fabric effective pore size range.

Appendices

Appendix A: Validation of bubble point experimentation

An ANOVA Single Factor Analysis of the data collected for the largest and smallest pore size calculations was performed, on the results of the 8 unused membranes samples. The point at which the largest pore pressure was recorded was elementary, as it was based on the pressure at which the first bubble stream was witnessed. Whereas, the smallest pore pressure was less straight forward, as it was less apparent when the entire membrane sample became completely fluidised. The different points at which the pressures were recorded for these two measures are illustrated in Chapter 4.1.1.1 The bubble gas transport method, Figure 19. The ANOVA was performed on both the largest and smallest pore measurements separately, as opposed to an average value of the two measurements.

The ANOVA Single Factor Analysis was performed at a 95% confidence level ($\alpha = 0.05$). The two tests were based on the following null and alternative hypothesises:

Null Hypothesis (H_0): $\mu_1 = \mu_2 = \cdots = \mu_n$

Alternative Hypothesis (H_a): $\mu_1 \neq \mu_2 \neq \cdots \neq \mu_n$

The results of the summarised ANOVA analysis can be seen in Table 7:

Table 7: ANOVA Single Factor Analysis of the bubble transport experimentation procedure.

Anova analysis	F_0	P-value	$F_{critical}$
Largest pore	0.654	0.707	2.657
Smallest pore	1.127	0.394	2.657

The table indicates the F-test statistic (F_0), the F-critical statistic ($F_{critical}$) and the P-value for both the largest and smallest pore size values recorded, for the 8 unused new membrane samples, where 3 repetitions were performed for each of the samples (8x3).

Based on the results of the ANOVA analysis performed, on the bubble transport experimental procedure, the Null Hypothesis cannot be rejected for both the largest and smallest pore size calculations. The implications of not being able to reject the Null Hypothesis is that the variation of the average pore size between the sample replications and between the samples was not significant. Therefore, the method and apparatus used to evaluate the membrane pore sizes calculated, using the 8 membrane samples, was sufficiently replicable based on a 95% confidence level.

Appendix B: Membrane to PVC frame glue strength evaluation

In this section the investigation used to select a glue for bonding the woven fabric material to the PVC membrane frame is covered.

B.1) Investigation

The formation of an effective bond between the PVC frame and the polyester woven fabric was an important step in the design of a woven fabric membrane. An insufficient bond between these two materials would result in potential membrane operation limitations that would not be as a result of limitations of the fabric, but rather limitations imposed by the membrane module design. The woven fabric flat sheet membrane module undergoes two different operation modes. The first mode is during membrane operation which applies a suction pressure to draw the permeate through the membrane pores. The second mode is positive pressure operation, as a result of a membrane backflush orientated defouling strategies. These variations in the forces being applied to the glue layer between the woven fabric and the PVC frame are indicated in Figure 57:

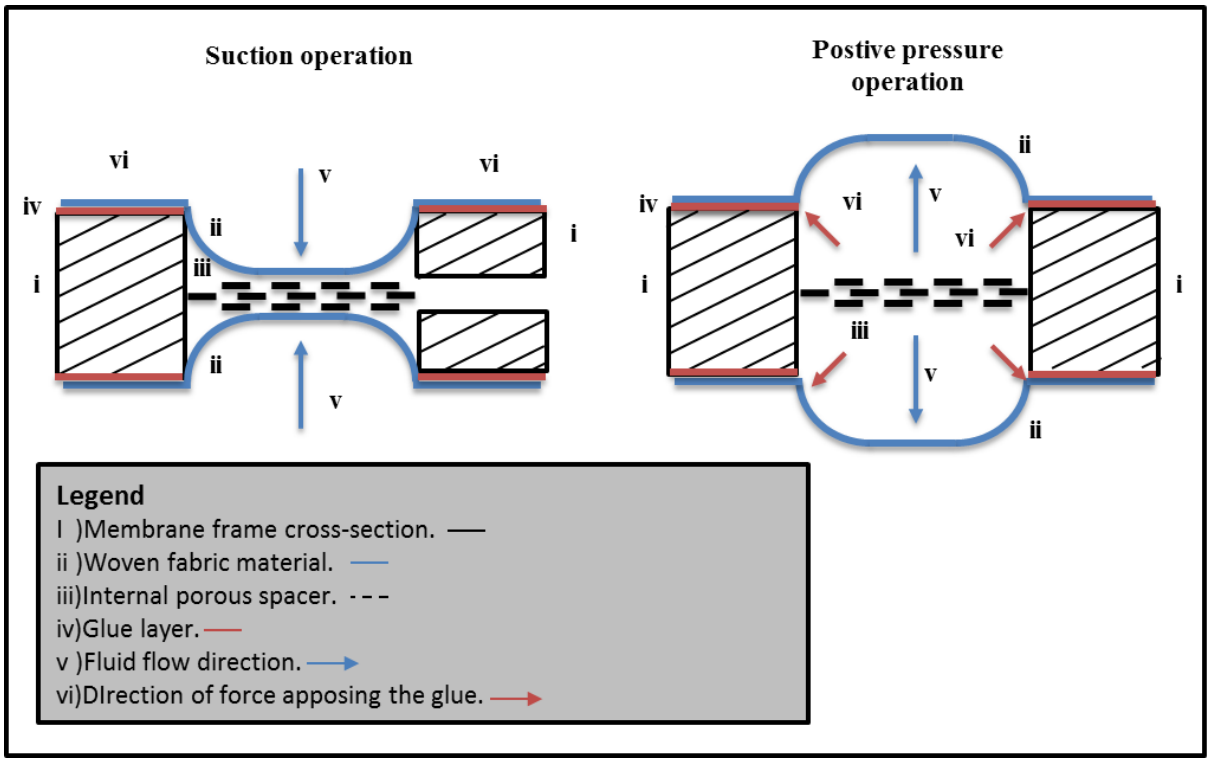

Figure 57: Force variations exerted on the woven fabric and glue binding layer during the different operation modes.

The woven fabric is flexible and deforms slightly under both positive and negative pressure loading as a result of the woven fabric not having a porous support layer impregnated in the

membrane material [1]. From the figure, it is apparent how the force being exerted on the membrane affects the surface area on which the glue acts. Under suction operation the membrane layers are 'pulled' towards the porous support layer, thus applying a force perpendicular to the glue layer over the entire width of the membrane frame whereas under positive pressure operation the membrane is 'pushed' away from the porous support layer, resulting in only an incremental portion of the glue layer taking the full force exerted on the membrane material. It was therefore proposed that the strength of the glues be tested under a positive pressure application, due to the higher pressure loading occurring on the glue layer during this operation mode.

Five different glues were tested for binding the woven fabric to the PVC frame: standard Hot Melt, Genkem VAW 739, MegaBond, No More Nails and Silicon Sealant Gasket Maker. These glues were selected for the following reasons: the standard hot melt was selected as it was the strongest of the water stable hot melts available, the Genkem VAW 739 and MegaBond were selected as they were competitive heat activated adhesive glues and the No More Nails and Silicon Sealant Gasket Maker were both silicon-binding agents advertised as having a relatively wide variety of suitable applications. The bind strengths were compared based on the force required to separate the woven fabric from the PVC frame.

The force was calculated by determining the positive pressure at which the woven fabric would became dislodged from the PVC frame. The force could then be calculated using Eq. (E.1):

$$F = P_{exerted} . A_{surface} \qquad \text{(E.1)}$$

where F is the force exerted on membrane sample and inversely on the glue layer binding the woven fabric and the PVC frame, $P_{exerted}$ the pressure exerted on the sample and $A_{surface}$ the surface area of the sample being pressurised.

B.2) Apparatus

A compressor was used to pressurise the membrane sample and a mercury manometer to monitor the pressure exerted. An illustration of the complete setup can be seen in Figure 58:

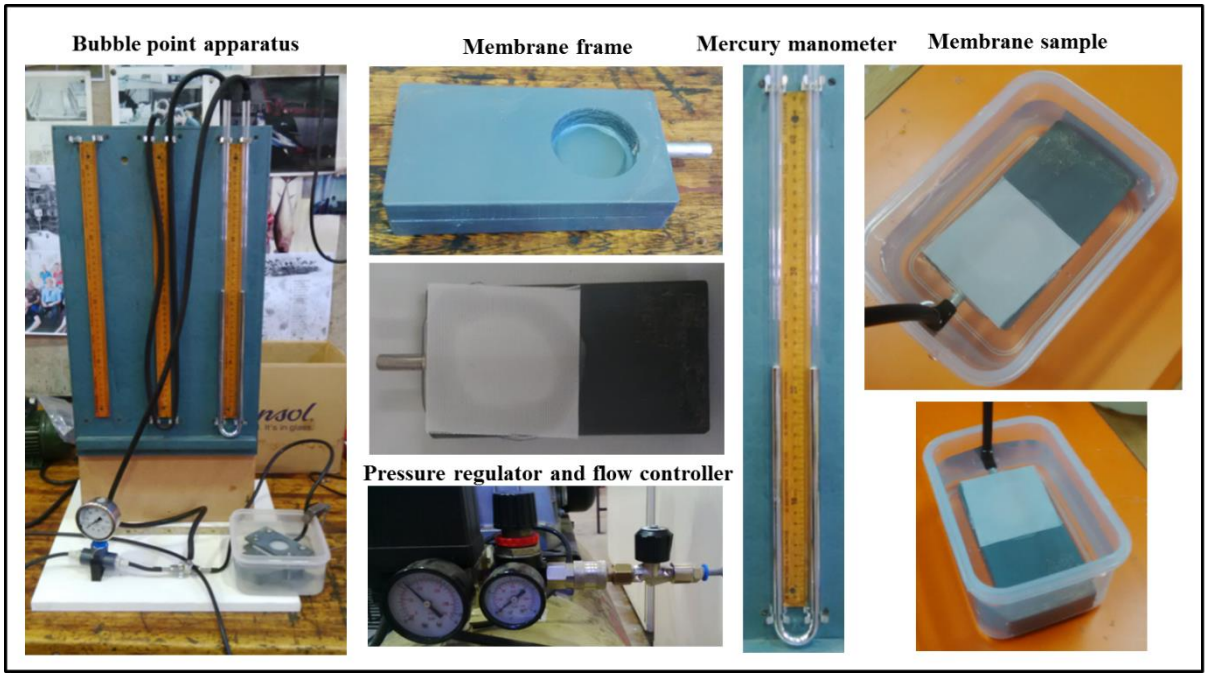

Figure 58: Glue strength comparison apparatus

The mercury manometer had an effective pressure range of 0-0.6 bar (430 mmHg) with an uncertainty of +-0.001 bar (1 mmHg). A secondary pressure gauge was used to indicate any possible pressure leaks in the apparatus. A 50x60 mm membrane sample size was secured to the membrane frame using the selected glues. The surface area of the membrane sample exposed to the force exerted by the compressed air was $0.8x10^{-3}m^2$.

The bubble point setup utilised both a pressure regulator and a flow controller. The purpose of this was to allow a particular pressure to be set, and then use the flow controller to control the rate of increase in pressure to the set pressure. This was used to pin point the pressure at which the membrane material became separated from the PVC frame, thus indicating failure of the glue layer to bind the wove fabric to the PVC frame.

B.3) Methodology

The membrane samples were prepared using the instructions as indicated on the packaging. The tests were performed by positioning the membrane sample in a clear dish, in such a way that there was 20 mm of water above the sample. The sample was allowed to soak for 5 minutes, to allow the membrane pores to be sufficiently wetted, before any pressure was applied to the membrane. The pressure regulator was set to 0.5 bar, with the flow controller closed. The flow controller was then set to allow a gradual pressure increase. The manometer

differential height was recorded at the point at which the woven fabric separated from the PVC frame. Each of the glues was tested twice using a new fabric sample each time.

B.4) Results and discussion

B.4.1) Experimental procedure validation

Due to the glue comparison tests utilising the same apparatus as was designed for the bubble transport procedure, investigated in 4.1.1 Membrane pore size, the validation of the experimental apparatus, for replicability, was not performed.

B.4.2) Comparison of results

From the results of the glue binding strength comparison tests it was apparent that the glues could be categorised into three distinct groups, namely: those that bonded better to the woven fabric only, those that bonded better to the PVC frame only and those that were able to bind to both the woven fabric and the membrane frame equally. A comparison of the variations in the binding layers can be seen in Figure 59:

Figure 59: Comparison of the glue binding layer favouring ether the woven fabric or the PVC frame

The figure indicates samples of the Silicon Gasket Maker, the No More Nails and the Hot Melt glue results. Starting from the right of the figure, the Silicon Sealer Gasket Maker can be seen to bind to woven fabric whereas the No More Nails has a significantly stronger bond to the PVC frame and the Hot Melt was able to bind evenly to both the woven fabric and the PVC frame.

The average glue failure force, in descending order, is summarised in Table 8:

Table 8: Average glue binding layer failure force.

Glues evaulated	Average glue failure force
Gemkem VAW 739	34.0
Mega Bond	24.4
Hot melt	17.1
Silicon Sealer Gasket Maker	12.1
No More Nails	5.6

The magnitude of the force was not important, but rather the order in which the glue appeared in the table. This was a result of the force exerted on the membrane being proportional to the surface area of the membrane material. Therefore, these glue failure forces could not be extrapolated to larger membrane modules. From the table above it is apparent how both the heat activated adhesive glues were the two strongest glues tested, with the Genkem and MegaBond having failure strengths of 34.0 N and 24.4 N respectively. The standard Hot Melt was third with a failure strength of 17.1 N and the two silicon based glues were fourth and fifth with the Silicon Sealer Gasket Maker and No More Nails having failure strengths of 12.1 N and 5.6 N respectively.

