FLUX PREDICTION IN HIGH-SHEAR MICROfiltration

by

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A thesis submitted in conformity with the requirements for the Degree of Doctor of Philosophy, Graduate Department of Chemical Engineering and Applied Chemistry

University of Toronto

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0-612-59095-X
ABSTRACT

Flux Prediction in High-Shear Microfiltration
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The use of membranes to treat effluents from the pulp and paper industry has been evaluated. Four applications related to the treatment and recycling of effluents were tested, and two were selected as having good industrial applicability: 1) microfiltration of old newsprint de-inking effluent containing flexographic ink; 2) treatment of an effluent from a mill using recycled old corrugated containers using a membrane bio-reactor.

The membrane system adopted in this research has a unique configuration. It consists of a bundle of hollow-fiber microfiltration membranes submerged in a tank into which the effluent is introduced. Vacuum is used inside the fiber lumen to create transmembrane pressure differential, and aeration to promote high-shear stress at the membrane surface to minimize concentration polarization and fouling. Periodic reverse filtration (backflushing) is also used.

A semi-empirical model to predict the permeate flux for this membrane configuration has been developed. The model was based on Darcy's law combined with a particle mass balance equation, and assumes that non-diffusive transport phenomena are the main mechanisms for the back-transport of particles from the membrane surface to the bulk solution during filtration. The model incorporates the use of reverse filtration.

The model has been validated by laboratory experiments using six different suspensions: flexographic ink ($\Phi_b = 0.005$ and 0.01); calcined alumina ($\Phi_b = 0.02$ and 0.03); and yeast ($\Phi_b = 0.01$ and 0.02), where $\Phi_b$ is the solid volume fraction in the suspension. The results showed good agreement between model prediction and experimental observations.
ACKNOWLEDGMENTS

I would like to thank everyone who contributed to the completion of this work.

It was a pleasure and a privilege to have Professor Douglas Reeve as my supervisor. His energy, intelligence and generosity provide an outstanding milieu for the people in his research group. It was a continuous great learning experience having his supervision.

I would like to express my sincere gratitude to my co-supervisor Professor Kim Woodhouse for her guidance and advice during this project. The valuable discussions and technical support she provided were crucial for the completion of this work.

Many thanks to Professors David Goring, David Kuhn, Grant Allen, Elizabeth Edwards and Dr. Michael Paleologou for their important advice and suggestions. I also would like to express my appreciation to Chris Smith, Hadi Husain, Hamid Rabie and Henry Behmann for their contributions for this work.

I am indebted to Esa Vilen, Wenshan Zhuang, He Yang, Jarmo Heinonen, Debby Repka, Luiz Castro, Jeff Beange, Paul Jowlabar, Rob Singh, Charles Mitchell and Dimitri Rubisov for their precious contribution for this work. I am also grateful to Atlantic Newsprint, Domtar Packaging, Millar Western Pulp, Repap, Flint Ink and Alcoa Alumina & Chemicals for providing samples. My special thanks to Cindy Tam and all my friends from the Pulp & Paper Centre. What a great working place! I am also grateful to Jacque, Gorette, Leticia, Sheila, Esther, Julie and friends from the Department of Chemical Engineering and Applied Chemistry.

The financial and technical support from CNPq - Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brazil, Zenon Environmental Inc., and Technology Ontario is very much appreciated.

Por fim, gostaria de prestar uma singela homenagem dedicando este trabalho à Ana Augusta, ao Juliano, à D. Orphila e à memória do meu grande amigo Helio Rezende. Obrigado.
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<td>$L$</td>
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\( Pe = \text{Peclet number} \)

\( r = \text{pore radius (L)} \)

\( r_p = \text{particle radius (L)} \)

\( R_{\text{e}}(n) = \text{recovery coefficient} \)

\( R_c = \text{cake resistance (1/L)} \)

\( \dot{R}_c = \text{specific cake resistance (1/L}^2) \)

\( R_c' = \text{specific cake resistance on mass basis} \)

\( Re = \text{Reynolds number} \)

\( R_f = \text{fouling resistance (1/L)} \)

\( R_m = \text{membrane resistance (1/L)} \)

\( s = \text{cake compressibility} \)

\( S = \text{internal surface area (L}^2) \)

\( Sc = \text{Schmidt number} \)

\( S_c = \text{internal surface area of cake (L}^2) \)

\( Sc_m = \text{Schmidt number at membrane surface} \)

\( Sh = \text{Sherwood number} \)

\( T = \text{absolute temperature, } ^\circ \text{K} \)

\( t = \text{time (t)} \)

\( t_f = \text{forward filtration time (t)} \)

\( t_b = \text{reverse filtration time (t)} \)

\( t_c = \text{time constant for cake growth} \)

\( U = \text{cross-flow velocity (L/t)} \)

\( U_{\text{sg}} = \text{superficial gas velocity (L/t)} \)

\( \frac{dU}{dy} = \text{velocity gradient} \)

\( V = \text{permeate volume (L}^3) \)

\( y = \text{distance from the wall (L)} \)

\[ \int_0^{t_f} k dt = \text{filtrate volume per unit area collected during forward filtration} \]
Greek Letters

\[ \beta = \frac{\Delta P_b}{\Delta P_f} = \frac{\text{reverse filtration transmembrane pressure}}{\text{forward filtration transmembrane pressure}} \]

\( \beta n \Delta b \) = filtrate volume per unit area lost during reverse filtration

\( \nu_{L,0} \) = inertial migration velocity (L/t)

\( \Delta P \) = transmembrane pressure (M/Lt²)

\( \Delta P_b \) = reverse filtration transmembrane pressure, (M/Lt²)

\( \Delta P_f \) = forward filtration transmembrane pressure differential, (M/Lt²)

\( \delta \) = boundary layer thickness (L)

\( \delta_c \) = cake layer thickness (L)

\( \eta \) = dynamic viscosity (M/Lt)

\( \tau \) = pore tortuosity

\( \gamma \) = shear rate in the boundary layer (t⁻¹)

\( \varepsilon \) = surface porosity

\( \rho \) = fluid density (M/L³)

\( \rho_s \) = mass density of solids comprising the cake (M/L³)

\( \mu \) = kinematic viscosity (L²/t)

\( \Phi_b \) = solid volume fraction of the suspension

\( \Phi_c \) = solid volume fraction in the cake

\( \varepsilon_c \) = void fraction of the cake

\( \nu_L \) = lift velocity of particles, (L/t)

\( \alpha_0 \) = constant related to size and shape of particles

\( \eta_f \) = pure fluid viscosity (M/Lt)

\( \tau_r \) = relative shear stress (M/Lt²)

\( \tau_w \) = shear stress at membrane wall (M/Lt²)

\( \tau_{wI} \) = shear stress at membrane wall at initial feed concentration (M/Lt²)

\( \Delta \chi \) = membrane thickness (L)
ABBREVIATIONS

AOX – Adsorbable organic halogen
BCTMP - Bleached-chemi-thermo-mechanical pulping
BKPM – Bleached kraft pulp mill
BME - Bipolar membrane electrodialysis
BOD - Biochemical oxygen demand
BPE - Bleach plant effluent
COD - Chemical oxygen demand
CTMP - Chemi-thermo-mechanical pulping
DO - Dissolved oxygen
ED – Electrodialysis
Eo - stage - Bleaching extraction stage
HC - High consistency
HRT - Hydraulic retention time
Kg/adt – kilograms per air dried ton of pulp
LC - Low consistency
MBR - Membrane bio-reactor
MF - Microfiltration
MLSS - Mixed Liquor Suspended Solids, mg/l
MWCO - Molecular weight cutoff
NF - Nanofiltration
NTU - Nephelometric turbidity unit
OCC - Old corrugated container
ONP - Old newsprint
OUR - Oxygen uptake rate
RO - Reverse osmosis
SCFM - Standard cubic feet per minute
T - Temperature
TDS - Total dissolved solids
TKN - Total Kjeldahl nitrogen
TMP – Thermo-mechanical pulping
TOC – Total organic carbon
TOX – Total organic halogen
TSS - Total suspended solids
UF - Ultrafiltration
VDS - Volatile dissolved solids
VRF - Volume reduction factor
VSS - Volatile suspended solids
Membranes are porous structures that can separate particles from a suspension or solute molecules from a solution. They have different physical and chemical properties, and transport through a membrane is driven by a pressure, concentration or temperature difference. Membrane processes are typically characterized by the pore size: microfiltration, pore size from 0.1 to 10 μm; ultrafiltration, pore size from 0.02 to 0.1 μm; nanofiltration, molecular weight cutoff (MWCO) from 500 to 20,000 Daltons; reverse osmosis MWCO less than 500 Daltons.

Membrane separation technology is not new. Ferry presents an interesting review of the early developments of membrane separations (Ferry, 1936). It was reported that, in 1855, Fick recorded dialysis experiments through artificial membranes. Ultrafiltration appears to have been first referred to by Schmidt who, in 1856, used an animal membrane to filter a solution of protein. In 1907, Bigelow and Gemberling prepared flat membranes from an ether-alcohol solution, and, in 1918, Zsigmondy and Bachmann patented a process to manufacture a graded series of membranes that became commercially available in Germany in 1927. A breakthrough in the use of membranes in industry was the development of asymmetric membranes by Loeb and Sourirajan in the early 1960’s (Cheryan, 1986; Mulder, 1991).

Membrane-based technology has some advantages over other separation processes. Energy consumption is generally low, the process is often easily integrated into existing equipment, membrane properties can be adjusted to different separation purposes, and normally no additives are required. Moreover, the development of new membranes with broader chemical and physical properties and their commercial availability has made this technology more attractive to industry.

However, there are still important challenges facing membrane technology. The performance of membrane processes can be measured by two parameters: selectivity, and permeation rate or flux. Fouling is the main limitation of membrane separation. It leads to severe flux decline, which has a significant negative influence on the process economics. In
order to minimize the adverse effects of fouling, many approaches have been pursued, such as membrane surface modification, improvement of the hydrodynamics of the system, and use of effective cleaning methods.

The pulp and paper industry is undergoing significant changes in its processes and environmental practices in order to meet stringent new regulations and respond to competitive markets. The industry is now looking at alternative separation technologies to reuse and conserve water. Therefore, membrane technology may have find important applications within the pulp and paper industry, particularly in the treatment of liquid effluents. While the use of membranes in the pulp and paper industry has potential, this technology is still not fully accepted by industry, principally because of technical concerns such as process efficiency, uncertainties in process integration, and for economic reasons. In addition, mill personnel are generally not familiar with membrane processes.

The characteristics of pulp and paper effluent makes the use of membranes in this industry a particular challenge. As mentioned previously, fouling is a critical parameter influencing the flux through a membrane. Pulp and paper process streams include many substances with high fouling potential, such as pulp fibers, high dissolved organic and inorganic concentration, hydrophobic substances and inks.

This thesis describes the development of a model originally designed for use in pulp and paper applications. The model predicts flux in high-shear microfiltration systems. In order to developed such a model, a preliminary investigation was undertaken to select a membrane system which would have the potential to manage the types of effluents originated in pulp and paper processes, and to select applications which could be used to evaluate the model.

The membrane system was chosen with the assistance of Zenon Environmental Inc. Zenon manufactures membranes and membrane systems, primarily for the treatment of domestic and industrial water and wastewater, and developed the membrane system adopted in this research. It consists of a submerged hollow-fibre microfiltration module. It is a unique configuration that consists of a bundle of hollow-fibre microfiltration membranes submerged in a tank into which the effluent is introduced. It uses a vacuum inside the fiber lumen to create the transmembrane pressure differential. Air is injected at the bottom of the
membrane fibres to promote high-shear stresses at the membrane surface to minimize concentration polarization and fouling, thus improving the filtration performance. The membranes have excellent chemical, thermal and mechanical stability. The use of periodic reverse filtration, i.e., backflushing, is commonly applied in industrial systems. These characteristics make the use of this particular system appropriate to the pulp and paper mill effluents.

The first part of this thesis is an evaluation of the potential use of membranes to treat effluents and facilitate recycling in different applications in the pulp and paper industry. The second part of the thesis is a development of a semi-empirical model to predict the permeate flux for this membrane configuration. Several models have been proposed in the literature for a variety of systems, based on distinct separation principles and sometimes on the combination of different theories, as described in Chapter 2 of this thesis. The present model was developed not only from previous models, but also from the observation of this particular system behaviour. The model was validated using laboratory experiments with synthetic suspensions.

The objectives of this work are:
1. to evaluate the use of microfiltration to treat effluents and facilitate recycling in different applications in the pulp and paper industry;
2. to formulate a semi-empirical model for flux prediction in high-shear systems;
3. to validate the model by laboratory experiments using synthetic suspensions.
2.1 Membrane Separation

A membrane is a selective barrier that separates components of a mixture. Membrane separation is based on the principle that a mixture can be partially fractionated by passing it through a porous structure, which tends to retain larger components while allowing smaller components to pass through (Gutman, 1987; Zydney and Colton, 1986).

Membranes can have different physical and chemical properties. Figure 2.1 shows a schematic flow sheet of the classification of membranes according to their nature and morphology. In this project more attention will be given to organic porous membranes.

![Figure 2.1 - Schematic flow sheet membrane classification](image)

Porous membranes contain pores that range from 0.1 to 10 μm for microfiltration and 2 to 100 nm for ultrafiltration. The selectivity of these membranes is determined mainly by the dimensions of the pores (Mulder, 1991). Typically, pressure-driven membrane processes are characterized according to pore size. The molecular weight cutoff (MWCO), reported in Daltons, indicates the size of the molecules that would likely be rejected by the membrane (Zydney and Colton, 1986; Gutman, 1987).
The dimensions of the pores, rather than the type of membrane material, determine the separation characteristics. The type of material is of crucial importance for chemical, thermal and mechanical stability, but not for flux and rejection (Mulder, 1991). Table 2.1 presents the filtration spectrum and the pressure range commonly used.

Table 2.1 - Filtration spectrum

<table>
<thead>
<tr>
<th>SPECTRUM</th>
<th>POR SIZE (µm)</th>
<th>MOLECULAR WEIGHT (Daltons)</th>
<th>PRESSURE (KPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microfiltration</td>
<td>0.1 - 10</td>
<td>-</td>
<td>70 - 350</td>
</tr>
<tr>
<td>Ultrafiltration</td>
<td>0.002 - 0.1</td>
<td>-</td>
<td>170 - 850</td>
</tr>
<tr>
<td>Nanofiltration</td>
<td>-</td>
<td>500 - 20,000</td>
<td>500 - 1500</td>
</tr>
<tr>
<td>Reverse Osmosis</td>
<td>-</td>
<td>&lt;500</td>
<td>3500 - 5000</td>
</tr>
</tbody>
</table>

The performance or efficiency of a given membrane is determined by two parameters: selectivity; and flux or permeation rate, which indicates the flow through the membrane.

Selectivity is generally expressed by the retention \((R)\), given by

\[
R = \frac{c_p - c_b}{c_p},
\]

where \(c_p\) is the solute concentration in the permeate, and \(c_b\) the solute concentration in the bulk suspension.

The flux or permeate rate \((J)\) is given by

\[
J = \frac{Volume}{Membrane \cdot area \times Time}.
\]

Membrane performance depends not only on the characteristics of the membrane but also on the temperature, pressure, velocity and concentration of fluid at the membrane surface (Gutman, 1987). The pH is also reported to be an important parameter (Johnsson and Petersson, 1989; Buisson et al., 1992). Increasing the temperature tends to increase the flux. The flux also tends to increase when the pressure differential increases, but only up to a certain point. The fluid velocity also has an influence on flux: the higher the velocity, the
better the mixing of the fluid and the lower the concentration at the membrane surface, thus improving the flux rate (Gutman, 1987).

In many cases, the permeation rate through a porous membrane can be described by the linear equation (Mulder, 1991)

\[ \text{Flux} = \text{Proportionality factor} \times \text{Driving force}. \]  

(2.3)

This is a typical equation for porous membranes where volume flux is proportional to pressure difference. The Hagen-Poiseuille equation is a simple illustration of this type of model; it describes the membrane structure as a number of cylindrical pores of the same diameter that are perpendicular to the membrane surface (Mulder, 1991). The equation is

\[ J = \frac{\varepsilon r^2}{8 \eta \tau} \cdot \frac{\Delta P}{\Delta x}, \]  

(2.4)

where \( J \) is the permeate flux through the membrane, \( \Delta P \) is the transmembrane pressure differential, \( \Delta x \) is the membrane thickness, \( \eta \) is the dynamic viscosity, \( \varepsilon \) is the surface porosity, \( \tau \) is the pore tortuosity (for cylindrical perpendicular pores, \( \tau = 1 \)), and \( r \) is the pore radius. This model gives a good description of transport through membranes but in practice, few membranes have this type of structure. Membrane pores are likely to be tortuous and of different dimensions (Gutman, 1987).

The Carman-Kozeny equation is based on a more realistic approximation of membrane structure. It models the membrane as a system of close packed spheres (Mulder, 1991). The equation is

\[ J = \frac{\varepsilon^3}{K_c \eta S^2 (1 - \varepsilon)^2} \cdot \frac{\Delta P}{\Delta x}, \]  

(2.5)

where \( S \) is the internal membrane surface area, and \( K_c \) is the Carman-Kozeny constant which is dependent on the shape and tortuosity.

The Hagen-Poiseuille and the Carman-Kozeny models suggest that the permeate flux increases in direct proportion to the transmembrane pressure and in inverse proportion to viscosity. This can be experimentally confirmed when pure water is used as a feed solution.
However, when the feed contains dissolved or suspended matter, it is observed that the permeate flux no longer depends solely on transmembrane pressure: there is a continuous development of resistance to the flux resulting from fouling.

Fouling can be defined as an accumulation of material onto and into the porous membrane, leading to a decrease in the permeate flux. Two classes of fouling may be defined (Davis, 1992c):

(i) *Internal membrane fouling*, which consists of the attachment of material within the internal pore structure of the membrane or directly to the membrane surface due to adsorption, precipitation, pore plugging, adhesion, etc.

(ii) *External cake fouling*, which consists of the formation of a stagnant cake layer on the membrane surface due to concentration polarization during filtration.

In membrane filtration, before the process reaches steady-state, the convective flow to the membrane surface is greater than the transport to the bulk solution due to diffusion. Consequently there is an accumulation of the rejected components (solute) at the membrane surface. This well-known phenomenon is called concentration polarization. Matthiasson and Sivik (1980) describe the negative effects due to concentration polarization:

(i) An increase in chemical potential at the surface decreases the driving force for filtration.

(ii) If the wall concentration of solute reaches the saturation concentration, precipitation or formation of a gel on the membrane surface increases the hydrostatic resistance.

(iii) High concentration of the solute at the membrane interface increases the risk of changes in the composition of the membrane material due to chemical attack.

(iv) The deposition of solute on the surface can change the separation characteristics of the membrane.

Fouling is in some cases an irreversible adsorption of macromolecules, while gel formation caused by concentration polarization in its true sense is reversible because there are no forces between the macromolecules in the gel (Matthiasson and Sivik, 1980). These phenomena are strongly dependent on the type of membrane process and the feed solution employed. Flux decline has a negative influence on the economics of a given membrane...
operation, and therefore, it is crucial to understand the problem in order to reduce its occurrence.

Several theories have been developed to describe concentration polarization and fouling, and mathematical models were proposed accounting for each. For pressure driven processes such as microfiltration, ultrafiltration and nanofiltration, the flux can be written as

\[
Flux = \frac{Driving\ force}{Viscosity \times Total\ resistance}
\]  

(2.6)

This is called the resistance model; it is based on the concept of resistance in series.

Depending on the design of the module, the flow of feed suspension can be directed perpendicular to the membrane surface, which is called dead-end or impact filtration; or it can be directed parallel or tangential to the membrane surface, which is called cross-flow or tangential filtration (Belfort et al., 1994). These are shown in Figure 2.2.

![Figure 2.2 - Schematic of dead-end and cross-flow filtration - Adapted from Redkar (1994)](image)

There are four basic types of module configurations, which are commonly used to house membranes (Mulder, 1991; Glutek et al., 1985):
(i) **Plate and Frame**: a flat sheet membrane is supported on plates, which are spaced apart using a frame. It operates at low to moderate pressures.

