Submerged hollow fiber membranes : hydrodynamic and fouling study

Yeo, Adrian Piah Song

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Submerged Hollow Fiber Membranes: Hydrodynamic and Fouling Study

Adrian Yeo Piah Song

**School of Civil and Environmental Engineering
College of Engineering**

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ABSTRACT

Abstract

Hollow fiber membranes have the advantage of the highest surface area to volume ratio of any type of membrane configuration. This thesis examines the interaction between the filtration performance and the bubble-induced hydrodynamics around submerged hollow fibers as well as physical characteristics such as fiber looseness and aerator configuration. Two non-invasive methods, Particle Image Velocimetry and Phase Contrast X-Ray MicroImaging (XMI) were used to characterize shear stresses, fiber movement and fouling. It was found that it was possible to provide the same level of performance with significantly less air flow by using smaller bubbles as opposed to larger ones. The standard deviations of the shear stress, and the acceleration of the fiber showed the best correlation with the performance for the tight fibers and loose fibers respectively. XMI was shown to be very useful for observing bubble formation and fouling within the membrane.

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LIST OF SYMBOLS

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B	Parameter as defined in Section 2.4.2
c_v	Cake voidage fraction
F_p	Flux with two phase flow
F_{p0}	Flux with only liquid flow
g	Acceleration due to gravity
h	Submerged height of fiber
J_{mi}	Average imposed flux based on inner diameter area
J_w	Clean Water Flux
l	Length of hollow fiber
$p(x)$	Pressure distribution along fiber
P_T	Transmembrane pressure
R_M	Membrane resistance
R_F	Fouling resistance
R_G	Concentration polarization resistance
s_v	Solids volume fraction
x	Distance along hollow fiber

Greek Letters

α	Parameter as defined in Section 4.3.3
λ	Defined by $s_v/(1-c_v-s_v)$
ρ_L	Density
τ_a	Shear stress due to axial flow
τ_b	Shear stress due to bubbling
τ_{ba}	Shear stress due to the interaction of axial flow and bubbling
τ'_a	Standard deviation of shear stress due to axial flow
τ'_b	Standard deviation of shear stress due to bubbling
τ'_{ba}	Standard deviation of shear stress due to the interaction of axial flow and bubbling
τ_{total}	Total shear stress
τ'_{total}	Total standard deviation of shear stress
ρ_L	Density
τ_{max}	Maximum shear stress in two phase flow

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τ_{min}	Minimum shear stress in two phase flow
μ	Viscosity

CHAPTER ONE

CHAPTER ONE INTRODUCTION

1.1 Introduction

Traditional methods of water and wastewater treatment are gradually being replaced by membrane filtration. The main advantages of water filtration using membrane processes are:

1. Invariable quality of produced water
2. Considerably lower amount of chemicals
3. Compact and small foot print
4. Maintaining the process without interruption under mild environmental conditions
5. Potentially lower consumption of energy as compared with other treatment techniques.

Compared to tubular, flat sheet, or spiral wound membrane modules, hollow fiber modules have the advantage of high specific membrane area. The increase in specific membrane area can be in the order of 10 to 100 times compared to the other types of membrane module. However, one disadvantage of the commercially used types is the difficulty of increasing mass transfer by means of turbulence promoters such as spacers, static mixing elements or baffles. Thus, to take full advantage of the high specific membrane area in applications such as submerged systems, it is important to understand the hydrodynamics of the submerged membrane hollow fiber membranes. This thesis seeks to increase the understanding of submerged systems with respect to the hydrodynamic conditions.

The most common way of improving the hydrodynamic situation around submerged hollow fiber membranes is gas sparging, or the introduction of bubbles around the hollow fibers. These bubbles induce mixing around the fibers and have been proven to improve flux. However, the effects of bubble size and frequency on the performance of hollow fibers have not been well documented. Moreover, the effect of aeration on a single fiber,

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and on a bundle of fibers would be different and would be affected by conditions such as the geometry and looseness of the fibers.

Another aspect of hollow fiber operation which is not well understood is the nature of flow within the lumen. Non-invasive, real-time observations of deposition on and within the membrane and the flow conditions in the lumen are particularly important in the quest for improving performance. Ideally information would be obtained at the micron to submicron scale as real time images.

1.2 Objectives

The objectives of this study are to:

- Determine the filtration behavior of submerged hollow fibers under different bubbling conditions;
- Determine the hydrodynamics within the hollow fiber module using Particle Image Velocimetry (PIV) techniques;
- Use X-ray Micro-Imaging (XMI) and PIV for non-invasive study of the fouling of hollow fiber membranes.

1.3 Organization of the Thesis

The thesis structure is as follows:

Chapter One gives a brief introduction to the problems studied. The objectives determined in this chapter aim to resolve the research problem

Chapter Two reviews the relevant theoretical and empirical literature concerning hollow fiber membrane systems.

Chapter Three describes the methodology used to conduct the experiment and well as providing details of the equipment used in the experiments.

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Chapter Four discusses the studies done on a model bundle of submerged hollow fibers. Information from filtration studies and PIV are combined to link hydrodynamics to fouling. Bubbles of different sizes and frequencies were used.

Chapter Five reports on the studies done on a single hollow fiber membrane, held tightly and with varying degrees of looseness. Again, the data from filtration and PIV are compared under different bubbling conditions.

Chapter Six introduces a new technique, XMI, for the observation of membrane phenomena. The application of this technique to observe fouling and flow phenomena is described.

Chapter Seven summarizes the conclusions from this study and gives recommendations for future research.

Appendix A A brief report is included as an appendix of the application of membrane filtration to disaster relief. This was carried out by the author in response to the Tsunami on December 26th, 2004.

Appendix CD-ROM Selected Experimental Results and Shear Stresses measured by PIV and XMI movies

CHAPTER TWO

CHAPTER TWO LITERATURE REVIEW**2.1 Introduction**

The membrane family is large and can be categorized according to their processes. Pressure driven membranes, such as microfiltration (MF) and ultrafiltration (UF) membranes, remove contaminants by various mechanisms depending on pore size. Solute transfer membranes, such as electrodialysis or dialysis membranes, remove contaminants by electrochemical, diffusive or preferential solubility effects. Thermally driven membranes, such as membrane distillation (MD) membranes, require a heated feed, and the liquid water undergoes phase change in order to pass through the membrane. Hybrid membrane processes are processes which couple one of the above membrane methods with another unit process such as adsorption, ion exchange or coagulation.

Pressure driven membranes are divided according to their pore size. Microfiltration membranes typically have pore sizes of 0.1 to 1.0 microns. They provide high removals of suspended solids including most bacteria, as well as partial removal of viruses and macromolecules. Ultrafiltration membranes have pore sizes ranging from 0.1 micron to less than 5nm. These membranes provide high removals of viruses and macromolecules including reasonable removal of extracellular polymeric substances (EPS). Nanofiltration membranes (NF) have pores that are approximately 2nm or less in size and retain most species, except for certain monovalent ions and low molecular weight organics. Reverse Osmosis (RO) membranes are essentially pore-free (or <1nm size) and give high retention of most species including monovalent ions.

There are several types of configuration of membranes. Common modules are tubular modules, plate modules, spiral-wound modules and hollow fiber modules. Tubular membranes are membranes cast on the inside of porous paper or plastic inserts with varying internal diameters and lengths. Plate type membrane modules consist of a flat rigid plate on which a flat sheet of membrane is placed. Often, a net like material is placed between the membrane sheet and the plate to provide the channel for permeate flow.

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Spiral wound modules consist of two flat sheets placed together with their active sides facing away from each other. They are separated from each other by a thin mesh like spacer material and are glued together on three sides. The fourth open side is fixed around a perforated center tube. Another mesh-like spacer of the required thickness is placed on one side of this envelope and the whole assembly rolled around a center tube in a spiral configuration.

2.1.1 Hollow Fiber Membranes

Hollow fiber membranes are similar to tubular membranes. However, they have smaller diameters (typically $< 2\text{mm}$) and are self supporting. Membrane modules with hollow fiber membranes usually have smaller sizes as compared to other modules with the same capacity. Hollow fibers are used extensively in the water industry. Perhaps the most well known uses of hollow fibers are in water treatment to remove turbidity and pathogens and in membrane bioreactors (MBRs) for wastewater treatment.

Membranes can be incorporated into the design of wastewater bioreactors as the solids/liquid separation stage in two ways – in an external loop or integrated into the bioreactor as submerged membranes. In the external loop design, the feed is circulated through a membrane module and the transmembrane pressure (TMP) is generated by the pump. In submerged systems the membranes are located in the mixed liquor tank, and bubbling is applied to produce surface shear. As the permeate is removed by suction, this insures a low TMP. Hollow fibers have been successfully used in submerged bioreactors and studied for more than a decade (Chiemchaisri & Yamamoto., 1994; Shimizu et al., 1996; Bouhabila et al, 1998; Yeom et al., 1999; Nagaoka et al., 1996; Gunder et al., 1998). Submerged membranes are also used in water treatment and pretreatment to RO and in these applications the solids loading is relative low so the bubbling is usually restricted to a cleaning phase at the end of a filtration cycle.

While the use of hollow fibers has many advantages, the disadvantage of using hollow fibers is their higher propensity to foul due to the poor hydrodynamics associated with the

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modules. One of the main methods for limiting fouling in membrane bioreactors is the use of aeration. The beneficial effects of aeration are widely known, however, there have not been any reported studies done on optimizing the airflow rate by varying the bubble size and frequency. Thus, at a given air flow rate and module design, there should intuitively be a combination of bubble size and frequency that generates the best cleaning effects on the surface of the membrane.

To ensure that hollow fiber membranes are used efficiently, it is important to design the module in such a way that the fibers foul evenly. The importance lies in the differing critical fluxes of each individual fiber in a hollow fiber membrane bundle due to the different hydrodynamic situations experienced.

In MBR experiments operating at constant flux, the trans-membrane pressure (TMP) increases slowly before there is a sudden TMP jump (Cho & Fane, 2002). There have been several reasons suggested for this transition to rapid fouling. One of the explanations is that uneven deposition occurs and this leads to self accelerating fouling. Hollow fiber modules would not be immune from this as demonstrated in Chapter 5 of this thesis.

2.2 Some Basic Concepts

This section briefly introduces some basic concepts of membrane filtration that apply to hollow fiber modules studies in this thesis.

2.2.1 Fouling, Blocking and Concentration Polarization

The reduction of flux passing through a hollow fiber membrane can be due to one of three processes. Concentration polarization refers to a build up of a higher concentration of particles or solutes on the surface of a membrane. Fouling refers to adsorption and deposition of particulates on and within the membrane. Inter-fiber blocking is a problem

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unique to hollow fiber membrane bundles. While both concentration polarization and fouling reduce flux, they have opposing effects on rejection.

Figure 2.1 Concentration Polarization

Concentration polarization (Figure 2.1) is a complication that arises when relatively large solutes or particles are filtered. Initially, as a result of convective transport of solute to the membrane, solute buildup will cause a steep concentration gradient within the boundary layer. This causes a back-transport of solute to the bulk because of diffusion. Eventually the two phenomena balance each other. Depending on the type of particle, this layer could be fairly viscous or gelatinous. This causes further resistance to the flow of permeate and flux is adversely affected.

Fouling is characterized by the attachment or adsorption of particles on or in the membrane. Operationally, there are three types of particle attachments to surfaces of membranes. Strong attachments refer to particles still present after the surface is cleaned. Weak attachments describe particles that are still absorbed at the surface when the filtration process is stopped, but would be removed by cleaning. Dynamic accumulation is the most diffuse and the weakest type of attachment. It comprises particles in the polarized layer and at the surface as a result of absorption due to high surface

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concentration during polarization. This type of attachment is reversible – once the filtration process is stopped, the particles diffuse away.

There are four mechanistic models used to describe fouling (Bolton et al., 2005). These models are based on how the particles interact with the membrane pores, or the surface of the membrane. Complete blocking occurs when particles seal off the pore entrance and prevent flow through the pore. When the particles obstruct the entrance of the pore, but do not seal it completely, it is referred to as intermediate blocking. Cake filtration occurs when the particles are too large to enter the pore, but build up a layer on the membrane surface. Standard blocking models the pores as straight cylindrical holes, and the particles accumulate within this cylinder.

Figure 2.2 Blockage due to cake accumulation (a) totally dispersed (b) partially accumulated (c) surface clogging

Figure 2.2 shows the different types of blockages that occurs in hollow fiber bundles (Kiat et al., 1992). For a closely packed bundle of hollow fibers, it has been observed that the spaces between the fibers get filled with particulates, and as more and more of the spaces are blocked, there are changes in the liquid flow distribution. This has the effect of significantly reducing the total flux.

The control of concentration polarization and fouling is the main objective in membrane operation. The use of crossflow, either driven by a pump or bubble induced, causes shear

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stress of the surface of the fiber, enhances back diffusion and the removal of attached particles. Other methods to combat pore blockage include periodic backwash.

2.2.2 Darcy's Law

Membrane fouling behaviour can be described by the “resistance in series” model (Darcy's Law) that is common in heat transfer and electric circuits. For an ideal membrane in pure water, the flux can be modeled as

$$J_w = \frac{P_T}{\mu R_M}$$

Where J_w is the clean water flux of the membrane, P_T is the trans-membrane pressure and R_M is the intrinsic membrane resistance. However, R_M is not a good indicator of membrane performance under real operating conditions, as fouling will occur. If significant membrane fouling occurs because of membrane-solute interactions, then this is accounted for by adding another resistance term because of fouling (R_F) to the model.

$$J = \frac{P_T}{\mu(R_M + R_F)}$$

In general, R_F is relatively unaffected by operating parameters as it is due to physicochemical reactions. To account for the build up of cake, or the concentration polarization layer, an additional resistance term, R_G is added to the equation.

$$J = \frac{P_T}{\mu(R_M + R_F + R_G)}$$

This resistance model has been validated by many studies (Chaing et al, 1986; Yeh et al., 1997), and gives an accurate result when modeling flat sheet membranes.

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2.2.3 Critical Flux Concept

In the ideal case, there are two forces acting perpendicularly on a particle experiencing filtration in a membrane system. The first is the force exerted due to the movement of water towards the surface of the membrane, the force due to filtration. The other is the lift force caused by cross flow along the surface of the membrane. Thus, in the ideal case, there are three possible situations. If the filtration force is less than the lift force, then no particles will accumulate on the surface of the fiber. If the filtration force is greater than the lift force, then particles will start to accumulate on the surface of the fiber. There must exist a point where the filtration force exactly balances the lift force, and the flux at which this condition occurs is termed the critical flux. Hence, the critical flux is understood to be the flux at which no particle deposition occurs on the surface of the membrane, and hence no flux decline occurs (Field et al., 1995)

Figure 2.3 Effect of Aeration Intensity on Critical Flux (Yu et al., 2003)

The hydrodynamic conditions including the aeration intensity can affect the critical flux (Figure 2.3). Yu et al. (2003) identified 3 different zones for flux. At “sub-critical flux”, the MBR can be operated for long periods without large increase in pressure. Within the “supra-critical” flux, the pressure increased rapidly, and operation was unsustainable.

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Between these two is the “critical zone”. Increasing the aeration intensity generally causes the critical flux to increase due to higher shear stress generated on the surface of the fiber.

2.3 Filtration Performance of Submerged Hollow Fiber Membranes

There are several factors that affect the performance of submerged hollow fibers. They include the physical properties of the membrane, such as the length, diameter and looseness, as well as hydrodynamic properties such as the bubbling rate and the cross flow velocity.

2.3.1 Fiber Diameter/Length

The fiber diameter can affect the filtration performance of the hollow fiber by influencing the pressure drop along the fiber. It also has an effect on the hydrodynamics and movement of the fiber.

Studies have reported high pressure drops within a hollow fiber membrane operating from shell to lumen (Chang et al., 2002). This is due to the small internal diameter of the hollow fiber. Due to higher suction pressures present nearer the top of the fiber, there would be higher local fluxes nearer the suction point of the fiber. Thus, the fiber both experiences an average total flux, as well as a local flux. If the fiber is long, it is possible that even though the average total flux is below the critical flux, there would be a region of the fiber where the local flux is higher than the critical flux (Chang et al., 2002; Cho & Fane, 2002). This could be one of the explanations for TMP jumps experienced in some MBRs. Increasing either the fiber length or decreasing the fiber diameter causes the pressure drops to be more pronounced, which is generally detrimental to the performance of the fiber.

The effect of fiber diameter also depends on whether fluid flow is driven by pump recirculation or airlift (Chang et al., 2001). With pump recirculation, at low cross-flow

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velocities, the effect of diameter on flux decline is almost negligible. However, with an increase in cross-flow velocity or injection of air, the effect of diameter increases. A significantly lower flux decline occurred for filtration with bubbling with the smaller diameters. This was due to the fact that performance enhancement was closely related to the movement of the fiber. Smaller fibers were more flexible than the larger fibers, and moved more in response to the hydrodynamic situation (Wicaksana et al., 2006).

With air lift driven crossflow, performance is sensitive to changes in fiber diameter, and flux decline appears to increase with an increase in fiber diameter. Comparison between the two modes of operation indicated that although the flux was higher for pumped recirculation, the difference between the two diminished with a decrease in fiber diameter.

Experimental results indicate that the effect of fiber diameter on filtration increases with an increase in turbulence around the fibers (Chang & Fane, 2001). For filtration with bubbling, the performance was sensitive to the fiber diameter and a significantly higher flux was obtained with the smaller fiber.

When the fiber is operated by applying suction to the inside of the fiber, due to the distribution of pressure along the fiber, which is higher at the end suction is being applied, the equation of the pressure distribution along the fiber is as follows: (Chang & Fane, 2001)

$$p(x) = (h + l - x)g\rho - \frac{\mu\lambda R_m J_{mi} l}{e^{\lambda l} - e^{-\lambda l}} (e^{\lambda l} + e^{-\lambda l})$$

Due to this pressure distribution, the flux will also be distributed locally along the fiber.

$$J(x) = \frac{\lambda l J_{mi}}{e^{\lambda l} - e^{-\lambda l}} (e^{\lambda l} + e^{-\lambda l})$$

The explanation of the symbols is given in the List of Symbols.

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The hollow fiber is therefore producing an average flux below the critical flux, but locally, the flux along the fiber could exceed the critical flux. If the maximum local flux is smaller than that of the critical flux, then no build up of particles occurs.

However, if the maximum local flux is higher than the critical flux, then deposition will occur locally along the fiber. In constant flux operation, this will lead to a pressure increase as the flux is distributed along the fiber. A detailed explanation regarding this phenomenon can be found in Chang & Fane (2001).

2.3.2 Fiber Looseness

In most applications, hollow fiber membranes are not held very tightly to allow for movement. Movement of hollow fibers in MBRs is generally caused by the introduction of air into the module. Looser fibers have greater degrees of movement, which can enhance back-diffusion into the solution. Fiber movement when multiple fibers are present can also result in physical contact between the fibers. This can enhance the permeate flux by mechanically eroding the foulant layer on the membrane surface (Berube and Lei, 2005). It is estimated that up to 40% improvement in filtration performance can be achieved by using loose fibers as opposed to tight fibers (Wicaksana et al., 2006). The use of loose fibers in MBRs can be considered to be one of the most important parameters in preventing fouling (Delgado et al., 2004).

However, there appears to be a limit to the looseness of the fiber with respect to improving the filtration performance. For example, Wicaksana et al. (2006) found that increasing the looseness of the fiber from 99% to 96% resulted in only a marginal improvement to the performance of the hollow fiber. In the same study, the role of fiber movement per se was examined in enhancing the performance of the hollow fiber. This was done by manually moving the fiber without bubbling. While there was an improvement in the performance of the hollow fiber with mechanical movement, it was much better to use bubbling to induce fiber movement. This indicates that bubbles play multiple roles in the control of fouling.

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2.3.3 Fiber/Module Arrangement

One of the main advantages of using hollow fiber membranes is the high membrane surface area to volume ratio, and the smaller footprint required compared to other membrane types. Hence, the importance of membrane packing density cannot be overlooked. Packing the membranes in a random manner causes serious mal-distribution in the flow within the hollow fiber bundle. It has been found that more than 40% of the flow within the bundle flowed through the largest 20% void area of the total module void area. The smallest 20% void area only carried 6% of the flow. Mal-distribution of the flow could cause differential fouling, and be detrimental to the performance of the fiber (Zheng et al., 2003).

The productivity of hollow fiber membranes is related to both flux as well as membrane packing density. A higher membrane packing density would increase productivity, as long as flux does not suffer greatly. Kiat et al. (1992) studied the effect of different packing densities on the flux decline and clogging mechanisms of hollow fibers. For a kaolin clay suspension, increasing the packing density decreased steady state flux to a minimum which then increased slightly. The flux did not decrease even when the packing density increased to the maximum possible. This suggests that the cake layer resistance formed on the bundle surface was negligible, and no surface clogging was observed. Kiat et al. went on to suggest that hollow fibers be operated based on the condition of no particle accumulation, i.e. below the critical flux. The productivity of the hollow fiber module as studied by Kiat et al. decreases to a minimum as packing density increased and then increased as packing density increases to the maximum possible. This suggests that packing density should be as high as possible to get maximum efficiency from a membrane plant. However, experiments conducted in this thesis have shown that an increasing packing density leads to poorer hydrodynamic conditions, and increased fouling.