Both the heat activated adhesives glues, Genkem and MegaBond, required 18 to 24-hour preparation and setting time. This was not suitable for the turnaround time required to achieve the number of tests required as part of this study. For this reason, the standard Hot Melt was selected as the desired glue for the investigations, as it had a setting and preparation time of 20 minutes in comparison to the 18-24 hour of the heat activated adhesive glues.

For relatively high positive pressure operation tests, which surpassed the failure force of the Hot Melt glue, a reinforcing frame was used to prevent the woven fabric from separating from the frame. The reinforcing frame was made from 10 mm PVC sheet, and secured using 12x M8x35 mm bolts as indicated in Figure 60:

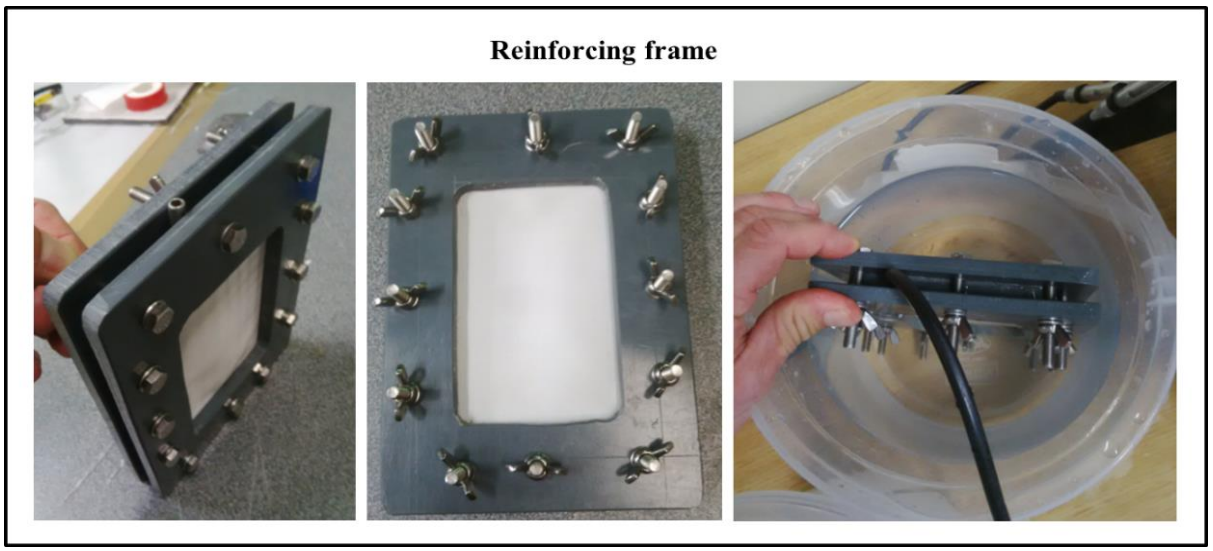

Figure 60: Reinforcing frame for relatively high positive pressure operation investigations.

The reinforced frame was only applicable to particular tests as a result of the reinforced frame affecting the inter membrane spacing and potentially hindering fluid flow within the flat sheet membrane apparatus.

B.5) Summary

The investigation indicated the following:

- The heat activated adhesive glues were able to out bind the woven fabric material to the PVC frame in comparison to the hot melt and silicon based glues tested.
- It was concluded that for permanent, non-reusable applications, the Genkem VAW 739 was most applicable as it had the highest binding failure force.
- For high quantity tests requiring new membrane modules, the hot melt was ideal as a result of its relatively high binding failure force and low preparation time.
- A reinforced securing frame was produced that secured the woven fabric to the frame for relatively high positive pressure applications.

B.6) References

[1] D.R. Machado, D. Hansson, R, Semiat, Effect of solvent properties on permeate flow through nanofiltration membranes. Part II: Transportation model, Journal of Membrane Science. 166 (2000) 63-69.

Appendix C: Flat sheet woven fabric membrane fabrication

In this section the design and fabrication of the flat sheet woven fabric membrane modules used in the investigations carried out as part of this study are covered.

C.1) Material selection

The flat sheet woven fabric membrane modules were made up of four distinct components, namely the PVC frame, the internal porous spacer, the permeate outlet and the woven fabric membrane sheets. The materials used for the respective components is discussed below:

The PVC frames were produced using one of two methods depending on the surface area requirements of the membrane module. For modules with membrane surface areas of approximately 0.05 m^2 or less, a single 8 mm PVC sheet was used with the centre milled out using a milling machine. For larger surface area membrane modules 20 mm strips were cut using a band saw, and glued using a PVC Cement compound to form the rectangular frame. This was to reduce material wastage.

The porous support layer was produced using multiple layers of a plastic mesh. The porous spacer was provided in a 5x1 m rolls.

The permeate outlet was produced using 6 mm stainless steel 306, extruded tubing. Stainless steel was used due to its low corrosion properties and its strength compared to plastic tubing. This allowed the tubing to be press fitted into the membrane frame to reduce the potential for leaks forming around this connection point.

C.2) Apparatus

The apparatus used to glue the membrane fabric to the PVC frame comprised of a purpose-built heating press and a general purpose hot melt glue gun. The heating press was required to sufficiently fluidise the hot metal glue to pass through the pores of the woven fabric. This resulted in a binding layer forming between the woven fabric and the PVC frames.

The heating press can be seen in Figure 61:

Figure 61: Membrane module fabrication heating press

The heating press comprised of two 350W insulated heating elements which were supported within two machined 8 mm aluminium plates. A layer of 2 mm Tufnell was used to insulate the heated aluminium plates from the mild steal securing frame. The temperature of the press was monitored using a thermocouple, and controlled using a rheostat. The heating press was able to heat both sides of the membrane module simultaneously when placed between the hinged aluminium plates.

C.3) Fabrication methodology

In this section only the fabrication of the membrane module using the standard Hot Melt glue sticks as selected in Appendix B: Membrane to PVC frame glue strength evaluation, is covered.

C.3.1) Porous support preparation

A series of preliminary investigations were performed to determine the effects of the porous support layer on the internal fluid flow within the membrane frame. This was performed by monitoring the TMP for different porous spacer designs and configurations. The finalised porous spacer design was selected based on the design that mimicked a spacer less module

most closely, in terms of the resultant membrane pressure drop. The step wise procedure followed for the fabrication of the woven fabric porous spacer can be seen below, with Figure 74 referencing the various steps:

a) The required number of porous spacer sheets were cut from the supplied roll of material.
b) The three parts of the spacer were then assembled, with a thin bead of the hot melt glue used to secure the different parts.

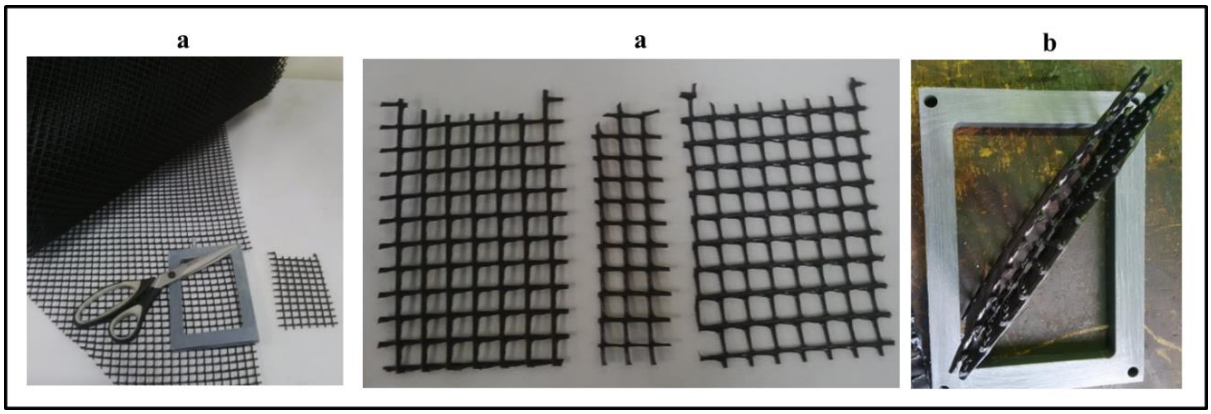

Figure 62: Woven fabric porous support layer preparation

C.3.2) Woven fabric sheet preparation

The woven fabric required preparation to take it from the 50m roll that it was supplied in, to the 150x130 mm woven fabric membrane sheets. The step wise procedure followed for the fabrication of the woven fabric membrane sheets can be seen below, with Figure 63 referencing the various steps:

a) The required number of membrane size sheets were cut from the roll of fabric.
b) A soldering iron was then used to seal the edges of the fabric to prevent it from unravelling during handling.
c) The woven fabric was then washed using a decreasing agent to remove any of the potential residual oils from the manufacturing process.
d) The sheets were rinsed and left to dry before being glued to the membrane frames.

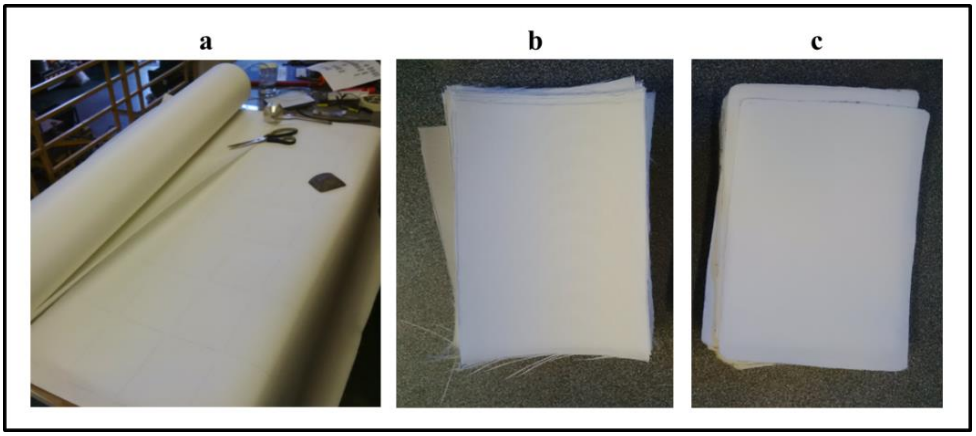

Figure 63: Woven fabric sheet preparation

C.3.3) Flat sheet membrane module fabrication

The step wise procedure followed for the fabrication of the woven fabric flat sheet membrane module can be seen below, with Figure 64 showing the various steps:

a) For reused frames, the old woven fabric membrane sheets were removed followed by the removal of any excess glue.
b) The frames required a key to be scotched into the surface to provide a coarse surface onto which the glue could bind.
c) The frame was then degreased using an acetone-based solvent, SuperSolve©.
d) A 2 mm bead of the hot melt glue was applied to one side of the membrane frame surface, thereafter rapidly applying the first sheet of the woven fabric onto the setting glue.
e) The porous spacer could then be positioned inside the membrane frame.
f) The second sheet of woven fabric was then glued to seal the membrane frame using again a 2 mm bead of the hot melt glue and rapidly applying the woven fabric sheet.
g) Once the heating press was within a range of 115-120^{0}C, the edges of the membrane module could be sealed using the heating press to reheat and 2 mm bead of glue, redistributing it over the 20 mm thickness of the frame.

It is important to note that the hot melt glue used was not thermosetting, therefore the hot melt gun was required only to create the 2 mm bead onto which the membrane sheet was lightly applied. The heating press was used to reheat the glue and adequately seal the membrane module.

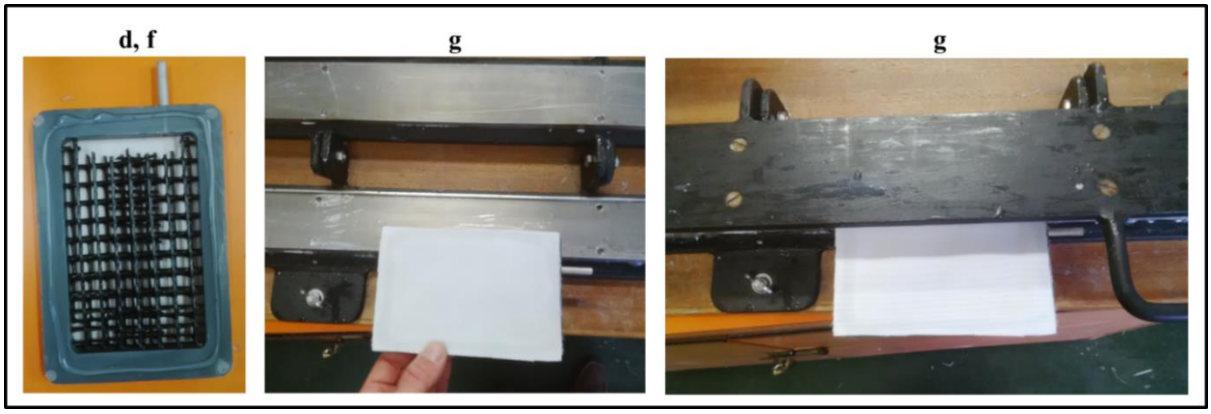

Figure 64: Membrane module preparation.