(ii) **Spiral Wound**: flat membrane sheets are sandwiched between mesh spacers with backing material and then rolled to form a tube. This type of module can operate at very high pressures and is often used for applications with a low level of suspended solids.

(iii) **Tubular**: this is a tube shaped membrane into which feed is pumped. The permeate is collected in a vessel enclosing the tube. It is used when the feed is viscous or has a high content of suspended solids.

(iv) **Hollow**: the membrane consists of many hollow fibres contained in a vessel; it operates at low to moderate pressures.

A submerged, hollow-fibre microfiltration module developed and patented by Zenon Environmental Inc. as ZeeWeed™ is used in this research. The module consists of a bundle of hollow-fibre microfiltration membranes submerged in a tank, into which the effluent is introduced. This configuration differs from conventional hollow-fiber filtration modules in that it uses a vacuum inside the fiber lumen to create the transmembrane pressure differential, and an aeration system that is used to promote turbulence to minimize concentration polarization and fouling, thus improving the filtration performance. A schematic diagram of the system is presented in Figure 2.3.
2.2 Use of Membranes in the Pulp and Paper Industry

2.2.1 Introduction

Membrane separation technology has not been widely used in the pulp and paper industry, principally because of technical concerns such as process efficiency and uncertainties in process integration, and for economic reasons. In addition, mill personnel are generally not familiar with membrane processes. Nevertheless, this technology has some advantages over other separation processes. Energy consumption is generally low, the process is sometimes easily integrated into existing process equipment, membrane properties can be adjusted to different separation purposes, and normally no additives are required. Moreover, the recent development of new membranes and their commercial availability has made this technology more attractive.
Membrane research in the pulp and paper industry has been carried out since the early 1970's, with emphasis on the treatment of effluents. The major concern in the early studies was to remove colour from E-stage bleaching effluents. In the 1980's the research direction shifted to the removal of toxicity from bleaching filtrates (Buisson et al., 1992). Today the greatest environmental challenge that the industry faces is the reduction and elimination of liquid emissions by means of recycling, the so-called closed-cycle mill. The use of membranes in closed-cycle systems is a promising field of research.

The use of membranes to concentrate liquid streams in pulping and papermaking processes also has potential; the driving force is energy consumption saving. The recovery of valuable products using membrane fractionation processes may also have a promising future in the pulp and paper industry. However, there are still problems remaining to be solved in order to overcome the drawbacks of membrane technology.

2.2.2 Treatment of Effluents

There are a number of different liquid streams in the mill, each of which has specific physical and chemical characteristics. The volume, suspended solids concentration, colour, organic and inorganic concentration, temperature, and pH of the stream can vary significantly. Accordingly, when in-plant control systems are used, each type of effluent has to be treated separately.

**Kraft Bleach Plants**

Among the many liquid streams discharged in a bleached kraft pulp mill (BKPM), bleach plant effluents account for the most significant volume and are a major source of dissolved organic matter, biochemical and chemical oxygen demand (BOD, COD), colour, and chlorinated organic compounds.

The use of membranes for treating effluents is perhaps most suited to the removal of colour from alkaline E-stage effluents. Of all membrane processes, ultrafiltration is the one that has been investigated most. There are at least two full-scale plants for the ultrafiltration of E-stage effluent, both of which are in Japan and began operations in 1981. At the Husum kraft mill in Sweden, an ultrafiltration pilot unit began treating Eo-stage effluent in 1986. In 1988
a full-scale test plant started operation using a cross-rotational filter, consisting of flat membrane cells stacked vertically with rotors between them was used to create high liquid velocity. The average flux during the first year of operation was 210 L/m²/h, including down periods due to fouling problems. Average COD and adsorbable organic halogen (AOX) removal rates of 54% and 59% were achieved, although large variations were observed. This plant ceased operation in 1992 because of increasing maintenance costs (Berglund et al., 1991).

The removal of colour, COD and AOX from E-stage discharges using ultrafiltration has been shown to be technically feasible. The efficiency of the removal process varies with the type of membrane used and the quality of effluent tested. Nonetheless, colour removal is normally over 85%, COD removal ranges from 55% to 88%, and 60% or more of the AOX is removed (Muratore et al.; 1983; Berglund et al., 1991; Ekengren et al., 1993).

Lundahl and Mansson (1980) described an ultrafiltration process used in the de-colouration of effluents from the alkali E-stage of a conventional kraft pulp mill. The process used polysulfone membranes arranged in a plate and frame module. The fluxes varied from 80 to 160 L/m²/h. The colour in the effluent was decreased by 87%, the COD by 70%, the biochemical oxygen demand (BOD) by 25%, and the acute toxicity by 50%.

Muratore et al. (1983) also conducted mill site ultrafiltration experiments. A three-stage industrial unit containing tubular membranes was used to process 2000 m³/h of filtrate from the alkaline extraction stage washer. Colour, COD, and BOD were reduced by 85%, 65%, and 40%, respectively, and the effluent was concentrated with respect to dissolved solids by a factor of 10. The average flux was 246 L/m²/h.

Researchers at Lund University, Sweden, carried out extensive studies using membranes to treat bleach plant effluents. Seven different types of membranes were tested to treat E-stage effluents (Jonsson, 1989). As expected, reverse osmosis had the lowest flux (50 L/m²/h) but very good COD reduction (95%). A polysulfone tubular membrane with a molecular weight cutoff (MWCO) of 8000 Dalton had the highest flux (210 L/m²/h). The COD and colour removal were 60% and 92%, respectively.
In another experiment, Jonsson and Petersson (1988) treated C-stage, and E-stage filtrates and mixed effluents using ceramic microfilter membranes with a pore diameter of 0.2 μm. Better fluxes were achieved when the effluents were treated separately. The COD removal of 25% for the mixed stream and for the acidic filtrates were considered poor. Better results were achieved for the E-stage filtrate where the COD removal was 45%.

Ekengren et al. (1993) conducted batch laboratory experiments to study ultrafiltration, nanofiltration and reverse osmosis filtration of effluents from a bleach plant. E-stage and C-stage effluents were mixed in standard proportions, and the pH was set to 7 by adding potassium hydroxide. COD and AOX removal efficiencies were 58% and 63%, respectively. The average flux was 100 L/m²/h. For the reverse osmosis experiments tubular and spiral wound membranes were tested. The average flux was 48 L/m²/h for the tubular module and 28 L/m²/h for the spiral wound membrane. The COD removal was over 80%, and the AOX removal was greater than 99% for both membranes. For the nanofiltration experiments, the COD removal was between 79% and 90%, and the AOX was decreased by 90% and 97%. The flux for the spiral wound membrane was 120-125 L/m²/h, and 90-120 L/m²/h for the tubular membrane.

More recently, the Commission of the European Community under the BRITE/EURAM program has supported research on membrane technology to treat pulp mill effluents (Pinho et al., 1995; Rosa and Pinho, 1995). Pinho et al. (1995) conducted lab and pilot scale experiments to study the application of nanofiltration to remove colour, COD, total organic carbon (TOC) and total organic halides (TOX) from E-stage effluents in order to recycle the effluent back to the system. Two polymeric membranes (tubular and spiral wound), and a tubular ceramic membrane were tested. Results were satisfactory except for the removal of chloride ions. Electrodialysis was used to remove the chloride. It was concluded that it is technically and economically feasible to optimize a sequence of pre-treatment, nanofiltration and electrodialysis in order to purify and recycle the E-stage effluent.

**Paper Mill Effluents**

Water consumption in paper mills varies from 8 to 23 m³/adt, depending on the type of paper and degree of closure of the mill (Panchapakesan, 1992). Research on membrane
ultrafiltration and nanofiltration has been carried out to treat different types of paper mill effluents, some of which is described below.

Paper mill effluent "whitewater" is normally rich in fibers, fillers and fines, resulting in a high suspended solids, and to a lesser extent COD and BOD, in mill emissions. The reuse of these effluents could bring meaningful environmental and economic benefits to the mill. A decrease in fresh water and chemical consumption, the minimization of fiber and filler losses, and the reduction in the end-of-pipe treatment of these effluents are some of the expected beneficial results of recycling. However, the recycling of such effluents without prior treatment can cause several problems during the process and thus affect paper quality. These problems are basically related to the accumulation of contaminants, which can have adverse effects such as poor drainage, plugging, corrosion, foaming, slime and bacterial growth, pitch, deposits and scale (Adams, 1994).

Nuortila-Joninken et al. (1995) studied the use of ultrafiltration and nanofiltration for the purification of whitewater on a pilot plant scale. Ultrafiltration studies were conducted on clear filtrate, that is, whitewater pre-treated in a fiber recovery system with disc filters. Two different types of effluents were tested, one from neutral and the other from acidic paper mills. The ultrafiltration removed close to 100% of total solids, colour, and polysaccharide content, mainly starch. The microbial content was also completely removed. However the fluxes obtained were not sufficient for an economical industrial application, ranging from 51 to 69 L/m²/h. Ions were not retained by ultrafiltration. The nanofiltration experiments were carried out using the filtrates from the ultrafiltration permeates. Two spiral wound modules were used in parallel. Very pure water was obtained using both membranes, with complete removal of COD, dissolved solids, polysaccharides and colour. The fluxes through the membranes were 5 and 6 L/m²/h/bar. Most measured substances and ions were best removed at acidic pH by the sulfonated polyester sulfone membrane.

Elefantisiotis et al. (1995) performed batch scale tests on two ultrafiltration membranes treating a simulated whitewater with a total dissolved solids concentration of 3000 to 4000 mg/L and a fatty acids concentration of 25 to 30 mg/L. The experiments indicated that the total dissolved solids, COD and TOC removals were in the range of 10% to 37%, and 25% to 45% of the resin acids were removed. Fatty acid removal ranged from 55% to 65%.
Ultrafiltration combined with biological treatment of these effluents resulted in a significant improvement and additional removal of all tested parameters.

The effluent from paper or paperboard coating processes does not represent a significant volume of the total paper mill effluent. However, due to its high solid concentration, it has an impact on the colour, COD, and suspended solids of the overall effluent. The effluent normally has the same chemical composition as the coating mixture applied, and the flow varies depending on the type of process and the process stability.

Pichon et al. (1992) tested the application of tubular polymeric ultrafiltration membranes to treat three different types of coating effluents, in laboratory and pilot scale trials. For a suspended solids content up to 10%, the average permeate flux ranged from 90 to 120 L/m²/h. For higher solids content, the flux rate decreased considerably. The flow velocity had a significant influence on the permeate flux: the higher the velocity, the higher the flux. The selectivity of the ultrafiltration membranes was excellent, and the permeates were clear and could be recycled as wash water. The concentrates could also be recycled provided they kept their original coating properties.

Other work on treating coating effluents was performed in Frovi, Sweden. Problems related to effluent colour at the ASSI FRÖVIFORS BRUK coated board mill led to a study on the use of a membrane process to recover and recycle coating effluents (Stridsberg et al., 1992). The pilot tests used a cross-rotation module with high shear forces. This module consists of a number of membrane cells stacked vertically, each cell with two filter membranes. Between the surfaces of these two membranes, there is a rotor that generates the cross flow. The results showed that the flux rates were about 200 L/m²/h. The permeate was free of COD and total solids. The concentrate had total solids up to 40%, and the flux was proportional to the viscosity. Therefore as the temperature increased, the flux increased by 20% to 30%.

Another potential use of membrane technology that has been tested in the paper industry is the treatment of flexographic de-inking newsprint effluent (Upton et al., 1995). Flexography is a relatively new printing technique and has several advantages over letterpress and lithography, common techniques used in printing. Flotation is widely used for de-inking
secondary fibers that have been printed by letterpress or lithography but it is ineffective in removing flexographic ink residues. Washing, on the other hand, is used effectively to remove the flexographic ink dispersion. However, this process results in liquid effluents rich in pigments that have an adverse effect on the environment.

Upton et al. (1995) investigated the use of ultrafiltration to treat such effluents; their aim was to recycle of the permeates. A polysulfone hollow fiber module with an MWCO of 500,000 D was tested in a lab scale apparatus to assess the feasibility of this treatment. The results showed that the permeate was free of ink pigments.

**CTMP and BTCMP Effluents**

The use of chemi-thermo-mechanical pulping (CTMP) and bleached-chemi-thermo-mechanical pulping (BCTMP) processes has grown significantly in recent years, particularly in Canada, the world leader in the production of high quality market mechanical pulp. Liquid emissions originating in CTMP mills are normally considered to be the most serious environmental concern. The BOD loads of these effluents are above 35 Kg/adt, and the COD loads are above 70 Kg/adt. Peroxide bleaching adds 15 to 25 Kg/adt to the BOD load and 25 to 40 Kg/adt to the COD load, depending on the peroxide charge. Effluent treatment costs represent about 5% of investment and of manufacturing costs (Malinen et al., 1985).

Beaudoin et al. (1992) evaluated the use of membranes to treat CTMP effluents. Two different types of effluents from different sources were characterized and tested, one from a washing stage between two refiners, and the other from the bleach plant. Several membrane types were tested in short-term trials that lasted 2 hours. Permeate quality and fluxes were measured, and membranes were chosen to be used for a long-term experiment. For the effluent from washers, an average flux of 210 L/m²/h was obtained. Dissolved solids, TOC and COD removal efficiencies were 36%, 55%, and 58%, respectively. The characteristics of the membranes used were not specified.

Ramamurthy et al. (1994) studied the process of fouling in hydrophilic ultrafiltration membranes used to treat screw press filtrates from CTMP mills. Tubular membranes manufactured by Zenon Environmental Inc. with MWCO of 10,000 D were used in this study. The membranes were previously soaked and compacted in distilled water and the
temperature was maintained constant at 55 °C. Effluent was pre-filtered using bag filters and pH varied from 5.7 to 6.1. Operating pressures investigated were 172, 345 and 517 KPa, linear velocities over the membranes were 3.53, 4.89, and 6.25 m/s. Fractional resistance due to the membrane, due to plugging and due to the fouling layer were calculated at various pressures. It was shown that at low pressures the effect of the membrane resistance was more significant than the other resistance. An increase in pressure resulted in an increase of the fouling layer resistance leading to a reduction of the flux. The contribution of the pore plugging resistance was relatively constant. High turbulence was found to be most effective in inhibiting fouling layer growth.

**Sulfite Pulping Process**

Although the use of the sulfite pulping process has declined, especially in North America, the mills still using it face serious environmental problems from the liquid effluent streams.

Sierka et al. (1992) evaluated membrane applications with effluents produced at a bleached sulfite mill. Tests were carried out to determine the rejection characteristics of commercially available ultrafiltration, nanofiltration and reverse osmosis filtration in terms of colour and organic material from the extraction stage (Eo-stage). Over 90% colour removal was obtained in all nanofiltration and reverse osmosis membranes, although only 27% to 53% of the TOC was removed by these membranes.

In Sweden, because of environmental restrictions, the MoDo Domşjö sulfite mill was forced to eliminate bleaching plant effluent discharge by recycling the effluent. This caused an increase in the concentration of resin in the system, and consequently there were problems with deposits and pulp quality. In 1985, an ultrafiltration unit was installed to treat the E-stage filtrates with the goal of removing resin. The concentrate goes to further concentration in a centrifuge, and the concentrate is then burned as a fuel. The permeate from the ultrafiltration unit is used as washing liquid in the bleach plant or as dilution for cooking liquor (Nordberg and Haggstom, 1988).
2.2.3 Fractionation and Recovery of Chemicals

Fractionation using membrane processes with the goal of recovering valuable byproducts has many potential applications within the pulp and paper industry. Paprican is carrying out several studies on the applications of membrane technology in the pulp and paper industry. One of its projects is the electrolysis of weak black liquor to remove a portion of the organic material from the spent liquor before it enters the recovery furnace (Paleologou et al., 1994a). The objective is to provide an incremental recovery capacity for the pulp mill. The use of bipolar membrane electrodialysis (BME) to recover caustic soda and sulphuric acid from sodium sulphate and caustic soda and chloric acid from sodium chlorate were proposed by Paleologou et al. (Paleologou et al., 1992; Paleologou et al., 1994a; Paleologou et al., 1994b).

The Finnish Pulp and Paper Research Institute has conducted studies on the ultrafiltration of kraft black liquor to remove alkali lignin, which can be utilized as feedstock for chemical processes. The fractionation of the liquor is carried out by ultrafiltration and diafiltration. (Jonsson and Wimmerstedt, 1985; Olsen, 1980). The ultrafiltration of spent sulfite liquor to separate lignosulfonate has been implemented in several places (Jonsson and Wimmerstedt, 1985).

2.3 Flux Prediction in Microfiltration Systems

2.3.1 Introduction

Several models have been developed to describe transport through a porous membranes. The models proposed for microfiltration have focused on concentration polarization and on the different mechanisms for the back-transport of particles from the membrane surface. A brief review of some important existing models is presented below.

2.3.2 Dead-end Filtration

In dead-end filtration the membrane and the cake layer formed on the surface by rejected particles may be considered as two resistance in series, as previously mentioned. Membrane
resistance can be estimated using the Hagen-Poiseuille or the Carman-Kozeny equation (Belfort et al., 1994; Davis and Grant, 1992). In practice, it is determined using flux measurements obtained in the absence of a cake layer, and its value may increase with time due to fouling (Belfort et al., 1994).

The resistance due to cake formation depends on the type of material formed, i.e., cake compressibility and porosity. When a cake is incompressible, its porosity and resistance are independent of the transmembrane pressure differential. The specific cake resistance may be estimated using the Carman-Kozeny equation (Belfort et al., 1994; Davis and Grant, 1992)

$$\hat{R}_c = \frac{K_c (1 - \varepsilon_c)^2 \cdot S_c^2}{\varepsilon_c^3},$$

where $\hat{R}_c$ is the specific cake resistance, $K_c$ is the Carman-Kozeny constant of the cake, $\varepsilon_c$ is the surface porosity of the cake, and $S_c$ the internal surface area of the cake.

On a mass basis, the specific cake resistance can be expressed by

$$R_c' = \frac{\hat{R}_c}{\rho_s \Phi_c} = \frac{\hat{R}_c}{\rho_s (1 - \varepsilon_c)},$$

where $R_c'$ is the specific cake resistance on a mass basis, $\rho_s$ is the mass density of solids comprising the cake, and $\Phi_c$ is the solid volume fraction in the cake.

When the cake is formed of a highly compressible material, the porosity and resistance are dependent on the transmembrane pressure differential. The effect of cake compressibility is a power-law function of the transmembrane pressure differential

$$R_c' = \alpha_c (\Delta P)^s,$$

where $\alpha_c$ is a constant related to the size and shape of particles, and $s$ is the cake compressibility (determined by measuring the specific resistance at various transmembrane pressures).

For flat membranes the cake resistance $R_c$ is proportional to the cake layer thickness $\delta_c$ as in

$$R_c = \hat{R}_c \cdot \delta_c,$$

The flux at any given time is given by
where \( R_m \) and \( R_f \) are the membrane and fouling resistances.

The rate of cake growth during dead-end filtration may be determined with the aid of a particle mass balance at the edge of the growing cake layer. The equation is

\[
\left( J + \frac{d \delta_c}{dt} \right) \cdot \Phi_b = \Phi_c \cdot \frac{d \delta_c}{dt},
\]

where \( \Phi_b \) is the solid volume fraction in the suspension, and \( \Phi_c \) is the solid volume fraction in the cake. The left-hand side of the equation represents the flux of particles into the surface of the cake layer, and the right-hand side represents the buildup of particles in the cake layer.