The optimization of packing density also depends on the permeate flux passing through the membrane (Serra, 1998). The membrane area available for filtering increases when

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the void fraction of the bundle or the fiber diameter is reduced; however, a reduction in these two parameters increases the pressure drop inside the fibers and in the bundle. A higher net flow rate tends to shift the optimum combination towards bigger diameters and more void volume.

2.4 Aeration

A comprehensive review on aeration and its application on transport processes pertaining to membrane filtration has been provided by Cui et al. (2003). In this review, several issues that the authors felt needed to be optimized were the bubble size and frequency, as well as to characterize the enhancement effect of the bubbles. Both of these issues will be addressed in this thesis.

2.4.1 Bubble Types and Regimes

Figure 2.4 Bubble Shapes and wakes (Miyahara et al., 1988)

Figure 2.4 show the shapes of various bubbles and their associated wakes. Small spherical bubbles are typically less than 1mm in diameter, and obey Stokes Law. As such, they do not induce any disturbance to the flow around them. Ellipsoidal bubbles range from 1.5 to 15mm. The bubble appears to rock back and forth, and leaves helical vortices in their wake. Spherical cap bubbles are larger than 15mm, and the primary wakes induced by these bubbles is about 4.5 times the volume of the bubble. A strong secondary flow effect can be created by these bubbles.

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Figure 2.5 Gas-Liquid two phase flow patterns (Whalley, 1987)

Increasing the gas hold-up in a column leads to different flow patterns in the column (Figure 2.5). At low gas flow rates, the column experiences bubble flow. Increasing the gas hold-up changes the flow pattern to that of slug flow. This is when the gas flows as large bullet-shaped bubbles approaching the diameter of the tube. Churn flow can be characterized by oscillatory, up and down motions of the liquid. As gas rates get even higher, the gas becomes the continuous phase. Liquid flows in an annulus around the edge of the pipe with liquid droplets mixed in the gas phase. This is the annular type of flow.

2.4.2 Effect of Aeration on Transport Processes

The more severe the fouling, the better the flux enhancement due to aeration. This has been shown to be true for both ultrafiltration (Cheng, et al., 1998; Bellara et al., 1996) and microfiltration (Sur & Cui, 2002). This would imply that aeration may be a cost effective (from an energy point of view) method of reducing fouling.

Several mechanisms have been identified by which aeration contributes to the enhancement of filtration processes. Bubble induced secondary flow disrupts the mass transfer boundary layer and promotes local mixing near the surface of the membrane. It

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was also found that that for microfiltration, the passage of bubbles causes a disruption of the deposited cake and could provide enhancements of up to 135% (Sur & Cui, 2002). The agitation of water could also induce particles in the water to collide, and aggregate forming larger particles which do not foul the membrane as readily (Choi et al., 2003).

For submerged fibers with unconstrained bubbles, the fiber experiences a fluctuating shear stress on its surface. This can be characterized by the mean shear stress, as well as some sort of measurement of the temporal and spatial variation of the shear stress. There is evidence that it is this variation in the shear stress that contributes greatly to the performance of the fiber. (Cabassud et al., 2001). This is evidenced by measurements which have shown that the peak shear force was substantially higher when bubbling is applied, as opposed to when bubbling was absent (Berube et al., 2006).

Using electrochemical methods, Ducom et al. (2002) illustrated that the performance of a flat sheet ultrafiltration membrane could be related to the relative magnitude of the shear variation in the two phase flow. They defined a parameter “B”, given by

$$B = \frac{(\tau_{\max} - \tau_{\min})_{\text{two-phase}}}{\tau_{\text{single-phase}}}$$

Where $\tau_{\max}$ is the maximum shear stress experienced in two phase flow, $\tau_{\min}$ is the minimum shear stress experienced in two phase flow, and $\tau_{\text{single-phase}}$ is the shear stress experienced in single phase flow.

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Figure 2.6 Link between flux enhancement and parameter B (Ducom et al., 2002)

Figure 2.6 shows the variation of parameter B with flux enhancement. The flux enhancement is characterized by the ratio of permeate flux with two phase flow, F_p and the permeate flux with only liquid flow, F_{p0} . This demonstrates the importance of shear transients for flux enhancement.

It has been suggested that (Cui et al., 2003) bubbles can produce three different effects on the submerged hollow fibers. The first is that the wake of the bubble induces a shear stress on the surface of the hollow fiber. The second is that the bubble physically impacts onto the fiber, and the last is that if the fiber is held loosely, the bubbles cause the fiber to displace and move back and forth (see Section 2.3.2 for Fiber Looseness).

When the fiber is operated from lumen to shell mode, aeration is usually applied within the lumen of the hollow fiber. Due to the small diameter, slug flow will usually result. Generally, this phenomenon is modeled by considering the shear stresses in three different zones of mass transfer (Ghosh & Cui, 1999; Smith & Cui, 2004). The zones are the film zone (where there is falling film between the wall and the bubble), the wake zone (a region of free turbulence) and the liquid slug zone. By calculating the mass transfer coefficients in each region, it has been shown that the mass transfer in the falling film

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region and the wake region is two orders of magnitude higher than in the liquid slug region. This shows the importance of shear stress on the filtration performance of the fiber.

2.4.3 Effect of Gas/Liquid Flows

Liquid flow has been shown to have a significant effect on the fouling behavior in membrane processes. A greater cross-flow velocity generally improves performance, by improving shear flows on the surface of the membrane. (Lin et al.; 2004; Liu, 2000). However, the use of two-phase flow is often more efficient than single phase flow. One reason put forward for this is that with a two phase flow, the cake structure is different as compared to a single phase flow. Increasing the air flow rate increases the cake porosity and reduces its specific resistance. Thus, two phase flow seems to expand the cake up to a limit where the notion of a cake is no more available (Cabassud et al., 2001). In addition, two phase flow has an intrinsic stochastic nature which increases instabilities which that can disrupt the boundary layers.

For submerged hollow fiber membranes, it was found that a modest gas flow enhances the performance of the membrane. However, enhancement quickly reaches a plateau at higher gas flow rates (Chang & Fane, 2001; Berube & Lei, 2005). Thus, increasing the aeration beyond a certain point would lead to little change in the performance of the membrane. Hence, the key to the optimization of aeration is to determine when diminishing returns for aeration sets in. The reason for this may be due to the effect of slugging gas flow on the liquid velocity in the falling film (Chang & Fane, 2000). Increasing the gas flow rate may cause the flow pattern to change to the slug flow regime (Figure 2.2).

Module design is very important when considering the effects of bubbling. If the bubbles are confined to regions near the hollow fiber, enhancements in the performance of the fiber would be more obvious (Ghosh, 2005). This is due to the bubbling impacting the hollow fiber more regularly, as well as a defined air-lift induced liquid flow being formed.

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Another strategy for reducing fouling is “air-cycling”. This involves introducing the bubbles sequentially on either side of a group of fiber bundles. With this strategy, the local bubbling intensity can be increased without increasing the total gas flow rate (Guibert et al., 2002). It was found that air-cycling could be used to induce a flow through the membrane. As the bubbles rise up one side of the membrane, water was drawn through the hollow fiber bundle, and this reduced the fouling. However, the effectiveness of this technique as the fiber bundle gets thicker has not been reported.

2.5 Non-invasive Methods

In order to better understand membrane processes, it is necessary to observe the membranes in operation. While measurements of the flux and pressure yield interesting results, they do not give an insight into the basic phenomena within the membrane, the polarization layer, or the bulk fluid. Non-invasive methods aim to fill the gap in this knowledge. A comprehensive review of non-invasive techniques has been provided by Chen et al. (2004).

2.5.1 Observing the Region Close to the membrane surface

The hydrodynamics near to the surface of the membrane has a strong relationship with the performance of the membrane. In particular, the shear on the surface of the membrane is a measure of the force that detaches a particle from the surface of the membrane. The movement of particles, as well as the rate and location of fouling can be determined by observing this region. Information on the rate or the location of fouling can lead to better designs of membrane modules.

There have been several innovative non-invasive methods used to study concentration polarization. These include refractometry techniques (Ethier and Lin, 1992), interferometry (Johnson, 1974) and the use of photo-interrupt sensors (McDonogh et al., 1995). While these methods provide very useful information, concentration polarization

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is not an important concern during microfiltration. The methods in the following section will be discussed with regard to their ability to convey an understanding of particle deposition and hydrodynamics near the surface of a membrane.

(i) Direct Observation Through the Membrane

One of the methods of direct observation is the Direct Observation Through the Membrane (DOTM) technique. It involves the use of an optical microscope with both transmitted and reflected light options. With the selected magnification of the microscope and a CCTV video camera attached to the microscope with the transmitted light source, particles larger than 1 micron can be clearly identified. This method was first reported by Hodgson et al. (1993). It has since been used to study membrane phenomena such as deposition on flat sheet membranes (Li et al., 1998) and, using a modified DOTM, hollow fiber membranes (Chang et al., 2002), as well as the efficiency of back-pulsing. (Mores and Davis, 2002). The use of DOTM techniques have yielded results about particle deposition phenomena at particular operating conditions such as particle size, crossflow, and permeate flux.

In particular, for hollow fiber membranes, when the flow was transverse to the hollow fiber, it was found, using the modified DOTM techniques that inertial deposition played an important role in the deposition of yeast on hollow fibers. Due to the inter-fiber vortices, the front face of all the down stream fibers experienced decreased deposition (Chang et al., 2002). Some of the flow induced by bubbling also occurs axially with respect to the membrane, and the distribution of the flow that penetrates into the bundle affects the performance of the hollow fibers tremendously.

Though a powerful technique, DOTM methods have several drawbacks. The first is that it is generally necessary to use a transparent membrane to give a clear image. Transparent membranes are either very thin, or very fragile, and great care and patience is required. Being an optical technique, the feed solution must also be relatively clear. The difficulty

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of applying this technique to observe concentration profiles, or the hydrodynamics in the boundary layer should be apparent.

(ii) Electrochemical methods

The measurement of velocity and cake deposition can also be done using electrochemical methods. In these methods, micro-sensors are used to detect the changes in the diffusion coefficient of an active species (Miyagi et al., 2000). The local mass transfer is measured using a voltage step method (L'Hostis et al., 1996). Without the presence of the cake, the velocity map at the surface of the micro-sensor can be mapped from the electrical response.

In the presence of a cake layer, calibration by measurements through bulk suspensions is required. For membranes, a distribution of a large number of microelectrodes on the surface of a flat sheet membrane can provide information on the velocity distribution at the surface of the membrane (Gaucher et al., 2002a). It was found that suction pressure enhanced the shear stress, and also stabilized the velocity gradient with reduced local fluctuations.

With electrochemical techniques, microelectrode tips, which extend to the membrane surface, will alter local flux, and flow distribution. Thus, these methods are termed quasi-non-invasive. Boundary layers next to the surface of the membrane are typically very thin (in the order of less than 1mm) and the intrusion of a probe into this layer would potentially disturb the boundary layer. Hence, this method is not very suitable for studying regions close to the membrane.

Using embedded electrodes with bubbling in an electrolytic mixture of potassium ferricyanide, potassium ferrocyanide and potassium chloride, Ducom et al. (2002) measured the wall shear stress on the surface of a flat sheet membrane. His results were previously discussed in Section 2.4.2.

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(iii) Constant Temperature Anemometry (CTA)

Besides using electrochemical means, heat transfer can also be used to monitor the flow. In constant temperature anemometry, the loss of heat from a thin wire is monitored using a voltage response mechanism. Velocity measurements have been made using hot-wire probes in a submerged membrane tank (Cao et al., in preparation). However, CTA suffers from the same problem as electrochemical techniques, that is, dependence on the presence of a probe in the region of interest. The presence of the probe would tend to disturb the flow.

2.5.2 Observing the Fouling Characteristics

Besides particles that attach onto the membrane surface, particulates that are smaller than the pores of the membrane can penetrate into the membrane structure, or block the pores. This is especially prevalent in membrane bioreactors, where there are large amounts of fine colloids in the feed. The build up of particulates is detrimental to the performance of the membrane, and forms a large part of the non-reversible fouling experienced by the membrane.

Impedance Spectroscopy

Impedance spectroscopy involves the application of varying frequencies and amplitudes of current to a membrane system. The amplitude and phase difference that is developed across the membrane is then measured, and the dispersions of the conductance and capacitance can be used to distinguish between single and multilayer systems, the porosity of the membrane as well as the changes in the electrochemical process (Coster et al., 1996). By modeling the membrane as an interconnected system of capacitances and resistances, changes in the measured capacitance or resistance can indicate where fouling or structural changes are taking place (Watkins & Pfromm, 1999).

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2.6 Particle Image Velocimetry

The general principle of operation of PIV is to illuminate tracer particles in the flow field of interest with a plane sheet of light, and to acquire two images of the flow field with a known time separation. By cross-correlating the positions of the particles obtained from the image, the displacement of the particles is determined. With a known time separation, it is possible to obtain the velocity field. In PIV, the displacement field is determined within interrogation areas within the image plane.

For illumination, a laser is usually used, as laser beams can be formed into sheets with a cylindrical lens. For PIV applications, Neodym-YAG lasers are commonly used. This is because YAG crystals (yttrium-aluminum-garnet) have high amplification and good mechanical and thermal properties.

PIV techniques measure particle velocity as opposed to fluid velocity. Hence, it is important that fluid mechanical properties of the particles be checked in order to avoid significant discrepancies between fluid and particle motion. In order to ensure good tracking of the fluid motion due to density differences, one has to use small diameter particles. On the other hand, if the particles are too small, they will not scatter enough light and will produce weak images.

In order to get a useful image when 2 or more phases are present in the solution, fluorescent particles such as Rhodamine B (fluorescent) particles have to be used as tracers. Florescent particles are typically used in near wall measurements due to the fact that they absorb the green light (532nm) and emit red light at a wavelength of 590nm. This is necessary as there is a large amount of light reflecting off the wall surface. If normal glass beads are used to track the flow, the light diffracted by the glass beads would enter the camera together with the reflected light, causing the particles close to the surface to become indistinguishable. When the Rodamine B particles are used, a red filter is placed in front of the camera. In this way, only the fluorescent light that is from the particles can enter the camera, while reflected light off the surface is filtered out.

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A novel method to determine wall shear stress was proposed by Kertzcher et al. (2001). They proposed using the light sheet to illuminate the particles near the wall only. This was achieved by using a fluid that allowed light to penetrate only partially into the fluid. In their experiments, water was mixed with black ink such that light was able to penetrate about 1mm into the fluid. They successfully measured the expansion flow when the flow from a 2mm pipe entered a 4mm pipe.

Wall shear stresses were successfully measured in biological applications to represent blood flows (Atkinson et al., 2001; Heise et al. 2003). It was possible to obtain a velocity vector about 90 microns from the wall surface. Stress impacts resulting from a fluid impacting a wall can also be determined (Kiesow, 1998). In these cases, it is necessary to linearly interpolate the shear stresses by assuming that at the wall the velocities are zero (Huang et.al., 1997). This recommendation was used in this thesis.

2.6.1 PIV and Two Phase Flows

When the bubbles are present, spurious particles are present in the recorded image. This is due to the three different paths light emitted from the particles could take before reaching the camera. The first is the direct path that formed the regular images. The second is when light was reflected off the surface of the bubble. The third is when the light was refracted through the Taylor bubble. The images due to the third path is easily distinguished from the others, however, the spurious images due to the path taken by the second path is not so easily distinguished. (Dias et.al., 2000) In order to apply PIV techniques to two-phase flows, it is often imperative to determine the interface between the liquid and gas phase. Due to the above effects, PIV techniques are not suitable for the determination of interface positions.

Taylor bubbles are large bubbles that take up almost the entire cross section of the flow channel. The flow patterns inside liquid slugs that flow between these large bubbles can be measured using PIV techniques when no bubbles are present (Thulasidas et.al., 1997).

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To study the flow around the bubble as it rises up a channel, the velocity of the bubble has to be determined. This can be done by using laser diodes coupled with photocells to measure the speed of the bubble. To determine the interface positions, a combination of PIV and shadowgraphy techniques are carried out with the same camera

Light from a frequency doubled, doubled pulsed Nd:YAG laser illuminates the flow. Fluorescent tracer particles reflect part of the light, and emit light. An optical filter blocks the reflected light, while the fluorescent light passes the filter. A double-pulsed high powered LED array illuminates the multiphase flow for the recording of gas bubbles. The flow is back illuminated, and the gas bubbles produce a shadow. The shadow image passes through the optical filter and is recorded.

The information from both the tracers and the bubbles are recorded simultaneously and recorded on a PIV/shadowgraphy image. In standard PIV imaging, the background of the recording is as dark as possible. In this technique, it becomes necessary to use a gray background so as to enable two different kinds of information to be obtained. Three ranges of grey are observed. The first range corresponds to the images of the tracers (high range), the second corresponding to the light from the LEDs crossing the test section (intermediate grey) and the lowest grey level corresponds to the shadow of the bubble.

To determine the bubble interface, one can use the derivative of the grey levels by applying a filter (Lindken et.al., 2002). Alternatively, one can subtract the image obtained with a reference image with the background illumination. The complete shadow of the bubble is obtained after binarization of the image. (Nogueira et.al., 2003)

This method can be applied to bubble swarms (Lindken et.al. , 2002) as well as Taylor bubbles (Nogueira et.al., 2003)

Lain et.al. (2001) developed a method of studying the hydrodynamics in a bubble column by an imaging PIV/PTV system. The measurement of the liquid velocity was done by

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adding fluorescent particles. Images of the bubbles and tracer particles were acquired by two CCD cameras simultaneously. Images of the bubbles were recorded by a CCD camera fitted with an optical interference filter corresponding to the wavelength of the applied laser. Images of the bubbles recorded were shown as points due to the reflection on the surface of the bubbles.

2.6.2 Application of PIV to Membranes

PIV techniques have been used to determine the hydrodynamics in membrane systems for flat plate systems and rotating filters. Gaucher et al., (2002b) measured the shear rate distribution above a membrane plate. Titanium dioxide was used as the tracer particle. It was found that the distribution was heterogeneous, and this was responsible for inhomogeneous fouling. In their experiments, a Plexiglass surface was used in place of the membrane. Hence, filtration effects were not accounted for.

Wereley et al. (2002) used PIV techniques to determine the flow conditions in a rotating filter separator. Two types of particles were used; smaller particles with average diameters of 10 to 25 microns were used to follow the fluid flow, while larger particles of 150 to 300 microns were used to follow the particulate flow. By using a red filter, it was possible to differentiate between the light emitted from these 2 particles. The area of interest was about 10mm by 10mm. A recent application of PIV to membranes is the measurement of flow between two flat membrane surfaces to study hydrodynamic effects on concentration polarization (Gimmelshtein & Semiat, 2005). There has to date been no reported application of PIV to submerged hollow fibers, and this is a topic of this thesis (Chapters 4 and 5).

2.7 Synchrotron Light Source

A synchrotron is a particular type of cyclic atomic particle accelerator in which the magnetic field (to turn the particles so they circulate) and the electric field (to accelerate the particles) are carefully synchronized with the traveling particle beam. By increasing

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these parameters appropriately as the particles gain energy, their path can be held constant as they are accelerated.

Phase Contrast XMI is a novel technique that has been made possible by the high coherence and low divergence of the light source in third generation synchrotron light facilities, such as the Singapore Synchrotron Light Source. XMI was pioneered by Bonse and Hart (1965) and has been used in such fields as biomedicine (Takeda, 2005) and the study of crack propagation (Toda, et al., 2004).

The advantage of XMI is that it can provide information about the internal structure and morphology of samples. With suitable conditions the information can be obtained rapidly ($<1s$) over a small area ($\sim 1mm$) so that non-invasive, real time observation of internal phenomena become possible. This opens up the possibility to obtain insights into membrane process phenomena. This is also a topic of this thesis.

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2.8 Summary

In this chapter, literature relevant to this thesis is reviewed. While submerged hollow fiber membranes have great potential for water and waste water treatment, the problem of fouling has to be addressed. Physical characteristics, such as fiber length and diameter affect the performance of the module. Just as important are hydrodynamic properties, such as the airflow rate, and the distribution of flow around the hollow fiber bundle. The interactions of the physical and hydrodynamic properties are not well understood and mathematical modeling of a hollow fiber system, with bubbling from the outside, is very complicated.

To study this interaction, several experimental techniques, such as DOTM, and CTA are discussed. In this thesis, PIV and XMI will be used due to their intrinsic benefits.