Appendix D: Preliminary investigation of flux enhancement methods

In this section the preliminary investigation into the effects of flux enhancement defouling methods is covered.

D.1) Investigation

The purpose of this preliminary investigation was to determine the validity of particular flux enhancement defouling methods for use on a woven fabric flat sheet membranes. The tests evaluated the effects of the flux enhancement methods at two operation levels as summarised in Table 9:

Table 9: Flux enhancement: upper and lower levels investigated.

Method	Levels tested	
Backflush duration (continuous)	5 minute	10 minute
Backflush duration (pulsed)	5 minute	10 minute
Air scour intensity (continuous)	5 L/min	10 L/min
Relaxation duration	5 minute	10 minute
Backflush + periodic air scour	5 minute 5 L/min	10 minute 5 L/min
Feed solution	50/50 D_{50} 5µm lime stone D_{50} 75µm bentonite	

The purpose of utilising two levels of testing was to better understand the defouling phenomena that was occurring.

The backflush (continuous) was performed by turning off the peristaltic pump after 10-minutes of operation, and opening the valves numbered 1 and 2 and closing valves 3 and 4 as indicated in the second image to the left in Figure 66, labelled 'Manifold and valve bank'. The backflush reservoir was topped up to maintain a constant level within the reservoir. The period that valves 1 and 2 were left open was dependent on the duration being tested, either 5-minute or 10-minute periods.

The backflush (pulsed) tests were performed by turning off the peristaltic pump after 10 minutes of operation, and opening the valves numbered 1 and 2 and closing valves 3 and 4 as indicated in the second image to the left in Figure 66, labelled 'Manifold and valve bank'. The backflush reservoir was topped up to maintain a constant level within the reservoir. The period that valves 1 and 2 were left open was 2 minutes. Then valves 1 and 2 were closed, and 3 and 4 reopened with the pump turned on for 1 minute. This process was then repeated by turning the pump off and reopening valves 1 and 2 and closing valves 3 and 4. The number of times this was repeated was dependent on the backflush duration investigated, either 5 minutes or 10 minutes. The purpose of turning the pump on during the defouling process, was to remove a small portion of the backflush fluid from within the membrane modules, such that when the backflush was recommenced it would create a low frequency pulsing effect.

The air scouring intensity (continuous) was altered by varying the needle valve, linked to the air blower to 5 L/min, or 10 L/min. The air scouring intensity was set at the beginning of the particular test and left so for the duration of the test.

The relaxation was tested by turning off the peristaltic pump after 10-minutes of operation and opening valves 1 and 2 and closing valves 3 and 4 in the same manner as the backflush was performed with the exception that valves 1 and 2 were left open for only 1 minute, then closed. The purpose of this was to relieve the built up pressure within the membrane module to allow the membrane material to return to a non-stressed state. The duration of the relaxation was dependent on the particular test, either 5 or 10-minutes.

The backflush and periodic air scouring tests combined a backflush with periodic air scouring. The procedure for the backflush was the same as that of the previous backflush (continuous) tests, except for the addition of the periodic air scour. The needle valve was adjusted before the start of the investigation to make sure that the correct flow rate was achieved through the air scour unit. The air blower was turned on at the start of the backflush

and then turned off once the backflush was completed for the particular cleaning cycle. A 5-minute backflush was performed with a coupled periodic 5 L/min air-scour intensity.

The feed solution used was a combination of 50/50 5 µm limestone and 7.5 µm bentonite to produce a feed solution with a turbidity of 100 NTU. An initial pure water flux of 50 LMH was used for these tests, and an operation period of 2 hours was maintained for each test. Therefore, the period for which the membranes were operated under suction was 2 hours overall for each test. The cleaning time added to the overall testing time, but did not take away from the 2-hour operating period of the membrane.

The flux enhancement tests evaluated the portion of the initial flux that remained at the end of the test as a result of the method applied. The results were presented as a percentage remaining of the initial flux value recorded. The percentage initial flux was calculated using Eq. (D.1):

$$P_J(t) = \left(1 - \frac{J_v(t_5) - J_v(t)}{J_v(t_5)}\right) x100 \qquad \text{(D.1)}$$

where $P_J(t)$ is the percentage remaining of the initial flux at time t, $J_v(t_5)$ is the initial flux value recorded 5 minutes after commencing the membrane operation, and $J_v(t)$ the flux recorded at time interval t. The purpose for selecting the initial flux recording after the first 5 minutes of operation, was as a result of the apparatus taking 4 minutes to draw the remaining air from the membrane modules.

D.2) Apparatus

The laboratory scale apparatus utilised for this phases of the laboratory scale testing, can be seen in Figure 65:

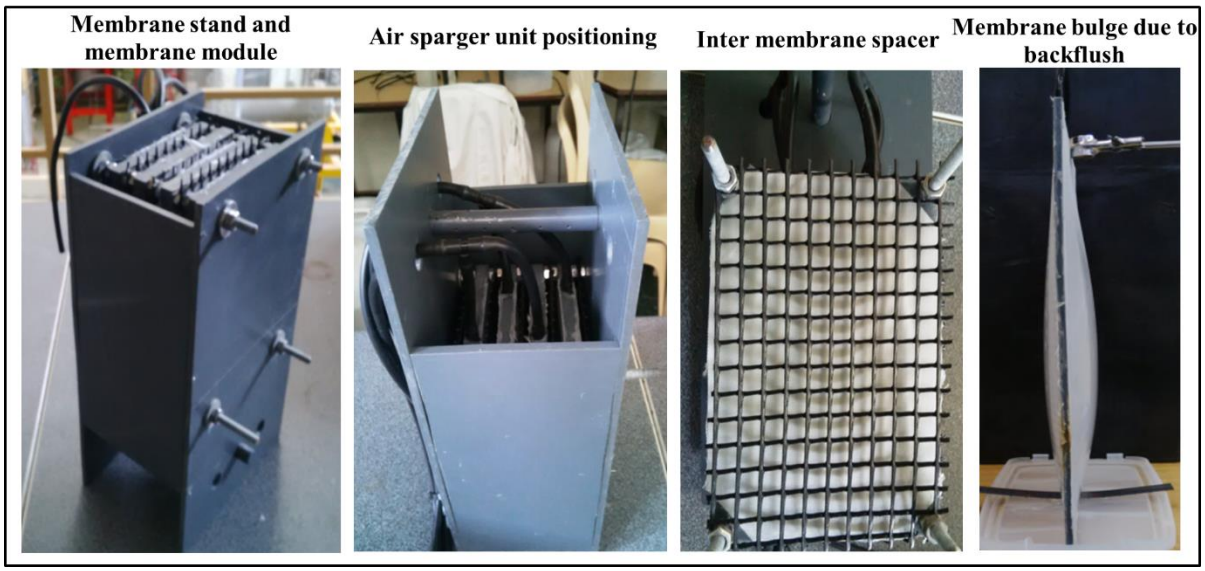

Figure 65: Preliminary flux enhancement laboratory scale membrane stand and module design.

The unit comprised of four 150x130x10 mm membrane modules. To counter the effects of membrane swelling when performing a backflush, a porous material was inserted between the membrane modules. This prevented the membrane modules contacting each other when performing a backflush. The membranes were interlinked to a central manifold, to which the peristaltic pump was connected. The A_d/A_r ratio for the unit was approximately 4, with a membrane-membrane spacing of 12 mm, and a membrane-spacer spacing of 5 mm (the spacer material was 2 mm thick). The underflow and overflow areas incorporated into the membrane apparatus, for improved hydrodynamics, can be seen illustrated in the first image to the left in Figure 65 above. The cutaway sections at the bottom and top of the membrane stand allow the fluid to circulate within the apparatus, promoting the induced two-phase fluid flow as a result of air scouring.

This was the first study to date, to the best of our knowledge, that investigated incorporating an in-situ backflush to a woven fabric membrane [1]. The incorporation of an in-situ backflush was only plausible due to the addition of the inter-membrane porous support layer as illustrated in Figure 65 ‘Inter-membrane spacer’. This inter-membrane porous support layer must not be confused with the porous spacer that is used within each of the flat sheet woven fabric membrane modules, which prevents the membrane sheets from contacting one another when under suction. An illustration of the preliminary membrane apparatus, incorporating the peristaltic pump, holding vessel, gravity backflush reservoir, manifold and valve bank can be seen in Figure 66:

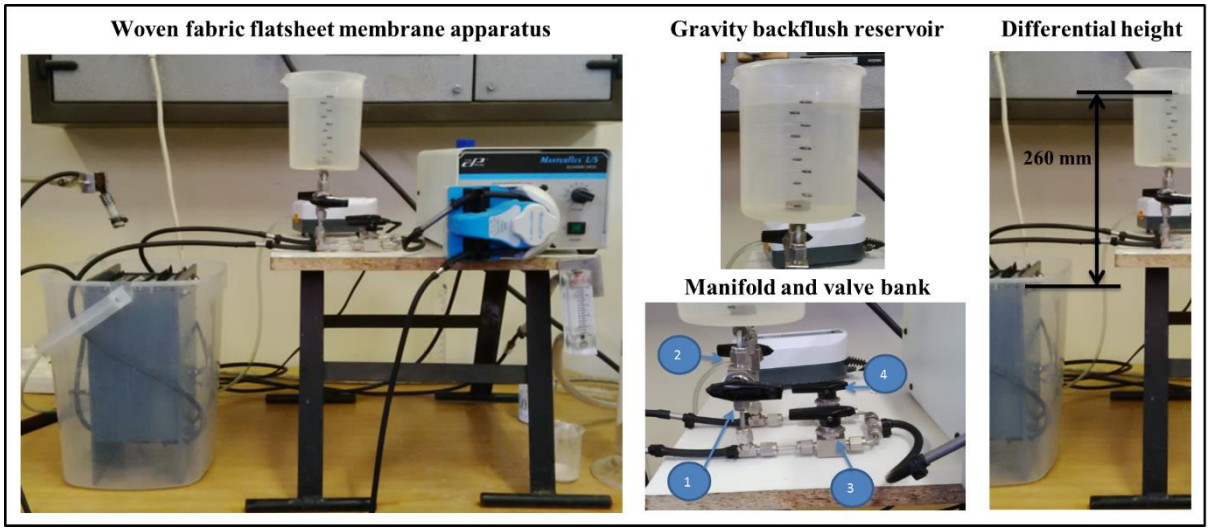

Figure 66: Preliminary flux enhancement, woven fabric flat sheet membrane apparatus.

D.3) Methodology

The operation procedure consisted of two parts, namely the initiation procedure and the operation procedure:

Step 1: Initiation procedure:

1) The membrane modules were tested for leaks prior to being utilised.
2) The holding vessel was filled with the RO water.
3) The membrane stand was lowered into the holding vessel and left for 10 minutes to allow the pressure within the membrane modules to equalise.
4) The pump speed was set to the desired flow and left to stabilise for 10 minutes before the first permeate flux was recorded.
5) If the pure water flux recorded was 50±2 LMH, the pump was turned off and the holding vessel drained. If it was lower or greater than 50±2 LMH, the pump flow was altered and the flow left to stabilise as before, before the new permeate flux was recorded. This was repeated until the desired flow was achieved.

Step 2: Operation procedure:

1) The holding vessel was filled with the prepared 5K limestone and bentonite feed suspension.
2) The membrane stand was lowered into the holding vessel and left for 10 minutes to allow the pressure within the membrane modules to equalise.
3) The pump flow was not altered.

4) The permeate turbidity and flux were recorded after 5 minutes of operation.
5) The tests were performed as described above.

Each of the cleaning trial tests were repeated, with a non-cleaning test (dead end operation test), performed between these repetition tests. These were compared to previous dead end tests to ensure that the membrane modules were sufficiently defouled and to indicate any possible membrane leaks that may have resulted from the cleaning method tested. This also randomised the testing order between repeat tests.

D.4) Results and discussion

D.4.1) Relaxation

The results of the relaxation tests performed can be seen in Figure 67:

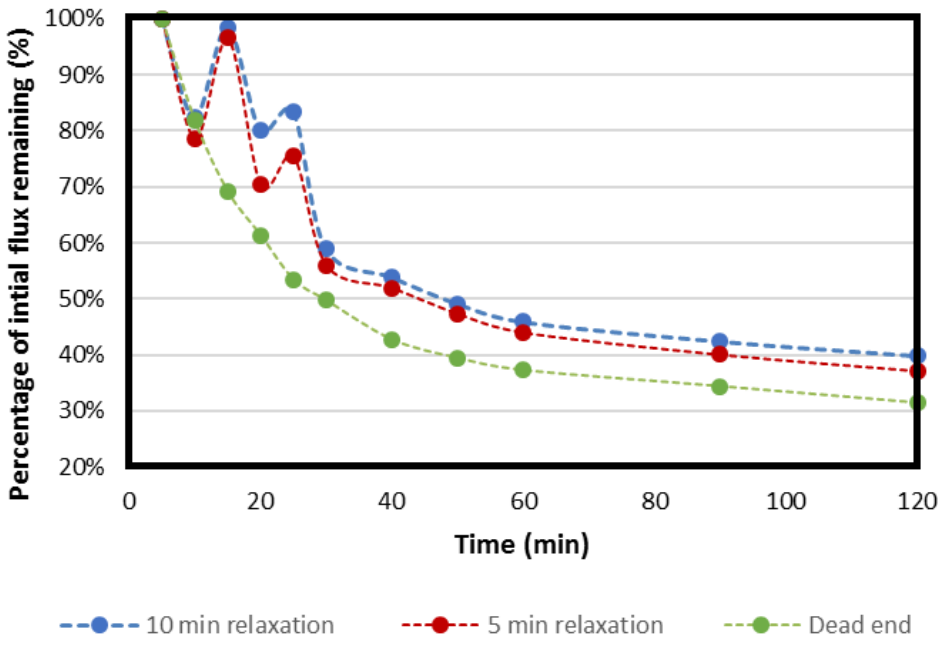

Figure 67: The effects of relaxation on flux recovery.