Combining Equations 2.11 and 2.12 yields

\[
\frac{d \delta_c}{dt} = \Phi_b \frac{J}{\Phi_c - \Phi_b} = \frac{\Phi_b \Delta P}{(\Phi_c - \Phi_b) \cdot \eta \cdot (R_m + \hat{R} \delta_c)},
\]

(2.13)

Keeping the pressure differential constant, Equation 2.13 can be integrated to give

\[
R_m \cdot \delta_c + \hat{R} \delta_c^2 = \frac{\Phi_b \Delta P}{(\Phi_c - \Phi_b) \cdot \eta} \cdot t,
\]

(2.14)

If the membrane resistance \( R_m \), the solid volume fraction in the cake \( \Phi_c \), and the specific cake resistance \( \hat{R} \) are all assumed to be constant, it can be deduced that

\[
\delta_c(t) = \frac{R_m}{\hat{R} \cdot \eta \cdot R_m^2} \left[ 1 + \frac{2 \hat{R} \cdot \Phi_b \cdot \Delta P \cdot t}{(\Phi_c - \Phi_b) \cdot \eta \cdot R_m^2} \right] - 1.
\]

(2.15)

Combining Equations 2.11 and 2.15:

\[
J(t) = J_o \left[ 1 + \frac{2 t \cdot \hat{R} \cdot \Phi_b \cdot \Delta P}{(\Phi_c - \Phi_b) \cdot \eta \cdot R_m^2} \right]^{-\frac{1}{2}},
\]

(2.16)
where $J_0$ is the initial flux. Integrating Equation 2.16 with respect to time gives

$$
\left( \frac{A}{V} \right)_t = \frac{\eta \cdot \dot{R}_c \cdot \Phi_b}{2(\Phi_c - \Phi_b) \cdot \Delta P} \left( \frac{V}{A} \right) + \frac{\eta \cdot R_m}{\Delta P},
$$

(2.17)

where $A$ is the external membrane surface area, and $V$ is the permeate volume.

Plotting $\left( \frac{A}{V} \right)_t \times \frac{V}{A}$ allows the specific cake resistance and membrane resistance to be determined from the slope $\left( \frac{\eta \cdot \dot{R}_c \cdot \Phi_b}{2(\Phi_c - \Phi_b) \cdot \Delta P} \right)$ and the intercept $\left( \frac{\eta \cdot R_m}{\Delta P} \right)$, respectively.

2.3.3 Cross-Flow Filtration

During the past two decades, cross-flow filtration has been increasingly used as an alternative to dead-end filtration. In cross-flow filtration, the bulk suspension flows tangentially to the membrane surface, exerting a high shear at the surface that prevents the increasing buildup of a cake layer. Thus, the cake layer remains thin, allowing higher permeate fluxes over extended periods of time (Belfort et al., 1994). Theoretical research has focused on various mechanisms by which the tangential shear prevents cake layer growth, leading to different models for predicting the permeate flux. A review is presented of the models which predict steady-state permeate flux during cross-flow microfiltration.

The Concentration - Polarization Model (Film Theory Model)

One of the simplest and most widely used models for predicting permeate flux is the concentration - polarization, or film theory model. The principle behind this theory is that the retained solute accumulates at the membrane surface, forming a thin layer. After a given period of time, when the steady-state condition is established, the convective solute flow is balanced by the solute flux through the membrane plus a diffusive flow back towards the bulk feed due to the concentration differential (Mulder 1991). The equation illustrating this principle is

$$
J \cdot c + D \cdot \frac{dc}{dx} = J \cdot c_p,
$$

(2.18)
where \( c \) is the solute concentration, \( c_p \) is the solute concentration in permeate, and \( D \) is diffusivity.

The boundary conditions are

\[
x = 0 \Rightarrow c = c_m \\
x = \delta \Rightarrow c = c_b,
\]

where \( \delta \) is the boundary layer thickness, \( c_m \) is the solute concentration at the membrane surface, and \( c_b \) is the solute concentration in the bulk suspension.

Assuming \( D \) to be constant, the integration of Equation 2.18 results in

\[
J = \frac{D}{\delta} \ln \frac{c_m - c_p}{c_b - c_p} = k \cdot \ln \left( \frac{\Phi_c}{\Phi_b} \right),
\]

(2.19)

where \( k = D/\delta \) is the mass transfer coefficient.

For laminar flow, the length averaged mass transfer coefficient is determined using Lévêque’s correlation for thin boundary layers (Porter, 1972) as in

\[
\langle k \rangle = 0.81 \left( \frac{\gamma D_p^2}{L} \right)^{\frac{1}{3}},
\]

(2.20)

where \( \langle k \rangle \) is the length averaged mass transfer coefficient, \( L \) is the tube or channel length, \( D_p \) is the particle diffusivity, and \( \gamma \) the shear rate in the boundary layer.

Combining Equations 2.19 and 2.20 leads to

\[
\langle J \rangle = 0.81 \left( \frac{\gamma D_p^2}{L} \right)^{\frac{1}{3}} \ln \frac{\Phi_c}{\Phi_b},
\]

(2.21)

where \( \langle J \rangle \) is the length-averaged permeate flux.

The Brownian diffusivity of a particle of radius \( r_p \) is given by the Stokes-Einstein relationship
\[ D_b = \frac{K_B T}{6\pi \eta r_p}, \]  
(2.22)

where \( D_b \) is the Brownian diffusivity, \( K_B \) is the Boltzmann constant equals to \( 1.38 \times 10^{-16} \) g.cm\(^2\)/s\(^2\) .K, \( T \) is the absolute temperature, and \( r_p \) is the particle radius.

Combining Equations 2.21 and 2.22 yields

\[
\langle J \rangle = 0.114 \left( \frac{\eta K_B T^2}{\eta^2 r_p^2 L} \right)^{\frac{1}{3}} \ln \frac{\Phi_E}{\Phi_b}. 
\]  
(2.23)

The prediction of the average length permeate flux gives good results when sub-micron-sized particles are considered using Stokes-Einstein diffusivity. However it is found to be inaccurate when larger (micron-sized) particles are considered: the predicted permeate flux is one or two orders of magnitude less than that observed in practice (Blatt et al., 1970). This phenomenon is referred to, by Green and Belfort, as the "flux paradox for colloidal suspensions" (Green and Belfort, 1980). Several alternative mechanisms have been proposed to explain this discrepancy and will be presented in the discussion that follows.

The Shear-Induced Diffusion Model

Zidney and Colton (Zidney and Colton, 1986) developed a model incorporating the shear-induced diffusivity of large particles that arises from induced velocity fields in the shear flow of concentrated suspensions. Shear-induced hydrodynamic diffusion of particles occurs because of particle-particle interactions in a flowing suspension that leads to lateral migration of the particles (Belfort et al., 1994). Two types of motion contribute to these interactions: a rotary motion that creates a velocity field exerting a drag force on other particles; and a translation motion causing particles moving on faster streamlines to capture particles on slower streamlines (Sethi, 1994).

Eckstein et al. (Eckstein et al, 1977) determined experimentally the shear-induced diffusivity for two cases:

For \( 0 < \Phi_b < 0.2 \):

\[ D_{si} = 0.02 r_p^2 \gamma; \]  
(2.24a)

for \( 0.2 < \Phi_b < 0.45 \):

\[ D_{si} = 0.025 r_p^2 \gamma, \]  
(2.24b)
where \( D_{si} \) is the shear-induced diffusivity.

Incorporating the value of the shear-induced diffusivity \( D_{si} = 0.025r_p^2 \gamma \) into Lévéque's correlation for the mass transfer coefficient as presented in Equation 2.20, gives

\[
\langle J \rangle = 0.078 \left( \frac{r_p^4}{L} \right)^{\frac{1}{3}} \gamma \ln \left( \frac{\Phi_c}{\Phi_b} \right).
\]  

(2.25)

Comparing Equation 2.20 with Equation 2.25, it can be noted that the length-averaged permeate flux increases in proportion to the applied shear rate when the shear-induced theory is considered, whereas in the film theory, the increase is proportional to the \( 1/3 \) power of the shear rate.

To derive Equation 2.25, some assumptions were made:

(i) The shear-induced diffusivity \( D_{si} \) is assumed to be constant. However, Leighton and Acrivos (Leighton and Acrivos, 1986) showed that for rigid particles, \( D_{si} \) increases with concentration.

(ii) Lévéque's solution is based on a linear velocity profile, which is true only if the viscosity \( \eta \) is constant.

Based on the earlier work of Eckstein et al., but employing a different apparatus, Leighton and Acrivos determined the coefficient of self-diffusion (Leighton and Acrivos, 1986):

\[
\hat{D}_{si}(\Phi) = \frac{1}{3} \Phi_b^2 \left( 1 + 0.5 \cdot e^{8.8\Phi_b} \right),
\]

(2.26a)

and

\[
D_{si} = \gamma \cdot r_p^2 \cdot \hat{D}_{si}(\Phi),
\]

(2.26b)

where \( \hat{D}_s(\Phi) \) is a dimensionless function of \( \Phi \).

**The Inertial Lift Model**

In an attempt to explain the phenomenon of the "flux paradox", researchers have proposed a model which considers a lateral migration of particles due to inertial lifting augmenting the back diffusion of particles away from the membrane surface (Belfort et al., 1994). This
theory assumes that particles migrate across the fluid streamlines if the inertial lift velocity is large enough to neutralize the opposing permeate velocity.

The inertial migration velocity of spherical particles under laminar flow conditions in diluted suspensions, where particle-particle interactions are negligible, is given by

\[ v_{L,o} = \frac{\rho_p^3 \gamma^2 f(y)}{\eta}, \]  

(2.27)

where \( v_{L,o} \) is the inertial migration velocity, \( \rho \) the fluid density, and \( y \) is the distance of the particle from the wall.

The Surface Transport Model

In the back-diffusion models presented so far, it was assumed that the cake layer is stagnant. However, some researchers have suggested that particles roll or slide along the membrane surface (Belfort et al., 1994; Lojkine et al., 1992). The basic concept of the surface transport model is to consider the force balance for a spherical particle on the membrane surface and determine whether it will adhere or be transported along the surface (Belfort et al., 1994).

The permeate flux after the cake layer has reached its steady-state thickness is given by

\[ J = 2.4 \cdot r_p \cdot \gamma \cdot (r_p^2 \cdot \hat{R_c})^{2/5} \cdot \cot \theta, \]  

(2.28)

where \( \theta \) is the angle of repose which depends on the surface morphology.

Based on the surface transport of the cake, Fane (1984) deduced the equation

\[ J = n_1 \cdot c_p^{n_2} \cdot U^{n_3}, \]  

(2.29)

where \( n_1, n_2, n_3 \) are constants and \( U \) is the cross-flow velocity.

They found that this model gave good correlations for suspensions containing rigid and deformable solids and droplets.

The Shear-Controlled Model

Razavi and Harris proposed a model for the ultrafiltration of the aqueous extract of soy flour, a complex suspension of proteins, lipids and carbohydrates, where the foulant layer was the major resistance (Razavi and Harris, 1996). The mass transfer model was not
applicable to this suspension, so the model was based on the shear stress on the membrane surface, which is given by

\[ J = J_0 \cdot \tau_r^n \cdot a \cdot e^{-bc}, \]  

(2.30a)

and

\[ \tau_r = \frac{\tau_w}{\tau_{wm}}, \]  

(2.30b)

where \( \tau_r \) is the relative shear stress, \( \tau_w \) is the shear stress at membrane wall, \( \tau_{wm} \) is the shear stress at membrane wall at initial feed concentration, \( n \) is a variable, \( a \) and \( b \) are constants, and \( c \) is the solute concentration.

Combined Models

As discussed previously, cross-flow membrane filtration depends on many variables including transmembrane pressure, cross-flow velocity, suspension composition, and membrane and module configuration characteristics. A global model should take into account all these factors (Lojkine et al., 1992).

Schulz and Ripperger proposed a model that incorporates both back-transport and resistance theory (Schulz and Ripperger, 1989). They assumed that back-transport by diffusion was an inadequate mechanism for cross-flow microfiltration and that membrane resistance can be neglected. The back-transport of particles is proportional to both the shear rate on the membrane surface and the layer thickness of the cake. From mass balances at steady-state, they derived the following expressions.

For turbulent flow:

\[ J = \sqrt{\frac{k_2 \cdot \Delta P \cdot (\rho_s - c_b) \cdot \rho \cdot U \cdot \eta}{\hat{R}_c \cdot c_b}}, \]  

(2.31)

and for laminar flow:

\[ J = \sqrt{\frac{k_3 \cdot \Delta P \cdot (\rho_s - c_b) \cdot U \cdot \eta \cdot \hat{R}_c \cdot c_b}{\eta \cdot \hat{R}_c \cdot c_b}}, \]  

(2.32)

where \( k_2 \), \( k_3 \) are filtration constants, and \( U \) the cross-flow velocity.
Another model that combines the resistance model and back-flux model was proposed by Shimizu et al. (Shimizu et al., 1996b). It is composed of two equations, one based on the resistance model and the other based on the back-flux model. The equations are

$$J^* = \frac{\Delta P}{\eta \cdot (R_m + R_f)},$$

and

$$v_L = k_L \cdot U \cdot c_b^{-0.5} \cdot (2r_p)^{0.67} \cdot \eta^{-1},$$

where $R_f$ is the fouling resistance, $v_L$ the lift velocity of particles, and $k_L$ a filtration constant.

If $J^* > v_{L(min)}$, then $J = v_{L(min)}$.
If $J^* \leq v_{L(min)}$, then $J = J_0$.

The resistance equation considers the membrane resistance and the resistance due to pore plugging (internal fouling). The back-flux model, based on the lift theory, considers cross-flow velocity, particle concentration, particle size and viscosity. The criterion for choosing the appropriate equation was determined by comparing the value of the initial filtration flux (after a rapid decline) with the flux or “lift velocity” given by the back-flux model. If the initial filtration flux is greater than the minimum lift velocity of the particles, then a cake layer is expected to form, and the predicted steady-state flux is given by the lift velocity equation. In contrast, if the initial filtration flux is equal to or smaller than the lift velocity of the particles, a cake layer is not expected to form, and the predicted steady-state flux is given by the resistance model.

The Use of Dispersed-Phase Turbulence Models

The main limitation of microfiltration is membrane fouling. Several methods have been applied to reduce membrane fouling, including the use of turbulence promoters.

The modeling of membrane filtration systems that operate under a turbulent regime has not been extensively studied compared with those systems that operate under laminar flow conditions. Turbulent mass transfer correlations can be applied directly to the concentration polarization model (film theory) with Brownian diffusion and cake limited resistance. However, this approach is not expected to be appropriate for the shear-induced or the
inertial-lift mechanism, since transverse particle motion depends directly on the local flow fields (Davis, 1992a).

The mass transfer coefficient $k$ depends strongly on the hydrodynamics of the system. It is related to the Sherwood number, which is equal to the dimensionless concentration gradient at the surface, and it provides a measure of the convective mass transfer occurring at the surface. The application of the Sherwood correlation to membrane operation was thoroughly reviewed by Gekas and Hallstrom (Gekas and Hallstrom, 1987).

The use of a dispersed phase to create cross flow, in particular air bubbles, is of great interest in the present study, since the ZeeWeed™ system that was tested employs this method to combat concentration polarization. The introduction of a dispersed phase that creates turbulence at the membrane surface has been shown effective in enhancing the permeation flux in ultrafiltration and microfiltration systems.

Cui and Wright carried out experiments with dextran solution using tubular ultrafiltration membranes, vertically installed (Cui and Wright, 1994). An increase of 70% to 250% in the permeate flux was observed when air was injected with the feed stream. The effect was more pronounced at higher feed concentration, and also at higher transmembrane pressure. Lee et al. used air slugs to improve the filtration of bacterial suspensions (Lee et al., 1993). An interesting result of this study was that the use of air had a stronger effect on the permeate flux when ultrafiltration membranes with lower nominal pore size were used.

Very little research has been done on the mathematical modeling of membrane filtration systems that possess a dispersed phase to promote turbulence on the membrane surface. Parvatiyar and Goving developed a mathematical model to explain the formation of concentration polarization in a membrane tube under turbulent flow conditions created by the use of a dispersed phase (Parvatiyar and Goving, 1995). The model was based on the concept of the energy spectrum of turbulence flow field. Their equation was

$$CP = \exp\left[\frac{Pe}{Sh^{(c)} + Sh^{(de)}}\right], \quad (2.34)$$

in which,
\[ Sh^{(e)} = 0.063 \cdot Sc^{\frac{1}{3}} \cdot Re_{dm}^{\frac{1}{32}}, \]  

(2.35)

and

\[ Sh^{(de)} = \frac{0.05316 \cdot Sc^{\frac{1}{3}} \cdot Re_{dm}^{\frac{5}{16}} \cdot Re_{dp}}{C_1^{\frac{1}{2}}}, \]  

(2.36)

where \( CP \) is the concentration polarization, \( Pe \) is the Peclet number, \( Sh^{(e)} \) is the Sherwood number accounting for energy dissipation, \( Sh^{(de)} \) is the Sherwood number accounting for energy dissipation due to dispersed-phase, \( Re_{dm} \) is the Reynolds number based on membrane tube diameter, \( Re_{dp} \) is the Reynolds number based on particle diameter, \( Sc \) is the Schmidt number, and \( C_1 \) a variable.

### 2.3.4 Reverse Filtration

A method for minimizing and controlling the adverse effects of concentration polarization is the use of reverse filtration. Reverse filtration is the counter-flow of the permeate through the membrane produced by changing the direction of the transmembrane pressure. There are two modes of operation for reverse filtration: backpulsing, which refers to high-frequency reverse filtration, i.e., several backpulses per second; and backflushing, which refers to low-frequency reverse filtration, i.e., one backflush every several seconds (Redkar, 1994). In backflushing, a cake is formed but removed periodically by the reverse flow. For backpulsing, the frequency of reverse filtration is so high that a stagnant cake layer never forms. Figure 2.4 presents a schematic for a reverse filtration operation.

Redkar developed the following mathematical models for high frequency backpulsing and low frequency backflushing (Redkar, 1994).

**High Frequency Backpulsing**

For a particular reverse filtration time, the average dimensionless flux would increase with the forward filtration time according to the equation

\[ \langle \frac{J}{J_o} \rangle = \frac{\langle J \rangle}{J_o} = \frac{t_f - \beta t_b}{t_f + t_b}, \]  

(2.37)
where \( \langle J \rangle \) is dimensionless average permeate flux (per cycle), \( \langle J \rangle \) is the average permeate flux (per cycle), \( t_f \) is the forward filtration time, \( t_b \) is the reverse filtration time, and

\[
\beta = \frac{\Delta P_b}{\Delta P_f} = \frac{\text{reverse filtration transmembrane pressure}}{\text{forward filtration transmembrane pressure}}.
\]

Figure 2.4 - Forward and reverse filtration operation

Low Frequency Backflushing

Redkar's model for low frequency backflushing is based on Romero and Davis's equation (Romero and Davis, 1988), which describes the initial permeate flux decline due to cake buildup during dead-end filtration. It gives

\[
J = J_o \left(1 + \frac{t}{t_c}\right)^{-0.5},
\]

(2.38)

where \( t \) is the time elapsed after the start of filtration, and \( t_c \) is the time constant for cake growth, as reported by Davis and Grant (1992) and is given by
It is assumed that the cake is instantly raised from the membrane wall and then removed by the axial flow during cross-flow filtration. Also it is assumed that there is no significant internal membrane fouling and that the membrane resistance is constant for reverse and forwarded filtration.

The average flux in one cycle is given by

\[
\langle j \rangle = \frac{\int_0^{t_f} J dt - \beta J_0 t_b}{t_f + t_b} = \frac{2J_0 t_c}{t_f + t_b} \left[ \left( \frac{t_f}{t_c} \right)^{0.5} - 1 \right] - \frac{\beta J_0 t_b}{t_f + t_b},
\]

(2.40)

where \( \int_0^{t_f} J dt \) is the total filtrate volume per unit area collected during forward filtration of duration \( t_f \) and \( \beta J_0 t_b \) is the total filtrate volume per unit area lost during reverse filtration of duration \( t_b \).
3.1 Introduction

To select a feasible and attractive application for membrane separation in the pulp and paper industry:
(i) criteria were established for application selection,
(ii) lab-scale tests were performed for four applications,
(iii) the best application was selected.

3.2 Criteria for Successful Application

Membrane separation processes can be applied in numerous unit operations within pulp and paper mills. Table 3.1 summarizes the studies as reported in the literature (Chapter 2) of different applications in the pulp and paper industry. Twelve applications from the literature search were thought to have good potential. They are listed in Table 3.2.

Three general criteria were taken into account in the selection of applications for subsequent lab-scale tests:
(i) physical-chemical properties of the stream,
(ii) types of membranes which achieve the separation goal and are suitable for mill integration,
(iii) market potential.