CHAPTER THREE

CHAPTER THREE METHODOLOGY**3.1 Introduction**

This chapter describes the filtration equipment and protocols as well as the two major characterization techniques employed, PIV and XMI.

3.2 Filtration Experiments

A system was set up to perform both filtration and flow characterization experiments relevant to single submerged hollow fibers.

3.2.1 Single Hollow Fiber Experiments**(i) Measurements on a Tight Fiber**

Two sets of experiments were conducted. The first experimental set done for PIV (details in next section) measurements was conducted using a 1mm diameter glass optical fiber in place of the hollow fiber membrane. It should be noted that the radial velocity of the liquid towards the fiber under filtration was typically 100 to 1,000 times less than that of the axial flow. Thus, the effects of using glass rods on the shear stress measurement were small. The second set of experiments involved the use of an actual hollow fiber in filtration experiments to determine the performance of the fiber under different bubbling conditions.

The flow channel in both experimental sets was 20mm by 20mm and 1m in length. The lengths of the glass rods and fibers used in the experiment were 800mm. A diagram of the experimental setup is shown in Figure 3.1. The bubble size and frequency was controlled by a combination of an air pump, a solenoid valve, pressure relief valve and metering valve, connected to a needle. This needle was situated adjacent (<1mm) from the fiber or glass rod.

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Figure 3.1 Experimental setup for single fiber experiments

Three different types of bubble were studied. The first type was the small (wobbly) bubble. This bubble was about 6mm in diameter, and was of an elongated shape. The next type was the medium (cap) bubbles. These bubbles took up the entire cross section of the channel, and were about 5mm in thickness. The last type of bubble was the large (slug) bubbles. These bubbles also took up the entire cross section of the flow channel but were thicker at 20mm. The volume of each bubble was approximately 0.11cm^3 , 10cm^3 and 40cm^3 respectively. The features of these bubbles mimic those used in real systems.

In the second set of experiments, a feed solution with 2g/l of bentonite was used; the average particle diameter was 5 microns. The fibers were made of polyacrylonitrile (PAN), with an inner diameter of 0.8 mm and an outer diameter of 1.0 mm. The nominal

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pore size of the membrane was 0.5 microns and the clean water permeability of the fibers was about $1.7 \text{ l/m}^2/\text{hr/kPa}$, and varied about $\pm 8\%$. To compare the performance of the membranes, they were run at flux of $25 \text{ l/m}^2/\text{hr}$, and the rate of increase of transmembrane pressure (TMP) was determined over the initial 30 minute period. The permeate was removed by a Masterflex peristaltic pump, and the permeate side pressure was measured with a pressure transducer, and data logged into a computer.

(ii) Measurements on a Moving Fiber

When loose fibers were used, it was necessary to use a larger sized channel to prevent the fiber from impacting the side of the walls. The channel was 50x50mm as opposed to 20x20mm for the tight fiber. Besides the differences in the size of the channel, the setup was broadly similar to the setup when tight fibers were tested.

The details of the experiment for both the tight and loose fibers are shown in Table 3.1 below. The looseness of a fiber is defined as $[1 - (\text{distance between fixed ends}) / (\text{length of fiber})] \times 100$.

Table 3.1 Experiment series for single fibers

No.	Expt.Name	Cross Flow Velocity (m/s)	Looseness (%)	Bubble Type	Bubble Frequency (Hz)
Filtration and PIV Experiments					
1-5	SPX Series	0, 0.016, 0.08, 0.16, 0.32	0	None	None
6-10	SPS Series	0	0	Small	0.1, 0.5, 1.0, 2.0, 5.0
10-14	SPC Series	0	0	Cap	0.05, 0.1, 0.5, 1.0, 2.0
15-19	SPL Series	0	0	Slug	0.05, 0.1, 0.5, 1.0, 2.0
20-24	SPLS Series	0.016	0	Small	0.1, 0.5, 1.0, 2.0, 5.0
25-29	SPLC Series	0.016	0	Cap	0.05, 0.1, 0.5, 1.0, 2.0
30-34	SPLL Series	0.016	0	Slug	0.05, 0.1, 0.5, 1.0, 2.0
35-39	SPFS Series	0.16	0	Small	0.1, 0.5, 1.0, 2.0, 5.0
40-44	SPFC Series	0.16	0	Cap	0.05, 0.1, 0.5, 1.0, 2.0
45-49	SPFL Series	0.16	0	Slug	0.05, 0.1, 0.5, 1.0, 2.0

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Filtration Experiments Only					
50-55	SL0%S Series	0	0	Small	0.1, 0.5, 1.0, 2.0, 3.0, 5.0
56-60	SL0%M Series	0	0	Medium	0.1, 0.5, 1.0, 2.0, 3.0
61-63	SL0%L Series	0	0	Large	0.1, 0.5, 1.0, 2.0
50-55	SL0.5%S Series	0	0.5	Small	0.1, 0.5, 1.0, 2.0, 3.0, 5.0
56-60	SL0.5%M Series	0	0.5	Medium	0.1, 0.5, 1.0, 2.0, 3.0
61-63	SL0.5%L Series	0	0.5	Large	0.1, 0.5, 1.0, 2.0
50-55	SL1.0%S Series	0	1.0	Small	0.1, 0.5, 1.0, 2.0, 3.0, 5.0
56-60	SL1.0%M Series	0	1.0	Medium	0.1, 0.5, 1.0, 2.0, 3.0
61-64	SL1.0%L	0	1.0	Large	0.1, 0.5, 1.0, 2.0
65-67	AL0%S	0	0	Small (Alt)	0.5, 2.0, 5.0
68-70	AL0.5%S	0	0.5	Small (Alt)	0.5, 2.0, 5.0
71-73	AL1.0%S	0	1.0	Small (Alt)	0.5, 2.0, 5.0
74-76	AL2.0%S	0	2.0	Small (Alt)	0.5, 2.0, 5.0
PIV Experiments Only					
77-79	SL0%S Series	0	0	Small	0.5, 2.0, 5.0
79-80	SL0%M Series	0	0	Medium	0.5, 2.0
81-82	SL0%L Series	0	0	Large	0.5, 2.0
83-85	SL0.5%S Series	0	0.5	Small	0.5, 2.0, 5.0
85-86	SL0.5%M Series	0	0.5	Medium	0.5, 2.0
87-88	SL0.5%L Series	0	0.5	Large	0.5, 2.0

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89-91	SL1.0%S Series	0	1.0	Small	0.5, 2.0, 5.0
91-92	SL1.0%M Series	0	1.0	Medium	0.5, 2.0
93-94	SL1.0%L	0	1.0	Large	0.5, 2.0

3.2.2 Hollow Fiber Bundle Experiments

A series of experiments were conducted to determine the performance of the hollow fiber in a model 3 x 3 bundle (9 fibers). Both constant flux and constant pressure operation were tested.

(i) Constant Pressure Operation

As a preliminary test, the fibers were operated in a constant pressure mode, without aeration. The hollow fiber module had a flow channel that was 1m long, with a 20mm by 20mm cross section. The membrane and feed solution were identical to the single fiber experiments.

To obtain varying packing densities, metal plates perforated with holes at different spacing were made. The fibers were inserted into 2 plates located at both ends of the module and pulled taut. The fibers were sealed at one end, and individually connected to a length of tubing at the other. These tubings were connected to ports in a vacuum chamber, where plastic cylinders collected the flux from each fiber separately. The vacuum chamber was connected to a pump (from CAST Manufacturing Inc.) that could maintain a negative pressure of up to 60kPa. With the vacuum chamber arrangement, each fiber in the bundle experienced the same driving pressure. The fibers were numbered from 1 to 9 as shown in Figure 3.2. For the constant pressure mode, the aeration was applied solely from the needle at the centre of the bundle (in front of Fiber 8).

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Figure 3.2 Fiber numbers for bundle experiments

The experimental setup is shown in Figure 3.3. To determine how the flux changed with time, a 2mm hole extended from each port and the number of drops per minute discharged was counted. By dividing the total number of drops by the total volume in the cylinder, the volume per drop could be calculated. It was found that the average size of each drop was about 0.03 to 0.04 liters. A video camera was used to record the number of drops that fell from the port per minute. With this information, the rate of change of the flux could be determined. In all the experiments, the pressure was stepped from -10kPa to -50kPa in 10kPa decrements. The pressure was maintained for 1 hour, after which the process was stopped, and the cylinders taken out and weighed. After weighing, the cylinders were replaced and the pressure was decreased by 10kPa, and run for another hour, and so on. The vacuum chamber pressure was measured with a digital pressure gauge.

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Figure 3.3 Experimental setup for fiber bundle experiments

A total of six series of experiments were carried out (Table 3.2). A packing density of 44% corresponds to a 0.5mm distance between fiber surfaces, while a 28% packing density corresponds to a 1mm distance between fiber surfaces. In each series, the recirculation velocity was varied using the Masterflex peristaltic pump.

Table 3.2 Constant Pressure Experiments

Series Name	Packing Density	No. of Fibers	Bentonite Concentration	No. of Tests	Considers the effects of
HFBX	44%	9	1.0g/l	6	Cross Flow
HFBY	28%	9	1.0g/l	6	Packing Density
HFBH	44%	9	2.0g/l	4	Feed Concentration
HFBL	44%	9	0.5g/l	4	Feed Concentration
HFBubble	44%	9	2.0g/l	1	Bubbling
HFSingle	Single	1	1.0g/l	4	

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(ii) Constant Flux Operation

The setup for constant flux operation is similar to the one when constant pressure is used. The flow channel was 50mm square and 450mm long.

A bundle of nine fibers arranged into a 3x3 square array was used in each experiment. In order to be able to differentiate between the fibers, the fibers were again numbered from 1 to 9 as shown in Figure 3.3. They were mounted onto two plates held 400mm apart. The plates held the fibers in the middle of the flow channel. The fibers were glued individually to pipes of equal length, and these were attached to the top of the vacuum chamber.

It was a challenge to come up with a method of determining the individual fluxes of a hollow fiber membrane bundle, whilst maintaining a constant average flux condition. The following innovative idea was used. First, feed solution was pumped into the module at a constant rate, causing the level of water in the module to rise. The vacuum pump was then switched on, and this drew permeate from the fibers into the vacuum chamber. If the level of the water in the module remained the same, then the permeate flux would be equal to the influx of the feed solution. However, if the level of water changed, the trans-membrane pressure (i.e. vacuum box pressure) was adjusted to increase the permeate flux until the level became constant. In order to ensure quick response to any changes to the level of the water, two precautions were taken. The first was to attach a sight glass to the module. The sight glass was a glass cylinder, 5mm in diameter. The second was to reduce the size of the flow channel near the top of the module with clay blocks. A reduction in the size of the flow channel caused the water level to be more sensitive to changes in the volume of liquid in the channel.

The permeate from the fibers traveled through the tubes into the vacuum chamber. The tubes were attached to fittings with needles, that allowed the permeate from each of the fibers to fall into graduated measuring cylinders. Readings from the cylinders were taken at 5 minute intervals for 180 minutes.

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Three types of bubble of different sizes were used in the experiments. The small-sized bubbles were roughly spherical, and had a volume of about 0.09 cm^3 , the medium and large sized bubbles were spherical cap bubbles, and had volumes of 1.13 cm^3 and 8.36 cm^3 respectively. The bubble size and frequency were controlled by a valve system, with a solenoid valve, a release valve, a metering valve and a needle in series. To examine any relationship between the location of bubble injection and the performance of individual fibers and the overall bundle, two bubbling positions, centre-line, and off-centre were examined (Figure 3.3). To ensure reproducible results, all filtration experiments were repeated at least twice. The experimental conditions are listed in Table 3.3.

Table 3.3 Constant Flux Experiments

No.	Series Name	Bubble		Crossflow (m/s)	Length (m)	Aeration Inlet
		Type	Frequency (Hz)			
1	CX	-	-	0	0.4	-
2	C1	-	-	0.0067	0.4	-
3	C4	-	-	0.0267	0.4	-
4	BS0.5	Small	0.5	-	0.4	Centre
5	BS2	Small	2	-	0.4	Centre
6	BL0.5	Large	0.5	-	0.4	Centre
7	BL1	Large	1	-	0.4	Centre
8	BS1 Loose	Small	1	-	0.404	Centre
9	BL1 Loose	Large	1	-	0.404	Centre
10	BS0.5 OC	Small	0.5	-	0.4	Off-Centre
11	BS2 OC	Small	2	-	0.4	Off-Centre
12	BL0.5 OC	Large	0.5	-	0.4	Off-Centre
13	BL1 OC	Large	1	-	0.4	Off-Centre

For the constant flux experiments, PIV measurements were also made in order to correlate the hydrodynamic situation to the performance of the hollow fiber membrane. To represent the hollow fibers, optical fibers were used (details of the PIV setup are given in section 3.3) If real hollow fibers had been used, the light sheet from the laser would

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have been blocked. The cladding of the hollow fibers was removed in these experiments. The refractive index of the optical fibers was 1.4. The difference between the refractive index of the water and the refractive index of the optical fiber would have caused the light sheet to diffract when it strikes the fiber. This would have caused particles that were not in the plane of the light sheet to become illuminated, resulting in accuracy in the calculated velocities. Hence it was necessary to match the refractive indices of the fluid and the hollow fiber.

In order to have unobstructed particle image velocimetry measurements in his experiments for complex turbomachinery with multiple blades, Uzol et al. (2002) used a 64% by weight solution of sodium iodide (NaI) in deionized water as the working fluid in their tests which matched with that of polymethyl methacrylate (PMMA), the acrylic material which their apparatus was made of. In this series of experiments, a similar solution was used. The refractive index of the NaI solution ranged from 1.49 to 1.50 at a temperature of 24 – 25°C with Nd:YAG laser wavelength of 532 nm passing through it. Full optical access was possible to both the laser sheet and the camera. The solution had a viscosity of about $1.1 \times 10^{-6} \text{ m}^2/\text{s}$, which was similar to that of water and this enabled operation at enough high Reynolds numbers. However, care had to be taken when handling the solution as I_3^- ions form in the NaI solution due to exposure to oxygen and light. Also, they absorb light in part of the visible range including green light, which complicates the PIV images.

3.3 Particle Image Velocimetry Setup

The PIV system was a Dantec FlowMap system. The illumination was from a dual-cavity frequency doubled Q-switched pulsed mini Nd:Yag laser. The energy level was 25mJ and the pulse duration was about 7ns. The emitted light sheet was green, at a wavelength of 532nm. The thickness of the light sheet was 1mm with a divergence angle of 32°. A Hi-Sense MkII camera was used to capture the images. The camera used a high-performance progressive scan interlines CCD chip. This chip includes 1280 by 1024 light-sensitive cells and an equal number of storage cells.

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Rhodamine B (fluorescent) particles, with a diameter of 10 microns, were used as the particle tracers. Florescent particles are typically used in near wall measurements due to the fact that they absorb the green light (532nm) and emit red light at a wavelength of 590nm. This is necessary as there is a large amount of light reflecting off the wall surface. If normal glass beads were used to track the flow, the light diffracted by the glass beads would enter the camera together with the reflected light, causing the particles close to the fiber surface to become indistinguishable. When the Rodamine B particles are used, a red filter is placed in front of the camera. In this way, only the fluorescent light that is from the particles can enter the camera, while reflected light off the surface of the fiber is filtered off.

The data acquisition was performed at 5Hz. Thus, a pair of images were captured every 200ms during the measurement period. The final interrogation cell size was 32x32 pixels with 50% overlap in Interrogation Area (IA) the fiber was obtained. The top and bottom 10 rows of velocity were ignored as the velocity vectors in those regions could have been affected by particles moving into or out of the image area.

3.3.1 Velocity Measurements near Curvilinear Surfaces

The columns of velocity vectors determined by PIV were averaged, and a single velocity profile extending from the edge of the fiber was obtained. From this profile, the velocity gradient at the surface of the fiber was determined based on 3 velocity readings within a 1mm boundary layer. The shear stress could then be determined from the resultant velocity gradient.

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Figure 3.4 Non zero velocities at the surface of the fiber

However, when measuring the velocity near to a highly curved surface, such as a hollow fiber, one of the issues is that the thickness of the laser light sheet can be comparable to the radius of curvature and hence the flow can be varying along the depth of the light sheet. This is a limitation inherent to the PIV system. The laser light sheet is typically 1mm thick. With the averaging effect, what is measured is not the exact velocity along an infinitely thin section of the flow, but rather an average within a thickness of 1mm. This is shown pictorially in Figure 3.4.

It is difficult to get an optical system to produce a very thin light sheet. Moreover, if the light sheet is too thin, it increases the possibility of the particles dropping out of the light sheet, which causes a lot of inaccuracy in the system.

Simulations done by Sridharan (2006) indicate that there are several conditions when the laser light sheet is not aligned with the fiber. This was not a problem when the fiber was held tightly, as it was possible to carefully align the fiber with the laser light sheet. However, when the fibers are moving in and out of the plane of fiber movement, the margin for error would be much greater. It was found that for a laser light sheet thickness of 1mm, in line with the fiber, the difference in the velocities nearest the fiber could be overestimated by approximately 30%. The differences in velocity would reduce further from the fiber as the velocity gradients would be reduced. As the shear stress was calculated using linear regression of 4 points nearest the fiber, this worked out to an

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overestimate of about 6% in the shear stress. The calculations in this paper are adjusted according to these guidelines.

3.3.2 PIV Measurements of Moving Fibers

Using PIV methods on a single fiber that was not in motion was relatively straight forward as the laser could be aligned directly with the fiber. However, applying PIV to loose fibers in bubbly flow was more complicated. The main problem was that the bubbles caused the fibers to move in and out of the plane of the light sheet. Thus, in some of the captured images, the fibers were sometimes behind the light sheet, and in others, the fiber was in front of the light sheet. In the case of moving (loose) fibers, it was necessary to sort the data and analyze only the data that was within the light sheet.

To help with the sorting of the data, fluorescent particles were glued onto the hollow fiber. If the fiber was within the plane of the light sheet, then the particles would fluoresce. The data were sorted manually, and images were selected according to the following criteria:

1. The particles glued onto the surface of the fiber must be fluorescing brightly.
2. The right hand side of the image must be darker than the left one (as the light is coming in from the left)
3. There are no particles in front of the fiber

As turbulence in bubble columns is a chaotic and irregular process, it became necessary to capture a large number of images to ensure that the average of the flow was monitored. For each bubbling condition, approximately 1500 images were collected. After the data were collected, the images were analyzed with the computer, and the velocity vectors resolved.

3.3.3 Mean and Standard Deviation

The mean and standard deviations were determined and used to study the variation of the shear stress. The mean is simply the average of all 1000 readings of the shear stress taken.

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The standard deviation is a measure of the spread of the data, and is known as the second central moment of the distribution. In this study, the standard deviation was normalized to the mean, and should be regarded as such throughout this study.

$$\text{Mean} = \frac{\sum x}{n}$$

$$\text{Standard Deviation (S.D.)} = \sqrt{\frac{\sum \left(\frac{x_i}{x} - 1 \right)^2}{n}}$$

3.4 Synchrotron X-Ray Micro Imaging (XMI)

The setup used for XMI is shown in Figure 3.5. Synchrotron Radiation from the electron beam of the Helios 2 storage ring at the Singapore Synchrotron Light Source is extracted from pole 16 on dipole D2 and travels about 15.21m along the beam pipe of the phase contrast imaging beam line. The beam exits through a 500 μm thick polished beryllium window into air and reaches the sample after about 1m distance. The Be window and the drift space in air are the only optical interactions the photon beam undergoes before striking the sample. A total distance from source to sample of 16.76m yields a spatial resolution of about 1 μm . Upon traversing the sample, both the amplitude and wave vector of the wave field are modulated by scattering and absorption. Downstream of the sample, the X-ray photons strike a CdWO_4 scintillation foil perpendicularly where they are converted to visible light which is reflected by 90° using a Si wafer into a magnifying objective (5x) mounted on a Roper Scientific CoolSNAP CCD camera (6x zoom). The distance between the sample and scintillation foil is typically in the range of 2-3cm.

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Figure 3.5 Setup of the Phase Contrast Imaging and Tomography (PCIT) at the Singapore Synchrotron Light Source

One of the constraints imposed in using the PCI facility at the SSLS is the photon beam attenuation in water. As the photon energies do not exceed 10 keV the attenuation length in water is less than 2mm. This required special considerations in setting up the filtration systems.