The figure indicates how the flux decreased over the two-hour period for all three tests, with the dead end test decreasing at a relatively higher rate, followed by the 5-minute relaxation test and then the 10-minute relaxation test, with the following percentage remaining flux values, after the 2-hour period: 31, 37 and 40% respectively. For the first 30 minutes of the operation period, the flux was recorded in 5-minute time increments such that the initial flux recovery after the relaxation was captured. This indicated how the relaxation was able to recover a decreasing portion of the initial flux with time. The decreasing nature was as a

result of the fouling layer formed becoming increasingly resilient to being removed with an increase in the operation period. This correlated with literature where an ineffective defouling measure resulted in the formation of irreversible fouling [2-4]. The irreversible nature of the fouling layer is due to the compounding of the reversible fouling layer.

The 10-minute and 5-minute relaxation periods followed a similar decreasing flux trend, with the 10-minute period decreasing at a relatively smaller percentage with time, than the 5-minute test. The fact that the 10-minute relaxation test only marginally outperformed the 5-minute relaxation test indicated the presence of a maximum relaxation period, whereafter the effects of an increasing relaxation period were negligible, in comparison to the loss in operation period.

D.4.2) Gravity backflush (continuous and pulsed)

The results of the backflushing tests performed can be seen in Figure 68:

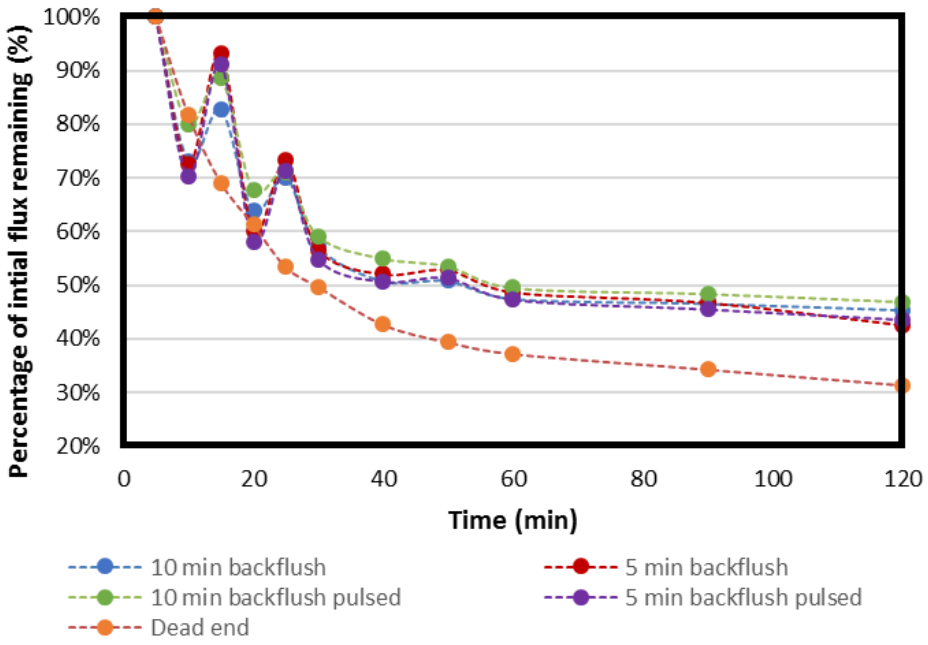

Figure 68: The effect of continuous and pulsed gravity backflush on flux recovery.

The figure indicates that the flux decreased over the two-hour period for all five tests, with the dead end test decreasing at a relatively higher rate, followed by the 5-minute continuous backflush test, the 5-minute backflush pulsed test, the 10-minute continuous backflush test and then the 10-minute backflush pulsed test, with the following percentage flux decrease values after the two-hour period: 31, 43, 44, 45 and 47% respectively. For the first 30-minutes of the operation period, the flux was recorded in 5-minute time increments such that

the initial flux recovered after the particular backflush performed could be observed. This indicated that the backflush methods, both pulsed and continuous, were able to recover a decreasing portion of the initial flux with time.

The decreasing nature was as a result of the fouling layer formed becoming increasingly resilient to being removed with time using the various backflush methods. This was the result of a compounding effect occurring in the fouling layer as a result of the propagation of the fouling layer, due to the ineffective defouling method. This correlated with literature where an ineffective defouling measure resulted in the formation of irreversible fouling [2-4]. The irreversible nature of the fouling layer is due to the compounding of the reversible fouling layer.

The 10-minute and 5-minute relaxation periods can be seen to follow a similar decreasing flux trend, with the 10-minute decreasing at a relatively smaller percentage with time, during both the continuous and pulsed tests. The fact that the 10-minute backflush only marginally outperformed the 5-minute relaxation test indicated the presence of a maximum backflush period whereafter the effects of increasing backflush duration are negligible in comparison to the loss in operation period.

The backflush methods tested were able to outperform the relaxation techniques tested as a lower rate of decrease in flux was achieved. The pulsed backflush compared to the continuous resulted in only marginal gains in the defouling potential of the membrane when compared to the same continuous duration test. These results correlated with literature as to the varying degrees of effectiveness of the methods tested [3,5].

D.4.2) Continuous air scouring

The results of the air scouring tests performed can be seen in Figure 69:

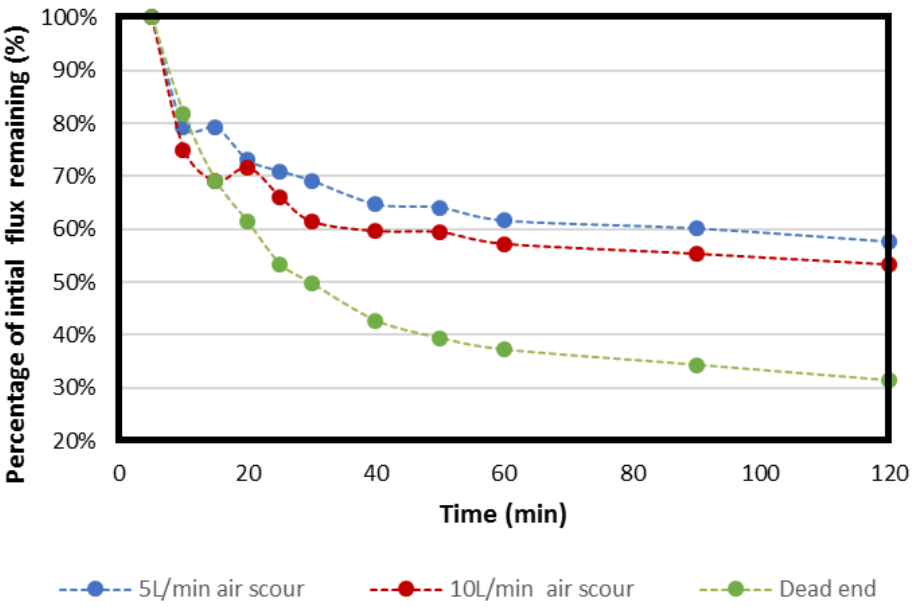

Figure 69:The effects of continuous air scouring on flux recovery.

The figure indicates how the flux decreased over the two-hour period for all three tests, with the dead end test decreasing at a relatively higher rate, followed by the 10 L/min air scour test and then the 5 L/min minute test, with the following percentage flux decrease values after the two-hour operation period recorded as 31, 53, and 58% respectively. The 5 L/min and 10 L/min air scour intensities tested followed a similar decreasing flux trend, with the 5 L/min minute decreasing at a relatively smaller percentage with time.

The results indicated in the figure were counter intuitive, as an increase in the energy to the membrane, in the form of an increased air flow rate through the air diffuser, would be expected to increase the flux enhancement efficiency of the air scouring. However, the results indicated that the lower 5 L/min tests resulted in relatively improved defouling efficiency compared with the 10 L/min. This phenomenon was also witnessed by M. Cele [1] who noted, as part of his study into improved IMBR hydrodynamics, that as the air scouring intensity was increased, by increasing the air flow rate, a point was reached whereafter the increased air flow resulted in coalescence of the bubble stream to a single stream. This greatly affected the distribution of the bubbles across the membrane surface and hampered the defouling efficiency due to the resultant air scouring ‘dead zones’ which formed. This would indicate a potential apparatus based limitation to the flux enhancement, as a result of the sparger design and not as a result of the woven fabric.

An important consideration during this study was that the formation of a fouling layer was required to improve the product quality achieved by the membrane. Therefore, a balance between completely defouling the membrane surface and providing sufficient measures to control the propagation of the fouling layer was required. A graphical representation of the effects of performing a backflush and air scouring on the product quality can be seen in Figure 70:

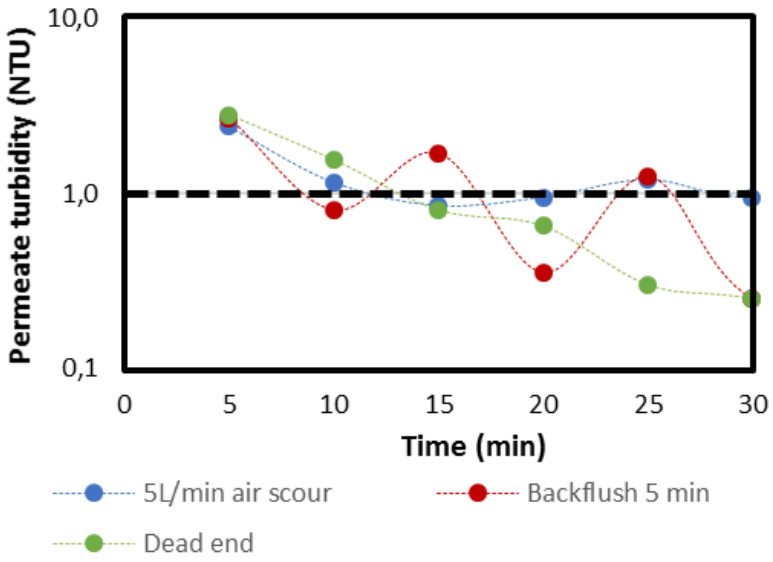

Figure 70: Comparison of the effects of air scouring and backflush on permeate turbidity.

The figure indicates the permeate turbidity for the first 30-minutes of the 5 L/min continuous air scouring test, the 5-minute backflush test and a dead end operation test. The figure indicates that all three tests started off with a turbidity in the range of 3 and 4 NTU. As the tests proceeded the product quality of the 5 L/min continuous air scouring test can be seen to oscillate around 1 NTU as a result of the air scouring reducing the propagation of the fouling layer. The backflush test can be seen to decrease to below a product turbidity of 1 NTU, and then increase after 10 minutes. This was as a result of the backflush removing a portion of the fouling layer that was allowed to form between backflush cycles. The dead end test product quality decreased with time with no oscillation or periodic increase with time. This was as a result of the fouling layer being left to propagate with no flux enhancement controlling measure. This indicated the importance of the fouling layer for product quality purposes.

The preliminary study indicated the trade-off between achieving a high product quality and maintaining a low fouling rate for a dynamic membrane process. This phenomenon correlates with the literature on the operation of a dynamic membrane process [6].

D.4.2) Combined effects

The results of the coupled periodic air scouring and backflushing, as well as the 5 L/min continuous air scouring, the 5-minute backflush and a dead end test for comparison purposes can be seen in Figure 71:

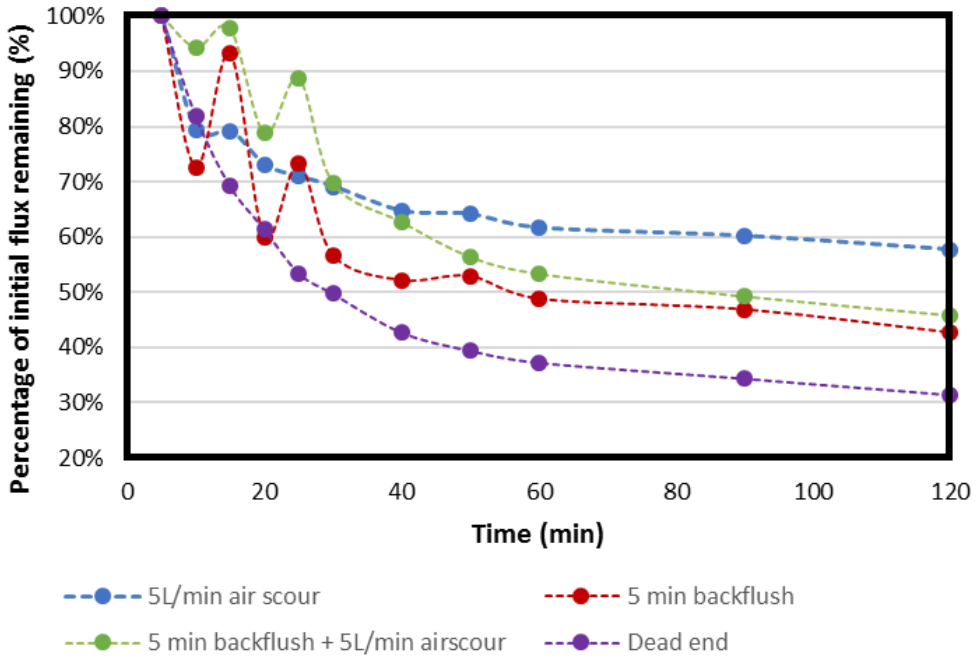

Figure 71:The effects of coupled gravity back flush and periodic air scouring on flux recovery.