The physical-chemical characteristics of each effluent are of great importance in choosing the appropriate separation process and selecting the right type of membrane to be used. Table 3.3 presents the main characteristics of each liquid stream shown previously in Table 3.1. The data shown in Table 3.3 was based on published literature and was discussed in Chapter 2 and summarized in Table 3.1. The concentration levels are presented as (*), (**) and (***) and are based on the following criteria:
**TSS:** Low concentration (*) < 200 mg/L  
Medium concentration (**) 200 – 1000 mg/L  
High concentration (***) >1000 mg/L

**TDS:** Low concentration (*) < 500 mg/L  
Medium concentration (**) 500 – 1000 mg/L  
High concentration (***) >1000 mg/L

**COD:** Low concentration (*) < 500 mg/L  
Medium concentration (**) 500 – 3000 mg/L  
High concentration (***) >3000 mg/L

The pH and temperature are also very important parameters. Current membranes and membrane modules can operate only in a certain range of pH and temperature. For many effluent streams in pulp and paper mills the pH must be adjusted, and temperature cooled prior to filtration. Chlorine, chlorine dioxide and hydrogen may have an adverse effect on the membrane. Normally, these substances are found only in bleaching effluents.

Table 3.4 summarizes the types of membranes that can be used in each application and the potential for mill integration. The data presented was also based on the literature review presented in Chapter 2, and on the known characteristics of each stream. The use of membranes as a pre-treatment was not considered in the analysis.

Table 3.5 shows the potential market for each application. This potential market was based on the 1993–1996 Canadian Pulp and Paper Capacity report published by the Canadian Pulp and Paper Association and on the 1996 American Forest and Paper Association report and also in private conversation with experts in the pulp and paper field.
### Table 3.1 - Summary of membrane applications in the pulp and paper industry

<table>
<thead>
<tr>
<th>APPLICATION &amp; PURPOSE</th>
<th>STREAM</th>
<th>MEMBRANE (*)</th>
<th>REFERENCES</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Treatment of Effluents:</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>- Whitewater</td>
<td>- UF, NF</td>
<td>- Nuortila-Joniken, J. et al., 1995; Elefsiniotis, P. et al., 1995</td>
</tr>
<tr>
<td></td>
<td>- De-inking</td>
<td>- UF</td>
<td>- Upton, B.H. et al., 1995, Chabot, B. et al., 1997</td>
</tr>
<tr>
<td></td>
<td>- BCTMP/CTMP</td>
<td>- UF</td>
<td>- Beaudoin, L. et al., 1992; Ramamurthy, P. et al., 1995</td>
</tr>
<tr>
<td><strong>Concentration:</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fractionation:</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Lignin/Soda</td>
<td>- Weak Black Liquor</td>
<td>- ED</td>
<td>- Paleologou, M. et al., 1994a</td>
</tr>
<tr>
<td>- HClO₃/NaOH</td>
<td>- Sodium Chlorate</td>
<td>- BME</td>
<td>- Paleologou, M. et al., 1992; 1994a ; 1994b</td>
</tr>
<tr>
<td>- Lignosulfonate/Liquor</td>
<td>- Sulfite Spent Liquor</td>
<td>- UF, RO</td>
<td>- Olsen, O., 1980; Wagner, J. et al., 1989</td>
</tr>
</tbody>
</table>

(*) UF= ultrafiltration, NF= nanofiltration, RO= reverse osmosis, ED= electrodialysis, BME= bipolar membrane electrodialysis
Table 3.2 - Potential applications for membrane separation processes

<table>
<thead>
<tr>
<th>TYPE OF MILL</th>
<th>LIQUID STREAM</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>PULP MILLS</strong></td>
<td>1. Kraft Acidic Bleach Plant Effluent (BPE)</td>
</tr>
<tr>
<td></td>
<td>2. Kraft Alkaline Bleach Plant Effluent (BPE)</td>
</tr>
<tr>
<td></td>
<td>3. Sulfite Acidic Bleach Plant Effluent (BPE)</td>
</tr>
<tr>
<td></td>
<td>4. Sulfite Alkaline Bleach Plant Effluent (BPE)</td>
</tr>
<tr>
<td></td>
<td>5. Kraft Pulping Liquor</td>
</tr>
<tr>
<td></td>
<td>6. Sulfite Pulping Liquor</td>
</tr>
<tr>
<td></td>
<td>7. Bleached Chemi-thermo-mechanical Pulping (BCTMP) Effluent</td>
</tr>
<tr>
<td></td>
<td>8. Thermo-mechanical Pulping (TMP) Effluent</td>
</tr>
<tr>
<td><strong>PAPER MILLS</strong></td>
<td>9. Whitewater</td>
</tr>
<tr>
<td></td>
<td>10. Coating Effluent</td>
</tr>
<tr>
<td><strong>RECYCLING MILLS</strong></td>
<td>11. Old Newsprint (ONP) De-inking Mill Effluent</td>
</tr>
<tr>
<td></td>
<td>12. Old Corrugated Container (OCC) Recycling Mill Effluent</td>
</tr>
</tbody>
</table>
Table 3.3 - Physical-chemical characteristics of different pulp and paper liquid streams

<table>
<thead>
<tr>
<th>APPLICATION</th>
<th>TYPICAL VOLUME</th>
<th>CONCENTRATION</th>
<th>T</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(m³/d)</td>
<td>(m³/adt)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>TSS</td>
<td>TDS</td>
<td>COD</td>
<td></td>
</tr>
<tr>
<td></td>
<td>org.</td>
<td>inorg.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kraft Acidic BPE</td>
<td>12000-25000</td>
<td>10-20</td>
<td>*</td>
<td>**</td>
</tr>
<tr>
<td>Kraft Alkaline BPE</td>
<td>8500-25000</td>
<td>5-20</td>
<td>*</td>
<td>***</td>
</tr>
<tr>
<td>Sulfite Acidic BPE</td>
<td>12000-40000</td>
<td>10-40</td>
<td>*</td>
<td>**</td>
</tr>
<tr>
<td>Sulfite Alkaline BPE</td>
<td>8500-25000</td>
<td>5-20</td>
<td>*</td>
<td>***</td>
</tr>
<tr>
<td>Kraft Pulping Liquor</td>
<td>7000-25000</td>
<td>8-15</td>
<td>*</td>
<td>***</td>
</tr>
<tr>
<td>Sulfite Pulping Liquor</td>
<td>5000-10000</td>
<td>10-20</td>
<td>*</td>
<td>***</td>
</tr>
<tr>
<td>BCTMP Effluent</td>
<td>4000-20000</td>
<td>10-30</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>TMP Effluent</td>
<td>4000-20000</td>
<td>15-30</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>Whitewater</td>
<td>4000-20000</td>
<td>10-25</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>Coating Effluent</td>
<td>240-480</td>
<td>0.2-1</td>
<td>***</td>
<td>*</td>
</tr>
<tr>
<td>ONP De-inking</td>
<td>5000-15000</td>
<td>50-70</td>
<td>***</td>
<td>*</td>
</tr>
<tr>
<td>OCC Recycling</td>
<td>5000-15000</td>
<td>50-70</td>
<td>*</td>
<td>**</td>
</tr>
</tbody>
</table>

**LEGEND:** * = Low  ** = Medium  *** = High  nd = non-detectable  na = not available

TSS = total suspended solids, TDS = total dissolved solids, COD = chemical oxygen demand
Table 3.4 - Membrane type and mill integration potential

<table>
<thead>
<tr>
<th>APPLICATION</th>
<th>MEMBRANE TYPE</th>
<th>MILL INTEGRATION</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MF</td>
<td>UF</td>
</tr>
<tr>
<td>Kraft Acidic BPE</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>Kraft Alkaline BPE</td>
<td>*</td>
<td>**</td>
</tr>
<tr>
<td>Sulfite Acidic BPE</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>Sulfite Alkaline BPE</td>
<td>*</td>
<td>**</td>
</tr>
<tr>
<td>Kraft Pulping Liquor</td>
<td>**</td>
<td>*</td>
</tr>
<tr>
<td>Sulfite Pulping Liquor</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>BCTMP Effluent</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>TMP Effluent</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>Whitewater</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>Coating Effluent</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>ONP De-inking</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>OCC Recycling</td>
<td>**</td>
<td>**</td>
</tr>
</tbody>
</table>

**LEGEND:** Potential for use:  * = Low  ** = Medium  *** = High

MBR = membrane bio-reactor
<table>
<thead>
<tr>
<th>APPLICATION</th>
<th>MILLION TONNES OF PULP &amp; PAPER PER ANNUM</th>
<th>PULP &amp; PAPER PRODUCT GROWTH POTENTIAL</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CANADA</td>
<td>USA</td>
</tr>
<tr>
<td>Kraft Acidic BPE</td>
<td>10.0</td>
<td>30.0</td>
</tr>
<tr>
<td>Kraft Alkaline BPE</td>
<td>10.0</td>
<td>30.0</td>
</tr>
<tr>
<td>Sulfite Acidic BPE</td>
<td>0.8</td>
<td>1.4</td>
</tr>
<tr>
<td>Sulfite Alkaline BPE</td>
<td>0.8</td>
<td>1.4</td>
</tr>
<tr>
<td>Kraft Pulping Liquor</td>
<td>11.0</td>
<td>51.0</td>
</tr>
<tr>
<td>Sulfite Pulping Liquor</td>
<td>0.8</td>
<td>1.4</td>
</tr>
<tr>
<td>BCTMP Effluent</td>
<td>3.5</td>
<td>na</td>
</tr>
<tr>
<td>TMP Effluent</td>
<td>5.5</td>
<td>7.0</td>
</tr>
<tr>
<td>Whitewater</td>
<td>18.0</td>
<td>42.0</td>
</tr>
<tr>
<td>Coating Effluent</td>
<td>0.9</td>
<td>8.3</td>
</tr>
<tr>
<td>ONP De-inking</td>
<td>1.3</td>
<td>na</td>
</tr>
<tr>
<td>OCC Recycling</td>
<td>1.2</td>
<td>11.0</td>
</tr>
</tbody>
</table>

**LEGEND:** Potential product growth: * = Low  ** = Medium  *** = High  na = not available
One potential application for microfiltration is the removal of flexographic ink from old newsprint (ONP) effluent in de-inking mills. Flexographic ink constitutes a serious problem in existing ONP recycling mills because it is water-based ink and cannot be efficiently removed by conventional flotation processes. If the water is then recycled, problems related to pulp brightness are often experienced. Given an increase in the use of flexographic ink in the printing industry, and given the increase in water reuse in mills, an adequate system is needed to remove flexographic inks from the wash water in de-inking facilities (Galland and Vernac, 1993; Upton et al., 1995). The market for ONP de-inked paper products seems to have high growth potential in North America. This application was selected for further lab-scale trials.

The use of microfiltration to concentrate and recycle coating effluent has good potential (Stridsberg et al., 1992). This application is attractive because of the relatively low flow of the effluent (0.2-1.0 m$^3$/adt of paper product); moreover it enables the recovery of a valuable by-product, the coating mixture. The production of coated paper and paperboard has been increasing over the last few years, and it seems that growth will continue due to the significant value added to the final product when surface coating is applied. This application was also selected for further investigation by lab-scale tests.

The use of high yield pulping processes such as chemi-thermo-mechanical pulping (CTMP) and bleached CTMP (BCTMP) has grown significantly in recent years, particularly in Canada, the world leader in the export of high quality market mechanical pulp. In this connection, it is also important to mention the two relatively new BCTMP mills in Canada that have pioneered totally closed-cycle water systems in their processes: Millar Western at Meadow Lake and Louisiana Pacific at Chetwynd. Both mills adopted mechanical recompression evaporators in order to concentrate the final effluent. The evaporator concentrate is combusted, and the condensates are sent for further treatment and then recycled (Young, 1994; Evans, 1992). The motivation for using membranes in this process is to concentrate the effluent prior to evaporation, thus saving energy. Because the recycled water must be of high quality, a system was proposed that consists of pre-treatment microfiltration followed by reverse osmosis. Lab-scale tests were conducted for this application.
The membrane bio-reactor combines a biological treatment system and a membrane separation unit; it is attractive for treating effluent from a mill which recycles old corrugated container (OCC). The OCC effluent contains relatively high levels of organic matter, and the degree of water recycling in this type of mill is extensive. There is also significant growth potential for OCC mills in North America. Environmental concerns (recycling mills are often associated with good environmental practices in the public perception) and the potential shortage of virgin fiber have led to increased recycling. Further investigation of this application was conducted in the laboratory as part of this thesis.

Applications related to sulfite pulping processes were not considered because of their limited growth potential in North America. Treatment of bleach plant effluents has been extensively studied, as detailed in the literature review. This stream was not chosen for further laboratory tests. Also, the concentration and fractionation of pulping liquors was not considered.

Hence, four applications were selected for further lab-scale tests:

(i) Application 1 - old newsprint de-inking effluent containing flexographic ink;
(ii) Application 2 - effluent from a paper coating operation;
(iii) Application 3 - effluent from a bleached chemi-thermo-mechanical pulp mill;
(iv) Application 4 - effluent from a board mill using recycled old corrugated container.

The first three applications were tested using a submerged microfiltration module, and the last using a submerged microfiltration membrane bio-reactor.

3.3 Evaluation of Four Microfiltration Applications

3.3.1 Objectives

The objectives of this work were to determine the efficiency and the feasibility of the use of microfiltration to treat effluents and to facilitate their recycling in the four previously selected applications. The specific objectives of the tests were to characterize each sample, and conduct tests to determine the main filtration characteristics and feasibility of each application.
3.3.2 Methodology

Analytical Methods

The following analyses were conducted in order to establish the effectiveness of the membrane separation process on the parameters pH, COD (chemical oxygen demand), TSS (total suspended solids), VSS (volatile suspended solids), TDS (total dissolved solids), conductivity, and colour. The methods used were based on the Standard Methods for the Examination of Water and Wastewater (AWWA, 1989). Samples were collected from the retentate and permeate. Permeate was collected after the flux was stabilized.

For Application 4, the following additional analyses were conducted on the feed effluent and the permeate of the membrane bio-reactor: BOD₅ (biochemical oxygen demand), phosphorus, and total nitrogen. The following analyses were carried out on the sludge: VSS (volatile suspended solids), DO (dissolved oxygen), OUR (oxygen uptake ratio), pH and temperature. The methods used were again based on the Standard Methods for the Examination of Water and Wastewater (AWWA, 1989).

Laboratory Apparatus

A submerged, hollow-fiber microfiltration module was used for Applications 1, 2, and 3. Figure 3.1 shows a schematic diagram of the membrane filtration system used. The module consisted of a bundle of hollow-fiber microfiltration membranes submerged in a tank, into which the effluent is introduced. The total membrane surface area was about 1.0 m². Each hollow-fiber was approximately 2 mm in outside diameter and approximately 500 mm in length. The tank had a diameter of 0.2 m and a volume of 20 L. The system was operated in a total recycle mode, i.e., the permeate was recycled to the tank, thus keeping the concentration and volume of the solution in the tank constant. Airflow was created by an air-blower, measured by an airflow meter and controlled by a valve. Polymeric hydrophilic membranes with a nominal pore size of 0.2 μm were used.

In Application 4, a similar system was used, except that the bio-reactor was composed of a 35-liter cylinder with a height of 1220 mm and a diameter of 250 mm. A microfiltration hollow-fiber module with a 0.5 m² filtration area was immersed in the bio-reactor containing
bio-mass. A polymeric hydrophilic membrane was used with a nominal pore size of 0.2 μm. In this case, an aeration system was used externally, not only to scour the membrane surface and so prevent fouling, but also to provide oxygen for the bio-mass. The system was operated in a feed and bleed mode, i.e., the system was continuously fed and permeate continuously discharged.

Figure 3.1 - Schematic diagram of filtration unit
Experimental

Samples were collected after the flux was stabilized. The tank was filled with 20 liters of effluent, and permeate was drawn through the membrane under a transmembrane pressure of -33.8 kPa (-10 in Hg). The airflow was kept at 0.94 L/s (2 SCFM). This corresponds to a superficial gas velocity of 0.0299 m/s. Superficial gas velocity ($U_{sg}$) is determined dividing the airflow rate by the cross-section area of the tank (0.03142 m$^2$). Experiments were conducted at room temperature.

In application 4, the permeate COD, the sludge VSS and the oxygen uptake rate (OUR) were the basic parameters used to establish the biological performance of the system. The system was kept at a constant temperature of 35°C and a pH of 7.5. The pH was controlled by automatically adding a 5% solution of sulfuric acid.

The membrane test procedures are summarized as follows:

(i) **Membrane Cleaning and Quality Check:** in order to determine the baseline conditions for the membrane used in the test, initial membrane cleaning was carried out. The tank was filled with de-ionized water, detergent and chlorine. The membranes were soaked for at least 24 hours in the solution with gentle aeration. The system was then drained and rinsed with water. The initial water flux test was conducted by circulating water through the system at transmembrane pressures of -16.9, -33.8, -50.8, and -67.7 kPa (-5, -10, -15, -20 in Hg), and the water flux was measured. No air was introduced during the water flux test.

(ii) **Flux Stability:** the tank was filled with 20 liters of the feed, and the permeate was recycled to the tank under a transmembrane pressure of -33.8 kPa (-10 in Hg). The airflow was kept at 0.94 L/s (2 SCFM). The permeate flow was measured until it stabilized. The duration of this phase was at least 24 hours at room temperature.

(iii) **Flux versus Transmembrane Pressure:** different transmembrane pressures of -16.9, -33.8, -50.8, and -67.7 kPa (-5, -10, -15, -20 in Hg) were tested and the permeate flow was measured. There was a fifteen-minute period between pressure changes. Airflow was kept constant.

(iv) **Flux versus Airflow:** transmembrane pressure was kept constant while different airflows were tested. They were 0, 0.47, 0.94, 1.42, and 1.89 L/s (0, 1, 2, 3, 4
These values correspond to a superficial gas velocity \((U_{sg})\) of 0, 0.0150, 0.00299, 0.0452, 0.0602 m/s, respectively. As explained before, the superficial gas velocity \((U_{sg})\) is determined dividing the airflow rate by the cross-section area of the tank \((0.03142 \text{ m}^2)\). There was a fifteen-minute period between airflow changes.

\((v)\) **Clean Water Flux versus Transmembrane Pressure without Membrane Cleaning:** In order to provide an idea of membrane flux reduction due to fouling, experiments to measure water flux versus pressure were conducted with the membrane uncleaned. No air was added to the system.

\((vi)\) **Membrane Cleaning and Quality Check:** As in the membrane cleaning and quality check described previously, the membrane was cleaned and the water flux test carried out.

When the system was operated to concentrate the effluent feed, the same procedures were carried out for every volume reduction factor \((\text{VRF})\). The VRF is defined as the volume of feed divided by the volume of concentrate generated.

### 3.3.3 Application 1: ONP De-inking Effluent Containing Flexographic Ink

**Effluent Characteristics**

The effluent used in this experiment was a mixture of flexographic ink and wash water obtained from a Canadian newsprint de-inking facility, located in Ontario. The mill used only recycled newsprint as feedstock. After pulping, the pulp passed through a high-density cleaner and subsequent coarse screening. The screened pulp was sent to a washing stage. The wash water was collected and sent to a flotation unit to remove suspended solids and ink particles after which it was recycled to the pulping vessel. Whenever flexographic ink is present, it remains in the wash water, i.e., it cannot be removed from the cycle by flotation, and so can cause low pulp brightness.

A schematic drawing of the de-inking process is presented in Figure 3.2. Samples were collected before water clarification, and flexographic ink was added prior to testing in order to simulate particularly adverse conditions.
Results and Discussion

The analytical characteristics of the retentate and permeate are presented in Table 3.6.