3.4.1 Operation from Lumen to Shell

To observe the fouling within a hollow fiber when a feed is pumped through the lumen, XMI was used to observe this phenomenon. A single fiber of PAN was used, having an inner diameter of 0.6mm, an outer diameter of 0.8mm and a nominal pore size of about 0.5 μm . In these observations, a single hollow fiber was held vertically (about 2cm) in front of the scintillation foil. The fiber was sealed at the bottom end and feed was pumped in dead-end mode into the lumen by a peristaltic pump. The pumping rate was adjusted so that the pressure within the fiber was kept at about 40kPa. The feed solution

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was 1% by weight of iron hydroxide. Bentonite was not used as it was not dense enough to provide a clear X-ray image. The size of the particles ranged from 0.1 to 10 μm . Images were captured automatically at 3 minute intervals. Scan times of about 1-10s were used to achieve an optimal signal to noise ratio in the image (note: the longer scan times occurred as the beam-line energy decayed). Due to filtration from the lumen-side water droplets tended to form on the outer membrane surface. To minimize signal attenuation the water was removed to maintain a 'dry' rather than a flooded permeate side.

3.4.2 Operation for the detection of bubbles in lumen

XMI was also used to examine the formation of bubbles in the lumen of a hollow fiber under suction.

Figure 3.6 Experimental Setup for XMI (Bubble Detection)

A single hollow fiber membrane was installed in a filtration cell which sat on a motorized stand. The stand could be remotely controlled by a computer. Figure 3.6 is the schematics of the in-situ filtration system which consists mainly of a feed tank, a filtration cell, a pressure transducer, and a suction pump. The filtration cell made of Perspex had 8 Mylar film covered observation slots (70mm x 6mm) arranged in two symmetric lines over the

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cell width as shown in Figure 3.6. A feed tank filled with MilliQ water was directly connected to the filtration cell. As the energies of the photon beam used for PCI at SSLS do not exceed 10 keV the attenuation length in water is less than 2 mm. To minimize the attenuation of the photon beam the thickness of the flow channel of the filtration cell was set at 1 mm. The length of the hollow fiber was 672mm and was arranged in a U-shape. The membrane was then installed in the cell and aligned with the observation slots. The membranes tested were 0.22 μm polypropylene hollow fibers (USFilter) with an inner/outer diameter of 0.39/0.65 (mm). The hydrophobic hollow fiber was wetted by pumping 70% ethanol solution into the fiber lumen for two hours before the start of the experiment.

The filtration experiments were carried out in constant permeate flow mode. A five head Gilson pump (Minipuls 3, Gilson) was used to extract permeate from one end of the U-shaped fiber. The suction pressure was monitored by the pressure transducer and the permeate flow was measured by the Toledo balance (PB-S, Mettler Toledo). The suction pressure and the weight of water collected were data logged into a computer.

3.5 Scanning Electron Microscope (SEM)

SEM images were taken for comparison to XMI Images. A scanning electron microscope (SEM, Hitachi Model S- 520, Japan) was employed to observe cross-section of the membranes. The membranes were cryogenically fractured in liquid nitrogen and then sputtered with gold.

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CHAPTER FOUR PERFORMANCE OF HOLLOW FIBER BUNDLES

4.1 Introduction

The performance of a single fiber under various hydrodynamic conditions provides valuable insight into the effects of hydrodynamics on the performance of submerged hollow fibers. Invariably, however, hollow fibers have to be packed into tight bundle to take advantage of their greatest asset – their high membrane surface area to footprint ratio. Hence, it is important to try understand the effects of hydrodynamics on the performance of a fiber bundle.

The productivity of hollow fiber membranes is related to both flux as well as membrane packing density. A higher membrane packing density would increase productivity, as long as flux is not detrimentally affected. The assumption is that each fiber within a bundle could develop different degrees of fouling, as each fiber in the bundle experiences different hydrodynamic conditions. As submerged hollow fibers become a more popular design concept, it becomes necessary to ensure that the productivity of the bundle is at its optimum level.

In this section, the performance of hollow fibers in a model 3x3 array (9 fibers) is studied under both constant pressure and constant flux conditions. PIV experiments were used to determine the hydrodynamic conditions within the membrane. Initial experiments were done under constant pressure conditions. Under these conditions, the effect of packing density, feed concentration, and cross flow were studied. Later experiments were conducted under constant flux conditions, and this focused on the effect of bubbling and fiber looseness. PIV techniques were used in the latter part of the study to characterize the hydrodynamics within the bundle and movement of the fiber.

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4.2 Experimental Protocols

The experimental protocols in this chapter can be found in Chapter 3.

4.3 Hydrodynamic Resistance

Before the results of the experiments conducted under constant pressure and constant flux are presented, the concept of hydrodynamic resistance is explored. The performance of a fiber that is held by itself within a large tank would not be the same as the performance of a fiber that is packed into a bundle of hollow fibers, even if no fouling is occurring. This is especially so if the fiber is at the center of a bundle of hollow fibers. One of the factors is the radial pressure drop experienced as the flow moves into the fiber bundle. The pressure drops in a hollow fiber module has been modeled (Brinket et al., 1994). It was found that pressure drops of up to 20% can occur in large hollow fiber bundles. However, this was for a hollow fiber module that was pressurized the shell side to 100kPa, with 19,290 fibers. Nevertheless, similar “bundle resistance” effects would occur in submerged modules. In addition, the “permeate competition” effect (described above) could play a role.

To model this phenomenon a new term is added to the “resistance-in-series” model. The “resistance-in-series” model for clean water is well known and is given by

$$J_w = \frac{P_T}{\mu R_M}$$

Where J_w is the clean water flux, P_T is the transmembrane pressure, μ is the viscosity of the fluid and R_M is the intrinsic membrane resistance.

A new resistance term, the hydrodynamic resistance (R_{HYD}), is added to R_M . The hydrodynamic resistance in this case would consist of both the “bundle resistance” as well as “permeate competition as defined previously.

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The hydrodynamic resistances for each position in our particular experimental setup were determined. Filtration was conducted with MilliQ water, and the resistance for each fiber was calculated. The membrane resistance of the fiber, held by itself in a large tank, was subtracted from the resistance of the fiber obtained when it was packed into a bundle. This represents the hydrodynamic resistance and was repeated at various permeate velocities. At each condition, the measurements were done 4 times and the results averaged.

Figure 4.1 Hydrodynamic resistances for various positions at different fluxes

Figure 4.1 shows the apparent hydrodynamic resistances experienced at the different fiber locations at different fluxes. To determine the relative magnitudes of the “bundle resistance” and “permeate competition, the centre fiber was left filtering while the other fibers did not experience any suction pressure. In this case, there would be no

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“permeate resistance”, as any increase in resistance would solely reflect the “bundle resistance”

It can be seen that for the range tested, the hydrodynamic resistance for each position remains relatively constant. The hydrodynamic resistance for the centre fiber was also much higher than the other positions. By comparing resistance of the center fiber when the fiber is in operation by itself, and when the all the fibers are in operation, it is observed that when all the fibers are filtering, the resistance increases. This is evidence of the contribution of the two components to the overall hydrodynamic resistance.

Based on these resistance values, the flux from each fiber can be calculated at different TMPs and this allows estimation of the different average bundle fluxes. It was found that, for the experiments conducted, the intrinsic standard deviations of fluxes would be approximately 0.24.

The standard deviation caused by the inclusion of hydrodynamic resistance, and the standard deviation at which pressure rise starts to occur are similar. This is an indication that once the distribution of flux becomes slightly different from the intrinsic distribution, pressure rise will occur.

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4.4 Operation in Constant Pressure Mode

Under the constant pressure condition, the following experiments were carried out to determine the effects of cross flow, packing density, feed concentration and bubbling. As a control, the performance of a single fiber was also used as a comparison. The experimental conditions were described in Section 3.2(i). A packing density of 44% corresponds to a 0.5mm distance between fiber surfaces, while a 28% packing density corresponds to a 1mm distance between fiber surfaces. To study the effect of feed concentration, experimental series HFBH and HFBL were conducted using different feed concentrations. To observe the effects of bubbling, a single experiment was done where bubbling was passed at a rate of 1 L/min through a need at the base of the module. As a reference, the detailed experimental conditions are reproduced in Table 4.1.

Table 4.1 Constant Pressure Experiments

Series Name	Packing Density	No. of Fibers	Feed Concentration	No. of Tests	Considers the effects of
HFBX	44%	9	1.0g/l	6	Cross Flow
HFBY	28%	9	1.0g/l	6	Packing Density
HFBH	44%	9	2.0g/l	4	Feed Concentration
HFBL	44%	9	0.5g/l	4	Feed Concentration
HFBubble	44%	9	2.0g/l	1	Bubbling
HFSingle	Single	1	1.0g/l	4	

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4.4.1 Performance of Bundle

To determine the performance of the bundle as a whole, the average flux from the fibers in the bundle was compared to the performance of a single fiber (series HFSingle in Table 4.1) under the same conditions.

Figure 4.2 Comparison between flux from a single fiber and average flux from a fiber bundle at a cross flow velocity of 0.05m/s

In Figure 4.2, the differences in the fluxes under various conditions, at a cross flow velocity of 0.05m/s are compared. At lower pressures, the performance of the single fiber is similar to the performance of the average of the fiber bundle. It must be noted in that for series HFBL, the concentration of the bentonite solution is lower (0.5g/l) as compared to the bentonite solution when a single fiber is tested (1.0g/l). This is why the average flux for series HFBL is higher than the flux from a single fiber at lower pressures. At lower pressures, increasing the pressure increases the flux obtained from

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the fibers. However, at higher pressures, increasing the pressure causes the average flux to fall.

As this is a constant pressure technique, the fouling is self-limiting. Thus, as the fouling layer builds up, the flux obtained from the fiber would fall as the pressure remains constant. When the packing density is 44%, there is a marked drop in the flux obtained when the pressure exceeds 30kPa. (Series HFBX, HFBH and HFBL). This shows that operating at higher pressures does not necessarily mean that a higher flux will be obtained.

Figure 4.3 Comparison between flux from a single fiber and average flux from a fiber bundle at a cross flow velocity of 0.33m/s

Increasing the cross flow velocity reduces the differences between the performance of a single fiber and the performance of a fiber bundle (Figure 4.3). In this case, there are no longer much differences between different conditions. Thus, neither changing the concentration or the geometry of the bundles has a significant effect on the average

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flux. The fluxes obtained at a crossflow velocity of 0.33m/s are also higher as compared to at lower crossflows. At higher crossflow velocities, increasing the pressure beyond 30kPa no longer brings about a fall in the average flux obtained. This is evidence that a higher cross flow velocity retards fouling.

Figure 4.4 Comparison between flux from a single fiber and average flux from a bundle at 50kPa

At 50kPa (Figure 4.4), and 0.02m/s crossflow velocity, the flux obtained when packing the bundle at 44% (HFBX) dropped by 25% as compared to a single fiber. At a packing density of 28% (HFBY), the flux obtained dropped by about 10%. At a high feed concentration (HFBH), the flux obtained dropped by almost 75%. As cross flow velocity increases, the fibers in the bundle start to behave more like single fibers.

The difference in performance between the fibers packed into a bundle and a single isolated fiber can be related to 2 factors. The first factor is the hydrodynamic effect (this effect is discussed in Section 4.3) and the second is the interference of the cake

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layers in the bundle. At higher packing densities, there will be more stagnant areas within the bundle. These stagnant areas cause the shear stress experienced by the fibers in the bundle to be less than that of a single fiber. This causes a build up of cake, and rapid fouling to occur. This problem is exacerbated by low cross flow velocity, which hinders fluid from penetrating into the fiber bundle and the effect is accelerated as local blocking or obstruction occur. The cake layers would also build up rapidly under low crossflow velocities. As these cake layers get thicker, they would start to touch each other, then start to merge, and clogging (interstitial blockage) would occur. This is detrimental to the performance of the bundle. The effect of hydrodynamics was further studied in the following sections with PIV techniques. Increasing the concentration of the feed causes the fibers to foul much more rapidly, which would explain the poor performance of the HFBH series of experiments.

At higher crossflows, there was no significant difference between the average fluxes under different geometries. Thus, introducing turbulence and flow into a column enable higher fluxes to be drawn.

4.4.2 Standard Deviations

The fibers were positioned on a 3x3 matrix and numbered from one corner, so that fibers 1,3,7 and 9 occupied corners, 2,4,6 and 8 were sides and 5 was central. This can be seen in as Figure 3.2 in Chapter 3. Although fibers 1, 3, 7 and 9 (corner) as well as fibers 2, 4, 6 and 8(side) were in the same position in the bundle, the flux from those fibers were, in some cases, dramatically different from each other. The standard deviation of the fluxes is a measure of the differences in fouling among the fibers. The higher the standard deviation, the more uneven the fouling on the different fibers in the fiber bundle. Differential fouling has been found to precede TMP jump under constant flux conditions (Section 4.5.1.4).

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Figure 4.5 Standard deviations of the fluxes for series HFBX (Packing density 44%, Feed concentration 1g/L)

Figure 4.5 shows the standard deviations of the fluxes under the experimental conditions of series HFBX. At lower axial flow velocities (between 0.005m/s and 0.04m/s), the standard deviation increases as the pressure increases. As the pressure increased beyond 30kPa for the lower axial flow velocities, the standard deviations increased. This is indicative that that uneven fouling has occurred. The increase in standard deviation occurs at lower pressures for the lower axial velocities. Thus, increasing the axial flow rate can delay the uneven fouling fibers in a hollow fiber bundle. Increasing the axial flow velocity to 0.08m/s or more causes the standard deviation of the fluxes to remain low throughout the experiment. Hence, a critical axial flow velocity exists, at which shear stress caused by the axial flow is sufficiently well distributed to prevent differential fouling on fibers.

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Figure 4.6 Standard deviations of the fluxes for series HFBY (Packing density 28%, Feed concentration 1g/L)

In Figure 4.6, at a lower packing density of 28%, the standard deviation did not vary much at any cross flow velocity or pressure and was less than 0.2. The same trend was also seen at very high feed concentrations. Thus at a feed concentration of 2g/l, the standard deviation was also stable at about 0.2, and no pattern could be discerned. Although the standard deviations were similar, the absolute value of the flux obtained was much lower when the feed concentration was high. By spacing out the fibers, or having a higher feed concentration, it is possible to reduce differential fouling in fiber bundles.

From Section 4.3 the effect of hydrodynamic resistance, in pure Milli-Q water, gives rise to an intrinsic standard deviation of approximately 0.24. Generally, the standard deviations are lower when filtration is done with bentonite solution. One of the reasons could be that the intrinsic standard deviation was determined under constant flux

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conditions, while the operation in this case was under constant pressure. As the bundle is small, the pressure drop within the bundle under constant pressure conditions would be low. This would lead to a lower intrinsic standard deviation. Moreover, when a fouling agent is present, the fibers with higher flux would foul rapidly, while the fibers with lower fluxes would foul less rapidly. This would lead to a more even distribution of flux.

There were differences in the fluxes in different fibers of up to 40% in some cases. Such large deviations occurred reproducibly but not necessarily in the same locations. The reason for these differences is believed to be the way cake builds up on the fiber. Observations after the experiments were conducted show that some fibers were stuck together, and were surrounded by a dense layer of cake, while other fibers are not.

The build up of cake is not likely to be uniform throughout the fiber surface. This is due to the complexity of flow as it enters the bundle. Hence, it is possible that some of the fibers would get stuck together as their cake layers merge, and then these fibers would foul rapidly, while the other fibers would not foul as rapidly. This merging of cake layers has been observed when filtering activated sludge (Kiat et al., 2002). When feed concentration is high, the standard deviation is lower than when the feed concentration is low. This could be due to the fact that cake layers build up rapidly when the feed concentration is high. As the cake layers grew, they encompassed the entire fiber bundle, resulting in equally poor performance for all the fibers. In contrast, at lower feed concentrations, the cake layers do not grow as rapidly and this leads to the possibility of preferential growth of the cake in certain directions, which would cause the performance of certain fibers to degrade and not others. It should be noted that local blockage is a self-accelerating phenomenon as an incipient blockage will reduce local flows which would encourage further deposition. A description of this process is given in Figure 4.21.

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4.4.3 Comparisons within the bundle

In the 3 by 3 matrix of fibers, there were only 3 unique positions, the centre fiber (Fiber 5), the corner fibers (Fibers 1, 3, 5 and 9) and the side fibers (Fibers 2, 4, 6 and 8). Besides the flux obtained from the fibers after an hour, the rate at which the flux changed on a minute by minute basis was also determined for fiber 5, representing the centre fiber, fiber 6, representing the side fiber, and fiber 7, representing the corner fiber. A typical flux-time graph is shown in Figure 4.7. Due to the protocols of this experiment, it was not possible to determine the change of flux over time with the other fibers, unlike for the constant flux experiments.

Figure 4.7 Flux-Time relationship for series HFBX, Crossflow velocity of 0.005m/s

To aid in the analysis, a parameter was defined to characterize the performance of a given fiber relative to all fibers;

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$$\alpha = \frac{\text{Flux from specified fiber}}{\text{Average flux of all fibers in bundle}}$$

α is a measure of how much flux a particular fiber is producing as compared to the average of the fiber bundle. If the α value is low, the fiber is producing less flux as compared to the other fibers, if the α value is higher, the fiber produces more flux as compared to the other fibers.

Figure 4.8 α relationship for series HFBX, Centre fiber

In Figure 4.8, the variation of α of the centre fiber with at different pressures and axial flow velocities is examined. As the pressure increased, or as the axial flow velocity decreased, the α value of the centre fiber fell. Increasing either the pressure (and hence the flux) or the decreasing the axial flow velocity encouraged the growth of a cake layer on the surface of the fibers. This led to more severe blocking of the fiber bundle.

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As Fiber 5 is in the centre of the bundle, it is surrounded on all sides by fibers, and would tend to perform the worst.

Figure 4.9 α relationship for series HFBX, Side fiber

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Figure 4.10 α relationship for series HFBX, Corner fiber

Figures 4.9 and 4.10 show the α values for the side and corner fibers respectively. The side fibers were surrounded by fibers on 3 sides, and had α values of between 0.8 and 1. The corner fibers have α values of between 1 and 1.4, as they are surrounded by fibers on 2 sides.

This shows the importance of the outer fibers in a fiber bundle in maintaining the total flux of a membrane bundle. Compared to fibers at the centre, fibers at the rim of the bundle produce 20% to 40% more flux. This is significant in smaller bundles, but would probably be less so in larger bundles.

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Figure 4.11 α relationship at 50kPa, Centre fiber

Figure 4.11 examines how α changes under different packing densities and feed concentrations. At low axial flow rates and a packing density to 44% (HFBX), the α value was 0.60. This meant that the middle fiber was only contributing 60% of the average flux of the fibers, and remained low even at high axial flow rates. At a high feed concentration (HFBH), there was no contribution to the flux at all, in effect the fiber had been completely fouled and contributed nothing to the performance of the bundle. For a packing density of 28% (HFBY), the alpha value was about 0.9, and at low feed concentrations for the higher packing density (HFBL), the α value was 0.78. In all cases, the alpha values approached unity as cross flow velocity increased.

The side and corner fibers would have experienced the main flow over much of their surface, and would have had a higher shear rate over those surfaces. This should have caused the performance of these fibers to be better than that of the centre fiber, and this

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was true for all the cases studied. The corner fibers had slightly more surface exposed to the main flow as opposed to the side fiber, and this explains why the corner fibers performed somewhat better than the side fibers. Moreover, it has been found (Figure 4.25) that the velocity within the hollow fiber bundle is much lower than the velocity outside it.

For the centre fiber, the performance would degrade significantly once cake accumulates in such a way that it blocks flow towards the fiber. This is the self-accelerating phenomenon referred to earlier. As pressure increases, the cake layer grows, and blocking occurs more quickly. This was also true for low cross flow velocities. This would cause the performance of the centre fiber to degrade more quickly than the corner or side fibers.

By spacing the fibers further apart, it is possible to slow this process, or even avoid it altogether, as shown in series HFBY (Figure 4.10). It can be seen that the performance of the centre fiber was close to that of the other fibers in this case. At high feed concentrations (HFBH), coupled with low cross flow velocities, the centre fiber was completely blocked and no flux was measured. These observations are different from the early work of Kiat et al (1992), probably due to the larger average fluxes observed in the current study.

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4.4.4 Effects of Bubbling

Figure 4.12 Effect of bubbling on flux

When bubbling was applied, there was a marked improvement in the performance of the bundle. Comparing the average of flux from the fibers, at 50kPa, there was a more than sixfold increase in the flux. (Figure 4.13) Furthermore, the standard deviations between the fibers in the same position fell to less than 0.1. The alpha values for all the fibers were between 0.95 and 1, eliminating the differences between them.

Whilst the overall benefits of bubbling are well established (Cui et al., 2004), these results show dramatically the effect at the individual fiber level in the bundle. The effect of bubbling is discussed in detail in Section 4.4.

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4.5 Operation in Constant Flux Mode

As established in Section 4.4.4, the role of bubbling on the performance of hollow fiber bundle is very important in improving the performance of the bundle. In this section, the setup of the experiment has been improved, and operation is now in constant flux mode. Most filtration operations are conducted in constant flux mode to provide a constant flow of treated water.