The figure indicated the flux decrease over the two-hour period for all four tests. The dead end test decreased at a relatively higher rate followed by the 5-minute backflush test, the coupled 5 L/min periodic air scour and 5-minute backflush and the 5 L/min continuous air scour. The following percentage flux decrease values were recorded after the two-hour period: 31, 43, 46 and 58% respectively. The 46% decrease in flux percentage over the 2-hour period of the coupled defouling measure marginally outperformed the 10-minute backflush cleaning method which had a flux decrease percentage of 45% over the 2-hour period.

The coupling of the periodic air scour with a backflush effectively halved the cleaning time required to achieve approximately similar defouling efficiency as a 10-minute backflush did. Combining periodic air scouring and backflushing required further investigations to determine to what extent the membrane could benefit from such a hybrid defouling measure.

D.5) Conclusion

D.5.1 Relaxation

The effects of performing membrane relaxation as a flux enhancement method, for two relaxation periods, indicated minimal recovery of the permeate flux lost to the effects of fouling. Both tests resulted in a relatively smaller percentage recovery of the flux in comparison to the dead end operation test. This was concluded to be a result of the relaxation defouling method applying no shear force or opposing entrainment force to the particulate matter entrained within the membrane pores. The portion of flux recovered by the relaxation method decreased overtime as a result of the fouling layer formed becoming increasingly resilient to being removed with an increase in the operation period. This was concluded to be the result of the formation of irreversible fouling due to an ineffective defouling measure.

D.5.2 Gravity Backflush

The effects of performing both continuous and pulsed membrane backflush as flux enhancement methods, for two operation periods, indicated minimal recovery of the permeate flux lost to the effects of fouling. Both tests resulted in a relatively smaller percentage recovery of the flux in comparison to the dead end operation test. The backflush techniques tested were able to outperform the relaxation technique tested as a lower rate of decrease in flux was achieved by the backflush methods compared to the relaxation method. This was concluded to be due to the membrane backflush applying an opposing force to the entrained particulate matter in the membrane pores.

The pulsed backflush compared to the continuous resulted in only marginal gain in the defouling potential for the membrane when compared to the same continuous duration test. The marginal performance increase of the pulsed method was concluded to be due to the majority of the pulse energy being absorbed by the expansion of the woven fabric material, as opposed to being focused solely on the entrained particulate matter.

D.5.3 Continuous air scour

The effects of performing continuous air scouring as a flux enhancement method, for two operation flow rates levels, indicated an higher flux recovery compared to the other methods tested. The investigation indicated the lower air scouring flow selected as having a greater effect on defouling the membrane modules as opposed to the higher flow test. This was deduced to be as a result of the coalescence of the bubble column to the centre of the

membrane modules at the higher flow. However additional investigation was required to investigate this phenomenon further. This additional investigation was performed as part of Chapter 6.

D.5.4 Combined effects

A gravity backflush combined with periodic air scouring was investigated to indicate whether the addition of the periodic air scouring would improve flux recovery of the gravity backflush. The 5-minute gravity backflush coupled with the periodic air scouring effectively halved the cleaning time required to achieve the same flux enhancement defouling efficiency as the 10-minute backflush did. The gravity backflushing coupled with the periodic air scouring was concluded to require further evaluation to determine to what extent the membrane could benefit from such a hybrid defouling measures.

D.5.5 Overall

During the evaluation it was concluded that the different flux enhancement measures affected the dynamic operation of the woven fabric membrane. The flux enhancement methods evaluated were unable to completely reduce the effects of fouling, however slight recovery was indicated by the different methods. The defouling potential of these methods was concluded to compromise the product quality achieved. Therefore, it was proposed that further revision of the membrane design was required to determine the full extent of these flux enhancement method, in an attempt to better control and suit the required dynamic operation of the woven fabric membrane.

D.6) References

[1] M. Cele, Development and evaluation of woven fabric immersed membrane bioreactor for treatment of domestic waste water for re-use, Department of Chemical Engineering, University of Natal, (2014).

[2] Z. Wang, Z. Wu, X. Yin, L. Tian, Membrane fouling in a submerged membrane bioreactor (MBR) under sub-critical flux operation: Membrane foulant and gel layer characterization, Journal of Membrane Science. 325 (2008) 238-244.

[3] A. Drews, Membrane fouling in membrane bioreactors- Characterisation, contradictions, cause and cures, Journal of Membrane Science. 363 (2010) 1-28.

[4] M. Kraume, D. Wedi, J. Schaller, V. Iversen, A. Drews, Fouling in MBR—what use are lab investigations for full scale operation?, Desalination. 236 (2009) 94–103.

[5] Z. Wang, J. MA, C.Y. Tang, K. Kimura, Q. Wang. X. Han, Membrane cleaning in membrane bioreactors: A review, Journal of Membrane Science. 468 (2014), 276-307.

[6] V.L. Pillay, C.A. Buckley, The operation of a cross-flow microfilter, Pollution Research Group, Department of Chemical Engineering, University of Natal, (1999).

Appendix E: Air scour design revision

The use of air scouring and membrane backflush orientated flux recovery methods were indicated as capable of recovering varying portions of flux lost as a result of the effects of fouling. However, a revision of the membrane apparatus hydrodynamics was required to better suit these flux enhancement methods to the woven fabric membrane. The bubble column formation of the membrane apparatus utilised in Chapter 6.1 Preliminary evaluation of flux enhancement methods, can be seen in Figure 72:

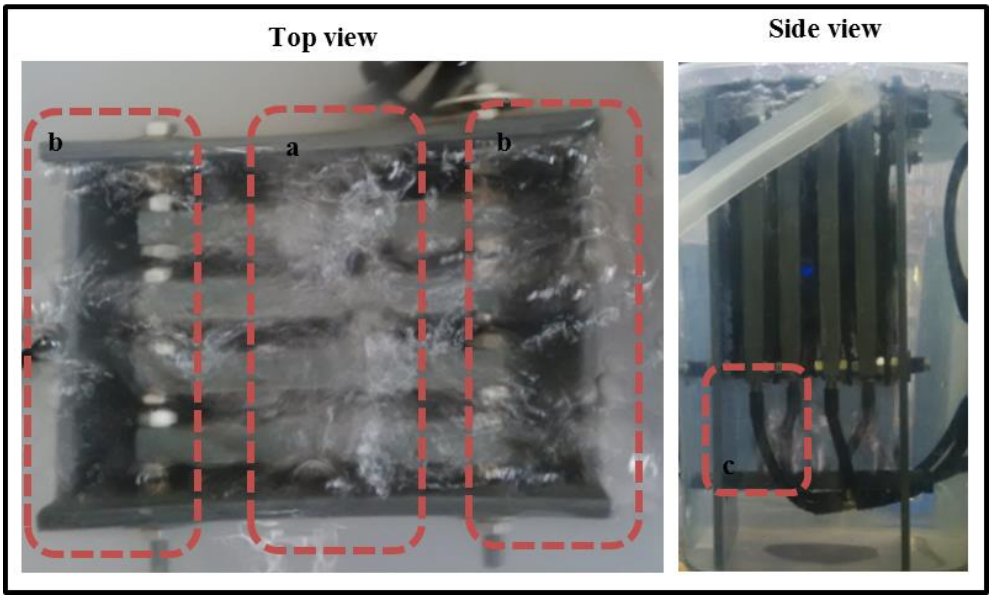

Figure 72: Preliminary membrane apparatus air sparger evaluation.

The figure was used to demonstrate some of the noted shortcomings of the preliminary membrane apparatus to the use of air scouring as a means of flux recovery. The combination of the air sparger design and hydrodynamic dimensions selected for the preliminary apparatus, resulted in a large portion of the bubble stream coalescing to the centre of the membrane modules. This was prominent for the relatively higher air flow rates tested. The coalescence of the bubble column to the centre of the membrane modules was indicated in the figure by the letter ‘a’. The figure indicated a portion of the bubble stream that was able to bypass the membrane modules, resulting in wasted defouling potential. This was as a result of the open area between the membrane modules and the membrane stand, as illustrated in the figure by the letter ‘b’. The sparger design utilised was noted as resulting in interference between the adjacent bubble streams, creating poor distribution of the bubble columns between the membrane modules. This is indicated in the figure by the letter ‘c’. From the

figure it is apparent that a revision of the preliminary design is required to better investigate the air scouring flux enhancement potential to the woven fabric membrane.

E.1 Review of the woven fabric flat sheet membrane apparatus

E.1.1 Investigation

A series of experiments were performed to better select an appropriate air scouring diffuser design as well as to fine tune the hydrodynamics of the membrane apparatus. The investigation was performed by evaluating the effects the following variables had on the bubble column formation in the membrane apparatus:

- The air sparger diffuser design.
- The height between the air sparger diffusers and the bottom of the membrane modules.

An effective distribution of the bubble column was required to enhance the two-phase fluid flow over the membrane's surface within the membrane apparatus. The preliminary membrane design was found to result in high coalescence of the bubble stream, which promoted air scouring 'dead-zones' that lowered the defouling potential of the air scouring method. Therefore, a revision on the apparatus design was required to ensure effective bubble column formation within the membrane apparatus. For this investigation, the bubble column dispersion was monitored laterally in both the x-direction and the y-direction, as shown in Figure 73:

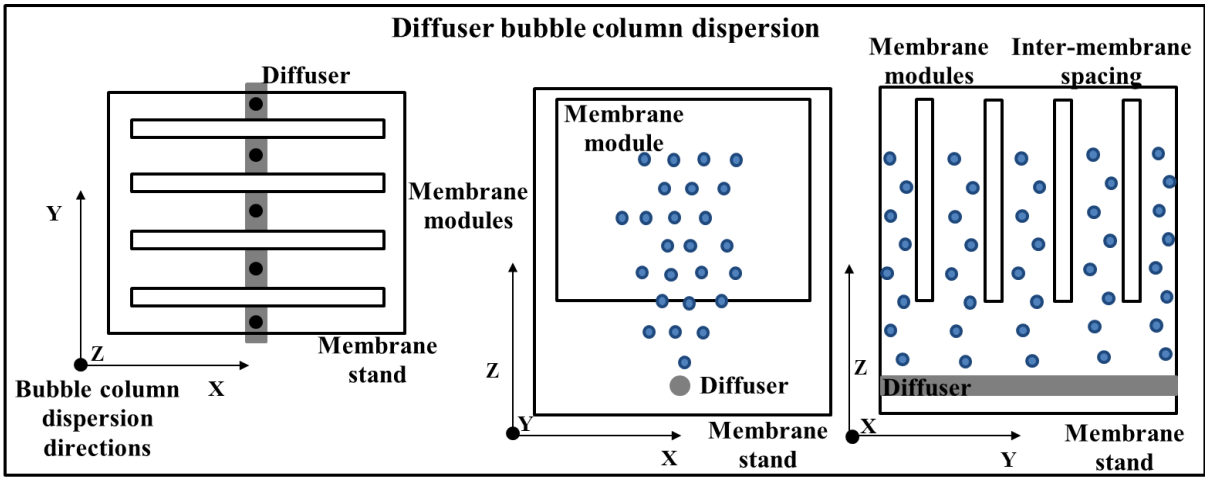

Figure 73: Bubble column lateral dispersion reference axes.

The x-direction indicates the dispersion of the bubble column over the membrane surface, whereas the y-direction indicates the dispersion between the inter-membrane spacing of adjacent membrane modules. The air sparger diffuser selection was based on the design that resulted in a relatively large dispersion of the bubble column in the x-direction and the height between the air sparger diffusers and the bottom of the membrane modules was selected based on height that resulted in the lowest dispersion in the y-direction. This was to ensure that the bubble columns entered the correct inter-membrane spacing, with relatively low interference between the adjacent bubble columns.

As part of the investigation, the design variables were to be selected for both a fine and coarse air scouring diffuser unit. It was expected that the design parameters would vary between the coarse and fine air scouring, and therefore the investigation was performed for both designs separately. Inspection of the actual bubble size formation was not performed as part of this investigation. It was indicated in literature that a diffuser pore size of larger than 1 mm produced bubbles that coincide with coarse bubble air scouring (> 5 mm) and diffuser pore sizes of smaller than 1 mm coincide with fine bubble air scouring (< 5 mm) [7]. The reason for testing both coarse and fine air scouring, lies in the potential for a higher dispersion of the bubble column being achievable at lower fine air scouring flow rates, compared to the coarse air scouring, thus making it possible to investigate lower air scouring flow rates, whilst maintaining the required degree of bubble column dispersion. The effects of varying the A_d/A_r ratio were not evaluated. Literature indicated the results of previous studies performed on both woven fabric membranes and other flat sheet membrane processes. These studies indicated the benefits of an A_d/A_r ratio of 2 or greater, for improving the hydrodynamics within a flat sheet membrane process [1-6].