Table 3.6 - Analytical characterization of ONP effluent containing flexographic ink

<table>
<thead>
<tr>
<th>STREAM</th>
<th>SOLIDS (mg/L)</th>
<th>COD (mg/L)</th>
<th>pH</th>
<th>Conduct. (µS/cm)</th>
<th>Colour (mg/L)</th>
<th>Turbidity (NTU)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TSS</td>
<td>VSS</td>
<td>TDS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Retentate</td>
<td>6490</td>
<td>4260</td>
<td>3590</td>
<td>6340</td>
<td>6.8</td>
<td>2.9</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
<td>0*</td>
<td>3160</td>
<td>860</td>
<td>8.5</td>
<td>2.7</td>
</tr>
</tbody>
</table>

0* = assumed to be very small since the membrane nominal pore size is 0.2 µm
nd = not determined

The ONP effluent is rich in organic suspended solids (VSS = 66% of the total suspended solids), consisting mainly of pulp fibers. The suspended solids were completely removed by the membranes, and the COD, mostly contributed by the solids, was also substantially removed (>85%) by filtration. However, microfiltration had little effect on the dissolved solids and conductivity.

Flexographic ink was removed by filtration, as shown by the decrease in colour from the retentate to the permeate. The colour of the feed stream was measured after filtering the
sample with an 8.0 μm pore size paper filter. The observed increase in the pH, from 6.8 in the retentate to 8.5 in the permeate, was most likely due to system aeration, which strips CO₂. The quality of the permeate is such that this stream can be recycled to the de-inking process. The flexographic ink was removed by microfiltration. The retentate could be further concentrated and used as a soil conditioner, or disposed of by incineration.

Figure 3.3 shows the flux versus time using microfiltration membrane at a transmembrane pressure of -33.8 kPa (-10 in Hg), an airflow of 0.94 L/s (2 SCFM) and a temperature of 10°C.

![Flux stability of ONP effluent containing flexographic ink](image)

The initial flux was 59 L/m²h; flux decreased over the first 16 hours, and thereafter was stable at 35 L/m²h. This final flux corresponds to a permeation rate of 1.0 L/m²h/kPa (3.5 L/m²h/in Hg).

During the membrane quality check, the flux of de-ionized water was measured at different transmembrane pressures. The same test was carried out with effluent, with the addition of 0.94 L/s (2.0 SCFM) of air injected into the tank. After the effluent test, the membrane was rinsed and the water flux test was repeated. The membrane was then cleaned and the water...
flux test conducted once more. Figure 3.4 is a plot of the flux behavior versus transmembrane pressure during these experiments.

![Diagram showing flux behavior versus transmembrane pressure]

Transmembrane pressure has a significant effect on the permeate flux. During effluent filtration, an increase of 66% permeate flux was observed after increasing the transmembrane pressure from -16.9 to -33.8 kPa (-5 to -10 in Hg). An increase of only 10% was observed after increasing the pressure from -33.8 to -50.8 kPa (-10 to -15 in Hg). This can be explained by an increase in the cake resistance as the transmembrane pressure exceeds -50.8 kPa (-10 in Hg). After cleaning, the membrane recovered its filtration capacity, as shown in Figure 3.4. The resistance due to membrane fouling reduced the water flux by more than 40%.

Figure 3.5 shows the effect of airflow variation on flux during effluent filtration. In this experiment, the transmembrane pressure was kept constant at -33.8 kPa (-10 in Hg).
Figure 3.5 - Airflow versus flux of ONP effluent containing flexographic ink

The results show that in this system, airflow had a significant effect on flux. Air scouring and agitation of the membrane fibers seem to prevent concentration polarization, thus increasing the flux substantially from 28.2 to 42.6 L/m²h. However, the curve also suggests that there is a limit to this increase, after which the increase in airflow does not have an effect on flux.

3.3.4 Application 2: Effluent from a Paper Coating Operation

Effluent Characteristics
Two types of effluents were used in this application: one taken under normal operating conditions (coating normal); and the other during a paper break (coating break). The "coating break" effluent is produced during paper breaks or paper machine start-up. The effluents were expected to have the same composition of suspended matter but different concentrations. The effluents were obtained from a Canadian mill. A schematic of the coating system is presented in Figure 3.6.
Figure 3.6 - Schematic layout of the coating system

Results and Discussion
Table 3.7 presents the analytical characteristics of the retentates and permeates of the “coating normal” effluent. Analyses were conducted at two different solids concentrations of the retentate.

This effluent has a high suspended solids content, which in turn has a high concentration of inorganic solids (85%). The amount of total dissolved solids is low, and consequently the conductivity is low. The removal of the suspended solids by microfiltration induced a dramatic reduction in the COD level (>99%). An increase in the pH was observed again due again to aeration. The permeates were low in colour and had zero turbidity (crystal clear).

The characteristics of the retentates and permeates of the “coating break” effluent are shown in Table 3.8. Analyses were conducted on samples taken from trials with retentates having two different solids concentrations.
Table 3.7 - Analytical characterization of "coating normal" effluent

<table>
<thead>
<tr>
<th>STREAM</th>
<th><strong>PARAMETER</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SOLIDS (mg/L)</td>
</tr>
<tr>
<td></td>
<td>TSS</td>
</tr>
<tr>
<td>Retentate</td>
<td>6980</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
</tr>
<tr>
<td>Retentate</td>
<td>16200</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
</tr>
</tbody>
</table>

0* = assumed to be very small since the membrane nominal pore size is 0.2 μm
nd = not determined

Table 3.8 - Analytical characterization of "coating break" effluent

<table>
<thead>
<tr>
<th>STREAM</th>
<th><strong>PARAMETER</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SOLIDS (mg/L)</td>
</tr>
<tr>
<td></td>
<td>TSS</td>
</tr>
<tr>
<td>Retentate</td>
<td>66700</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
</tr>
<tr>
<td>Retentate</td>
<td>165400</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
</tr>
</tbody>
</table>

0* = assumed to be very small since the membrane nominal pore size is 0.2 μm
nd = not determined

The "coating break" effluent has a concentration of suspended solids ten times higher than that of the "coating normal" effluent. More than 80% of the total suspended solids are inorganic. As expected, there was no significant change in conductivity. The COD levels were reduced considerably (>99%) with the removal of the solids.

After the "coating break" effluent was treated, the permeates were again very low in colour and turbidity, and an increase in pH was observed. According to the mill personnel, at present, coating effluent at their mill represents more than 30% of the total suspended solids
in the final effluent, despite its relatively low volumetric flow. The recycling of this stream would improve the final effluent quality significantly.

Although the recovery of the concentrate at a consistency of 16.5% is feasible, in practice it would be easier for the operation to recycle a stream with a higher concentration, preferably as high as the coating mixture, which is about 50 to 60% solids. With the type of microfiltration configuration used, it is impossible to reach the desired concentration, because of flux limitations. Therefore another system would have to be used for further concentration.

Figure 3.7 shows flux versus time for both "coating normal" and "coating break" effluents. The transmembrane pressure was kept at -33.8 kPa (-10 in Hg) and the airflow injection at 0.94 L/s (2 SCFM). The temperature of the samples during testing was 16° C.

![Figure 3.7 - Flux stability of coating effluents](image)

For the "coating normal" effluent (VRF = 1), the flux decreased from 32.1 to 29.4 L/m²h after stabilization. After the normal coating effluent was concentrated two times the initial
concentration (VRF = 2), the flux was initially 25.5 L/m²h and stabilized at 22.7 L/m²h. The flux for the "coating break" effluent (VRF = 1) decreased from 22.2 to 9.3 L/m²h. After concentrating two times the initial concentration (VFR = 2), the flux decreased from 6.8 to 5.3 L/m²h. This final flux corresponds to a permeation rate of 0.157 L/m²h/ kPa (0.53 L/m²h/in Hg).

Figure 3.8 is a plot of the flux versus transmembrane pressure for the coating effluents at two different concentrations (VRF = 1 and VRF = 2). The airflow was kept constant at 0.94 L/s (2 SCFM) during filtration, and the temperature was maintained at 16 °C.

In this application fluxes were extremely small especially for the most concentrated stream, i.e., the coating break effluent. The flux reduction observed when comparing "water before testing" with "water without membrane cleaning" shows that over 40% of the flux reduction was most likely due to membrane plugging or fouling. After membrane cleaning, the flux recovered substantially. Transmembrane pressure did not have a significant effect on the permeate flux, and this occurred particularly during filtration of the most concentrated streams.

![Figure 3.8 - Flux versus pressure of coating effluents](image-url)
Figure 3.9 shows the flux versus airflow for the coating effluents. An increase in permeate flux was observed with increasing airflow. This phenomenon seems to be more significant for less concentrated streams. For example, an increase in the permeate flux of 6.3 L/m²h per L/s of applied air was observed for the "coating normal" effluent at VRF = 1, while an increase of only 1.14 L/m²h per L/s of applied air was observed for the "coating break" effluent at VRF = 2.

![Flux versus airflow of coating effluents](image)

Figure 3.9 - Flux versus airflow of coating effluents

3.3.5 Application 3: Effluent from a Bleached Chemi-thermo-mechanical Pulp (BCTMP) Mill

**Effluent Characteristics**

The effluent used in this experiment was obtained from a Canadian BCTMP mill. In this mill the liquid effluent is treated and recycled in a closed-cycle fashion. The effluent originating in the mill is clarified in a flotation unit and then sent to mechanical recompression evaporators, where the concentrate is burned in a chemical boiler and the
condensates are treated and recycled. Figure 3.10 presents a partial layout of the process, and shows the sampling point.

![Diagram](image)

Figure 3.10 - Sampling point of BCTMP mill effluent

A proposed system for this application consists of a microfiltration pre-treatment followed by a reverse osmosis unit. Because of technical problems, the reverse osmosis experiment was not carried out.

Results and Discussion

Table 3.9 presents the analytical characteristics of the BCTMP effluent. Analyses were conducted at four different solids concentration of the retentate.

This effluent has high levels of total dissolved solids (50% organic) and high conductivity. The suspended solids level of the effluent, initially at 3320 mg/L, was concentrated up to 23490 mg/L. The effect of microfiltration on COD was not significant. The COD level of the permeates was high (12000 mg/L). An increase in pH was also observed in this application, most likely due to aeration.

In order to allow mill integration the subsequent reverse osmosis experiment would have to be conducted. The quality of the permeate from the microfiltration experiment does not allow permeate recycling, i.e., dissolved solids are not retained by microfiltration.
Table 3.9 - Analytical characterization of BCTMP effluent

<table>
<thead>
<tr>
<th>STREAM</th>
<th>SOLIDS (mg/L)</th>
<th>COD (mg/L)</th>
<th>pH</th>
<th>Conduc. (µS/cm)</th>
<th>Colour (mg/L)</th>
<th>Turbid (NTU)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TSS</td>
<td>VSS</td>
<td>TDS</td>
<td>VDS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Retentate</td>
<td>3320</td>
<td>2580</td>
<td>nd</td>
<td>nd</td>
<td>12950</td>
<td>6.8</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
<td>0*</td>
<td>14330</td>
<td>6570</td>
<td>12440</td>
<td>7.9</td>
</tr>
<tr>
<td>Retentate</td>
<td>7870</td>
<td>5920</td>
<td>nd</td>
<td>nd</td>
<td>27040</td>
<td>8.4</td>
</tr>
<tr>
<td>Permeate</td>
<td>0*</td>
<td>0*</td>
<td>14360</td>
<td>6730</td>
<td>11680</td>
<td>8.6</td>
</tr>
<tr>
<td>Retentate</td>
<td>15580</td>
<td>11290</td>
<td>nd</td>
<td>nd</td>
<td>37520</td>
<td>8.0</td>
</tr>
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<td>0*</td>
<td>14350</td>
<td>6470</td>
<td>12620</td>
<td>8.7</td>
</tr>
<tr>
<td>Retentate</td>
<td>23490</td>
<td>16590</td>
<td>nd</td>
<td>nd</td>
<td>60400</td>
<td>8.2</td>
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<tr>
<td>Permeate</td>
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<td>0*</td>
<td>13370</td>
<td>6660</td>
<td>11760</td>
<td>8.4</td>
</tr>
</tbody>
</table>

0* = assumed to be very small since the membrane nominal pore size is 0.2 µm
nd = not determined

Figure 3.11 plots flux versus time for the BCTMP effluent at different concentrations. The transmembrane pressure was kept at -33.8 kPa (-10 in Hg) and the airflow at 0.94 L/s (2 SCFM). The temperature of the sample during testing was 16°C.

For effluent with VRF = 1, the flux decreased from 30.1 to 16.1 L/m²h after stabilization. For effluent with VRF = 8, the flux stabilized at 8.5 L/m²h. This final flux represents a rate of 0.251 L/m²h/kPa (0.85 L/m²h/in Hg).
Figure 3.11 - Flux stability of BCTMP effluent

The flux versus transmembrane pressure is shown in Figure 3.12. The airflow was kept constant at 0.94 L/s (2 SCFM) during the effluent filtration, and the temperature was maintained at 16°C. In this application fluxes were extremely small, and transmembrane pressure did not have a significant effect on flux, particularly during filtration of the most concentrated stream.

After membrane cleaning, the flux did not recover completely as in the previous applications. This is probably due to a significant internal membrane fouling. It is important to note that the cleaning procedures were the same for every application.

Figure 3.13 shows the flux versus airflow for the BCTMP effluent. For this range of tested airflows, the permeate flux increased at a rate of approximately 1.26 L/m²·h per L/s of air.
Figure 3.12 - Pressure versus flux of BCTMP effluent

Figure 3.13 - Airflow versus flux of BCTMP effluent
3.3.6 Application 4: Effluent from a Board Mill Using Recycled Old Corrugated Container (OCC)

Effluent Characteristics
The effluent used in this application was obtained from a Canadian linerboard mill using old corrugated cardboard (OCC). A schematic layout of the process including the sampling point is presented in Figure 3.14. At present, a portion of the whitewater is recycled to the pulping unit, and a portion is discharged as wastewater into a municipal wastewater treatment system.

![Schematic Layout of Linerboard Mill and Sampling Point](image)

Figure 3.14 - Partial Schematic Layout of Linerboard Mill and Sampling Point

System Start-up
Twenty liters of activated sludge from the Burlington municipal treatment facility were put into the reactor and kept aerated. Enough effluent was fed in daily to keep bio-activity. After three weeks the system was stable, and from then on it was fed continuously with an effluent flow of 2 mL/min, equivalent to a load of approximately 0.3 kg COD/m³d. This load was increased gradually over a 40-day period up to 30 mL/min, which provided a load of 4.3 kg COD/m³d and a hydraulic retention time (HRT) equal to 19 hours.
After increasing the feed to 4 mL/min, a pH increase from 7 to 9 was observed. Sulfuric acid was added automatically to the reactor to keep the pH at 7.5. Approximately 0.42 L of the 5% sulfuric acid was consumed per kg COD. After increasing the feed up to 16 mL/min, excessive foaming occurred. Occasionally, adding de-foamer to the reactor solved this problem. During the experiment, the level of dissolved oxygen in the reactor was maintained at a concentration higher than 4 mg/L. The concentration of VSS in the reactor increased to 8000 mg/L, and the oxygen uptake ratio, OUR, was around 54 mg/L/h after bio-mass acclimatization was achieved.

Results and Discussion

Table 3.10 shows the characteristics of the effluent, permeate, and sludge and the overall removal efficiency of the bio-reactor. These data represent an average of values collected after stabilization of the system, during the last two weeks of the tests. The removal efficiencies for COD and BODs were 95% and 99%, respectively. The removal efficiency was extremely good, showing complete acclimatization of the bio-mass to the effluent. This enabled a high biodegradation of the dissolved organic material present in the effluent. Removal of only 20% of the true colour was observed. As expected, suspended solids were completely removed by microfiltration.

Table 3.10 - Analytical characterization of the bio-reactor

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>SOLIDS (mg/L)</th>
<th>COD (mg/L)</th>
<th>BOD (mg/L)</th>
<th>Colour (mg/L)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TSS</td>
<td>SSV</td>
<td>TDS</td>
<td>SLUDGE</td>
<td>nd</td>
</tr>
<tr>
<td>FEED</td>
<td>127</td>
<td>123</td>
<td>3000</td>
<td>3500</td>
<td>2000</td>
</tr>
<tr>
<td>PERMEATE</td>
<td>0*</td>
<td>0*</td>
<td>1250</td>
<td>180</td>
<td>12</td>
</tr>
<tr>
<td>REMOVAL (%)</td>
<td>100</td>
<td>100</td>
<td>58</td>
<td>95</td>
<td>99</td>
</tr>
</tbody>
</table>

0* = assumed to be very small since the membrane nominal pore size is 0.2 μm
nd = not determined

A reduction of more than 90% in COD and BOD levels and the complete removal of suspended solids allow the recycling of this stream to the process. According to the mill
personnel a permeate with TDS concentration less than 1500 mg/L and TSS concentration less than 100 mg/L could be used as chemical dilution water, seal water for pumps, agitators and in wet end showers. As in any biological system, sludge is produced and needs to be handled and disposed of. However, the bio-reactor works in an extended aeration mode, which minimizes sludge production, and does not require an extra sludge unit.

Membrane tests were performed with two different hydraulic retention times (HRT): first with an HRT of 29 hours and then with an HRT of 19 hours. Figure 3.15 shows the flux versus time at a transmembrane pressure of -33.8 kPa (-10 in Hg), an airflow of 0.94L/s (2 SCFM) and a temperature of 35°C.

![Flux stability of the bio-mass](image)

During the HRT=29 h experiment, the initial flux of 120.5 L/m²h decreased over 42 hours to 90.0 L/m²h. The stabilized flux corresponds to a rate value of 2.65 L/m²h/kPa (9.0 L/m²h/in Hg). During the experiment with HRT=19 h, the flux decreased from 100 to 52.6 L/m²h, after which it stabilized. It is important to mention that the same membrane was used for both tests, and the membranes were not cleaned between experiments. As expected,
the phenomenon of fouling during the first test had a significant effect on the second experiment. This was probably the main reason for the observed decrease in flux during the experiment at HRT=19 h, as compared to the first trial with HRT=29 h.

During the membrane quality check, the flux of de-ionized water was measured at different transmembrane pressures. The same test was carried out for the bio-reactor with HRT=19 h and with HRT=29 h. The results of flux versus transmembrane pressure from these experiments are shown in Figure 3.16.

![Figure 3.16 - Pressure versus flux of bio-mass](image)

An increase in the transmembrane pressure had a significant effect on the flux. During effluent filtration with HRT=29h, an increase of 61% permeate flux was observed after increasing the transmembrane pressure from -16.9 to -33.8 kPa (-5 to -10 in Hg). An increase of only 21% was observed after increasing the pressure from -50.8 to -67.7 kPa (-15 to -20 in Hg). During effluent filtration with HRT=19h, an increase of 52% permeate flux was observed after increasing the transmembrane pressure from -16.9 to -33.8 kPa (-5 to -10 in Hg). An increase of only 11% was observed after increasing the pressure from -50.8 to
-67.7 kPa (-15 to -20 in Hg). This reduced increase at higher transmembrane pressure can be explained by an increase in the cake resistance as the transmembrane pressure increases.

Figure 3.17 shows the airflow versus the permeate flux during the experiment. The effect of the airflow on the permeate flux was small compared with the results obtained for the same test with the other applications.

![Figure 3.17 - Flux versus airflow of bio-mass suspension](image)

3.4 Conclusions

(i) Application 1: Old Newsprint (ONP) De-inking Effluent Containing Flexographic Ink

The treatment of this effluent using microfiltration proved to be technically feasible, and it has good potential for industrial application.

(ii) Application 2: Effluent from Paper Coating Operation

The removal of suspended solids by microfiltration led to good quality permeates. The filterability of these effluents was poor, especially for the “coating break” effluent. The
recovery of the concentrate at a consistency of 16.5% is possible. However, considering the flux limitation of this stream, Application 2 was not considered appropriate for this type of membrane configuration.

(iii) Application 3: Effluent from Bleached Chemi-thermo-mechanical Pulp (BCTMP) Mill

The filterability of this effluent was very poor, and the membrane did not completely recover its water flux capacity after cleaning. The reverse osmosis experiment has not been carried out, and an analysis of mill integration is difficult without these results. However, the flux limitation encountered in the microfiltration experiments suggests that this hollow-fibre configuration is not appropriate to treat this effluent.