The role of bubble size and frequency are examined in this section. PIV experiments are conducted to determine the hydrodynamic conditions within the bundle. The experimental conditions are as described in Section 3.2 of Chapter 3. As a reference, the experimental conditions are reproduced in Table 4.2.

Table 4.2 Constant Flux Experiments

No.	Series Name	Bubble		Crossflow (m/s)	Length (m)	Aeration Inlet
		Type	Frequency (Hz)			
1	CX	-	-	0	0.4	-
2	C1	-	-	0.0067	0.4	-
3	C4	-	-	0.0267	0.4	-
4	BS0.5	Small	0.5	-	0.4	Centre
5	BS2	Small	2	-	0.4	Centre
6	BL0.5	Large	0.5	-	0.4	Centre
7	BL1	Large	1	-	0.4	Centre
8	BS1 Loose	Small	1	-	0.404	Centre
9	BL1 Loose	Large	1	-	0.404	Centre
10	BS0.5 OC	Small	0.5	-	0.4	Off-Centre
11	BS2 OC	Small	2	-	0.4	Off-Centre
12	BL0.5 OC	Large	0.5	-	0.4	Off-Centre
13	BL1 OC	Large	1	-	0.4	Off-Centre

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4.5.1 Filtration Experiments

4.5.1.1 Standard Deviation of the Fluxes

Figure 4.13 Flux obtained from individual fibers under constant flux conditions in the absence of axial flow or bubbling (Series CX, Table 4.2)

A typical flux distribution among the fibers is shown in Figure 4.13. In this example, it took some 45 minutes to adjust the average flux to the desired value of 18 l/m²/hr. However, at all times the applied transmembrane pressure was the same for all 9 fibers. The relatively long start up period was due to the time needed for the permeate to move through the tubes and be collected by the measuring cylinder.

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Figure 4.14 Flux obtained from the corner, side and centre fibers when small bubbles are used at a frequency of 0.5 Hz (no liquid flow, BS 0.5, Table 4.2)

Figure 4.14 shows the average fluxes obtained from the corner (C1, C3, C7, C9), side (C2, C4, C6, C8) and middle fibers (C5) under small bubbling conditions at a frequency of 0.5 Hz. The bubbling location was from the centre. The performance of individual fibers under the constant flux conditions was similar to the performance under constant pressure conditions. The centre fiber produced the least amount of flux as compared to the other fibers, and declined more rapidly. The side fibers performed better than the centre fiber, but the fibers at the corner positions performed the best. The differences in performance are attributed to an apparent “hydrodynamic resistance” in the bundle, which we assume to have two components. One component is due to the bundle resistance, which is related to the need for lateral flow into the bundle structure. The other component is due to permeate competition, which we define below. The “bundle resistance” occurs centre fiber is surrounded on all sides; the side fibers had fibers on 3 sides, while the corner fibers had neighboring fibers on only 2 sides. The fibers were spaced closely together (inter-fiber gap was 0.5mm) so there was more

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sheltering around the centre fiber which would naturally lead to a weaker flow around this fiber.

However, the centre fiber has an additional disadvantage which is “permeate competition.” The flux into a fiber generates a radial velocity profile normal to the fiber surface. This means that radial velocity vectors will decline from a value (u_o) at the fiber surface (fiber radius r_o) to a value of $u_o (r_o/r)$ at a distance $(r-r_o)$ from the fiber surface. Thus, for fibers of 0.5mm radius, the radial velocity vector will decline by 50% at a point 0.5mm from its surface. If another fiber is located at this point, it will experience this fluid vector away from its surface which will compete with its own flux generated radial vector towards its surface. For an array of fibers, those that are completely surrounded by other fibers (in our case, fiber 5) will experience the greatest permeate competition and those at the corner of an array will experience the least permeate competition.

Overall the contribution of “bundle resistance” and “permeate competition” results in an apparent “hydrodynamic resistance” within the bundle matrix. This explains the differences in the initial fluxes shown in Figure 4.14. In other words, the sheltered fibers experience slightly lower fluxes than fibers at the edges of an array or bundle. Usually lower fluxes mean less fouling, all other factors being equal. However, in our case, the lower flux central fiber shows more fouling (declines towards the end of the experiment) which must be caused by its less effective hydrodynamic environment (See Section 4.4.3).

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Figure 4.15 Variation of Standard Deviation with Time for no bubbling conditions

In order to quantify the homogeneity of fouling or blocking of the hollow fiber system, the standard deviation of the individual fluxes of each of the nine fibers was calculated. This is similar to the standard deviation in Section 4.4.2. The main difference is that for the experiments conducted under constant flux conditions, it was possible to obtain the standard deviation over a period of time, instead of at the end of every 1 hour. A high standard deviation would imply that there was a significant difference amongst the fluxes obtained from the individual fibers, and vice versa. The transient variation of the standard deviations when no bubbling was applied is presented in Figure 4.15. When there was no axial flow (line A in Figure 4.15) and at a small axial flow velocity of 0.0067m/s (line B in Figure 4.15) the standard deviation increased exponentially, after approximately 100mins, indicating that fouling or blocking had begun. These observations confirm that at low liquid flow velocity, the particle removal rates were negligible and fouling occurred progressively and preferentially around some fibers. Increasing the axial flow to 0.0267m/s (line C in Figure 4.15) caused a marked

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improvement in fiber performance. The standard deviation remained range-bounded between 0.05 and 0.15 after 3 hours.

A standard deviation of between 0.05 and 0.15 can be considered as the “background” standard deviation in our system due to the differences in the permeability of the individual fibers, as well as the “permeate competition” effect (discussed above) and the random build up of particles on the fiber surfaces. Observations of the trans-membrane pressure, which will be discussed in more detail in the following sections, showed that when the standard deviation was within this range the TMP did not rise significantly, indicating that the fibers were not subjected to excessive fouling or blocking.

Figure 4.16 Variation of Standard Deviation with Time when bubbling is introduced from the centre-line

Figure 4.16 shows the variations of the standard deviation when the bubbling was introduced from the centre of the module for the small bubbles introduced at 0.5 and

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2.0Hz and large bubbles introduced at 0.5Hz and 1.0Hz; no axial liquid flow was imposed. When small bubbles were used (lines A and B), the standard deviations increased more rapidly compared to large bubbles (lines C and D). This is due to the fact that individual large bubbles have stronger wakes, and induce more movement in the fluid. Stronger wakes are able to penetrate into the bundle and induce a stronger shear stress on the fiber surface to remove the particulates that have been deposited. This would retard the build up of particles on the surface of the fiber, and also promote more uniform fouling. Similar reasoning explains why increasing the bubbling frequency led to a lower standard deviation.

Figure 4.17 Variation of Standard Deviation with Time when medium bubbles are introduced from the centre-line

When medium sized bubbles were used, the effect of increasing the frequency using the same size of bubble can be clearly observed. At a very low frequency (0.1 Hz, line A in Figure 4.17), the fiber performs similarly to when the fiber is in stagnant water. This

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indicates that at such a low frequency, the action of the bubbles is insufficient to prevent fouling. Increasing the frequency causes the standard deviation to become more stable. At 0.5Hz (line B in Figure 4.17), the standard deviation of the fluxes of the fibers is less, indicating the fouling of the fibers is more even. At the higher frequencies of 1.2Hz and 2.0Hz, the standard deviations are more or less constant over 180mins (lines C and D in Figure 4.17). This shows that there is a limit to the aeration intensity required to stabilize the standard deviation. Increasing the intensity of aeration cannot improve the performance indefinitely.

Figure 4.18 Variation of Standard Deviation with Time when bubbling is introduced from the off-centre

The location of the bubble injection had a significant impact on bundle performance. Thus, when bubbling was applied from the off-centre position, the variations of the standard deviations with time were similar regardless of bubble type or frequency (Figure 4.18). This is in contrast with the results in Figure 4.15, where bubbling with small bubbles (applied from the centre-line) induced higher standard deviations. With

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the aeration now introduced further away from the bundle, the effects of bubbling were not as significant as compared to the central bubbling position. Hence, the differences between larger and smaller bubbles become less obvious. However, it is interesting to note that despite the differences in the size of the bubbles, there was little difference in the behavior of the standard deviation. This is likely to be due to the distribution of flow around the bundle and a detailed assessment on why the fibers foul more evenly when bubbles are introduced from the side is presented in the following sections.

4.5.1.2 Fouling Characteristics of Individual Fibers in a Bundle

Reference experiments under the base condition of no axial liquid flow and no bubbling were repeated in order to examine if there was a fouling pattern among the individual fibers in the experimental fiber bundle. In all the experiments, the middle fiber fouled rapidly, other fibers followed progressively. Some fibers which had higher fluxes in the first experiment performed worse in the second experiment and vice versa. This is similar to findings from the experiments conducted under constant flux conditions. In this series of experiments, it was possible to understand the rate at which the fibers foul.

In the absence of a particle removal mechanism, the initial build up of particles on an individual fiber becomes a random process, which explains the unpredictability of which fiber would foul first in a fiber bundle. However, when the cake started to build up on (central) fiber 5, it would connect with other cake layers building up on the other fibers, causing serious blocking. This is why the centre fiber consistently performs the worst in the bundle. However, the random effect for the other fibers may be, in part, due to slight discrepancies in the positions of the fibers. This phenomenon is discussed in the section on PIV techniques.

When the particle removal mechanism was effective, such as when the crossflow velocity was high (0.0267m/s) or when bubbling was efficient, a significant build up of

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particles on the surface of any of the hollow fiber membrane would not occur. As a result, fouling was even across the fiber, and there was little difference in the fluxes of the individual fibers (See line D, Figure 4.16, for large bubbles at 1.0Hz on the centre-line.)

When the large bubbles moved up the column, it was observed that the bubbles did not intrude into the bundle itself due to surface tension effects. Thus, as the bubble moved up around the bundle, the water within the bundle migrated due to drag forces. This created good flow agitation within the bundle with the net effect that fouling was kept to a generally low level. This is confirmed by PIV results (below) which show a high net flow when large bubbling is applied. The strong agitation caused by the large bubbles contributed to the low fouling observations, but at a cost of higher required air flow rate.

Figure 4.19 Fluxes obtained from individual fibers under constant flux when small bubbles at 2Hz are used (centre-line)

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There is an initial jump in measured flux in Figure 4.19 as the fluid takes time to travel through the pipes before falling into the measuring cylinders. When small bubbles were introduced into the module from the centre, we observed the unexpected, reproducible result that fiber 8 had one of the least fluxes, with the exception of the centre fiber (Figure 4.19). This was surprising because fiber 8 was the closest to the source of aeration (see Figure 3.2). Upon closer examination, it was observed that the behavior can be explained by the flow pattern inside the bundle. When the bubble moved up the column, it drew the surrounding fluid to flow laterally through the spaces between the fibers. A dead zone was then formed due to the sheltering by Fibers 2 and 5. This dead zone is a region where there is little or no fluid movement.

Figure 4.20 Dead zones when small bubbles are introduced from the centre

The liquid is induced to flow through the bundles as shown in Figure 4.20. As the bubble is introduced near fiber 8, and as the bubble rises, water is drawn across the fibers to replace the air that is moving upwards. In this case, a “dead zone” of little or no flow will be induced between fiber 5 and 8. Due to the lack of fluid movement,

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significant fouling on Fiber 5 initiated. The cake layer grew, and then connected with the surface of fiber 8 and caused the performance of fiber 8 to degrade next.

When the bubbles were introduced from the off centre position, the bubbles were further away from the fiber bundle. Although the strength of the wakes was weaker due to the distance, the turbulent flows caused by the wakes were able to distribute more evenly over the nine fibers due to the absence of dead zones. This gave rise to a lower standard deviation of fluxes when small bubbles were introduced from the off-centre as opposed to the centre position. The key point appears to be to provide sufficient surface shear as evenly as possible to maintain homogeneity of performance.

Another notable feature of inter fiber blocking with small bubbles is that the blocking always occurred initially in a 2 by 2 square. Based on replicated experiments, the 4 fibers with the lowest amount of flux produced were corner fibers 4,5,7,8 or 5,6,8,9 for centre-line injection, or the fibers furthest from injection for off-centre injection. From these observations, it is postulated that inter fiber blocking in a 3x3 bundle occurs in the following manner. A cake layer forms behind fiber 8 due to the weaker hydrodynamic conditions in that location. Cake also builds up unevenly on the various fibers, but most quickly on the centre fiber (fiber 5). Eventually, the cake layers from fiber 5 and fiber 8 merged, and the cake layers from fibers on either side of fiber 5 (fibers 4 or 6) would then merge into this cake layer. Finally, the fiber at the corner of the bundle would also start to be blocked, as it was surrounded by badly blocked fibers (Figure 4.21). When bubbles are released from the off-centre, the fouling always occurred further from the fiber. As there is now good distribution of flow within the fiber bundle, the fouling would occur further from the bubbling source as the effects of bubbling were felt more weakly there.

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Figure 4.21 Stages of fouling

4.5.1.3 Pressure

Table 4.3 Average Final TMP after 3 hours for all experiments (OC stands for off-centre bubbling)

Experiment Conditions	Average Final TMP after 3 hours (kPa)
No Axial Flow	76.95
Axial Flow = 0.0067m/s	78.40
Axial Flow = 0.0267m/s	13.79
Small Bubbles (0.5Hz)	78.12
Small Bubbles (2.0Hz)	63.99
Small Bubbles (5.0Hz)	50.44
Small Bubbles (0.5Hz) OC	50.80
Small Bubbles (2.0Hz) OC	42.68
Medium Bubbles (0.1Hz)	83.34
Medium Bubbles (0.5Hz)	50.44
Medium Bubbles (1.2Hz)	14.65
Medium Bubbles (2.0Hz)	12.42
Large Bubbles (0.5 Hz)	30.55
Large Bubbles (1.0 Hz)	11.17
Large Bubbles (0.5 Hz) OC	41.23
Large Bubbles (1.0 Hz) OC	30.34

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In the above experiments, when the suction pressure increased beyond 70 kPa, de-aeration of the water occurs. This is observed visually as bubbles appearing in the filtration line.

The suction pressure measurements (effectively the transmembrane pressure/TMP) in the vacuum box at the end of the experiments without bubbling are shown in Table 4.3. The final TMPs (after 3 hours of filtration) were similar for no axial flow, and an axial flow of 0.0067m/s. The final TMPs in these 2 experiments were almost 80kPa, which were close to the maximum negative pressure that the vacuum chamber could accommodate. Thus, a small increase in the axial liquid velocity was insufficient to improve the particle deposition on the fiber. However, increasing the axial flow velocity to 0.0267m/s brought about a large improvement in terms of pressure which reduced to 14 kPa.

When small bubbles were used, increasing the bubbling frequency, i.e. increasing the gas flow rate, led to a decrease in the final TMP. Moving the bubbling from the centre to the off-centre position also caused the pressure requirement to drop. The effect of changing the bubble location is best illustrated by comparing bubbling from the centre at 5.0Hz and bubbling from the off-centre at 0.5Hz. The suction pressures at the end of 180 minutes were similar for both cases. Thus, by simply changing the location of bubbling, it was possible to reduce the air flow by ten times to get the same suction pressure, for this particular module.

When large bubbles were used, the pressure obtained was much lower than that for the small bubbles. Increasing the bubbling frequency decreased the final pressure, while moving the bubbling location from the centre to the off-centre position caused the final pressure to increase. These phenomena will be discussed together with the PIV results.

When medium bubbles were used, we can see clearly the effect of increasing the bubble frequency. The difference between the final pressure at a low frequency of

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0.1Hz and when the water is stagnant is insignificant. This indicates that there must be a minimum frequency of aeration before there is an impact on the performance of the fibers. Increasing the frequency of the medium bubbles has a beneficial effect on the performance of hollow fibers in terms of TMP. However, when the frequency reaches between 1.2Hz and 2.0Hz, increasing the frequency does not appear to have much additional effect of the performance of the hollow fibers. This is an indication there is an optimum level of aeration, above which no significant improvement in the fouling behavior is observed.

4.5.1.4 Relationship between the Standard Deviation and TMP

Figure 4.22 Variation of pressure and standard deviation with time when there was an axial flow of 0.067m/s

The standard deviation of the fluxes for the array of fibers was closely related to the transmembrane pressure (TMP) applied to the bundle as illustrated by Figure 4.22. The

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rise in TMP in Figure 4.22 corresponded closely with the increase in standard deviation. The direct correspondence was observed irrespective of the presence of bubbling, the size of the bubbles, or the bubbling location. There was also evidence that the rise in standard deviation preceded the rise in suction pressure which could lead to a means of warning of imminent fouling or blocking problems. When the TMP did not increase during the course of the experiment, the standard deviations of the fluxes did not rise above about 0.20.

Figure 4.23 Variation of Final Pressure with Standard Deviation for all experiments

The correlation between the required TMP and the standard deviation for all the experiments is further elaborated in Figure 4.23. When the standard deviation was below about 0.15, the pressure was generally low. As the standard deviation increased beyond 0.2, the required pressure increased. This is strong evidence that once there is too much variation among the individual fibers, the performance of the hollow fiber bundle will deteriorate rapidly. Hence, this leads to an important conclusion that the bundle should be operated within a limited standard deviation of fluxes, which is about

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0.20 in this case. Beyond that, the bundle should be cleaned, either mechanically or chemically. A practical issue for large modules is how to determine the development of flux heterogeneity.

4.5.2 Particle Image Velocimetry

The preceding results point to the importance of liquid flow within the fiber array. In order to obtain insight into the local flow distributions, we have use Particle Image Velocimetry (PIV) to make non-invasive measurements of liquid movement. However to do this, we have had to simulate the hollow fiber bundle by using a bundle of optical fibers and refractive index matching as described in the experimental methods.

4.5.2.1 PIV- Axial Flow

Figure 4.24 PIV image of bubble and fibers

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PIV was used to determine the velocity profiles in-between fibers 4, 5 and 6. Figure 4.24 is a photograph of an image taken by the camera while carrying out PIV experiments. As the refractive indices of the bulk solution, as well as the optical fibers representing hollow fibers are matched, the boundary between the fiber and the fluid is not clearly defined. To allow for this, the positions of the fibers were first marked without solution.

Figure 4.25 Velocity profiles using PIV with different axial flows

The velocities were determined by averaging all the velocities in the x-direction, over 1000 sets of images. A typical velocity profile is shown in Figure 4.25 for two different axial flow rates. From the figure, we can see that the velocities close to the fiber tend to be close to zero. This is due to friction effects near the surface of the fiber. The apparent velocities within the bundle do not display this phenomenon. The reason for this is that at the centre of the bundle, the fibers maybe be slightly shifted from their position compared to the top and bottom of the bundle. The fibers are 1mm in diameter, and only 0.5mm (500 microns) apart. It is practically impossible to ensure that the

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fibers remain in their exact square position. As the velocities are taken as an average over a thickness of 1mm (the thickness of the light sheet), a slight shift in the fiber could have led to a slightly different reading.

It is probably because of this phenomenon that the fibers tend to foul in a random manner when there is no axial liquid flow or bubbling. The cake layer would grow preferentially on some fibers due to the slightly non-uniform placement of the fibers. Thus, repeating experiments in this scenario leads to different fibers being blocked first, depending on the exact geometry at that point in time. However, once a cake removal mechanism is present, there would be some sort of pattern to the fiber blockage. This implies that the effect of the non-uniformity of the fiber placement has little effect once a cake removal mechanism is present. However, it is clearly seen in Figure 4.25, that the velocity within the fiber bundle is much lower than the velocity outside the fiber bundle; this confirms the difficulty of the axial flow outside the fiber array penetrating inside the fiber bundle.

In principle, it would be possible to estimate the shear stresses based on the velocity profiles near the surface of the fibers, however it would be unwise to do so in this case. This is because, we cannot be completely certain of the exact location of the fiber with the present procedure. Moreover, the fibers are so close together, they could intrude into each others viscous laminar sublayers. In this case, the assumption that the shear stress can be determined from the velocity gradient of the flow would be invalid. Thus, the average velocity within the spaces between the fibers was determined as a measure of the penetration of the flow into the bundle.

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4.5.2.2 PIV – Centre-line Bubbling

Figure 4.26 Time series of the average velocity under the condition of 0.5 Hz small bubbling

Examining a random segment of the time series when small bubbles are applied at 0.5 Hz, we notice that there are periods when the velocity drops suddenly, as shown in Figure 4.26. In Figure 4.26, we examine the time interval between 179 and 185s. This is an indication of when the bubble is passing the window of observation. As the bubble moves upwards, at that instant, the fluid must be moving downwards due to mass balance. However, when a time series of an experiment using small bubbles at 2.0Hz is examined, this trend could not be observed. This is probably due to the fact that the sampling rate is 5Hz, while the bubbles were passing at 2Hz. Under these conditions, the sampling rate was not fast enough to capture the trend.