E.1.2 Apparatus

The apparatus designed for this investigation, consisted of a Perspex vessel, a height adjustable Perspex membrane simulator and a diffuser stand. The dimensions of the Perspex vessel and the membrane simulator can be seen in Figure 74:

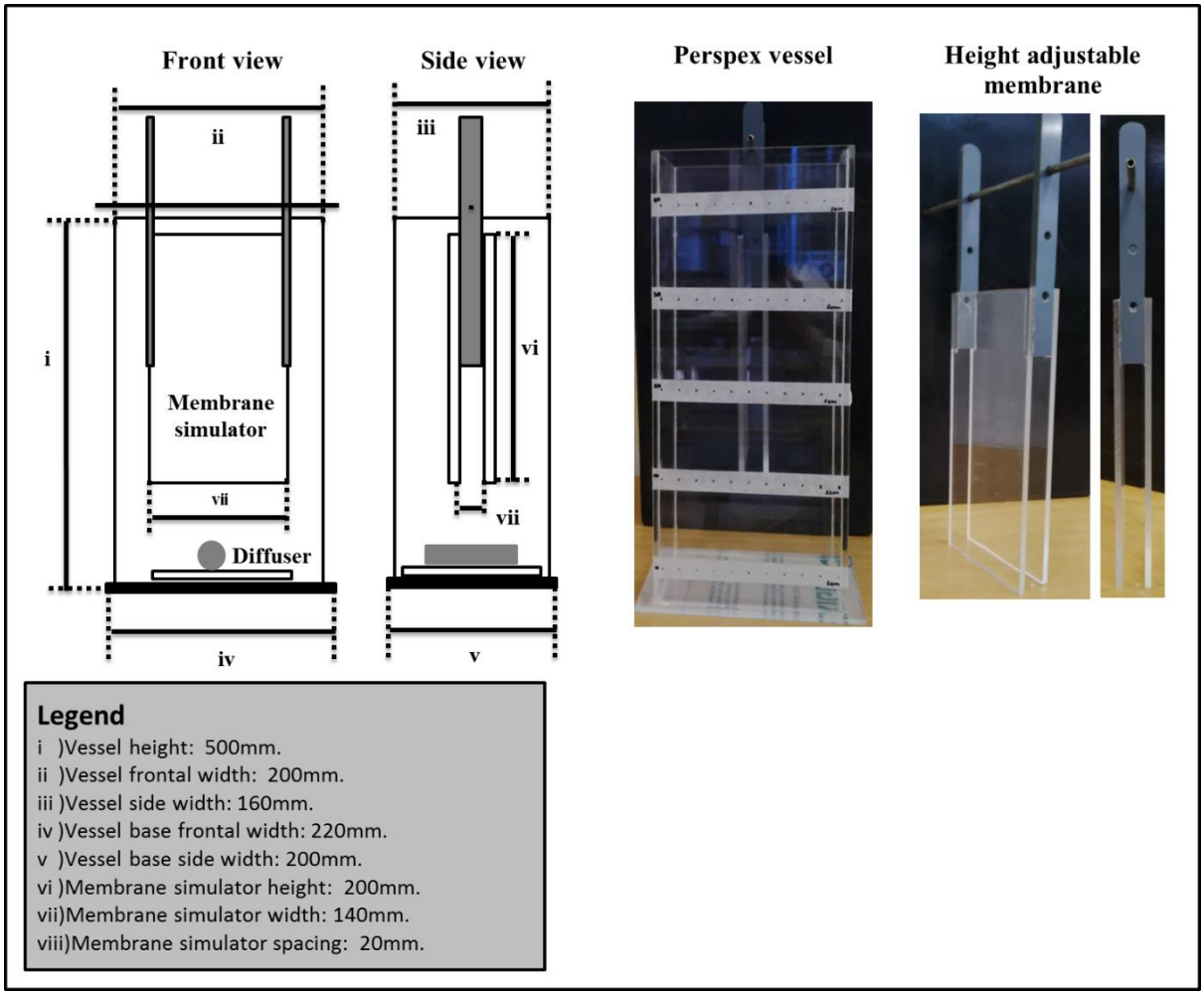

Figure 74: Air sparger design apparatus.

The membrane simulator was used to mimic the inter-membrane spacing of two adjacent membrane modules within a membrane stand. The use of membrane modules would have resulted in a visual hindrance of the bubble column within the inter-membrane spacing. The purpose of making the height of the membrane simulator over the diffuser unit adjustable, was to evaluate the effects this variable had on the bubble column dispersion in both the x- and y-directions.

Masking tape was used to add horizontal 20 mm increments to the front face of the Perspex vessel. The height increments started in line with the diffuser unit and were placed 100 mm apart, with the diffuser unit being referenced as the 0 mm mark. These were used to compare the bubble column dispersions from the centreline of the designs tested.

The air diffuser designs evaluated can be seen in Figure 75:

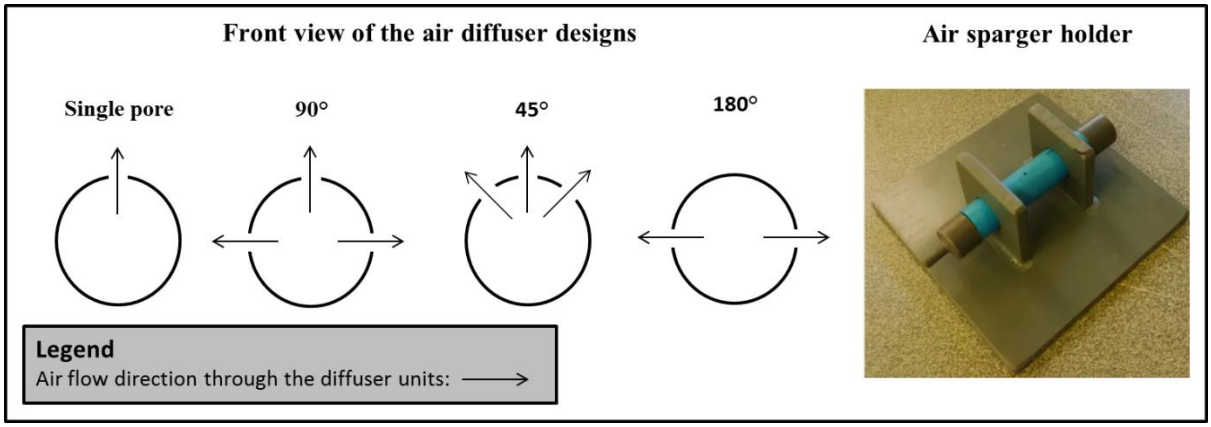

Figure 75: Air diffuser designs investigated for improved bubble column dispersion.

The figure indicates the pore orientation on the diffusers tested for both the fine and coarse air scouring investigations. A blower unit was used to feed the air sparger units and a variable flow controller was used to adjust the flow to the sparger between 0 and 10 L/min.

E.1.3 Methodology

The methodology used as part of the investigation follows a two-step procedure:

Step 1: Diffuser selection

a) The Perspex vessel was filled with RO water to the 400 mm mark.
b) The selected air sparger design, coarse or fine, was loaded into the sparger stand and lowered into the vessel, with the sparger unit perpendicular to the front of the vessel.
c) The blower flow was set to 2.5 L/min for coarse air scouring and 1.7 L/min for fine air scouring, depending on the test.
d) The bubble column was left to stabilise for 5 minutes.
e) Thereafter two pictures were taken of the bubble column dispersion in the x-direction.
f) The process was repeated for the four sparger designs.

Step 2: Membrane-diffuser height selection

a) The Perspex vessel was filled with RO water to the 400 mm mark.
b) The desired air sparger design was loaded into the sparger stand and lowered into the vessel, with the sparger unit parallel to the front of the Perspex vessel.
c) The height adjustable membrane simulator was placed within the Perspex vessel, with the unit positioned on the lowest height setting.

d) The blower flow was set to 2.5 L/min for coarse air scouring and 1.7 L/min for fine air scouring, depending on the test.
e) The bubble column was left to stabilise for 5-minutes.
f) Thereafter two picture of the bubble column dispersion in the y-direction were taken.
g) The membrane simulator was then raised by 50 mm and the process repeated for the three simulator height adjustments.

The images were processed using Microsoft Excel 360 to determine the average deflection for each of the air sparger designs tested. The average dispersion value was calculated by taking the average dispersion from the centreline of the two repeat runs, for each of the height increments measures.

An illustration of the two step procedure performed can be seen in Figure 76:

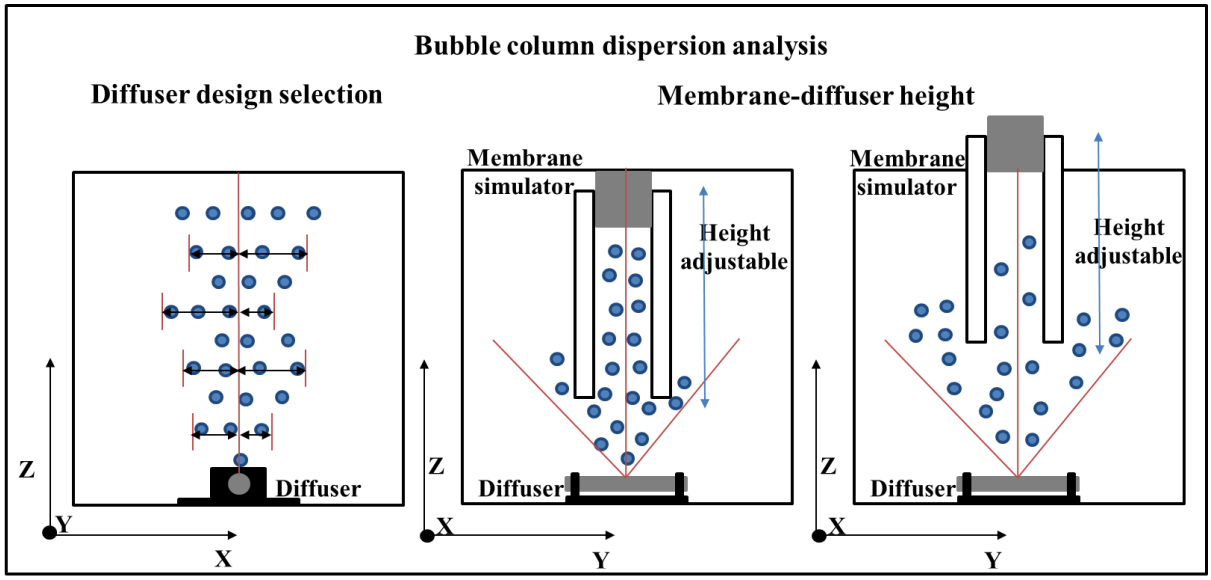

Figure 76: Bubble column dispersion evaluation.

E.1.4 Results and discussion

The results and discussion comprises two sections, namely the design of a coarse air sparger unit and the design of the fine air sparger unit.

E.1.4.1 Results and discussion of coarse air scouring

The results of the coarse air scouring investigation for evaluating the bubble column dispersion in the x-direction for the diffuser designs tested can be seen in Figure 77:

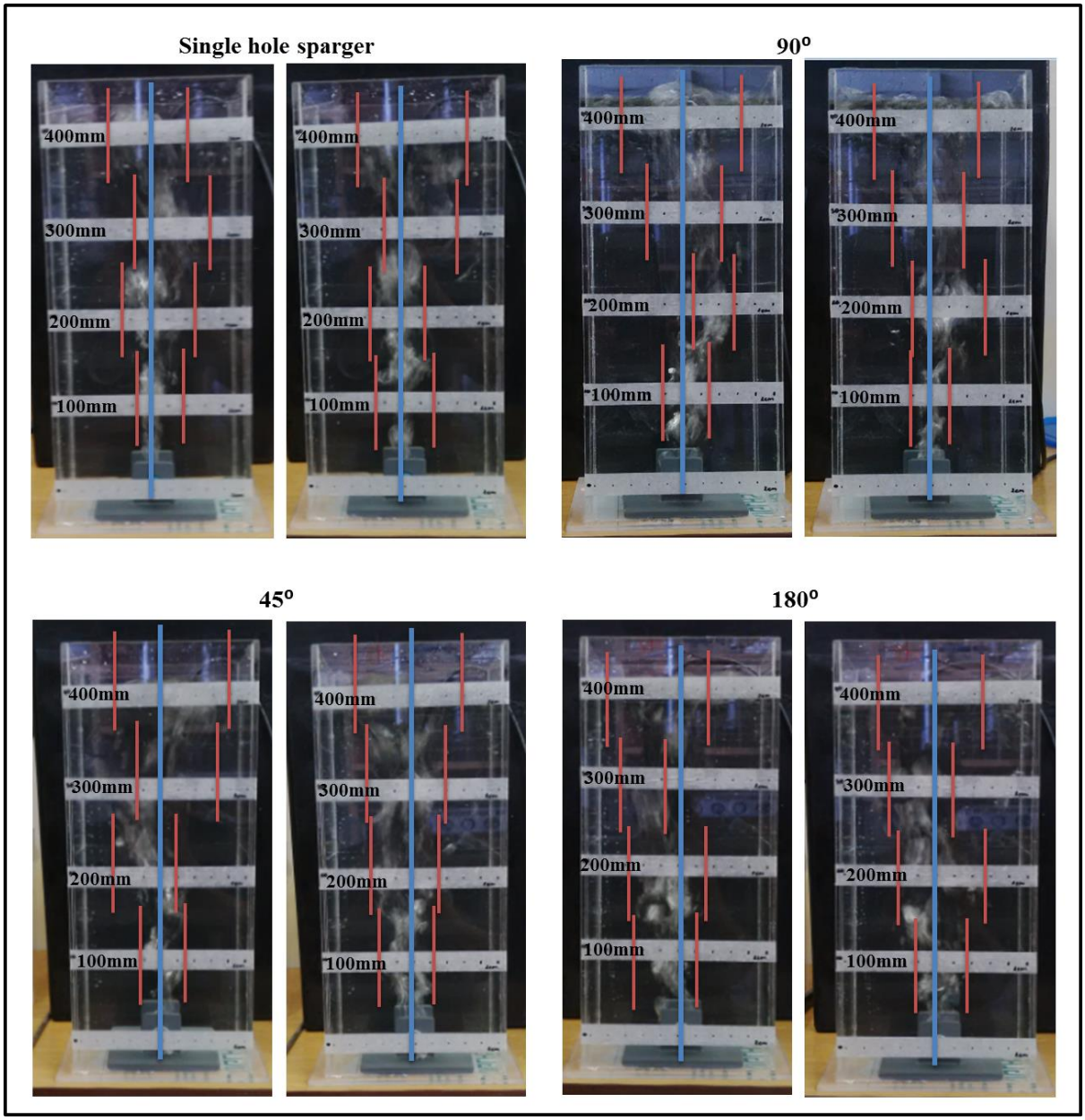

Figure 77: Bubble column dispersion evaluation for the sparger pore orientations investigated.