(iv) Application 4: Membrane Bio-Reactor Treatment of OCC Recycling Effluent

The effluent from the OCC mill has high levels of COD (3500 mg/L) and BOD$_5$ (2000 mg/L). A two-month period was necessary to acclimatize a population of microorganisms to the effluent. An overall removal rate of 95% of COD and 99% of BOD$_5$ was achieved after stabilization of the system. The permeate fluxes obtained during the filtration tests were high. The recycling of the treated effluent to the mill process is viable.

From the results of the screening tests, two applications using microfiltration were identified and have good potential industrial applicability. They were:

(i) Microfiltration treatment of ONP effluent containing flexographic ink;
(ii) Membrane bio-reactor treatment of OCC effluent.

This part of the work contributed new information about the general use of membranes in the pulp and paper industry and about the effectiveness of the membrane system tested.

The next part of the thesis consists of the development of a model for flux prediction in microfiltration. The bio-reactor system is much more complex and too difficult to model at this time.
4. DEVELOPMENT OF A MODEL FOR FLUX PREDICTION IN HIGH-SHEAR MICROFiltrATION SYSTEMS

4.1 Introduction

In the previous chapter, a preliminary evaluation of microfiltration of pulp and paper effluents was undertaken using a high-shear submerged hollow-fibre membrane configuration. In this chapter a development of a model for flux prediction for this particular membrane configuration is described. A model that enables the prediction of flux in microfiltration systems can be useful in designing and scaling up a given system. In order to complete this task, a literature review was undertaken (as presented in Chapter 2). The observation of the flux behavior in the preliminary tests, as shown in the Chapter 3 was useful in choosing the type of model that would be suitable for this particular system.

Significant progress has been made during the last few decades in understanding the dynamics of mass transfer in membrane separation processes. The models proposed for microfiltration have focused on concentration polarization, and on the different mechanisms of back-transport of particles from the membrane surface.

As discussed in the literature review, the mechanisms of back-transport of particles from the membrane surface can be divided in two categories: diffusion and hydrodynamics. Some of the models proposed in the literature are primarily based on diffusion mechanisms accounting for the back-transport of solute away from the membrane surface; these include the concentration-polarization model and the shear-induced diffusion model (Belfort et al., 1994; Mulder, 1991; Eckstein et al., 1977; Leighton and Acrivos, 1986; Zydney and Colton, 1986). The inertial lift model considers the lateral migration of particles due to inertial lifting that augments the back diffusion of particles (Belfort et al., 1994). Although these models consider diffusion as the main mechanism of the back-transport of particles in cross-flow ultrafiltration and microfiltration systems, some researchers assume that in systems where hydrodynamic shear forces take place at the membrane surface, the back-transport of particles is caused mainly by these forces, and diffusion can be neglected (Schulz and Ripperger, 1989; Ravazi and Harris, 1996).
Because the system we are working with is based on high-shear stresses at the membrane surface, a semi-empirical model is proposed assuming that non-diffusive transport phenomena are the main mechanisms for the back-transport of particles from the membrane surface to the bulk solution during filtration. The industry is often using reverse filtration as a mechanism to keep the membrane as clean as possible. So the model also takes into account the influence of backflushing.

Numerical simulations of this new model were carried out, and the influence of each variable in the model was analyzed. The model conforms to the anticipated behavior of its parameters.

**4.2 Model Development**

**4.2.1 Reverse Filtration Model**

As presented earlier, reverse filtration is a common practice in industrial-scale microfiltration systems. There are two modes of operation for reverse filtration: backpulsing refers to high-frequency reverse filtration, and backflushing refers to low-frequency reverse filtration (Redkar, 1994). Backflushing is accomplished by the counter-flow of permeate through the membrane produced by changing the direction of transmembrane pressure. This cleaning operation is cyclic, and it is usually performed before the steady-state or pseudo-steady-state flux condition is achieved.

Redkar proposed a backflushing model, which assumes that no internal fouling occurs and the resistance of the membrane during forward filtration and during reverse filtration are the same (Redkar, 1994). The model assumes that the flux is completely recovered after backflushing. As shown earlier in Chapter 2, the average flux in one cycle is equal to the total permeate volume per unit of membrane area collected during forward filtration minus the total filtrate lost during reverse filtration, divided by the sum of the forward and reverse filtration time as in
\[
\langle J \rangle = \frac{\int_0^t J\, dt - \beta J_0 t_b}{t_f + t_b},
\]

where \( \langle J \rangle \) is the average permeate flux per cycle, \( \int_0^t J\, dt \) the filtrate volume per unit area collected during forward filtration, \( \beta J_0 t_b \) the filtrate volume per unit area lost during reverse filtration, and \( t_f \) and \( t_b \) the forward and the reverse filtration time respectively.

Most of the existing models have been developed to predict permeate flux after the pseudo-steady state condition is achieved. However, a model that includes backflushing as a mechanism for minimizing concentration-polarization and fouling should be based on the transient or time-dependent flux.

4.2.2 Transient Flux

To describe the behavior of the flux in the transient region, Romero and Davis developed a theoretical model of cross-flow microfiltration that considers the time-dependent decline of permeate flux (Romero and Davis, 1990; Davis, 1992c). The model is based on the shear-induced hydrodynamic diffusion. It considers that the initial flux decline in cross-flow filtration due to cake buildup could be approximated by modeling it as in dead-end filtration.

In dead-end filtration, the flux is governed by Darcy's law (resistance model) and the cake resistance is determined by a mass balance at the edge of the growing cake as presented earlier in Chapter 2, and is summarized in Figure 4.1.
Dead-end Filtration

Mass Balance:

\[
\text{FLUX OF PARTICLES INTO THE SURFACE OF THE CAKE} = \text{BUILDUP OF PARTICLES IN THE CAKE}
\]

\[
\left( J + \frac{d\delta c}{dt} \right) \cdot \Phi_b = \Phi_c \cdot \frac{d\delta c}{dt}
\]

Figure 4.1 - Particle mass balance in dead-end filtration, where \( J \) is the permeate flux, \( \Phi_b \) is the solid volume fraction in the suspension, \( \Phi_c \) is the solid volume fraction in the cake and \( \delta_c \) is the cake layer thickness.

As presented earlier in Chapter 2, the transient flux for dead-end filtration was derived and given by

\[
J(t) = J_0 \left[ 1 + \frac{2 t \cdot \hat{R}_c \cdot \Phi_b \cdot \Delta P}{(\Phi_c - \Phi_b) \cdot \eta_o \cdot R_m^2} \right]^{-\frac{1}{2}},
\]

where \( J_0 \) is the initial flux, \( \Phi_b \) is the solid volume fraction in the suspension, \( \Phi_c \) is the solid volume fraction in the cake, \( R_m \) is the membrane resistance, \( \hat{R}_c \) is the specific cake resistance, \( \Delta P \) is the transmembrane pressure differential, and \( \eta_o \) is the dynamic viscosity.

4.2.3 A New Expression for the Transient Flux

In high-shear systems, however, it is observed that the high-shear stresses have a significant effect on the transient flux. When high shear stresses are applied to the liquid at the membrane surface, the decrease of flux with time due to the build-up of cake is less than that observed in absence of shear stress. This behavior suggests a mass balance for the high-shear system different from that of dead-end filtration. When high-shear stresses are applied,
a term must be included that expresses the transport away from the membrane due to these forces as shown in Figure 4.2.

![Diagram](image)

**Mass Balance:**

| FLUX OF PARTICLES INTO THE SURFACE OF THE CAKE | FLUX OF PARTICLES AWAY FROM THE SURFACE DUE TO TURBULENCE | BUILDUP OF PARTICLES IN THE CAKE |

Figure 4.2 - Particle mass balance in high-shear filtration systems

Considering that the back-transport of particles into the bulk stream is proportional to the velocity gradient on the membrane surface and also proportional to the cake layer thickness, as described by Schulz and Ripperger (1989), the second term of the particle mass balance of Figure 4.2 becomes

\[
\text{FLUX OF PARTICLES AWAY DUE TO TURBULENCE} = k_1 \cdot \delta_c \cdot \frac{dU}{dy},
\]

where \( \delta_c \) is the cake layer thickness, \( \frac{dU}{dy} \) the velocity gradient, and \( k_1 \) a constant.

The constant \( k_1 \) is an intrinsic characteristic of the particles. Its value will depend on the particle size and particle size distribution. It also will depend on other characteristics of the particles such as the potential for adhesion, which is the capacity of the particles to stick at the membrane surface. The new particle mass balance is then

\[
\left( J + \frac{d \delta_c}{dt} \right) \cdot \Phi_b - k_1 \cdot \left( \frac{dU}{dy} \right) \cdot \delta_c = \Phi_c \cdot \frac{d \delta_c}{dt}.
\]
Assuming that the cake resistance $R_c$ is equal to the specific cake resistance times the cake thickness, permeate flux was described by the resistance model as previously shown (Equation 2.11 of Chapter 2).

\[ J = \frac{\Delta P}{\eta_o \left( R_m + \hat{R}_c \cdot \delta_c + R_f \right)} \]  

(2.11)

where $J$ is the permeate flux, $\Delta P$ is the transmembrane pressure differential, $\eta_o$ is the dynamic viscosity of the permeate, $R_m$ and $R_f$ are the membrane and fouling resistances.

Using Darcy’s law and assuming that the membrane resistance and the fouling resistance are negligible compared with the cake layer resistance, the flux can be written as

\[ J = \frac{\Delta P}{\eta_o \cdot (\hat{R}_c \cdot \delta_c)} \]  

(4.3)

where $\hat{R}_c$ is the specific cake resistance, and $\delta_c$ is the cake layer thickness.

Combining Equation 4.3 and the particle mass balance given in Equation 4.2, leads to

\[ \frac{d\delta_c}{dt} = \frac{\Delta P \cdot \Phi_b}{\eta_o \cdot \hat{R}_c \cdot \delta_c (\Phi_c - \Phi_b)} - \frac{k_1 \cdot dU}{dy \cdot \delta_c} \]  

(4.4)

Given that the cake thickness is zero at the beginning of the filtration, i.e., $\delta_c = 0$ at $t=0$; assuming that the transmembrane pressure $\Delta P$ and the velocity gradient $dU/dy$ are kept constant over the period of filtration; and also assuming that $\hat{R}_c$ is constant, i.e., no significant change in cake compression occurs, then Equation 4.4 can be integrated to yield

\[ \delta_c(t) = \left( \frac{\Delta P \cdot \Phi_b}{\hat{R}_c \cdot k_1 \cdot \eta_o \cdot dU/dy} \right) \left( 1 - e^{\frac{-2k_1 \left( \frac{dU}{dy} \right) t}{(\Phi_c - \Phi_b)}} \right) \]  

(4.5)

From Equation 4.5 it can be noted that the cake thickness $\delta_c$ tends to increase with an increase in the transmembrane pressure, $\Delta P$, and also with an increase in the solid volume fraction of the suspension, $\Phi_b$. In contrast, the cake thickness decreases as the velocity gradient or the constant $k_1$ increases.

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Combining Equations 2.11 and 4.5, it is possible to write a new time-dependent expression for permeate flux as in

\[
J(t) = \frac{\Delta P}{\sqrt{\frac{\Delta P \cdot \Phi_b \cdot \eta_o \cdot \hat{R}_c}{k_1 \cdot \left(\frac{dU}{dy}\right)} \cdot \left[1 - e^{-\frac{2 \cdot k_1 \cdot \left(\frac{dU}{dy}\right)}{\Phi_c - \Phi_b}}\right] + \eta_o \left(R_m + R_f\right)}}.
\]

(4.6)

Note that the membrane and the fouling resistances are now considered. Although these resistances are considerably smaller than the cake resistance, they have an important effect on flux at the beginning of the filtration, when the cake is not yet formed. Equation 4.6 is the general expression that provides the permeate flux over time.

For a Newtonian fluid

\[
\frac{dU}{dy} = \frac{\tau_w}{\eta},
\]

(4.7)

where \(\tau_w\) is the shear stress at membrane wall and \(\eta\) is the suspension viscosity.

Assuming a Newtonian fluid and considering that the suspending colloids and particles may significantly change the viscosity of the suspension, then Equation 4.6 becomes

\[
J(t) = \frac{\Delta P}{\sqrt{\frac{\Delta P \cdot \Phi_b \cdot \eta_o \cdot \hat{R}_c}{k_1 \cdot \tau_w} \cdot \left[1 - e^{-\frac{2 \cdot k_1 \cdot \tau_w \cdot t}{\Phi_c - \Phi_b}}\right] + \eta_o \left(R_m + R_f\right)}}.
\]

(4.8)

It is important to consider that the microfiltration configuration used in this work is unique, and that the effects of the air bubbles on the membrane surface are difficult to estimate. The model takes into account the effect of shear stress on the membrane surface but does not distinguish the source of this shear stress. A correlation between shear stress and airflow might be established when airflow is expressed in terms of superficial gas velocity. Earlier investigators have suggested that there exist an average shear rate \(\dot{\gamma}_{av}\) in a bubble column,
which is proportional to the superficial gas velocity $U_{sg}$ supplied to the column (Nishikawa, M. et al., 1977; Allen and Robinson, 1991). Thus

$$\dot{y}_{av} = CU_{sg}, \quad (4.9)$$

where $C$ is a constant. Typically $C$ is equal to 5000 for $U_{sg}$ reported in m/s. Superficial gas velocity ($U_{sg}$) can be determined by dividing the air volumetric flow rate by the cross-section area of the tank.

In the development of the model, the specific cake resistance $\hat{R}_c$ was considered to be constant over time, which means that there is no significant change in cake compression. This is valid when the particles that form the cake are incompressible, so the cake porosity and its resistance are independent of the applied transmembrane pressure differential. However, many substances that form the cake are compressible, so Porter suggests that the effect of cake compressibility can be estimated by

$$\hat{R}_c = \alpha_0 \cdot \left(\Delta P\right)^s \cdot \rho_s \cdot \Phi_c, \quad (4.10)$$

where $\alpha_0$ is a constant related to size and shape of particles, $s$ the cake compressibility, and $\rho_s$ the mass density of solids comprising the cake (Porter, 1977).

A combination of Equations 4.8 and 4.10 leads to

$$J(t) = \frac{\Delta P}{\sqrt{\frac{\Delta P^{1+s}}{k_1 \cdot \tau_w} \cdot \Phi_b \cdot \eta \cdot \alpha_0 \cdot \rho_s \cdot \Phi_c \cdot \left(1 - e^{\frac{-2 \cdot k_1 \cdot \tau_w \cdot t}{\eta}}\right) + \eta_o \left(R_m + R_f\right)}}. \quad (4.11)$$

From Equation 4.11 it can be noticed that when assuming the membrane and fouling resistances negligible compared to the cake resistance, the transient filtration flux is directly proportional to the square root of the transmembrane pressure drop $\Delta P$ for incompressible cakes ($s \rightarrow 0$). Otherwise, for highly compressible cake ($s \rightarrow 1$), the transmembrane pressure drop $\Delta P$ will have an insignificant effect on flux. The behavior of transmembrane pressure described by Darcy’s model indicates that flux is directly proportional to the applied pressure gradient. However, other investigators have found that this relation is frequently violated since the increase in the applied pressure gradient also tends to increase the cake
resistance due to deposition of material on the membrane surface (Lojkine et al., 1992; Wakeman and Tarleton, 1991). From Equation 4.10 it is evident that the forward filtration flux is inversely proportional to the square root of the mass density of solids comprising the cake ρs.

The suspension viscosity η can be substituted by the expression given by Hiemenz (1986) that relates the viscosity of the suspension to the volume fraction of solids as in

\[ η = η_o \cdot \left(1 + 2.5Φ_b + k_o φ_b^2 \right) \quad (4.12) \]

where φb is the solid volume fraction of the suspension, and ko a constant which is equal to 10 for spheres.

The backflushing model proposed by Redkar was given by Equation 2.40. The integral

\[ \int_0^t J(t) \, dt \] using J(t) as in Equation 4.10 is complex but can be evaluated numerically.

4.2.4 Flux Decline due to Fouling

As discussed earlier, Equation 2.40 gives the average permeate flux per cycle, and it does not take into consideration the decrease in the flux after each cycle due to irreversible fouling or due to external fouling that is not completely removed by backflushing. In order to account for the effect of irreversible fouling Equation 2.40 can be modified by multiplying it to an empirical "recovery coefficient" lower than one that describes this flux decrease. So it can be written that

\[ \langle J_n \rangle = R(n) \cdot \langle J_1 \rangle, \quad (4.13) \]

where \( \langle J_n \rangle \) is the average permeate flux in nth cycle, \( \langle J_1 \rangle \) is the average permeate flux in the first cycle, and \( R(n) \) the recovery coefficient.

4.3. Numerical Simulations

In microfiltration separation processes, several variables may significantly affect the permeate flux. These variables can be divided into three categories: the operating conditions,
the suspension characteristics, and the membrane characteristics. The main operating conditions considered in this analysis of the model are the forward and reverse transmembrane pressures ($\Delta P_f$, $\Delta P_b$) and the shear stress ($\tau_w$) applied to the membrane surface. The solid volume fraction of the suspension $\Phi_b$ and the fluid viscosity $\eta_0$ are the parameters related to the suspension characteristics. The cake compressibility ($s$) and the solid volume fraction of the cake ($\Phi_c$), the constants $k_i$ and $\alpha_o$ are also directly related to the suspension characteristics.

Numerical simulations of the transient forward flux given by Equation 4.11 were carried out using nominal values of the constants obtained from the literature as follows. For spherical particles, the void fraction of a randomly packed cake is $\varepsilon_c = 0.4$, so the solid volume fraction of the cake $\Phi_c$ was assumed to be $(1-\varepsilon_c) = 0.6$ (Davis, 1992b). The dynamic viscosity of the fluid was taken to be $\eta_0 = 0.001$ kg/ms, and the dynamic viscosity of the suspension $\eta = 0.001054$ estimated indirectly by using Equation 4.12 and assuming the constant $k_o$ is equal to 10 for spherical particles (Davis, 1992b). The solid volume fraction of the suspension $\Phi_b$ is set equal to 0.02; the mass density of the cake solids $\rho_b$ is set equal to 3000 kg/m$^3$.

The shear stress at the membrane $\tau_w$ wall was set to be 10 kg/ms$^2$; the dimensionless constant $k_i$ is an intrinsic characteristic of the particles, which is assumed to have a value of $1 \times 10^6$. The constant $k_i$ is always multiplied by the shear stress $\tau_w$ in the model. The values of $k_i$ and $\tau_w$ were chosen randomly after a few computer simulation trials.

The cake compressibility $s$ varies from 0 to 1 (Belfort et al., 1994), and for the purpose of this study it was assumed to be 0.5. The constant $\alpha_o$ which is related to the size and shape of the particles was taken to be $7.64 \times 10^{10}$ ms$^2$/kg, estimated indirectly by using Equation 4.9, and assuming the specific cake resistance calculated using Carman-Kozeny Equation shown in Equation 2.7, and given by

$$\hat{R}_c = \frac{R_c}{\delta_c} = \frac{K \cdot (1-\varepsilon_c)^2 \cdot S_c \cdot \varepsilon_c^2}{\varepsilon_c^3},$$

(2.7)
where $\varepsilon_c$ is the void fraction of the cake, $K$ is a constant and $S_c$ the specific surface area. For rigid spherical particles of radius $r$, the specific surface area is $S_c = 3/r$, and the constant $K$ is reported by Grace to have a value of 5.0 (Grace, 1953).

Considering that the particle radius is equal to 0.1 $\mu$m and using these values in Equation 2.7, the specific cake resistance is estimated to be equal to $R_c = 2.53 \times 10^{16}/\text{m}^2$. The membrane resistance and the fouling resistance were assumed to be equal 6.3 $\times$ $10^{11}$/m each. The time in this analysis was set to be 360 seconds. Table 4.I summarizes the values of the variables considered for the numerical simulation.