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Figure 4.27 Average velocity measured inside the fiber bundle (Bubbling from the centre-line)

Within the gap between fibers, there are about 5 to 6 velocity measurements. To obtain the average velocity, the average of the all the data points within each gap is taken over the 1000 sets of readings. Figure 4.27 shows the variation of average velocity, measured within the bundle, with the bubble size and frequency. When small bubbles are used, increasing the bubbling frequency increases the average velocity. An increase in the bubbling flow rate of 4 times for the small bubbles (from 0.5 Hz to 2.0 Hz), leads to an increase in the average velocity of more than 4 times. This indicates that using small bubbles at a higher bubbling rate may have a non-linear effect on the performance of the bundle. The velocity induced for the medium sized bubbles is higher than that of the small sized bubbles (although not by much at 2.0Hz).

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Figure 4.28 Plan and side view of a large bubble moving up a bundle of fibers

For the large bubbles, increasing the bubble frequency actually causes the velocity within the bundle to fall. The reason for this is when the large bubbles move up along the fibers, they wrap around the fibers as depicted in Figure 4.28. Due to surface tension effects, the large bubbles tend not to move into the spaces between the fibers. The upward motion of the large bubbles causes fluid to move downwards within the bundle as shown in Figure 4.28. Under these conditions, increasing the frequency of bubbling, would lead to a lower average velocity induced within the bundle. However, as is shown later, the filtration performance is not very sensitive to the average liquid velocity, but is sensitive to the fluctuation of the velocity.

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Figure 4.29 Standard deviation of the average velocities (Bubbling from the centre)

As a measure of the fluctuation of the velocity, the standard deviation of the velocities was determined (Figure 4.29). It is evident that increasing the frequency or the bubble size increases the standard deviation of the average velocities. Increasing the bubble size or frequency causes stronger wakes in the fluid. The wake caused by the bubble is the main cause of variations in the flow and the wake induced, per bubble, by the small bubbles are inherently weaker than those of the large bubbles.

However, it should be noted that the “benefit” of the large bubbles is not conclusive. The approximate ratio of the bubble volumes small: medium: large is 1: 12.5: 93, so in our study, a single large bubble consumes 7x the airflow of a medium bubble and 93x the airflow of a small bubble. Figure 4.29 shows that medium bubbles at 2.0Hz generate similar standard deviations of velocity as large bubbles at 1.0Hz giving the same effect with only 30% of the airflow rate. Comparing the small bubble at 2.0Hz

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and the medium at 0.5Hz, we reach a similar conclusion, i.e. only 30% of the airflow is required with the smaller bubble.

4.5.2.3 PIV – Off-Centre Bubbling

Figure 4.30 Average velocity measured inside the fiber bundle (Bubbling from the off-centre)

As the bubbling is no longer symmetrical when the bubble is released from the off-centre position, the velocities of the left side of the module (between fibers 1,2,4,5,7 and 8) and the right side of the module(between fibers 2,3,5,6,8 and 9) are now compared separately in Figure 4.30. The velocities induced by the small bubbles in the off-centre position are much less compared to when the bubbles are introduced from the centre of the module. This is due to the fact that the bubbles are now released further from the fiber bundle, thus, the effect of bubbling is weaker. Increasing the frequency of bubbles had little effect on the induced velocities. At both frequencies, the velocity on the right side (nearer the source of aeration), is higher than that of the left

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side. As the bubbles are nearer to the right side, the effect of bubble passage would be felt more strongly there.

However, in this section, only the velocities in the axial direction of the fiber are considered in our study. It was not possible to determine the velocity of the fluid in the radial direction, i.e. normal to the plane of the light sheet. It is this movement of water that is expected to enhance the performance of hollow fibers when small bubbles are used.

When the large bubbles are introduced from the off-centre position, we observed that the velocities induced are all strongly negative, and the magnitudes are higher than when the bubbles are introduced from the centre. The reason for this is that the bubble has again wrapped around the fiber bundle as in Figure 4.28, however, since the bubbles are now introduced at the side of the bundle, there is no chance for small satellite bubbles to enter into the bundle. Thus, the higher the bubbling frequency, the greater the magnitude of the velocity in the negative direction to compensate for liquid upflow with the large bubbles.

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Figure 4.31 Standard deviation inside the fiber bundle. (Bubbling from the off-centre)

The standard deviations of the average velocities are compared in Figure 4.31. The standard deviations induced by the small bubbles were low due to the fact that the velocities induced were quite weak, while the standard deviations generated by the large bubbles in the off-centre bubbling mode are comparable to when the bubbling is introduced from the centre. However, as indicated previously, the velocities and standard deviations in the radial direction could not be measured. Since, the large bubbles that are introduced close to the array of fibers “wrap” around the fibers, the hydrodynamic situation, in terms of the standard deviation, within the hollow fibers should be quite similar.

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4.5.3 Relationship between hydrodynamics and performance

Figure 4.32 Variation of final TMP with average velocity

Figure 4.33 Variation of final TMP with standard deviation

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Figures 4.32 and 4.33 compare the variation of the final filtration TMP obtained with the average array velocities and standard deviations respectively. For the bubbles that are introduced from the off-centre, the average velocity and standard deviations of the left and right gaps were taken. From Figure 4.32 there does not appear to be any clear relationship between the average velocity and the final TMP measured. When the velocity is low, the pressure varies greatly. This would imply that low axial velocities per se have little or no effect on the performance of the hollow fiber, although, at the highest absolute average velocities measured, there is trend of lowering the required TMP. On the other hand, Figure 4.33 shows that increasing standard deviation improved the performance of the hollow fiber bundle in terms of the final suction pressure. It is expected that the type of hydrodynamic situation observed in this study to be mirrored inside hollow fiber bundles used in industry. If this is correct, one of the keys to improving the performance of tight hollow fiber bundles would be the generation of a large standard deviation in local velocity as opposed to increasing the average velocity. This may explain the benefit observed by Guibert et al. (2002) by intermittent bubbling on either side of a hollow fiber bundle.

When small bubbles are used, there is a decrease in final TMP as the bubbling location is moved from the centre to the corner of the fiber bundle (Table 4.3). This is counter-intuitive as the velocities induced within the fiber bundle are much lower when the bubbling is introduced from the corner of the fiber bundle. Moreover, the standard deviations are also comparable. The performance of an individual fiber is a function of the flow across its surface. For PIV measurements, only the velocity of the flow in the axial direction (y-axis) of the fibers could be measured, and movement of fluid in the x-axis could not be measured. Unlike bubbling from the centre, bubbling from the corner precludes the formation of a strong “dead zone”.

Moreover, the above finding is significant because it shows that bubbling closer to the bundle (centre) is not necessarily better than bubbling further away. This is despite the

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fact the closer one gets to the bubble, the stronger the local effect of the wake. Thus, when designing an aeration system for fouling control, it is important to understand how this aeration system will affect the distribution of flow and fouling within the bundle. If the bubbles are introduced incorrectly, it could lead to an increase in the probability of differential fouling across the bundle and then the bubbling would actually be detrimental to the performance of the hollow fibers. The problem of differential fouling would be particularly important under constant “average” flux conditions where fouling and module blocking are self-accelerating phenomena.

For the large bubbles, moving the bubbling from the centre to the corner of the fiber bundle actually caused the performance of the fiber bundle to degrade. When large bubbles are introduced from the centre, there is a sudden explosion of air into the flow channel very close to the surface of one of the fibers. When this happens, small satellite bubbles are introduced in the middle of the fiber bundle. These bubbles would do a good job scouring the surface of the fibers. However, there is much less chance of this when the bubbles are introduced from the corner of the fiber bundle.

4.5.4 Implications

Although this study has involved a small model array with bubbling introduced at two “arbitrary” locations, the results suggest that large bubbles are not necessarily most beneficial. For example, it should be recalled that medium bubbles at 2.0Hz generated the same standard deviations of velocity as large bubbles at 1.0Hz, giving similar effects with only 30% of the airflow. A similar comparison of small vs. medium bubbles also showed equivalent effects with about 30% of the airflow when small bubbles were used.

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Figure 4.34 Comparisons of air flow rates and performance

In order to compare the effects of the 3 bubbles sizes we have plotted the final TMP for 3 hour experiments versus the air flow rate. The data are shown in Figure 4.34, and it should be noted that the x-axis is a log-scale. For the small bubbles it was difficult to achieve high air flow rates due to the limitations of the solenoid valve which has an upper limit of 5.0Hz. Figure 4.34 shows that low final TMPs could be achieved with both large and medium bubbles, but the amount of air flow required for the medium bubbles was about an order of magnitude lower. The data tend for the small bubbles, particularly for the OC location, suggests that they may outperform the medium bubbles. Thus, many small or medium bubbles may be able to achieve the benefits (or better) of fewer large bubbles with a potential reduction in required air flow. While it is difficult to predict the potential savings in airflow rate, our results suggest the current trend for coarse bubbling in submerged membrane systems may not optimal. It is of interest to note that similar conclusions can be reached from the results of Sofia et al. (2004) using coarse and fine bubble aerators in an MBR.

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4.6 Comparison between Experimental and Industrial Systems

When comparing the experimental system in this study with real systems, one of the issues to be addressed is the difference between the numbers of fibers of the two systems. As this study focused more on the flux determination from individual fibers under constant pressure and constant flux conditions, only a small bundle was used for experimental purposes.

In larger bundles, the number of fibers in the bundle would increase proportionally to the square of the radius of the bundle, while the number of fibers in the periphery would increase proportionally to the radius of the bundle. Hence, in an industrial system, the number of “centre” fibers would greatly outnumber the fibers in the periphery.

In the beginning of the operation of a hollow fiber module, the fouling tendency of the fibers at the periphery would be the highest. This is because the radial flow towards the perimeter of the bundle would be approximately the summation of the flux of the entire bundle. However, this fouling tendency may be balanced by the higher rates of removal of the foulants due to favorable hydrodynamic conditions.

4.7 Conclusions

Initial work was done to determine the factors affecting the performance of hollow fiber bundles under constant pressure conditions. The combined effects of low cross flow velocity and high pressure causes the fiber bundle performance to be much worse than that of a single fiber. It also increases the standard deviation of the fluxes of the individual fibers. Lowering the packing density leads to a marked improvement in the bundle performance as measured against the single fiber. Also, the negative effects of low cross flow velocity and high pressure are not as acute when packing density is

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high. This is caused by the fibers being further apart, and hence the cake layers from the fibers cannot interfere with each other.

Increasing the feed concentration has a very negative effect on fiber performance. At a low cross flow velocity and high pressure, the centre fiber was completely blocked up and there was no flux from that fiber at all. This is caused by the cake layer growing rapidly to block up the fibers. The rapid growth of the cake layer contributes to the fact that the fibers in the same position have near uniform performance. At low feed concentration, the performance of the bundle is better. However, the cake layer builds up slowly, and this gives the opportunity for the fibers to get stuck together, which in turn causes the fibers to have a standard deviation.

The mechanics of fouling in a model bundle of a 3 by 3 array of hollow fibers were also studied under constant flux conditions. Two complementary experimental setups were used in this study. The first was used to determine the individual fluxes produced by each fiber in “average” constant flux conditions. The second setup was used to determine the likely hydrodynamic characteristics within the bundle using Particle Image Velocimetry.

Since the rate of fouling was different for each fiber, the standard deviation of the fluxes of each fiber increased as the experiment progressed. When the standard deviation of fluxes and the suction pressure were compared over time a good correlation could be observed. As the standard deviation increased, the pressure required to maintain the constant flux increased in tandem. There is evidence that the increase in standard deviation of fluxes precedes the increase in suction pressure, pointing to a possible early warning of fouling. When a fiber fouled more seriously than the other fibers, then the decrease in the flux of that particular fiber must be matched by an increase in the flux of the other fibers to maintain a constant flux condition. The phenomenon is self-accelerating as the flux increases in the other fibers, which then causes them to foul. For the experiments conducted, it was found that an

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increase in the standard deviation of fluxes above about 0.20 would cause the pressure to increase significantly.

The apparent hydrodynamic resistance for each position in the hollow fiber array was determined. It was found that the standard deviation of the fluxes as predicted by the hydrodynamic resistance model was close to the standard deviation above which pressure increase started to occur. This meant that once the distribution of the flux became slightly different from the initial/intrinsic distribution, pressure rise would start to occur.

This study also demonstrated the importance of ensuring that no “dead zones” are present in the fiber bundle. When small bubbles were introduced from the centre of the module, a dead zone was formed due to blockage between fibers 2 and 5. The blockage caused these two fibers to foul rapidly, and the standard deviation and the suction pressure to increase. Applying aeration from off-centre resulted in reduction of the dead zones, and better performance in terms of suction pressure rise. This is in spite of the fact that the bubbles were closer to the bundle during centre bubbling.

In terms of the hydrodynamics, it was found that the bubble passage typically caused a sudden drop in liquid velocity. The average induced liquid velocity in the bundle could not be correlated to the final suction pressure, but the standard deviation of the liquid velocities had a strong influence on the performance of the fiber bundle. This suggests that operation of fiber bundles would be improved by strategies that increase unsteady-state flow conditions. The use of smaller sized bubbles was found to be more economical in these experiments. Small bubbles released from the corner of the bundle had a final TMP that was comparable to larger bubbles with a 10 times greater air flow rate.

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CHAPTER FIVE PERFORMANCE OF TIGHT AND LOOSE SINGLE FIBERS

5.1 Introduction

In the previous chapter, the potential of PIV techniques for the study of the hydrodynamics in fiber bundles was demonstrated. In this chapter, we use PIV to characterize the influence of bubbles on the shear stress and the movement of loose and tight fibers.

5.2 Experimental Protocols

Details of the experimental facilities and protocols used are given in Chapter 3.

5.3 Tight Fibers

In this section, the effect of bubbling on the performance of tight hollow fibers is examined. PIV techniques are also used to characterize the shear stress induced on the surface of the fibers due to bubbling.

5.3.1 Velocity Measurements

Figure 5.1 Velocity Vector Map (a) without bubbling (b) with bubbling

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Figures 5.1a and 5.1b show the typical instantaneous velocity vector maps without bubbling and with bubbling respectively. The length of the arrows indicates the magnitude of the velocity at that point. When there was no bubbling, the velocity vectors were uni-directional and increased with distance from the surface of the fiber. When bubbling was introduced, wake effects in the flow field could be observed.

5.3.2 Mean Shear Stresses

Figure 5.2 Mean shear stress at different bubble sizes and bubbling frequencies (Liquid Velocity, $V_a = 0\text{m/s}$)

When no imposed axial liquid flow was present, the shear stresses varied with the bubble frequency as shown in Figure 5.2. The shear stresses also varied with bubble size, with the shear stresses induced by small bubbles much less than those of the cap bubbles. They were, in turn, smaller than those for the slug bubbles. For the small bubbles, the shear stress increased almost linearly with the bubbling frequency. For the cap and slug

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bubbles, the shear stress increased linearly until about 1Hz, and then started leveling off at higher frequencies.

Figure 5.3 Mean shear stress at different bubble sizes and bubbling frequencies ($V_a = 0.016 \text{ m/s}$)

The trend for the shear stresses when imposed axial flow was present was similar to the case of no imposed axial flow, except that the values were generally higher (Figure 5.3). With imposed axial flow, the shear stress was present even without bubbling. Thus, unlike the case without bubbling, when the bubbling frequency was zero, there was still some positive value for the mean shear stress. With bubbling, the mean shear stress increased more or less linearly with the bubbling frequency for the small bubbles. For the cap and slug bubbles, the shear stress increased with the bubbling frequency at even faster rates though there was a tendency towards a leveling off effect as the frequency increased. This will be discussed in greater detail in the following sections.

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5.3.3 Standard Deviations of Shear Stress

Figure 5.4 Standard deviation of the shear stress at different bubble sizes and bubbling frequencies ($V_a = 0$ m/s)

Figure 5.5 Standard deviation of the shear stress at different bubble sizes and bubbling frequencies ($V_a = 0.016$ m/s)

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Figure 5.6 Standard deviation of the shear stress at different bubble sizes and bubbling frequencies ($V_a = 0.16\text{m/s}$)

The trends for the standard deviations were similar to those for the mean shear stresses. The standard deviation under the conditions of no axial flow, 0.016m/s of axial flow and 0.16m/s of axial flow are shown in Figures 5.4, 5.5 and 5.6 respectively. Without axial flow, the standard deviation increased linearly as the bubbling frequency increased for the small bubbles. For the cap and slug type bubbles, the standard deviations increased quickly when the bubbling frequencies were low, but began to level off at higher bubbling rates. With the low axial flow (0.016m/s), the standard deviations were very similar to no axial flow.

The behavior of the standard deviation was distinctly different with the axial flow of 0.16m/s . For the small and cap bubbles, the standard deviations reduced initially with bubbling frequency before increasing slightly and leveling off at about 1Hz . The standard deviation of the slug bubbles also showed the initial reduction before increasing to roughly the same value as when there was no bubbling present. Thus, the stronger the axial flow, the fewer the fluctuations were experienced in the flow.

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5.3.4 Effect of Bubble Size and Frequency on Induced Shear Stresses

The shear stress is a measure of the force acting on the surface of the membrane. Bubbling causes an increase in the shear stress as it induces the movement of fluid on the membrane. Since a larger bubble will induce more vigorous movement in the liquid, the slug bubbles would generate higher shear stresses than cap bubbles, followed by the small bubbles.

The mean shear stress can be separated into three components, the shear induced by the bubble, τ_b , the shear induced by the axial flow, τ_a , and the interaction between the flow and bubble, τ_{ba} as follows.

$$\tau_{total} = \tau_b + \tau_a + \tau_{ba}$$

If the relationship between the τ_a and τ_b , is that of a simple superposition, where the mean shear stress in the presence of bubbling and axial flow is the addition of the mean shear stress when there is only axial flow and the mean shear stress when there is only bubbling, then τ_{ba} would be zero. However, the experiments appeared to suggest otherwise. τ_{ba} was in fact a positive number, and increased as the bubble frequency and axial flow velocity increased (Table 5.1). The reason is probably because with axial flow and bubbling together, the velocities of the gas bubbles and the liquid phase increased, leading to a corresponding increase in the mean shear stress.

Table 5.1 Comparison of Shear Stress and Standard Deviation of Shear Stress for Cap Sized Bubbles

S/N	Bubble Frequency (Hz)	Flow Rate (m/s)	τ_{total} (Pa)	τ_a (Pa)	τ_b (Pa)	τ_{ba} (Pa)	τ'_{total} (Pa)	τ'_a (Pa)	τ'_b (Pa)	τ'_{ba} (Pa)
1	0.05	0.016	0.014	0.012	0.000	0.001	0.030	0.025	0.054	-0.050
2	0.1	0.016	0.030	0.012	0.003	0.015	0.063	0.025	0.066	-0.028
3	0.5	0.016	0.042	0.012	0.027	0.003	0.124	0.025	0.099	-0.001
4	1	0.016	0.080	0.012	0.050	0.019	0.165	0.025	0.149	-0.009
5	2	0.016	0.129	0.012	0.065	0.052	0.187	0.025	0.175	-0.013
6	0.05	0.160	0.164	0.155	0.000	0.009	0.196	0.196	0.054	-0.054
7	0.1	0.160	0.177	0.155	0.003	0.018	0.133	0.196	0.066	-0.128
8	0.5	0.160	0.195	0.155	0.027	0.013	0.126	0.196	0.099	-0.169
9	1	0.160	0.235	0.155	0.050	0.030	0.172	0.196	0.149	-0.173
10	2	0.160	0.279	0.155	0.065	0.060	0.166	0.196	0.175	-0.205

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Increasing the bubble size or frequency typically increased the standard deviation of the shear stress. This is because bigger bubbles and more bubbles would cause more wake and turbulence effects in the column.

Similar to the mean shear stress, one can separate the standard deviation into 3 components. The standard deviation due to the bubble, the standard deviation due to axial flow, and the interaction between the axial flow and the bubbling.

$$\tau'_{total} = \tau'_b + \tau'_a + \tau'_{ba}$$

For the standard deviation, it was found that τ'_{ba} is negative. This implies that the axial flow in the background caused the standard deviation to be lower than the superposition of the standard deviation of the bubbling and axial flow. When there was a strong background flow, the turbulence induced by the bubbles was dampened and the background flow was quickly re-imposed on the fiber surface. This reduced the fluctuations in the bubble column, and the standard deviation was reduced as a consequence (Table 5.1)

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5.3.5 Effect of Bubbling on the rate of increase of TMP (dP/dt)

Figure 5.7 Rate of TMP rise at different bubbling sizes and bubbling frequencies ($V_a = 0\text{m/s}$)

Figure 5.8 Rate of TMP rise at different bubbling sizes and bubbling frequencies ($V_a = 0.016\text{m/s}$)

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Figure 5.9 Rate of TMP rise at different bubbling sizes and bubbling frequencies ($V_a = 0.16\text{m/s}$)

The rate of increase of trans-membrane pressure (dP/dt) gives an indication on how quickly the membrane is being fouled. The dP/dt values observed were dependent on the frequency and the size of the bubbles and on the imposed liquid flow as shown in Figures 5.7 to 5.9. When no axial flow was present, the dP/dt fell as the bubbling frequency increased. The fall was greater the larger the bubbles (dP/dt [small] > dP/dt [cap] > dP/dt [slug]). The effect tended to level off at 0.5Hz, where any increase in the bubbling frequency for any bubble size did not bring a noticeable improvement in the performance of the fiber. The trend was repeated when there was an axial flow of 0.016m/s.