The figure indicates the distribution of the bubble column in the x-direction from the centreline. The centreline was indicated in the figure by the blue horizontal line and the noted bubble column dispersion was indicated in the figure by the red lines.

From the figure it was apparent how the lack of a dividing unit to designate the flow between a riser and downcomer section, resulted in turbulence close to the surface of the vessel. For this reason, the bubble column dispersion from a height of 350 mm to 400 mm was not considered for the design comparison. The results of the bubble column dispersion in the x-direction have been summarised in Table 10:

Table 10: Average coarse bubble column dispersion summary.

Sparger pore orientation design	Single pore	90°	45°	180°
Membrane-sparger height	Average bubble column dispersion from centerline (mm)			
100mm	30	23	28	33
200mm	37	31	39	48
300mm	40	40	45	34
400mm	49	61	64	56

The table indicates the average dispersion noted for the four diffuser pore patterns designs tested at 100 mm increments from the diffuser unit. From the table it is apparent that the 180^0 diffuser design resulted in the largest dispersion for a height range of 0-200 mm from the sparger, whereas the 45^0 diffuser design resulted in the largest dispersion for a height range of 200-400 mm. The 180° diffuser design was selected as the design to be incorporated into the woven fabric flat sheet membrane apparatus. This was for its effective bubble column dispersion between the 0-200 mm height range which limited the membrane stand size required.

The second step of the bubble column dispersion was performed using the 180° diffuser design. The results can be seen in Figure 78:

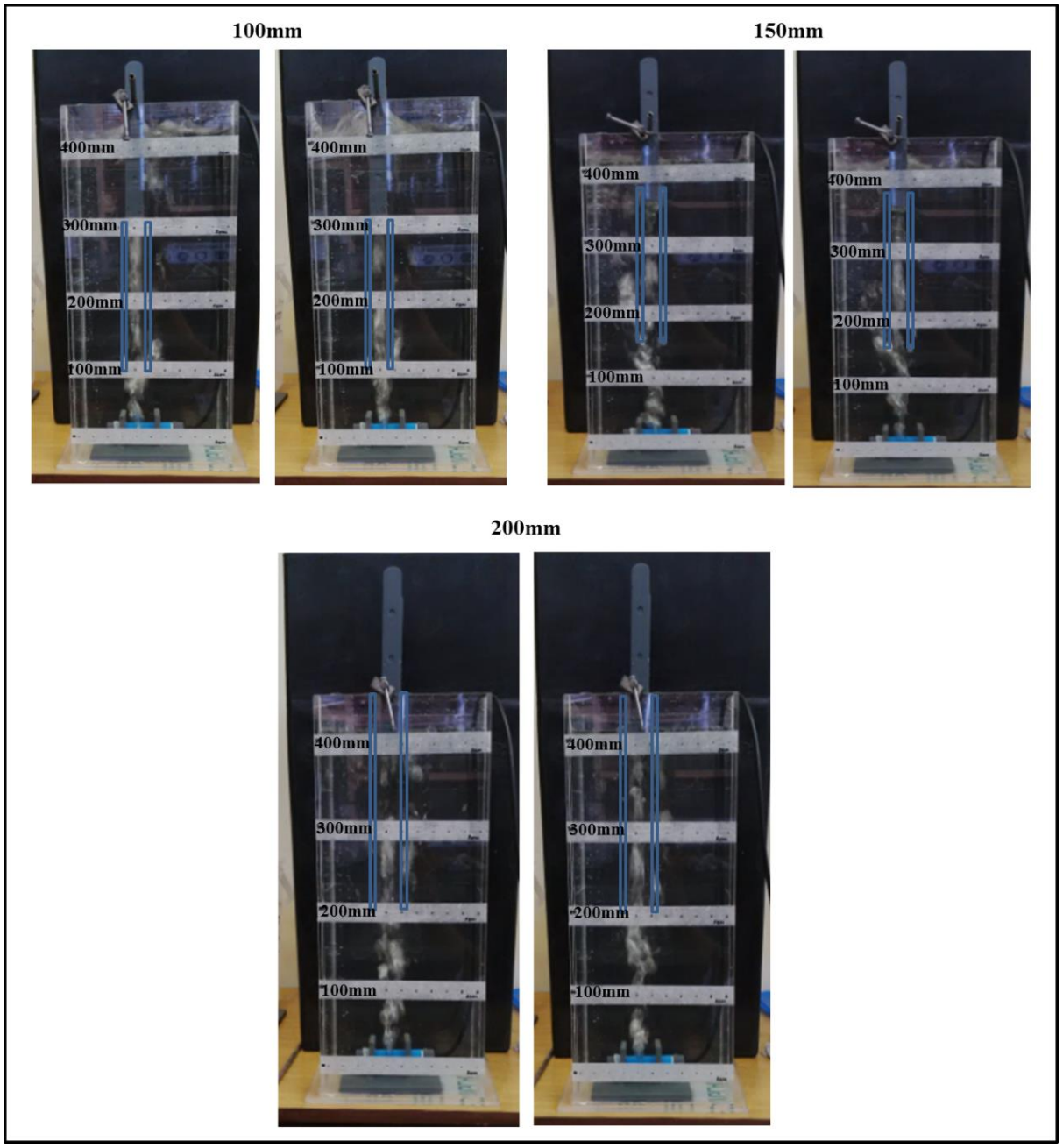

Figure 78: Membrane-sparger distance investigation for the selected 180° coarse sparger diffuser design.

Two photographs of each of the positions were taken to capture the fluctuations in the bubble column. From figure it was apparent how the bubble stream required a distance over which to stabilise, where after the dispersion in the y-direction was noticeably less. This can be seen by the decrease in the dispersion between the 150 mm and 200 mm height images as a result of the increased stabilisation of the bubble column over the additional 50 mm height travelled.

For practical purposes the 200 mm height is not applicable, due to the dispersion of the bubble column occurring between the heights of 100 mm and 200 mm before stabilising. This dispersion would prove problematic when multiple diffuser units were placed alongside due to the increased potential for interference between the adjacent bubble streams in this region. For this reason, the membrane-sparger height range, for the coarse bubble 180^0 diffuser design, was selected to be 50-100 mm.

E.1.4.2 Results and discussion of fine air scouring

The results of the fine air scouring investigation for evaluating the bubble column dispersion in the x-direction for the diffuser designs tested can be seen in Table 11:

Table 11: Average fine bubble column dispersion summary.

Sparger pore orientation design	Single pore	90°	45°	180°
Membrane-sparger height	Average bubble column dispersion			
100mm	28	30	30	33
200mm	24	27	30	40
300mm	29	32	33	33
400mm	44	43	27	42

The table summarises the distribution of the bubble column in the x-direction from the centreline, as a result of the fine air sparger diffuser pore designs tested.

From the table above, it is apparent how the 180° diffuser design resulted in the largest dispersion for a height range of 0-200 mm from the sparger, whereas the dispersion from a height range of 300-400 was very similar for the four designs tested. The 180° diffuser design was selected as the design to be incorporated into woven fabric flat sheet membrane apparatus. This was because of its effective bubble column dispersion in the 0-200 mm height range. This limited the membrane stand size required.

The second step of the bubble column dispersion investigation was performed, based on the selection of the 180° diffuser design. The test was performed by visually evaluating the dispersion noted in the images captured. The membrane-sparger height range for the fine bubble 180° diffuser design was selected to be 50-100 mm, based on the results of the test performed.

E.1.5 Finalised sparger design and membrane stand dimensions

The coarse and fine air sparger diffuser designs selected can be seen in Figure 79:

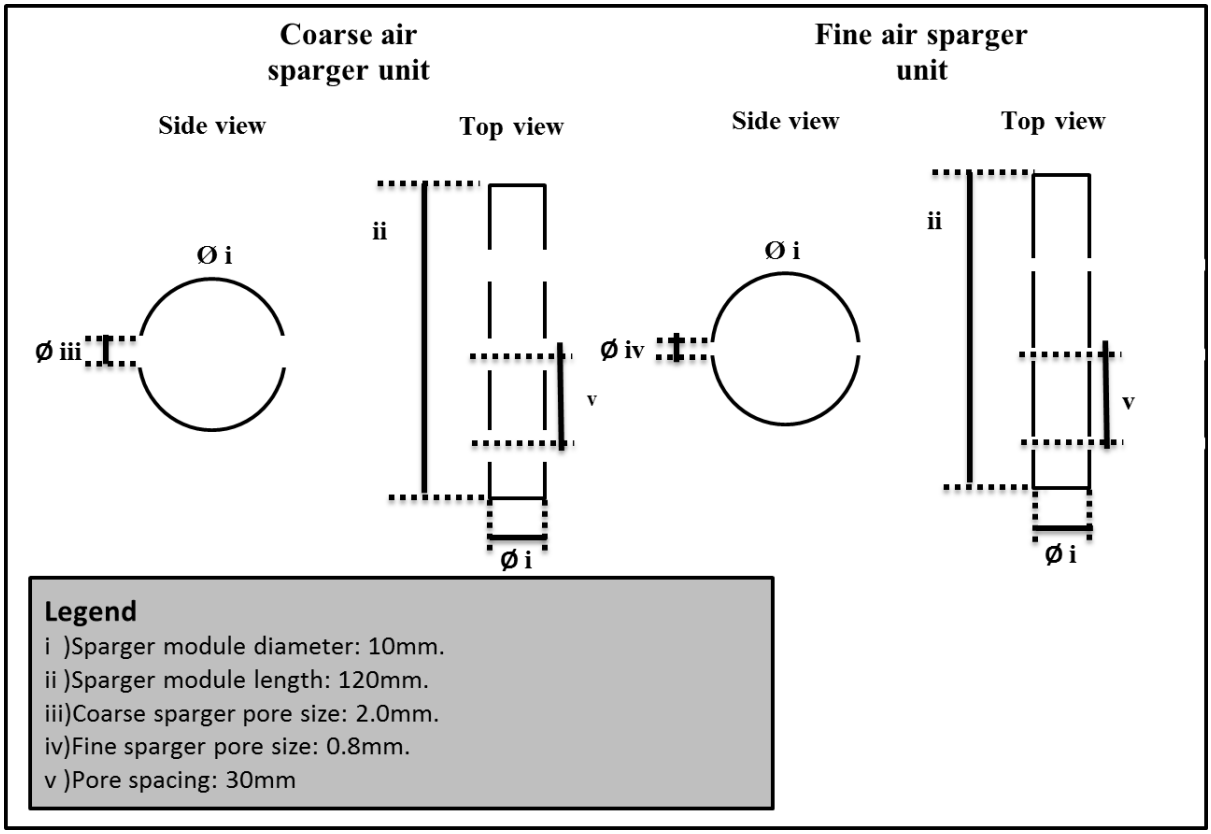

Figure 79: Revised air sparger design and configuration.

i) A sparger module diameter (10 mm) was selected to reduce the flow hindrance of the recirculating fluid flow between the sparger units.

ii) A sparger module length (120 mm) was determine by the membrane stand width.

iii) A coarse sparger pore size (2 mm) was selected based on literature indicating the cut-off pore size between fine and coarse air scouring as being 1 mm. 2 mm was selected to ensure the formation of coarse bubble air scouring.

iv) A fine sparger pore size (0.8 mm) was selected based on literature indicating the cut-off pore size between fine and coarse air scouring as being 1 mm. Pores smaller than 1 mm are considered as fine bubble air sparger diffusers.

v) Pore spacing of 30 mm was required to provide a dedicated bubble stream to each of the membrane modules. This was therefore dictated by the inter-membrane spacing utilised.

The coarse and fine air sparger diffusers were designed for a flow of 2.5 L/min and 1.7 L/min respectively per row of diffuser pores, therefore the diffusers were designed for a flow of approximately 7.5 L/min and 5 L/min respectively based on the diffuser units having three rows of pores.