4.3.1 Effect of Permeate Viscosity $\eta_0$ on Flux

The effect of the permeate viscosity, $\eta_0$, on flux is shown in Figure 4.3. Flux decreases with an increase in viscosity. In the low viscosity region, small changes in viscosity cause significant changes in flux, while in the high viscosity region, the changes are smaller. For example, increasing the permeate viscosity from 0.0004 kg/ms to 0.0024 kg/ms a decrease of 80 L/m$^2$h in flux is observed, while increasing the viscosity $\eta_0$ from 0.0024 kg/ms to 0.0074 kg/ms a decrease of only 12 L/m$^2$h in flux is observed. The behavior of flux over time for different values of viscosity is presented in Figure 4.4. The model predicts that the steady-state flux is reached in less time for high values of viscosity.
Table 4.1 - Values of the variables considered in the model simulations

<table>
<thead>
<tr>
<th>Variable</th>
<th>$\Delta P$ (kPa)</th>
<th>$\eta_0$ (kg/ms)</th>
<th>$\phi_c$</th>
<th>$\phi_b$</th>
<th>$k_o$</th>
<th>$\rho_s$ (kg/m$^3$)</th>
<th>$\tau_w$ (kg/ms$^2$)</th>
<th>$k_l$</th>
<th>$s$</th>
<th>$\alpha_o$ (ms$^2$/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal value</td>
<td>33.8</td>
<td>0.001</td>
<td>0.6</td>
<td>0.02</td>
<td>10</td>
<td>3000</td>
<td>10</td>
<td>$1 \times 10^{-6}$</td>
<td>0.5</td>
<td>7.64 x 10$^{10}$</td>
</tr>
<tr>
<td>Range of set values</td>
<td>0 - 200</td>
<td>0 - 0.009</td>
<td>0.1 - 0.8</td>
<td>0 - 0.2</td>
<td>-</td>
<td>-</td>
<td>0 - 200</td>
<td>-</td>
<td>0 - 1</td>
<td>-</td>
</tr>
</tbody>
</table>
Figure 4.3 - Flux as a function of permeate viscosity $\eta_0$ as predicted by the model

Figure 4.4 - Flux as a function of time at different values of permeate viscosity $\eta_0$ as predicted by the model
4.3.2 Effect Solid Volume Fraction in the Suspension $\phi_s$ on Flux

Flux decreases with increasing solid volume fraction in the suspension, as shown in Figure 4.5. This is in agreement with other models (Lojkine et al., 1992). Figure 4.6 shows flux as a function of time at different values of solid volume fraction of the suspension $\phi_s$ as predicted by the model.

Figure 4.5 - Flux as a function of solid volume fraction of the suspension $\phi_s$ as predicted by the model

Figure 4.6 - Flux as a function of time at different values of solid volume fraction of the suspension $\phi_s$ as predicted by the model
4.3.3 Effect Solid Volume Fraction in the Cake $\Phi_c$ on Flux

The effect of the solid volume fraction in the cake $\Phi_c$ on flux is shown in Figure 4.7. Increases in $\Phi_c$ results in decreases on flux. The model also predicts that flux tends to reach steady-state faster as the solid volume fraction in the cake $\Phi_c$ increases (Figure 4.8).

![Figure 4.7 - Flux as a function of solid volume fraction of the cake $\Phi_c$ as predicted by the model](image1.png)

![Figure 4.8. Flux as a function of time at different values of the cake solid volume fraction $\Phi_c$ as predicted by the model](image2.png)
4.3.4 Effect of Transmembrane Pressure $\Delta P$ on Flux

Figure 4.9 shows the behavior of flux versus transmembrane pressure $\Delta P$ for different values of solid volume fraction in the suspension $\Phi_b$. It can be noted that an increase in flux is observed with increasing transmembrane pressure. The effect of transmembrane pressure $\Delta P$ on flux is more significant for lower values of solid volume fraction in the suspension $\Phi_b$. For example, increasing $\Delta P$ from 20 kPa to 80 kPa at $\Phi_b = 0.02$ an increase in flux of 42.5 L/m²h is observed, while for the same increase in $\Delta P$ at $\Phi_b = 0.04$ an increase of only 30 L/m²h is observed.

![Figure 4.9](image)

**Figure 4.9 -** The effect of transmembrane pressure on flux for different solid volume fractions of the suspension as predicted by the model

4.3.5 Effect of Shear Stress $\tau_w$ on Flux

Figure 4.10 shows the behavior of flux versus shear stress at the membrane surface $\tau_w$. Increases in the shear stress results in higher permeate fluxes. A rapid increase in flux is
observed until a certain value of the applied shear stress, after which it increases moderately. This is in agreement with other models (Lojkine et al., 1992).

Figure 4.11 shows the behavior of flux versus time for different values of shear stress applied. Figure 4.12 illustrates the behavior of flux versus transmembrane pressure $\Delta P$ for different shear stresses applied. Flux increases with increasing of the transmembrane pressure. Flux also increases with an increase in applied shear stress, as observed previously in Figure 4.11. It is interesting to note that the curves presented in Figure 4.12 suggest that the effect of $\Delta P$ on flux is more significant when higher shear stresses are applied. For example, increasing $\Delta P$ from 20 kPa to 80 kPa at $\tau_w = 20$ kg/ms$^2$ an increase in flux of 56 L/m$^2$h is observed, while for the same increase in $\Delta P$ at $\tau_w = 10$ kg/ms$^2$ an increase of only 42.5 L/m$^2$h is observed. It is likely that the increase in the shear stress facilitates the action of the transmembrane pressure $\Delta P$.

![Graph of Flux vs. Shear Stress](image)

Figure 4.10 - Flux as a function of shear stress $\tau_w$ as predicted by the model
Figure 4.11 - Flux as a function of time for different values of shear stress as predicted by the model.

Figure 4.12 - The effect of transmembrane pressure on flux for different values of shear stress as predicted by the model.
4.4 Summary

A model has been developed using Darcy’s law and the particle mass balance of solutes at the surface of the membrane. It considers the shear stress as the main mechanism of back-transport of particles from the cake. The following assumptions have been made in the derivation of the model:

(i) Constant fluid properties with respect to time
(ii) Constant shear stress with respect to time
(iii) Constant specific cake resistance with respect to time
(iv) Negligible solute in the permeate
(v) Negligible back diffusion

Although several important characteristics related to the suspension such as particle size, particle size distribution, particle shape are not explicit in the model, their effect appear indirectly in the constants such as the cake compressibility $s$ and $k$. 

Numerical simulations of this new model conform to the anticipated behaviour of its parameters. The model predicts that:

(i) Assuming the membrane and fouling resistance negligible compared to the cake resistance, flux is directly proportional to the square root of the transmembrane pressure $\Delta P$ for incompressible cakes ($s \to 0$). Otherwise, for highly compressible cake ($s \to 1$), the transmembrane pressure drop $\Delta P$ will have an insignificant effect on flux.

(ii) Flux decreases with an increase in permeate viscosity $\eta_0$. In the low viscosity region, small changes in viscosity cause significant changes in flux, while in the high viscosity region, the changes are smaller.

(iii) Increases in the solid volume fraction in the suspension $\Phi_b$ result in decreases of the flux, and the effect of transmembrane pressure $\Delta P$ on flux is more significant for lower values of $\Phi_b$.

(iv) Increases in solid volume fraction in the cake $\Phi_c$ result in decreases of the flux. The model also predicts that flux tends to reach steady-state faster as the solid volume fraction in the cake $\Phi_c$ increases.
(v) Flux increases as shear stress $\tau_w$ increases. A rapid increase in flux is observed until a certain value of the applied shear stress, after which it increases moderately.

(vi) The effect of transmembrane pressure $\Delta P$ on flux is more significant when higher shear stresses are applied.
5. EXPERIMENTAL DEVELOPMENT

5.1 Introduction

In the previous chapter, a model was proposed for permeate flux prediction in high-shear microfiltration systems. The model is based on the assumption that non-diffusive transport phenomena are the main mechanisms for the back-transport of particles from the membrane surface to the bulk solution during microfiltration when high shear stresses are applied on the membrane surface. It also considered the use of reverse filtration, i.e., backflushing, to control and to minimize concentration polarization and fouling. An expression for the transient flux was derived from the mass balance of particles on the membrane surface and the resistance model. This equation was substituted into Redkar's backflushing model.

The objective of this chapter is to validate the model qualitatively by laboratory experiments. It would be difficult trying to verify the model using the ONP effluent, because of the complexity of determining some of the required variables for an effluent suspension that is composed of many substances. Therefore, it would be more appropriate to test the model using synthetic suspensions where the variables could be easily determined. Therefore, six suspensions were tested: flexographic ink \((\phi_b = 0.005 \text{ and } 0.01)\), calcined alumina \((\phi_b = 0.02 \text{ and } 0.03)\) and yeast \((\phi_b = 0.01 \text{ and } 0.02)\). In each case, the main characteristics of the filtration were determined. The effect of transmembrane pressure and the effect of airflow (shear stress) on the permeate flux during forward filtration was studied.

Flexographic ink was chosen for its industrial importance in connection to the chosen application as described in Chapter 3. Calcined alumina is composed basically of aluminum oxide and it was chosen because of its uniform particle size distribution. Yeast was also chosen because of its uniform particle size distribution, and because of its fouling characteristics as a biological material.

The equipment used for the tests was similar to the one used previously in Chapter 3, except for its size. The membrane fibres length were deliberately chosen to be the same as the ones
used in industrial-scale modules. So the results would be closer to what is expected in an industrial module.

5.2 Materials

5.2.1 Equipment Design

A submerged microfiltration hollow-fiber module was used for the filtration experiments. This system consists of a bundle of hollow-fiber microfiltration membranes submerged in a tank, into which the suspension is introduced. Each hollow-fiber was approximately 2 mm outside diameter and 1.5 m long. The total membrane area was 2 m². Membrane fibers were assembled in such a way that they were free to move. Transmembrane pressure was provided by an external pump, which created a vacuum inside the fiber lumen, generating a flux from the outside to the inside of the hollow fiber. Aeration was used to promote turbulence to minimize concentration polarization and fouling. Air is injected using an air blower. The tank had a diameter of 0.4 m and a volume of 230 L. The system was operated in a total recycle mode, i.e., the permeate was recycled to the tank, thus keeping the concentration and volume of the suspension in the tank constant. A picture of the laboratory membrane system is presented in Figure 5.1. Polymeric hydrophilic membranes with a nominal pore size of approximately 0.1 μm were used.

The experimental apparatus design allows changes in transmembrane pressure and airflow. Backflushing is carried out by reversing the flux of the permeate. This is accomplished by changing the flux automatically using solenoid valves and a timer. A temperature controller was installed at the permeate tank to keep the temperature constant. A schematic diagram of the system operation is shown in Figure 5.2.
Figure 5.2 - Schematic diagram of system operation
5.2.2 Suspension Characterization

Three different suspensions were tested: flexographic ink, calcined alumina, and yeast.

(i) Flexographic ink suspensions
Flexographic ink suspensions were obtained by mixing de-ionized water with flexographic ink. The ink, *Arrowflex Black KZ-K075/5*, was supplied by Flint Ink. The median particle size of the flexographic ink was determined to be approximately 0.2 μm, as shown in Appendix 1, which also contains the particle size distribution. Suspensions with two different solid volume fractions were tested: \( \phi_b = 0.005 \) and 0.01. These fractions correspond to approximately 0.6 and 1.2 weight percent, respectively.

(ii) Calcined alumina suspensions
Calcined alumina suspensions were obtained by mixing de-ionized water with calcined alumina (aluminum oxide), product A-16SG from Alcoa Industrial Chemicals. The median particle size was 0.6 μm with a particle size distribution shown in Appendix 1. Suspensions with two different solid volume fractions were tested: \( \phi_b = 0.02 \) and 0.03. These fractions correspond to approximately 6.6 and 10 weight percent, respectively.

(iii) Yeast suspensions
Yeast suspensions were obtained by mixing de-ionized water with Fleischmann's dry yeast (*Saccharomyces cerevisiae*). The median particle size of the yeast was determined to be approximately 5 μm, as shown in Appendix 1. Suspensions with two different solid volume fractions were tested: \( \phi_b = 0.01 \) and 0.02. These fractions correspond to approximately 0.93 and 1.86 weight percent, respectively.

5.3 Methods

5.3.1 Membrane Cleaning and Quality Check

In order to determine the baseline conditions for the membrane used in the test, initial membrane cleaning was carried out. The tank was filled with de-ionized water, detergent
and chlorine. Typically, the membranes were soaked for 24 hours in the solution with gentle aeration. The system was then drained and rinsed with water. An initial water flux test was conducted by circulating water through the system at different transmembrane pressures and measuring the flux. No air was introduced during this test.

5.3.2 Determination of Membrane Resistance

The membrane resistance $R_m$ was determined by running de-ionized water through the clean membranes at different pressure drop. Then $R_m$ was calculated from

$$R_m = \frac{\Delta P}{\eta \cdot J_o},$$

where $J_o$ is the initial flux, $\Delta P$ is transmembrane pressure differential, and $\eta$ is the dynamic viscosity.

5.3.3 Effect of Transmembrane Pressure and Backflushing on Flux

For flexographic ink suspensions with transmembrane pressures of -16.9, -33.8 and -50.8 kPa (-5, -10 and -15 in Hg), the permeate flow was measured. Calcined alumina suspensions were tested at transmembrane pressures of -16.9, -27.1 and -33.8 kPa (-5, -8 and -10 in Hg). Yeast suspensions with solid volume fraction of 0.01 were tested at transmembrane pressures of -16.9, -33.8 and -50.8 kPa (-5, -10 and -15 in Hg). Yeast suspensions with solid volume fraction of 0.02 were tested at transmembrane pressures of -33.8, -50.8 and -67.7 kPa (-10, -15 and -20 in Hg). Temperature was maintained at 25°C.

The system was operated by backflushing for 30 seconds after every 360 seconds of forward filtration. The backflushing cycle time was chosen in accordance with Zenon's industrial experience. For flexographic ink suspensions and yeast suspensions, the backflushing transmembrane pressure was maintained at 13.5 kPa (4 in Hg). For calcined alumina suspensions, the backflushing transmembrane pressure was maintained at 6.3 kPa (2 in Hg). These backflushing transmembrane pressures were chosen after preliminary trials showed that they achieved suitable membrane cleaning. Flux was measured over time.
5.3.4 Effect of Airflow on Flux

The transmembrane pressure was kept constant for all suspensions tested while different values of airflow were tested. For flexographic ink suspensions, these values were 0, 0.47, 0.94, 1.42, 1.89, 2.36 L/s (0, 1, 2, 3, 4, 5 SCFM). These values correspond to a superficial gas velocity \( U_{sg} \) of 0, 0.0037, 0.0075, 0.0113, 0.0150, 0.0188 m/s, respectively. Superficial gas velocity \( U_{sg} \) was determined dividing the airflow rate by the cross-section area of the tank. For calcined alumina suspensions, airflows were 0, 0.94 and 1.42 L/s (0, 2, and 3 SCFM), and \( U_{sg} \) of 0, 0.0078, 0.0113 m/s. For yeast suspensions, airflow values of 0, 0.47, 0.94, 1.42, 1.89, 2.36 L/s (0, 1, 2, 3, 4, 5 SCFM) were tested. These values correspond to a superficial gas velocity of 0, 0.0037, 0.0075, 0.0113, 0.0150, 0.0188 m/s, respectively.

5.3.5 Statistical Analysis

The errors were calculated using the standard deviation of the average of the three cycles. The values shown are \text{MEAN} \pm \text{STANDARD DEVIATION}.

5.4 Results and Discussion

5.4.1 Membrane Resistance

The system was operated using de-ionized water, and flux was measured at different transmembrane pressures. Using Equation 5.1, and assuming that the dynamic viscosity of pure water is \( \eta_0 = 0.001 \text{ kg/m/s} \), the forward and reverse membrane resistances were determined; the results are presented in Table 5.1.

It should be noted that the membrane is asymmetric: the resistance in the forward filtration direction is larger than in the reverse filtration direction. It should also be noted that the same membranes were used in all tests. Membrane resistance tends to increase due to permanent fouling, i.e., deposits which could not be removed using the standard cleaning procedure.
Table 5.1 - Membrane resistance prior to suspension filtration

<table>
<thead>
<tr>
<th>Suspension to be tested</th>
<th>Solid Volume Fraction in the Suspension ($\phi_b$) to be tested</th>
<th>Direction</th>
<th>Membrane Resistance $R_m$ (m$^2$) ($\times 10^{11}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcined Alumina</td>
<td>0.02</td>
<td>forward</td>
<td>4.59</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
<td>1.71</td>
</tr>
<tr>
<td></td>
<td>0.03</td>
<td>forward</td>
<td>5.12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
<td>2.61</td>
</tr>
<tr>
<td>Flexographic Ink</td>
<td>0.005</td>
<td>forward</td>
<td>8.73</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
<td>3.97</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>forward</td>
<td>10.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
<td>3.97</td>
</tr>
<tr>
<td>Yeast</td>
<td>0.01</td>
<td>forward</td>
<td>9.37</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
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</tr>
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<td>9.37</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reverse</td>
<td>4.16</td>
</tr>
</tbody>
</table>

5.4.2 Effect of Transmembrane Pressure and Backflushing on Flux

**Flexographic Ink**

For suspensions of flexographic ink the system was operated by backflushing for 30 seconds after every 360 seconds of forward filtration. The backflushing transmembrane pressure was maintained at 13.50 kPa (4 in Hg). Flux was measured over time. The forward transmembrane pressures tested were -16.9, -33.8 and -50.8 kPa (-5, -10, -15 in Hg).

A typical graph of flux versus time is shown in Figure 5.3. Other graphs of flux versus time for different transmembrane pressures are presented in Appendix 2. The average forward filtration flux during three cycles for airflow values of 0, 0.47, 0.94, 1.42, 1.89 and 2.36 L/s (0, 1, 2, 3, 4 and 5 SCFM) are shown in Table 5.2 for ink suspension at $\phi_b = 0.005$, and in Table 5.3 for ink suspension at $\phi_b = 0.01$. 