When the axial flow was increased to 0.16m/s, the dP/dt did not change when bubbling with small bubbles. This implies that the use of small bubbles was not effective in controlling the fouling of the hollow fiber under high axial flows. For the cap and slug bubbles, no effect was observed when bubbling was below a frequency of 1Hz. However, when the frequency increased to 1Hz and beyond, there was a marked fall in dP/dt .

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The action of bubbling on submerged hollow fibers can be divided into 2 stages. The first stage is when the bubble is in contact with the fiber. In the second stage, the fiber experiences the wake of the bubble, and when the wake dissipates, the fiber then experiences the background flow (if it exists). The first stage would definitely be present for the cap and slug bubbles, since the bubbles take up the entire column area as they rise. However, the small bubbles may or may not touch the fiber surface. Visual observations during the experiments suggest that the small bubbles seldom moved close to the fiber. For the second stage, the effects of the wakes depend on the bubble type, the background flow as well as the distance of the fiber from the bubble. Different sizes of bubbles give rise to different wake effects (Ducom et al., 2002). Hence, it is useful to know which stage is more important in controlling fouling.

The analysis of the two stages is similar to when bubbling is applied to the inside of a tubular membrane (Cui et al., 2002). However, unlike the case where the bubbling is traveling up a well defined tube, the paths of bubbles in a submerged membrane module are more tortuous and the effects would be less certain. Thus, the fiber may experience both stages, or just the wake stage, depending on the bubble's motion.

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Figure 5.10 Rate of increase of TMP at different gas flow rates ($V_a = 0\text{m/s}$)

Figure 5.11 Rate of increase of TMP at different gas flow rates ($V_a = 0.016\text{m/s}$)

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Figure 5.12 Rate of increase of TMP at different gas flow rates ($V_a = 0.16\text{m/s}$)

In the previous chapter, it was demonstrated that it was possible use a much lower airflow rate (up to 10 times less) to achieve a particulate fouling rate for a tight fiber bundle by using smaller bubbles. A comparison of the effects of gas flow rates on controlling dP/dt for various axial flows is shown in Figures 5.10 to 5.12. The gas flow rates for the small bubbles are usually much lower than the cap and slug bubbles due to the intrinsic small size of the bubble. Without axial flow (Figure 5.10), dP/dt fell as the gas flow rate increased at a more or less constant rate, and a dP/dt of 2kPa per minute can be achieved with a gas flow rate of 0.01 liters per minute using small bubbles or 0.1 liters per minute using cap or slug type bubbles. For the smaller bubbles, there is much less contact of the bubble with the fiber as compared to the larger bubbles. The fact that the same performance can be achieved with much less air flow provides evidence that it is the wake of the bubbles, rather than contact with the fiber that determines the performance of submerged hollow fibers. Interestingly, when the gas flow rate was between 0.05 to 0.3 liters per minute, it did not matter whether cap or slug type bubbles were used. The performance of the fiber in this case only depended on the gas flow rate.

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At an axial flow rate of 0.016m/s (Figure 5.11), the performance, in general, was better (lower dP/dt) than without the axial flow. Hence, a slow axial flow enhances the performance over a stagnant column for all bubble types. Again, it was possible to achieve the same dP/dt at a much lower gas flow rate with small bubbles as compared to using large bubbles. When axial flow is present, comparing the cap and slug bubbles, there is now a difference in the performance of the fiber at the same gas flow rates. Thus, when axial flow is present, it is now important to take into account the type of the bubble. At gas flow rates between 0.05 and 0.3 liters per minute, cap bubbles yielded better performance than slug bubbles. This phenomenon will be discussed in the next section. At high axial flow rates (Figure 5.12), using a gas flow rate of below 0.08 liters per minute does not result in any improvement in dP/dt , only using high gas flow rates and bubbling frequencies can one improve the performance of the fiber.

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5.3.6 Statistical Parameters of Shear Stress and the Performance of the Fiber

5.3.6.1 Mean and Standard Deviations

Figure 5.13 Mean shear stress at different axial flow rates and bubble regimes

Figure 5.14 Standard deviations of the shear stress at different axial flow rates and bubble regimes

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The mean shear stress and standard deviations are compared at different gas flow rates in Figures 5.13 and 5.14 for cap and slug bubbles. When there was no axial flow, the magnitude of the mean shear stress and standard deviations of cap and slug bubbles were comparable. However, once axial flow was present, the mean shear stress and standard deviations became different, with higher values for cap bubbles than slug bubbles at the same gas flow rate, which may explain the better performance of the cap bubbles at the same gas flow rates.

In the presence of a high axial liquid flow rate (0.16m/s), the small bubbles did not have a significant effect on the mean shear stress or the standard deviations of the shear stress within the range of bubbling frequencies tested. The mean shear stress was much higher with high axial flows due to the intrinsic high shear stress of the liquid flow itself. For cap and slug bubbles, enhancement occurred but only when the bubbling frequency was above 0.5Hz. The reduction in dP/dt corresponded to the rise in shear stress at 0.5Hz. However, the magnitude of the standard deviation was comparable to when there was little or no axial flow and remained more or less the same for all bubble types and frequencies (Figure 5.6). Under these conditions (high liquid flow) the performance of the fiber was dominated by the mean shear stress as opposed to the standard deviation of the shear stress.

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Figure 5.15 Effect of mean shear stress on rate of increase of TMP

Figure 5.16 Effect of standard deviations of the shear stress on the rate of increase of TMP for tight fibers

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Figures 5.15 and 5.16 show the effects of the mean shear and standard deviation on dP/dt . It is evident that the graph for the mean shear stress can be split into 2 parts with a discontinuity in plots. The part on the left contains data for no axial flow and $V_a = 0.016\text{m/s}$ while the parts on the right is when $V_a = 0.16\text{m/s}$. Thus the mean shear stress is not the sole factor that influences the performance of the fiber. Although there is a definite relationship between the mean shear stress and enhancement, it is possible to achieve good enhancement without the need for high mean shear stresses. However, when the relationship between dP/dt and standard deviation is examined (Figure 5.16), there is a distinct trend that an increase in the standard deviation decreases the rate at which the pressure increases. Hence, the standard deviation of the shear stress is also an important factor in determining the performance of the hollow fiber.

The results show that for a submerged hollow fiber in bubbly flow, both the mean and the standard deviation of the shear stress affect the performance of the fiber. When the mean shear stresses are low, such as in cases when there is little or no axial flow, it is possible to induce high standard deviations to improve performance. In this case, the performance of the fiber is dominated by the standard deviation. As the mean shear stress increase, by increasing the axial flow or other methods, the standard deviation plays a less important role in reducing fouling, and the performance is now dominated by the mean shear stress. It is interesting to note that Cabassud and co-workers (Ducom et al., 2002a, Ducom et al., 2002b) came to similar conclusions for two phase flow applied to a flat sheet nanofiltration module. They were able to correlate flux enhancement to the relative magnitude of shear stress variations.

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5.4 Loose Fibers

5.4.1 Movement of Fibers

The position of the hollow fiber at its mid-point was monitored by observing the fluorescent particles fixed to the fiber. With the pictures captured at a frequency of 5 Hz, it became possible to determine the pattern of movement of the fiber. Each picture frame provided one data point for the position of the fiber which was measured from the edge of the picture. The average reading of each set of data was taken, and the distance the fiber was from this average position was taken as the displacement of the fiber. The velocity of the fiber movement was also determined from the displacement of the fiber versus time, and the acceleration of the fiber was in turn determined from the rate of change of velocities.

Figure 5.17 A sample of the velocity graph when small bubbles are used at 0.5 Hz – fiber was 1.0% loose

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Figure 5.17 shows the time variation of the velocity of a fiber that is 1.0% loose. In the experiment, small bubbles at 0.5 Hz (every 2 seconds) were introduced into the module. Inspection of the velocity variations shows that about every 2 seconds, there was a significant movement in the position of the fiber in the negative direction (towards the left hand side of the picture). At a frequency of 0.5Hz, the bubble caused the fiber to “curl” as it passed. From the pictures, it was observed that the sharp movement in the fiber coincided with the passage of the bubble at that point. With this movement, similar spikes in the velocity and the acceleration can be observed. This pattern was repeated for all the PIV measurements of bubbling at 0.5Hz. When the bubbles were introduced at a frequency of 2.0Hz, this phenomenon was not obvious. There could be several reasons for this. The first is that the frequency of sampling was 5Hz, and the expected phenomenon had an expected frequency of 2Hz. The frequency of sampling may have been insufficient to capture the patterns of movement (similar to Section 4.4.3.2) The second is that at high frequencies, the fiber did not return to its original position before other bubbles passed so successive bubble effects overlapped.

The movement of the fiber as the bubbles passed was always in one direction. For the case above, the movement was in the negative x-direction (to the left in the picture). When aeration was introduced at a frequency of 0.5 Hz, at any bubble size or fiber looseness, the fiber always displaced in the same direction. Thus, bubbling at low frequencies caused the fiber to move in more or less the same way. This was generally in the opposite direction to the of air injection.

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Figure 5.18 Variation of standard deviation of fiber position versus air flow rate at various looseness

Figure 5.18 shows the standard deviation of the displacement of the fiber with relation to the air flow rates for looseness of 0.5 and 1.0%. It is an indication of how much the fiber moved under different conditions of bubbling. When small bubbles are used, the degree of movement of the fibers was the lowest compared to the medium and large bubbles. Increasing the bubbling rate for the small bubbles did not increase the degree of movement of the fiber significantly. By increasing the looseness of the fiber, the degree of movement of the fiber increased significantly for small bubbles. When medium or large bubbles were used, the movements of the fiber were greater compared to when small bubbles were used. However, increasing the air flow rate only increased the standard deviation slightly, while increasing the looseness caused a more significant increase. Increasing the air flow would probably not change the absolute amount the fiber moved significantly, but increasing the looseness would allow the fiber a larger range of motion. A similar effect was recently reported by Wicaksana et al. (2006).

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Figure 5.19 Variation of fiber velocity versus air flow rate at various looseness

The variation of the velocity of the fiber is examined in Figure 5.19. For small bubbles, the velocity of the fiber did not change significantly as either the looseness or the air flow rate increased. Thus, although the range of motion increased as the bubbling rate increased, the velocity at which the fibers moved did not increase proportionally. In other words, small bubbles caused the fibers to move at more or less constant rates, irrespective of looseness or rate of bubbling. Small bubbles were probably too small to induce a significant velocity in the fiber. For the larger bubbles, the velocity of movement was higher for looser fibers at lower gas flow rates.

When a low frequency of bubbling was applied, one can imagine the fiber to be moving from side to side. If the bubbles were sufficiently spaced apart (in time), a fiber could move to one side before returning to more or less its original position. Thus, one of the limiting factors in this case would be the degree to which the fiber could move. This would be directly affected by the looseness of the fiber. Hence, at low bubbling frequencies, increasing the looseness would increase the velocity of the fiber. However,

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as the bubbling frequency increases, the effect on movement would depend less on the looseness of the fiber, as the effects of the continuous bubbling would cause the fiber to “vibrate”, rather than move to an extremity and then return.

Figure 5.20 Variation of acceleration of fiber position versus air flow rate at various looseness

When the variation of the acceleration is examined in Figure 5.20, it is observed that for the looseness of 0.5%, the acceleration increased more or less linearly if small bubbles were used. When the looseness was increased to 1.0%, there was a sudden increase in the acceleration as the air flow rate increased. Increasing the looseness for the medium sized bubbles also caused the acceleration to increase at an increasing rate. The acceleration for the large bubbles also increased with the looseness. The relationship between acceleration and dP/dt is discussed below.

Acceleration is measured as a change in the velocity of the fiber. When bubbling at low frequencies was applied, the velocity of the fiber does not fluctuate very much, as the effect of the bubbles is a local effect. However, increasing the bubbling frequency would

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increase turbulence, and the fibers would “vibrate” as opposed to “sway”. This would increase the fiber acceleration.

5.4.2 Effect of Looseness on the Performance of the Fiber

Figure 5.21 Effect of bubbling frequency on rate of increase of TMP when medium bubbles are used

Figure 5.21 shows the effect of bubbling frequency on dP/dt for medium sized bubbles. It is evident that the best performance was obtained with 1.0% looseness. Generally similar trends were obtained for the small and the large bubbles.

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Figure 5.22 Effect of fiber looseness on rate of increase of TMP for 3 bubble sizes

The effect of looseness on dP/dt can be seen in Figure 5.22 for the 3 bubble sizes at the maximum frequency tested for each size. Increasing the looseness from 0% to 1.0% improves the performance of the fiber, but increasing the looseness further from 1.0% to 2.0% causes dP/dt values to increase. This suggests that there is an optimal level of looseness (1.0% in this study). The existence of optimal fiber looseness is not really surprising. At the limit of a tight fiber, the effect of bubbling is dampened, and for a very loose fiber, the displacement may move the fiber away from the influence of bubbles.

Figure 5.23 Effect of gas flow rate on rate of increase of TMP when the fibers at a looseness of 1.0%

Figure 5.23 shows how the dP/dt values (for a fiber looseness of 1.0%) changed by varying the air flow rates. The trends for the other looseness tested were similar. This figure shows that it was possible to achieve the lowest dP/dt at a low air flow rates using smaller bubbles. In these cases, the effect of looseness magnified the improvement of dP/dt using small bubbles. In some cases, it was possible to achieve the same dP/dt using 100 times less air using smaller bubbles. This was similar to the findings in our previous chapter for tight fibers.

5.4.3 Shear Stress Experienced on Loose Fibers

At the outset it should be noted that the values of the shear stress are only indicative values. While it was possible to determine the position of the fiber accurately from almost every frame captured by the camera, only frames in which the fiber was apparently in line with the laser were used to estimate shear stress. However, it was not possible to determine if the fiber was exactly in line with the laser. From simulations by Sridharan

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(2006), a small deviation in the laser position could result in significant inaccuracies in the measurement of shear stress.

Figure 5.24 Variation of mean shear stress with air flow rates for small, medium and large bubbles at various looseness

The average shear stresses measured on the surface of the fibers at different looseness are shown in Figure 5.24. In all cases, increasing the looseness of the fiber initially increased the average shear stress, but as the air flow rate increased, the differences between the average shear stress decreased. The behavior of the standard deviations of the shear stress was similar to the average shear stress (Figure 5.25).

Figure 5.25 Variation of standard deviation shear stress with air flow rates for small, medium and large bubbles at various looseness

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5.4.4 Relationship between the motion of the fiber, shear stress and the rate of increase of TMP

Figure 5.26 Variation of standard deviation of the shear stress with rate of increase of TMP for the range of bubble sizes and fiber looseness

For the tight fibers it was established (Figure 5.16) that the standard deviation of the shear stress (τ_{sd}) was a major factor influencing dP/dt . For the loose fibers the relationship is less clear cut, as seen in Figure 5.26 although by analogy with the tight fibers a decrease in dP/dt with standard deviation would be expected. The scatter in the data may have been due to the difficulties measuring shear stresses for the loose fibers, as explained above.

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Figure 5.27 Variation of the rate of increase of TMP with fiber acceleration for the range of bubble sizes and fiber looseness

However, performance should also be related to a characteristic of induced fiber movement. The best relationship was obtained by plotting dP/dt versus fiber acceleration (Figure 5.27); whereas “fiber velocity” showed no correlation with dP/dt . When the acceleration was low, dP/dt did not improve, however, as the acceleration increased, there was an obvious trend of improvement of dP/dt . A parallel can be drawn with the tight fibers in this case. When the accelerations are low, the performance of the fiber would be controlled by the standard deviation of shear stress. When the accelerations are higher, the performance of the fiber would be more controlled by the acceleration. Hence, from the trends observed, it is the bubble-induced instabilities, in the shear stress or the fiber movement that most affect the performance of the fiber as opposed to the absolute values.

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5.5 Conclusions

Particle Image Velocimetry can provide data on the shear stress, and characteristics of fiber movement of a submerged hollow fiber membrane in an idealized chamber. This can provide quantitative data relevant to filtration performance.

For tight fibers, it was possible to obtain similar dP/dt values by using an air flow rate that was 10 times lower by using smaller bubbles rather than big bubbles at higher air flow rates. Increasing the liquid axial flow velocity meant that smaller bubbles did not improve the performance of the fiber significantly. This was due to the fact that the performance of tight fibers was related to two parameters, the mean and standard deviation of the shear stress. When axial velocities were low, the mean shear stresses were low. Hence, the performance of the fiber was dominated by the standard deviation of the shear stress. At the same air flow rates, small bubbles were more efficient at inducing flow that produced larger standard deviations. Increasing the liquid axial flow velocity increases the mean shear stress on the fiber due to high intrinsic velocity applied but dampened the importance of the standard deviation in improving performance.

For loose fibers, increasing the looseness from 0% to 1.0% improved dP/dt for all the sizes of bubbles tested. Increasing the looseness from 1.0% to 2.0% resulted in higher dP/dt values for medium and large bubbles, and generally lowers dP/dt for the small bubbles. This suggests an optimum value of fiber looseness.

As with the case for tight fibers, it was also possible to use a much lower air flow rate to maintain the same dP/dt by using smaller bubbles. The performance of the loose fiber was strongly related to the bubble-induced acceleration of the fiber as well as the standard deviation of the shear stress.

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**CHAPTER SIX NON-INVASIVE OBSERVATION OF LUMEN PHENOMENA
USING XRAY MICROIMAGING****6.1 Introduction**

Non-invasive observation of membrane filtration processes can provide important physical insights that could lead to improved performance and efficiency. For microporous membranes, such as microfilters, the features of interest for observation are the bulk fluid flow phenomena, deposition on the surface and deposition within the membrane structure.

Phase contrast X-ray microimaging(XMI), which has a resolution of 1 μm , differs from previously reported computer-aided X-ray tomography which has a resolution of the order 500 μm . In this chapter, the use of phase-contrast X-ray imaging with synchrotron radiation for *in situ* observation of microporous membrane phenomena at 1 μm spatial and approximately 33 ms temporal resolution per frame is demonstrated (with possible improvements in both space and time domains). We also demonstrate that XMI can be used for tomography for the study of the distribution of fouling within a hollow fiber and its pores, to provide greater structural information.

6.2 Experimental Protocols

Details of the experimental facilities and protocols used are given in Chapter 3.

6.3 Comparison between XMI and SEM

The results presented are a series of images obtained under different conditions to illustrate the potential of the technique.

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Figure 6.1 Images of dry membrane fiber in transverse and longitudinal section. Correspondence between (a) and (c) the secondary electron images (SEI), and (b) and (d) the X-ray microimages (XMI) is evident.

In figure 6.1, images of a dry fiber made by scanning electron microscopy (SEM) (a,c) and by X-ray microimaging (XMI) (b,d) are compared. XMI show contrast wherever there is a distinct change of refractive index. Sharp dark lines which delineate the fiber-lumen interface and an interface between two wall layers are caused by refraction. While the correspondence of the images is obvious they convey quite different information. For the SEM pictures, a dry fiber was sectioned transversally (a) and longitudinally (c). Due to the short depth of focus of the SEM, the pictures show the surface of the cuts in topographic contrast.

For XMI, the fiber was not sectioned and pictures b and d show the respective views as a and c, but along and across the whole fiber in – mostly – refractive contrast. Note that the XMI pictures represent the integrated effect of refraction along the full path of the X-rays through the fiber. While picture d shows the wall and lumen structure of the fiber quite comparably to picture c, the apparent deformation of the circular cross section in picture b is caused by the picture being taken along a somewhat curved piece of fiber. The black lines delineating the inner wall from the lumen and separating two wall layers are due to refraction. Both methods, XMI and SEM, clearly show the porous structure of the walls which is the basis for the filtration capability.

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6.4 Operation from Lumen to Shell Mode

One of the unique applications of this technique is that it gives optical access to the lumen of the shell while filtration is taking place. This is done in a non-invasive manner. In this section, we demonstrate the ability of this technique to provide unique insights into the filtration behavior of the membrane.

Figure 6.2 XMI of a membrane fiber during progressive deposition of ferric hydroxide on the lumen wall after (a) 0 min (b) 5 min and (c) 30 min of operation.