From the above investigation the remaining membrane stand dimensions could be determined. These are summarised in Figure 80:

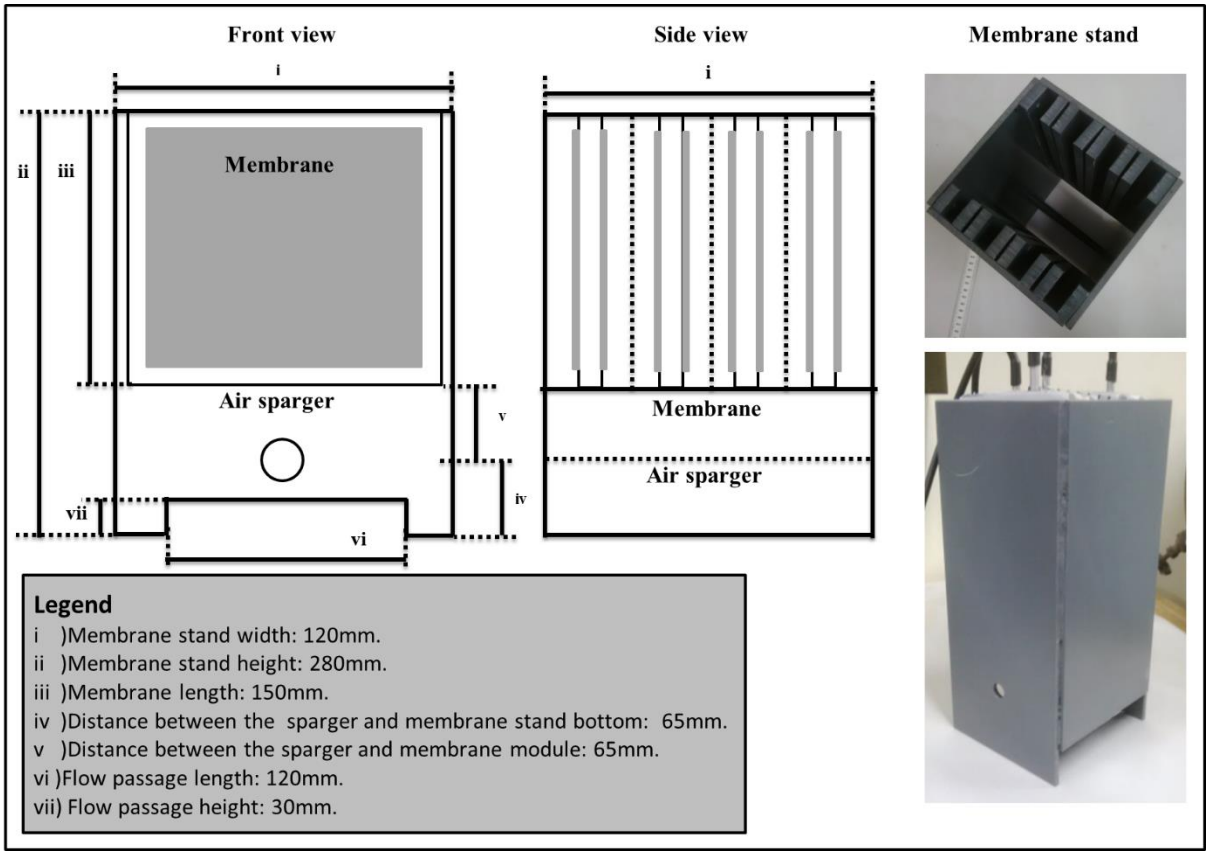

Figure 80: Revised membrane stand dimensions and configuration.

i) The membrane width (120 mm) was designed around the laboratory scale membrane module dimensions.

ii) The membrane stand height (280 mm) was designed around the laboratory scale membrane length, sparger-membrane spacing and the bottom of the membrane stand-sparger spacing selected.

iii) The membrane length (150 mm) was fixed based on the laboratory scale membrane module dimensions selected at the start of the investigation.

iv) The distance between the sparger and the membrane stand bottom (60 mm) was an important measure as it had an effect on the two-phase fluid flow. The distance between the bottom of the membrane stand and the air sparger acted as a reservoir for the fluid being entrained within the rising bubble column formed [1]. This measure was therefore maximised for the membrane stand height selected.

v) The distance between the sparger and membrane (60 mm) was selected based on the results of the experimentation carried out, which indicated an applicable range of 50-100 mm.

E.2 Membrane module design

The finalised flat sheet woven fabric membrane modules and their placement within the membrane stand can be seen in Figure 81:

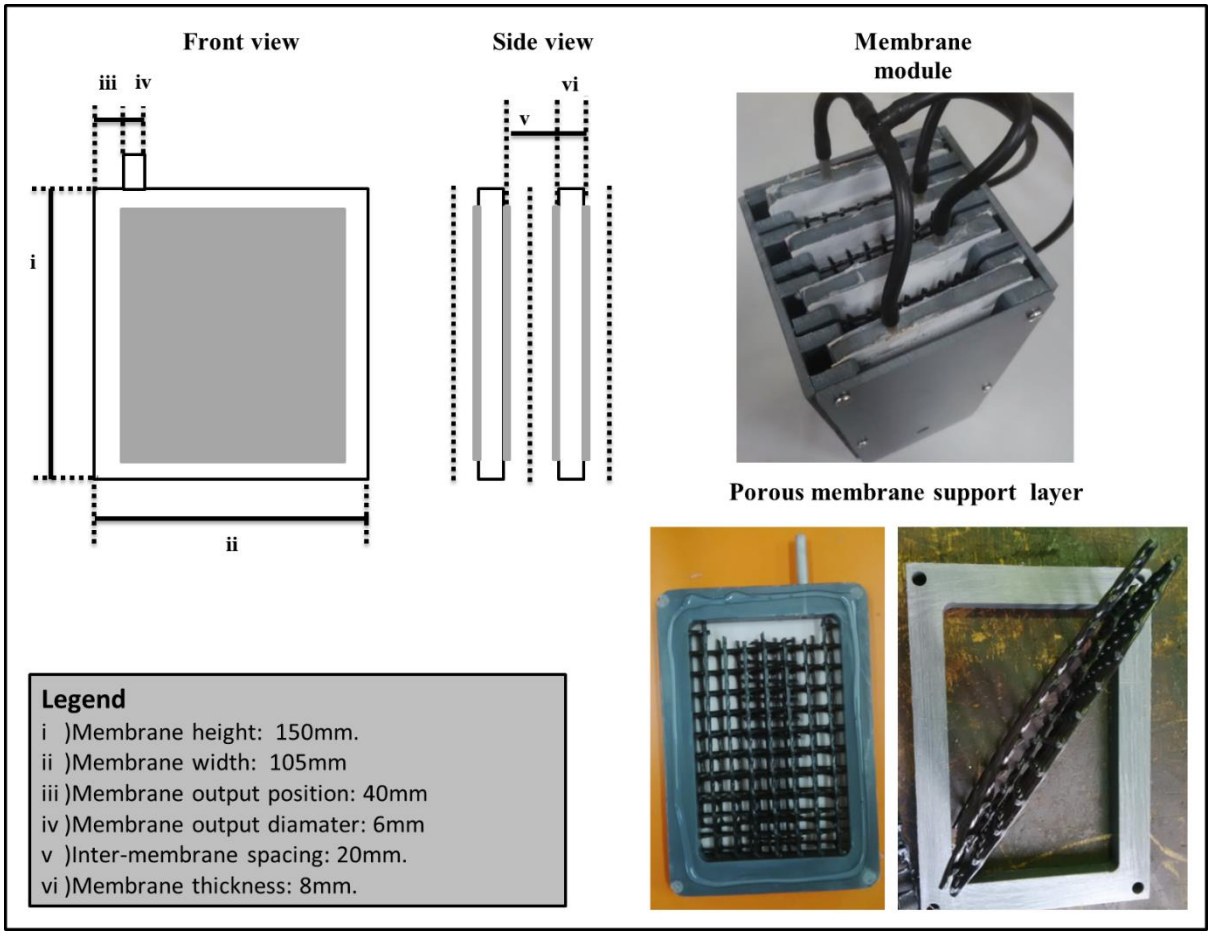

Figure 81: Membrane module design.

i) The membrane height (150 mm) and **(ii) The membrane width (105 mm)** were selected as the standardised laboratory scale membrane module dimensions.

iii) The membrane output (40 mm) was positioned close to the edge of the membrane frame to reduce the piping requirements between the manifolds and the membrane modules.

iv) The membrane output diameter (6 mm) was selected based on it being the largest diameter outlet that could be used with the 8 mm PVC membrane frames without compromising the membrane frame integrity.

v) The inter-membrane spacing (20 mm) was an important membrane design parameter as it affected the membrane packing density and the resultant hydrodynamics. An inter-membrane spacing of 20 mm was selected due to the inter-membrane porous spacer effectively halving the inter-membrane spacing. This resulted in a membrane to porous support spacing of 9 mm (the porous spacer is 2 mm thick). The 9 mm was within the specified spacing range of 5-12 mm discussed in literature [1-6].

The reason for the increased spacing was due to the woven fabric material expanding during positive pressure operation. A woven fabric flat sheet membrane module with dimensions of 150x105x8 mm has the capability of expanding outwards by approximately 9 mm during a backflush. Therefore, to prevent the adjacent membrane modules from contacting one another, during positive pressure operation, a relatively larger inter-membrane spacing was required with the addition of a porous support layer placed between the adjacent membranes. The preliminary laboratory scale woven fabric membrane apparatus, as represented in Figure 65, of Appendix D: Preliminary investigation of flux enhancement methods, utilised a 5 mm membrane to porous support spacing. This spacing was not sufficient to reduce the flow hindrance caused by the expansion of the woven fabric during positive pressure operation. The spacing was therefore increased to an inter-membrane spacing of 9 mm to improve on the flow characteristics between the membrane modules.

E.3 Revised laboratory scale membrane apparatus

A photograph of the revised woven fabric flat sheet membrane apparatus can be seen in Figure 82:

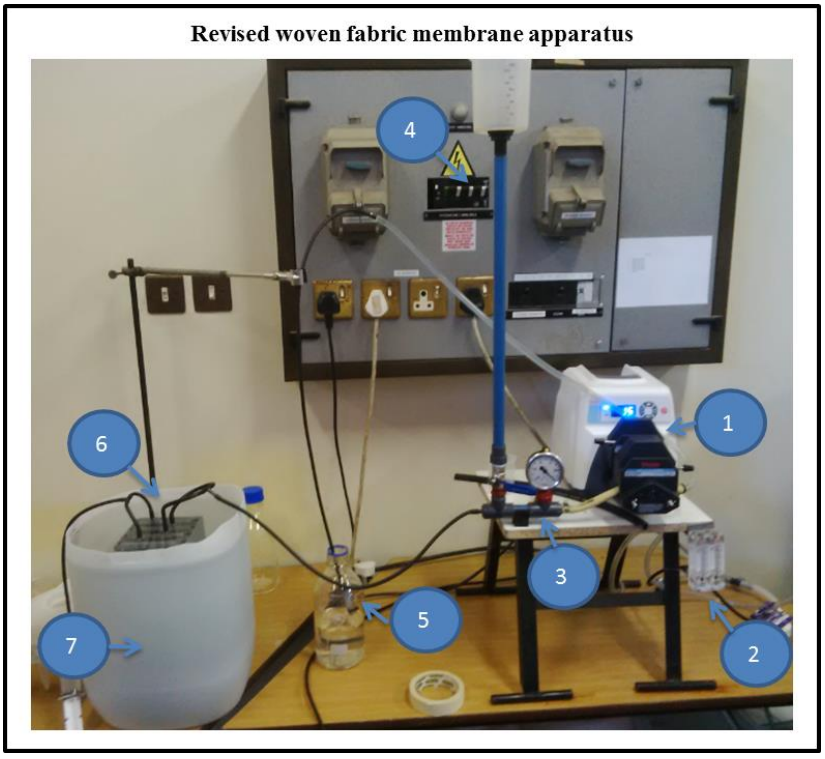

Figure 82: Revised laboratory scale woven fabric membrane apparatus.

The components that make up the complete membrane apparatus are numbered and illustrated in the figure, with the description of the components discussed below:

1: The permeate driving force was provided by a MasterFlex 6-600rpm, easy load, peristaltic pump.

2: The air scouring air flow rate was monitored using a rotary meter with a flow range of 0-10 L/min.

3: The TMP was monitored using a non-stepped vacuum gauge with a pressure range of -100-0 kPa.

4: A height variable backflush reservoir with a backflush pressure range of 3-10 kPa was used to vary the gravity backflush intensity.

5: The permeate was collected in a separate vessel and not recycled back to the membrane.

6: Indicates the membrane stand within the holding vessel.

7: A 25 L holding vessel was utilised.

The method used to determine the membrane TMP is outlined in Chapter 3.2.2 Resistance calculations. However, the increased surface area of the membrane modules utilised resulted in a negligible membrane TMP for the flow range utilised in the subsequent investigations.

E.4 Summary

From the membrane apparatus design review:

- A coarse and fine air sparger diffuser unit design was selected for the respective membrane apparatus.
- The hydrodynamics of the membrane apparatus was altered to improve the flux enhancement of air scouring and backflush orientated methods.