When the fiber is wetted (Figure 6.2 a-c), air in the pores is replaced by water, thus reducing the difference in refractive index between the PAN material and the pores by about one half and so diminishing the refractive contrast. The sharp refraction lines marking the inner wall surface and the interface between the two wall layers gradually

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disappear. From pictures a through c, a gradual increase in absorptive contrast due to deposition of iron hydroxide is visible. After 5 minutes filtration (Figure 6.2 b), the line that delineates the lumen and the inner wall has become more distinct again, but broader as well, because it is now due to the existence of an initial cake layer on the inner surface of the membrane. After 30 minutes filtration (Figure 6.2 c), a distinct layer of cake has accumulated. At this point, the membrane was allowed to dry and an XMI of the dry fiber collected (Figure 6.3).

Figure 6.3 XMI of the dried fiber after 30 min operation (compare with Figure 6.2(c)).

An SEM autopsy was also performed on the fouled membrane and is shown for comparison. The distribution of ferric hydroxide is clearly evident by XMI, but the SEM image provided less detail. The distribution of ferric hydroxide is clearly evident, with thicker regions having darker contrast. The lighter contrast are channels that developed for liquid transport. The SEM is superimposed (upper) but fails to show the same details as XMI. Moreover, as the cake layer had slightly impregnated the membrane, X-ray mapping in the SEM was also less successful than XMI in showing the distribution of deposition.

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Figure 6.4 XMI of the wet fibre in which large and small ferric hydroxide particles were used to cause fouling on the lumen surface and within the membrane pores respectively. (a) Initially fouling is confined to the inner surface and variation in deposition thickness is evident, but (b) after longer operation smaller particles have impregnated the wall pores.

XMI not only allows direct observation of cake build-up on membrane surfaces, but also allows imaging of the migration of fines deep into the substructure of the membrane. To illustrate this, a feed was used with a range of particle sizes from 0.1 - 2 microns allowing simultaneous fines penetration into the pores of the membrane as well as build-up of cake on the surface. Figure 6.4a shows a fiber that has been fouled with a feed solution containing this range of ferric hydroxide particle sizes. Although the fiber is wet, features of the substructure can be observed because ferric hydroxide particles have deposited within the pores making them visible with XMI. After some time, very serious fouling occurs and the surface and internal fouling can be seen as shown in Figure 6.4b. The extent of fouling was subsequently confirmed with SEM and microanalysis.

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6.5 Bubble Formation in the Lumen* (*this work was done in collaboration with Dr Sheng Chang, UNESCO Centre for Membrane Science and Technology, UNSW, Australia*)

Figure 6.5 XMI of two stationary air bubbles in the membrane lumen.

As XMI can identify two phase phenomena within the lumen, it is relevant to the increasingly important use of bubbling to control fouling in membrane processes. Figure 6.5 shows an air bubble in the liquid-filled lumen of the fibre. In this example the bubble was static.

Previous studies have shown that during the filtration of MilliQ water significant pressure fluctuations were observed. (Shang et.al., 2005). This was thought to be due to the formation of bubbles within the fiber lumen. The membranes tested were 0.22 μm polypropylene hollow fibers (USFilter) with an inner/outer diameter of 0.39/0.65 (mm) and the feed was MilliQ water. The fiber was completely submerged into a channel of 2mm thickness. Unlike the images captured in the previous section, the walls of the hollow fiber are not very obvious. In the previous section, feed was pumped through the

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lumen, while the outside of the fiber remained dry. The walls were not evident in the beginning, but as a fouling layer started to develop on the inner surface, the delineation between the wall and lumen became more evident. In this study, no fouling layer developed, hence, there the refractive contrast was diminished.

Figure 6.6 Fluctuations in TMP with MilliQ water as feed at different fluxes

Figure 6.6 shows the variation of TMP with time for different fluxes using MilliQ water as the feed. For the various fluxes tested, there were variations in TMP, which were similar to those described in other studies (Sheng et.al., 2005).

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Figure 6.7 Formation of bubbles within the lumen (a) 5 minutes (b) 6 minutes (c) 7 minutes (d) 8 minutes from the start of filtration

Figures 6.7 a, b, c and d show four consecutive images taken at time intervals of 60 seconds. In this experiment, the imposed flux was $25.9 \text{ L/m}^2/\text{hr}$, which corresponded to an averaged TMP of approximately 8.2 kPa . The image represents an area of $1 \times 0.75 \text{ mm}^2$ with the outline of the fiber wall identified by the solid white lines. The dark artifacts in the images are imperfections in the CdWO_4 scintillator. The observation identified two different types of bubble. Some bubbles were stagnant bubbles and are labeled (A) and the others were mobile bubbles and are labeled (B).

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6.5.1 Characteristics of Stagnant Bubbles

Figure 6.8 Stagnant Lumen of a hollow fiber membrane at a flux of 12.4 L/m²/hr at (a) 3 minutes (b) 4 minutes (c) 9 minutes (d) 27 minutes from the start of filtration

The stagnant bubbles were observed to attach to the internal surface of the membrane with a flat bottom completely merged with the outline of the fiber wall. In the range of TMP from about 4.5kPa to 16.1kPa, the stagnant bubble was always attached at a fixed location and no movement or detachment was observed under any conditions. These observations corresponded to the section closest to the suction end.

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At an imposed flux of $12.4 \text{ L/m}^2/\text{hr}$, which corresponded to an averaged TMP of about 4.5 kPa, the stagnant bubble was observed to be very long (approx 50mm). As shown in Figure 6.8, the shape of the bubble changed slowly. The bubble remained the same shape from the 3rd to the 8th minute after filtration started, then it appeared as if the bubble was “stretched” from its original shape during the period of 9 minutes to 27 minutes, and then returned to its original shape after that. This meant that either some part of the bubble broke off from the main bubble and traveled up the lumen or that the bubble contracted back to its original shape. Regardless, the change was very slow at the low TMP.

Increasing the TMP to 8.2kPa at an imposed flux of $25.9 \text{ L/m}^2/\text{hr}$, caused the bubble to change in shape more rapidly. Within 30 minutes, the bubble no longer oscillated, and became a stable bubble attached to the surface of the fiber. Figure 6.9 shows the transition of the bubble from the long unstable to the stable shape in six consecutive minutes at a TMP of 8.2kPa. This large bubble appeared to block most of the cross section of the fiber lumen, and stopped other bubbles from passing that point. After the stable shape was reached, there was no further change in the bubble shape until the imposed flux was increased to $48.8 \text{ L/m}^2/\text{hr}$.

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Figure 6.9 Transition of bubble to a stable shape at (a) 25 minutes (b) 26 minutes (c) 27 minutes (d) 28 minutes (e) 29 minutes (f) 30 minutes from the start of filtration

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Figure 6.10 Stable and Moving bubbles at a flux of $48.8 \text{ L/m}^2/\text{hr}$ at (a) 65 minutes (b) 67 minutes (c) 69 minutes (d) 71 minutes (e) 73 minutes from the start of filtration

Figure 6.10 are five consecutive images which were taken at two minute intervals and at an imposed flux $48.8 \text{ L/m}^2/\text{hr}$ (TMP of 16.1 kPa). In this figure we see a bubble approaching the stagnant bubble. The bubble moves past the stagnant bubble without merging with the bubble and appears to break up into smaller bubbles. The images captured by the XMI techniques represent a depth averaged image of the fiber and its

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lumen. Thus, although the bubbles appear to be overlapping, they are actually on different planes, with one bubble behind the other.

At the end of the experiment, the entire fiber length was observed using XMI to determine the distribution of bubbles along the fiber lumen. It was found that most of the bubbles in the lumen could be found in the region from the suction end to 5cm away from it.

6.5.2 Characteristics of mobile bubbles

The series of images in Figure 6.7 reflect the formation, growth, and detachment of small mobile bubbles at an imposed flux $25.9 \text{ L/m}^2 \text{ hr}$ with an averaged TMP of about 8.2 kPa. The figure consists of 4 consecutive images observed over 4 minutes. Small mobile bubbles with diameters about 20-50 μm can be seen in Figure 6.7(a). These bubbles formed at six points. Figure 6.7(b) shows that three of the bubbles had disappeared while the other 3 bubbles had started to grow in size. In Figure 6.7(c), part of a larger bubble can be seen moving up the lumen at the bottom of the image. The large bubble from downstream of the lumen broke apart as it approached the narrow channel caused by the stagnant bubble. Some of the small bubbles appeared to have been detached by the passing of the large bubble. However, the stagnant bubble remained where it was the entire time as shown in Figure 6.7(d).

The formation, growth and movement of the mobile bubbles were found to be dependent on the imposed TMP. At low TMP (4.3kPa), there was no mobile bubble formation in the observed region. However, increasing the TMP to 8.2kPa, the formation of mobile bubbles was found in the locations indicated in Figure 6.7. One of the important observations related to the mobile bubbles was that they were always observed to form at fixed locations. This suggests that the formation of the bubbles may be related to the special physicochemical or morphological features of the membrane materials at these points.

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Figure 6.11 Growth of Bubbles in Fiber Lumen at (a) 61 second (b) 80 seconds (c) 106 seconds (d) 110 seconds (e) 120 seconds after the start of filtration

The observations showed that the time scale for growth of the mobile bubbles could vary from several seconds to minutes, depending on the suction pressure. Figure 6.11 shows the images of bubbles taken at 61, 80, 106, 110, and 120 seconds at a TMP of 16.1 kPa. In the period from 61 to 106 seconds, small bubbles were attached to the fixed points within the fiber lumen that gradually increased in size under the action of the suction pressure. After 110 seconds, the three large bubbles detached from the wall and disappeared. At

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120 seconds, six new small bubbles were observed to start growing at the same locations the previous bubbles vacated.

Permeate flow would not be able to penetrate the regions where the bubbles are attached. It is possible that the stagnant bubble could be caused by regions of incomplete wetting, leaving some hydrophobic points at some local regions in the fiber lumen. Where such a hydrophobic point exists, bubbles will tend to form at that point. This leads to local obstructions in the fiber lumen as the liquid permeate flow will have to squeeze through the narrowed flow channel. This increases the local flow resistance and would result in an increased TMP in different sections of the fiber observed in previous studies (Sheng et.al., 2005). Bubble growth will cause TMP increases due to obstruction of the lumen, while detachment of the bubbles would produce relief in the TMP. As a result, the existence and change in bubble characteristics under suction conditions will cause an unstable flow and induce fluctuations in suction pressure.

6.5.3 Removal of Bubbles

In order to test the theory that incomplete wetting causes bubbles, which in turn cause TMP fluctuations, the fiber was completely wetted. This was done by pumping 50% ethanol solution into the suction end for 10 hours. This was repeated by pumping in the ethanol solution into the dead end for 10 hours. It was found that after undergoing this procedure, there were no longer any bubbles observable within the fiber lumen, and the TMP did not fluctuate significantly.

Figure 6.12 shows the behavior of the TMP after alcohol treatment with marked decreases in fluctuations in the TMP.

Figure 6.12 Fluctuations of TMP after treatment with alcohol

6.6 Conclusions

Phase contrast X-ray microimaging (XMI) with synchrotron radiation has been shown to be a powerful tool for non-invasive observation of phenomena occurring in membrane filtration processes. The technique is able to observe the deposition of particles as a cake inside the lumen of a hollow fibre membrane and also detect deposition and fouling within the membrane structure. The resolution of the observation technique is of the order of 1 μm which is superior to other available non-invasive techniques. In addition to non-invasive observation of the membrane in operation, XMI may be useful for membrane characterization, providing details of membrane morphology and skin layer thickness. It can also be used for autopsy of used membranes.

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It has been demonstrated that due to incomplete wetting, combined with certain surface features, stagnant and mobile bubbles may exist in the lumen of polypropylene hollow fiber membranes. The stagnant bubbles, whose shape changed with increases in suction pressure finally reached a stable geometry at high imposed TMPs. These bubbles were observed to be attached to certain local regions of the fiber lumen, while the small mobile bubbles were active, and their formation, growth, and detachment from the wall of the bubbles was observed. The behavior of the TMP suggests that the existence of bubbles in the lumen may have caused considerable fluctuations in the suction pressure during filtration. In addition, this study also confirmed the X-ray micro-imaging is a powerful tool for non-invasive observation of two-phase flow phenomenon inside a hollow fiber lumen.

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CHAPTER SEVEN CONCLUSIONS AND RECOMMENDATIONS**7.1 Conclusions**

Water is a critical issue facing the world today, and membrane technology is becoming increasingly important for water and wastewater treatment. In this thesis, the hydrodynamics of submerged hollow fiber modules and its effects on the filtration performance was studied. The influence of fiber looseness, packing density, and bubbling characteristics on the shear stresses and the movement of the fiber were determined.

Particle Image Velocimetry (PIV) was used extensively to determine the variation in the shear stresses on the fibers, as well as the flow distribution around a bundle of submerged hollow fibers. PIV was also used to characterize quantitatively the movement of a loose fiber under bubbling conditions. Particle Image Velocimetry can provide data on the shear stress, and characteristics of fiber movement of a submerged hollow fiber membrane. This can provide quantitative data relevant to performance.

In a model 3 x 3 array (9 fibers), it was found that the fiber in the middle of the bundle fouled the most severely. The fibers in the bundle did not foul evenly, and the standard deviation of the fluxes of each fiber increased as the experiment progressed. When the standard deviation of the fluxes and the required suction pressure were compared over time, there was a good correlation. As the standard deviation increased, the pressure required to maintain a constant flux increased as well. There is evidence that the increase in standard deviation of fluxes precedes the increase in suction pressure, pointing to a possible early warning of fouling. From these observations, a new term in Darcy's Law can be added specifically for hollow fiber bundles. The hydrodynamic resistance is the increase in resistance experienced when the fibers are packed tightly into a bundle as opposed to being by itself. The hydrodynamic resistance has two components, the bundle resistance and permeate competition. The presence of dead zones within a hollow fiber bundle can significantly increase the rate of fouling. The use of smaller sized bubbles was found to be favorable in this thesis. Up to 10 times less air flow could be used to maintain

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the same rate of fouling. This benefit may be due to the fact that they provide more numerous “events” that compensates for the modest impact for each bubble event.

For single fibers, it was also possible to obtain similar performances by using an air flow rate that was 10 times lower by using smaller bubbles at higher frequency. The performance of tight fibers was shown to be related to two parameters, the mean and standard deviation of the shear stress. The effect of bubbles was influenced by the presence or absence of a background liquid axial flow velocity. When axial velocities were low, the mean shear stresses were low. Hence, the performance of the fiber was dominated by the standard deviation of the shear stress. Small bubbles were more efficient at inducing flow that produced large standard deviations. Increasing the axial flow velocity increased the mean shear stress on the fiber due to the high intrinsic velocity applied. The importance of the standard deviation in improving performance in this case was now muted.

For loose fibers, increasing the looseness from 0% to 1.0% improved the performance of the fibers. However, increasing the looseness beyond 1.0% does not guarantee better performance. This suggest an optimum level of fiber looseness for a given bubble size. The performance of the loose fiber was strongly related to the acceleration of the fiber. It was important that the fiber be allowed to move freely in the module, and the design of aerators be modified to ensure that this happens.

A novel method, X-ray MicroImaging(XMI) based on the concepts of Phase Contrast Imaging (PCI) was used in this thesis. It has been shown to be a powerful tool for non-invasive observation of phenomena occurring in membrane filtration processes. The technique was able to observe the deposition of particles as a cake inside the lumen of a hollow fiber membrane and also detect deposition and fouling within the membrane structure. The resolution of the observation technique is of the order of 1 μm which is superior to other available non-invasive techniques.

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Using XMI techniques, it was also demonstrated that due to incomplete wetting, combined with certain surface features, stagnant and mobile bubbles may exist in the lumen during filtration from shell to lumen in the lumen. The behavior of the suction suggested that the existence of bubbles in the lumen may have caused considerable fluctuations in the suction pressure during filtration.

As a general observation, the increasingly popular submerged hollow fiber module is sensitive to prevailing hydrodynamics induced by bubbling. Further optimization of the concept seems possible based on a fundamental understanding of the complex interactions between two phase flow and imposed shear stress and fiber motion.

7.2 Recommendations

From this thesis, it has been shown that it should be possible to optimize the bubble size, bubble frequency, fiber looseness and the aerator design to provide the highest standard deviations of the shear stress and the acceleration of the fiber. The challenge now is to integrate what has been reported in this thesis into real world systems. Large bundles of fibers should be studied using PIV techniques developed in this thesis. PIV can also be used to study other membrane phenomena. Appendix B shows another way PIV can be used to understand fundamental membrane processes.

The study of differential fouling of fibers within the same fiber bundle can also be extended to large bundles. Of particular interest would be the possibility of developing a technique that can predict the onset of severe fouling by observing the differential rates of fouling of the fibers. In addition, novel bundle arrangements could be developed that improve homogeneity of performance, thereby increasing productivity.

XMI can be used to directly study the deposition of foulants within the membrane, and in the lumen. This should provide insights not available with any other methods. Extensions of the equipment used in this thesis could allow observation of fouling in real time.

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APPENDIX A

APPENDIX A MEMBRANES FOR HUMANITY

(This Appendix is a brief report of a project initiated and carried out by the author in response to the Tsunami on the 26th of December 2004)

Introduction

One of the main requirements of human survival is a clean and safe supply of water. The World Health Organisation estimates that 15 million children under the age of 5 die each year due to water born diseases. Most of these fatalities are due to one of six main diseases associated with water supply and sanitation. (Diarrhea, *Ascaris*, *Dracunculosis*, Hookworm and Trachoma). These diseases are caused by biological contamination of water.

The Tsunami which struck on the 26th of December 2004 devastated the west coast of Sumatra. Thousands were killed, and many more were left homeless. Overnight, many children became orphans.

Presently, people in the town of Meulaboh and the surrounding area are purchasing water from vendors, or boiling water from wells. Boiling remains the most commonly used technology to obtain water free of biological contaminants in many developing countries. With an average energy efficiency of 12% for most stoves, a family of 5 requiring approximately 35 litres of water a day, a total of 12kg of wood fuel is required per day, several times the daily requirement of wood for cooking. This requires that a large amount of money is spent on fuel (1% of Jakarta's GDP is spent by the residents of the city on boiling their drinking water). In places that cannot afford it, it becomes necessary to send people out to collect wood fuel. Gathering wood fuel for daily cooking is already a heavy burden on hundreds of millions of women and girls in the developing world. Moreover, greenhouse emission, deforestation and many other effects make boiling an economically unrealistic and unsustainable method to disinfect drinking water.

Membranes for Humanity

Membranes have been an integral part of water treatment processes for decades. Ultrafiltration is often used as a pretreatment for reverse osmosis processes for the removal of micro-organisms. Hollow fibers are one of the most common configurations of ultrafiltration membranes. The use of hollow fibers allows a large surface area of membrane to be packed into a small volume, allowing for the treatment of a large amount of water with a very small plant.

Advantages of membrane technology include

1. Water produced is CLEAN and SAFE (free of turbidity)

Water produced from membrane micro-filtration is clear, and has 99% removal rate of bacteria. Biological contamination of water is responsible for most of the fatalities due to unclean water.

APPENDIX A

2. Water produced is CHEAPER than purchasing from vendors

Water produced via the proposed system will be drinkable from the source (no boiling required) and will be available up to much less than purchasing from vendors.

3. Water production DOES NOT DEPEND ON ELECTRICITY

In emergency situations or in remote areas, electricity may or may not be available, hence, a hand pump is used to drive the water through the filter. This provides reliability in the most adverse situation.

4. Large volumes of water can be produced with a SMALL TRANSPORT VOLUME

The typical response to an emergency would be to fly in vast quantities of water into the affected areas. A membrane unit can produce up to 25 to 30 times its own volume of water in a day.

A Hollow Fiber Filtration System

In the aftermath of the Tsunami, 9 water treatment units were donated by Jurong International and the Nanyang Technological University. A PhD. Student, Adrian Yeo, went to Meulaboh with a Red Cross Medical Team, and installed the units. After receiving feedback from the users, the design has changed to make the units more user friendly and robust.

In October 2005, 10 more of the upgraded units donated by the World Federation of Diamond Bourses were installed in Meulaboh. Mercy Relief Singapore has also committed to a purchase of 10 of these units. With the exception of the units purchased by Mercy Relief Singapore, the units were placed at locations identified by a local NGO, KKI.

APPENDIX A

Technical Specifications

Membrane Type :	Polypropylene Hollow Fibers
Fibre Dimensions:	
Outside diameter	310 ± 15 µm
Wall Thickness .	33 ± 3 µm
Dimensions of slot pores (EM)	0.1 x 0.7 µm
Parameters:	
Porosity .	50 ± 4 %
Air Permeability.	90 ± 15 l/m ² s b
Water Permeability	300 l/m ² h bar
Collapse Pressure .	0.2 MPa
Burst Pressure	0.3 MPa
Ultimate Tensile Strength per one fibre	2 N
Flow Rate per module	200-300L/hr
Micro-organism removal	
Cryptosporidium	99.9%
Fecal Coliforms	99.9%
Total Coliforms	99.9%