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Figure 5.3 - Flux versus time for flexographic ink suspension at transmembrane pressure of -33.8 kPa and airflow of 1.89 L/s and $\phi_b = 0.005$
Table 5.2 - Flux during flexographic ink suspension filtration with $\Phi_b = 0.005$

<table>
<thead>
<tr>
<th>$\Phi_b$</th>
<th>Airflow</th>
<th>Cycle 1</th>
<th>Cycle 2</th>
<th>Cycle 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 L/s</td>
<td><strong>Cycle 1</strong></td>
<td>13.3</td>
<td>29.7</td>
<td>29.6</td>
</tr>
<tr>
<td>(0 SCFM)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.47 L/s</td>
<td><strong>Cycle 1</strong></td>
<td>29.0</td>
<td>41.4</td>
<td>29.6</td>
</tr>
<tr>
<td>(1 SCFM)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.94 L/s</td>
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<td>31.0</td>
<td>44.2</td>
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<tr>
<td>(2 SCFM)</td>
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<tr>
<td>1.42 L/s</td>
<td><strong>Cycle 1</strong></td>
<td>31.4</td>
<td>47.0</td>
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<tr>
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<tr>
<td>1.89 L/s</td>
<td><strong>Cycle 1</strong></td>
<td>32.8</td>
<td>47.0</td>
<td>32.0</td>
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<tr>
<td>(4 SCFM)</td>
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<tr>
<td>2.36 L/s</td>
<td><strong>Cycle 1</strong></td>
<td>32.6</td>
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<td>(5 SCFM)</td>
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AVERAGE PERMEATE FLUX DURING FORWARD FILTRATION (L/m²h)

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<th>$\Phi_b$</th>
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<th>-50.8 kPa</th>
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<td></td>
<td>55.4</td>
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<tr>
<td>(3 SCFM)</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td>1.89 L/s</td>
<td><strong>Cycle 1</strong></td>
<td></td>
<td></td>
<td>55.3</td>
</tr>
<tr>
<td>(4 SCFM)</td>
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<td></td>
</tr>
<tr>
<td>2.36 L/s</td>
<td><strong>Cycle 1</strong></td>
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<td>54.8</td>
</tr>
<tr>
<td>(5 SCFM)</td>
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</table>

Average Fluxes:

- 0 L/s: 29.4 ± 0.4, 42.1 ± 1.3, 51.5 ± 0.8
- 0.47 L/s: 31.1 ± 0.2, 44.4 ± 0.3, 53.9 ± 0.5
- 0.94 L/s: 31.4 ± 0.1, 46.2 ± 0.7, 55.8 ± 0.3
- 1.42 L/s: 32.4 ± 0.4, 46.7 ± 0.4, 56.4 ± 0.9
- 1.89 L/s: 32.6 ± 0.4, 49.1 ± 0.8, 56.2 ± 1.4
Table 5.3 - Flux during flexographic ink suspension filtration with $\phi_b = 0.01$

<table>
<thead>
<tr>
<th>$\phi_b$</th>
<th>Airflow</th>
<th>AVERAGE PERMEATE FLUX DURING FORWARD FILTRATION (L/m²h)</th>
</tr>
</thead>
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<td></td>
<td>Cycle 2</td>
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<td></td>
<td>Cycle 3</td>
<td>16.8</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>17.2 ± 0.8</td>
</tr>
<tr>
<td>0.47 L/s (1 SCFM)</td>
<td>Cycle 1</td>
<td>20.8</td>
</tr>
<tr>
<td></td>
<td>Cycle 2</td>
<td>21.8</td>
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<tr>
<td></td>
<td>Cycle 3</td>
<td>20.2</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>20.9 ± 0.7</td>
</tr>
<tr>
<td>0.94 L/s (2 SCFM)</td>
<td>Cycle 1</td>
<td>21.5</td>
</tr>
<tr>
<td></td>
<td>Cycle 2</td>
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<td></td>
<td>Cycle 3</td>
<td>22.8</td>
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<tr>
<td></td>
<td>Average</td>
<td>21.9 ± 0.4</td>
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<tr>
<td>1.42 L/s (3 SCFM)</td>
<td>Cycle 1</td>
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<td>Cycle 2</td>
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<tr>
<td></td>
<td>Average</td>
<td>21.3 ± 0.4</td>
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<tr>
<td>1.89 L/s (4 SCFM)</td>
<td>Cycle 1</td>
<td>22.0</td>
</tr>
<tr>
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<td>Cycle 3</td>
<td>20.9</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>22.1 ± 1.3</td>
</tr>
</tbody>
</table>

In Figures 5.4 and 5.5 the average permeate flux versus transmembrane pressure is plotted for airflows of 0, 0.47 and 2.36 L/s.
Figure 5.4 - Average permeate flux versus transmembrane pressure for flexographic ink suspension at $\phi_b = 0.005$ and different airflows

Figure 5.5 - Average permeate flux versus transmembrane pressure for flexographic ink suspension at $\phi_b = 0.01$ and different airflows
An increase in the permeate flux was observed when the transmembrane pressure was increased. The shape of these curves is similar to the shape of the curves generated by the model, and presented in Figure 4.12 of the previous chapter.

Backflushing had a significant effect on restoring the initial membrane flux. During three cycles of filtration of the flexographic ink suspension, the initial flux was almost completely restored, and the average permeate flux per cycle did not vary significantly, as shown in Tables 5.2 and 5.3. Long-term internal membrane fouling is expected to occur after only several cycles.

Figure 5.6 is a plot of the transmembrane pressure versus the average permeate flux for $\phi_b = 0.01$ and $\phi_b = 0.005$ at an airflow of 0.47 L/s (1 SCFM). Figure 5.7 plots the transmembrane pressure versus the average permeate flux for $\phi_b = 0.01$ and $\phi_b = 0.005$ at an airflow of 2.36 L/s (5 SCFM).

![Figure 5.6 - Average permeate flux versus transmembrane pressure for flexographic ink suspensions with $\phi_b = 0.01$ and $\phi_b = 0.005$ at an airflow of 0.47 L/s (1 SCFM)](image-url)
Figure 5.7 - Average permeate flux versus transmembrane pressure for flexographic ink suspensions with $\phi_b = 0.01$ and $\phi_b = 0.005$ at an airflow of 2.36 L/s (5 SCFM)

From Figures 5.6 and 5.7, it is clear that the flux decreases as the solid volume fraction of the suspension increases. The effect of transmembrane pressure is more significant for more diluted suspensions, i.e., for suspensions with lower values of solid volume fraction. This behaviour also conforms to the model presented in Figure 4.9 of the previous chapter.

**Calcined Alumina**

For suspensions of calcined alumina, the system was operated by backflushing for 30 seconds every 360 seconds of forward filtration. The backflushing transmembrane pressure was maintained at 6.3 kPa (2 in Hg). Flux was measured over time during operation of the system at transmembrane pressures of -16.9, -27.1, and -33.8 kPa (-5, -8, -10 in Hg). A typical graph of flux versus time is shown in Figure 5.8. Other graphs of flux versus time for different transmembrane pressures are presented in Appendix 2.
Figure 5.8 - Flux versus time for calcined alumina suspension at transmembrane pressure of -33.8 kPa and airflow of 1.42 L/s and $\Phi_b = 0.03$

Table 5.4 summarizes the flux data and shows a flux increase due to the increase in transmembrane pressure. The average values are the average of forward filtration permeate flux during three cycles.

In Figures 5.9 and 5.10 the transmembrane pressure versus the average permeate flux is plotted for airflows of 0, 0.94 and 1.89 L/s.
Table 5.4 - Flux during calcined alumina suspension filtration

| $\phi_0$ | Airflow | $\Delta P$ (kPa) | Cycle 1 | Cycle 2 | Cycle 3 | Average
|----------|---------|-----------------|--------|--------|--------|---------
| 0 L/s (0 SCFM) | Cycle 1 | 18.3 | 24.5 | 31.4 | 24.4 | 31.3 | 35.7 | 24.6 ± 0.4 | 31.5 ± 0.3 | 35.4 ± 0.4 | 0.02 |
| 0.94 L/s (2 SCFM) | Cycle 1 | 30.0 | 34.6 | 41.4 | 28.0 | 33.3 | 41.0 | Cycle 2 | 30.0 | 34.6 | 41.4 | Cycle 3 | 26.5 | 33.4 | 41.0 | Average | 28.2 ± 1.8 | 33.8 ± 0.7 | 41.1 ± 0.2 |
| 1.42 L/s (3 SCFM) | Cycle 1 | 14.8 | 22.6 | 26.6 | 18.6 | 25.1 | 30.6 | Cycle 2 | 18.6 | 25.8 | 30.4 | Cycle 3 | 18.6 | 25.8 | 30.4 | Average | 18.6 ± 0.1 | 25.6 ± 0.4 | 30.4 ± 0.2 | 0.03 |
| 0 L/s (0 SCFM) | Cycle 1 | 20.2 | 26.2 | 31.2 | 18.8 | 26.8 | 31.1 | Cycle 2 | 19.0 | 27.4 | 31.2 | Cycle 3 | 19.4 ± 0.8 | 26.8 ± 0.6 | 31.2 ± 0.1 | Average | 19.4 ± 0.8 | 26.8 ± 0.6 | 31.2 ± 0.1 | 1.42 L/s (3 SCFM) | 18.8 | 26.8 | 31.1 |
Figure 5.9 - Average permeate flux versus transmembrane pressure for calcined alumina suspension with $\Phi_b = 0.02$ and different airflows

Figure 5.10 - Average permeate flux versus transmembrane pressure for calcined alumina suspension with $\Phi_b = 0.03$ and different airflows
An increase in the permeate flux was observed when the transmembrane pressure was increased. Again, the shape of these curves is similar to the curves generated by the model and presented in Figure 4.12 of the previous chapter.

In this application, the backflushing transmembrane pressure was maintained at 6.3 kPa (2 in Hg), half of the value of 12.6 (4 in Hg) used during filtration of the flexographic ink suspensions, and still it had a significant effect on restoring the initial membrane flux. During three cycles of filtration of the calcined alumina suspension, the initial flux was almost completely restored, and the average permeate flux per cycle did not vary significantly.

Figure 5.11 plots the transmembrane pressure versus the average permeate flux for $\phi_b = 0.02$ and $\phi_b = 0.03$ at an airflow of 0.94 L/s (2 SCFM). Figure 5.12 plots the transmembrane pressure versus the average permeate flux for $\phi_b = 0.02$ and $\phi_b = 0.03$ at an airflow of 1.42 L/s (3 SCFM).

![Figure 5.11 - Average permeate flux versus transmembrane pressure for calcined alumina suspensions with $\phi_b = 0.02$ and $\phi_b = 0.03$ at an airflow of 0.94 L/s (2 SCFM)](image)
Figure 5.12 - Average permeate flux versus transmembrane pressure for calcined alumina suspensions with $\phi_s = 0.02$ and $\phi_s = 0.03$ at an airflow of 1.42 L/s (3 SCFM)

From Figures 5.11 and 5.12, it can be seen that the flux decreases as the solid volume fraction of the suspension increases. The effect of transmembrane pressure is more significant for more diluted suspensions, i.e., for suspensions with lower values of solid volume fraction. Again, this phenomenon agrees with the model as presented in Figure 4.9 of the previous chapter.

**Yeast Suspensions**

For yeast suspensions, the system was operated by backflushing for 30 seconds after every 360 seconds of forward filtration. The transmembrane pressure during backflushing was kept at 13.50 kPa (4 in Hg). Flux was measured over time. For yeast suspensions with solid volume fraction of 0.01 the forward transmembrane pressures tested were -16.9, -33.8 and -50.8 kPa (-5, -10 and -15 in Hg). For yeast suspensions with solid volume fraction of 0.02 the transmembrane pressures tested were -33.8, -50.8 and -67.7 kPa (-10, -15 and -20 in Hg). A typical graph of flux versus time is shown in Figure 5.13. Other graphs of flux versus time for different transmembrane pressures are presented in Appendix 2.
Figure 5.13 – Flux versus time for yeast suspension at transmembrane pressure of -33.8 kPa and airflow of 1.89 L/s and $\phi_b = 0.01$

The average forward filtration permeate flux during three cycles for airflow values of 0, 0.47, 0.94, 1.42, 1.89 and 2.36 L/s (0, 1, 2, 3, 4 and 5 SCFM) are shown in Table 5.5 for a yeast suspension with $\phi_b = 0.01$, and in Table 5.6 for a yeast suspension with $\phi_b = 0.02$.
Table 5.5 - Flux during yeast suspension filtration with $\Phi_b = 0.01$

| Airflow          | Cycle 1 | Cycle 2 | Cycle 3 | Average  
|------------------|---------|---------|---------|----------
| 0 L/s (0 SCFM)   | 12.1    | 29.9    | 28.4    |          
| 0.47 L/s (1 SCFM)| 21.1    | 42.2    | 41.2    |          
| 0.94 L/s (2 SCFM)| 20.9    | 39.3    | 48.4    |          
| 1.42 L/s (3 SCFM)| 21.3    | 43.0    | 48.5    |          
| 1.89 L/s (4 SCFM)| 22.4    | 43.5    | 49.9    |          
| 2.36 L/s (5 SCFM)| 25.0    | 42.8    | 49.2    |          

Average permeate flux during forward filtration (L/m² h)

-16.9 kPa | -33.8 kPa | -50.8 kPa
-22.0 | -43.1 | 47.7
-23.4 | 43.3 | 47.1
-24.6 | 43.1 | 48.0

0.1
Table 5.6 - Flux during yeast suspension filtration with $\Phi_b = 0.02$

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<th>Cycle 2</th>
<th>Cycle 3</th>
<th>Average</th>
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<tr>
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<td>24.1</td>
<td>27.4</td>
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<td>Average</td>
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<td>$24.8 \pm 0.6$</td>
<td>$27.4 \pm 0.1$</td>
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<td>28.1</td>
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<tr>
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<td>26.0</td>
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<td>Average</td>
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<td>$29.3 \pm 0.3$</td>
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<tr>
<td>2.36 L/s (5 SCFM)</td>
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<td>$24.4 \pm 0.6$</td>
<td>$30.1 \pm 0.7$</td>
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In Figures 5.14 and 5.15 the average permeate flux versus transmembrane pressure is plotted for airflows of 0, 0.47 and 2.36 L/s.
Figure 5.14 - Average permeate flux versus transmembrane pressure for a yeast suspension with $\Phi_b = 0.01$ and different airflows

Figure 5.15 - Average permeate flux versus transmembrane pressure for a yeast suspension with $\Phi_b = 0.02$ and different airflows
Again, an increase in the permeate flux was observed when the transmembrane pressure was increased. The shape of these curves is similar to the curves generated by the model, and presented in Figure 4.12 of the previous chapter.

Backflushing had again a significant effect on restoring the initial membrane flux. During three cycles of filtration of the flexographic ink suspension, the initial flux was almost completely restored, and the average permeate flux per cycle did not vary significantly, as shown in Tables 5.5 and 5.6.

In Figure 5.16 the average permeate flux versus transmembrane pressure is plotted for $\phi_b = 0.01$ and $0.02$ at an airflow of 0.47 L/s (1 SCFM). In Figure 5.17 the transmembrane pressure versus the average permeate flux is plotted for $\phi_b = 0.01$ and $\phi_b = 0.02$ at an airflow of 2.36 L/s (5 SCFM).

![Graph showing permeate flux versus transmembrane pressure for a yeast suspension with $\phi_b = 0.01$ and $0.02$ at an airflow of 0.94 L/s (2 SCFM).](image)

Figure 5.16 - Average permeate flux versus transmembrane pressure for a yeast suspension with $\phi_b = 0.01$ and $0.02$ at an airflow of 0.94 L/s (2 SCFM)
Again, it is observed that flux decreases as the solid volume fraction of the suspension increases. In this application, the effect of transmembrane pressure is again more significant for more diluted suspensions, i.e., for suspensions with lower values of solid volume fraction. This behavior conforms to the model presented in Figure 4.9 of the previous chapter.

5.4.3 Effect of Airflow on Flux

Flexographic Ink

Flux over time was measured at constant transmembrane pressures under different shear stresses by changing the airflow. Airflows of 0, 0.47, 0.94, 1.42, 1.89 and 2.36 L/s (0, 1, 2, 3, 4 and 5 SCFM) were tested. The graphs of flux versus time for different airflows are presented in Appendix 3. Figures 5.18 and 5.19 show the effect of the applied airflow (shear stress) on the average forward filtration flux at transmembrane pressures of -16.9, -33.8, and -50.8 kPa (-5, -10 and -15 in Hg).

Figure 5.17 - Average permeate flux versus transmembrane pressure for a yeast suspension with $\phi_s = 0.01$ and $\phi_s = 0.02$ at an airflow of 2.36 L/s (5 SCFM)
Figure 5.18 - Average permeate flux versus airflow for a flexographic ink suspension with solid volume fraction $\phi_b = 0.005$ and different transmembrane pressures

Figure 5.19 - Average permeate flux versus airflow for a flexographic ink suspension with solid volume fraction $\phi_b = 0.01$ and different transmembrane pressures
In general, an increase in the permeate flux was observed when the airflow was increased in the range of 0 to 0.47 L/s after which little or no increase was observed. This phenomenon conforms to the model predictions presented in Figure 4.10 of the previous chapter.

Calcined Alumina

Flux over time was measured at constant transmembrane pressures under different shear stresses by changing the airflow. Airflows of 0, 0.94 and 1.42 L/s (0, 2 and 3 SCFM) were tested. The graphs "flux versus time" for different airflows are presented in Appendix 3.

Figures 5.20 and 5.21 show the effect of the applied airflow (shear stress) on the flux at transmembrane pressures of -16.9, -27.1, and -33.8 kPa (-5, -8 and -10 in Hg).

Figure 5.20 - Average permeate flux versus airflow for calcined alumina suspension with solid volume fraction $\Phi_b = 0.02$ and different transmembrane pressures
An increase in the permeate flux was observed when the airflow was increased due to an increase in the shear stresses at the membrane surface, and this resulted in the minimization of cake formation. However, in this range of airflows tested, it was not possible to observe a rapid initial increase followed by a moderate increase in flux, which is predicted by the model and was found in the previous application. This is probably due to the fact that only three different airflows were tested, so what can be observed in Figures 5.20 and 5.21 is the initial rapid flux increase, after which a moderate increase would follow.

**Yeast Suspensions**

Flux over time was measured at constant transmembrane pressures under different shear stresses by changing the airflow. Airflows of 0, 0.47, 0.94, 1.42, 1.89 and 2.36 L/s (0, 1, 2, 3, 4 and 5 SCFM) were tested. The graphs of flux versus time for different airflows are presented in Appendix 3. Figures 5.22 and 5.23 show the effect of the applied airflow (shear stress) on the flux at transmembrane pressures of -16.9, -33.8, and -50.8 kPa (-5, -10 and -15 in Hg).
Figure 5.22 - Average permeate flux versus airflow for a yeast suspension with solid volume fraction \( \Phi_b = 0.01 \) and different transmembrane pressures

Again it is observed that in general the permeate flux increased when the airflow was increased in the range of 0 to 0.47 L/s, after which little or no increase was observed.
5.5 Summary

The objective of this chapter was to validate qualitatively the model for flux prediction proposed in the previous chapter. Six suspensions were tested: flexographic ink ($\phi_b=0.005$ and $\phi_s=0.01$), calcined alumina ($\phi_b=0.02$ and $\phi_s=0.03$) and baker’s yeast ($\phi_b=0.01$ and $\phi_s=0.02$). In each case, the effects of transmembrane pressure, aeration rate and suspension concentration on permeate flux were investigated. The main conclusions were:

(i) for all applications, an increase in the permeate flux was observed when the transmembrane pressure was increased

(ii) the effect of transmembrane pressure was more significant during the filtration of more diluted suspensions, i.e., for suspensions with lower values of solid volume fraction

(iii) permeate flux decreased as the solid volume fraction of the suspension increased

(iv) for all applications, backflushing had a significant effect on restoring the initial membrane flux. During three cycles of filtration of every suspension, the initial flux was almost completely restored, and the average permeate flux per cycle did not vary significantly. This is not expected to occur after several cycles because of the long-term internal membrane fouling that is not removed by backflushing

(v) generally, an increase in the permeate flux was observed when the airflow was increased, due to an increase in the shear stresses at the membrane surface. However, a rapid increase occurred until a certain value of applied airflow, after which only small changes were observed.

In general, the experimental filtration behaviour of the various suspensions conformed qualitatively to the model presented in the previous chapter.
6.1 Introduction

In the previous chapter, a qualitative validation of the model was carried out. The objective of this chapter is to validate quantitatively the model by substituting the suspension characteristics and operating variables into the model equation and comparing the predicted flux with the experimentally determined flux.

The experimental validation of the model, as it is shown in Equation 4.8, is difficult due to the fact that some variables such as the solid volume fraction of the cake $\Phi_c$, the shear stress at the membrane surface $\tau_w$, and the constant $k_l$ are difficult to determine. This is particularly true for the type of configuration used in this work. In order to facilitate the experimental verification of the model, some variables were grouped to form lumped parameters that were easier to determine.

6.2 Methodology

In order to validate the forward transient flux of the model two lumped parameters “$a$” and “$b$” were derived from

$$a = \frac{\hat{R}_c}{k_l \cdot \tau_w},$$  \hspace{1cm} (6.1)

and

$$b = \frac{\hat{R}_c}{\eta \cdot (\Phi_c - \Phi_b)},$$  \hspace{1cm} (6.2)

where $\hat{R}_c$ is the specific cake resistance, $k_l$ is a constant, $\tau_w$ is the shear stress at membrane wall, $\eta$ the dynamic viscosity, and $\Phi_b$ is the solid volume fraction in the suspension, $\Phi_c$ is the solid volume fraction in the cake.

Substituting these expressions in Equation 4.8 of Chapter 4, and neglecting the fouling resistance $R_f$, we get
The parameter "a" can be determined experimentally: when the value of t is high, and the membrane resistance $R_m$ is negligible, $J(t)$ becomes constant, equal to the pseudo steady-state flux. So "a" can be calculated from

$$J = \frac{\Delta P}{\sqrt{\Delta P \cdot \Phi_b \cdot \eta \cdot \alpha}}. \quad (6.4)$$

The parameter "b" can also be determined experimentally by means of dead-end filtration theory. In order to simulate a dead-end filtration condition, the system was operated without applying air. The transient flux during dead-end filtration is given by

$$J(t) = J_o \left[ 1 + \frac{2 \cdot \hat{R}_c \cdot \Phi_b \cdot \Delta P}{(\Phi_c - \Phi_b) \cdot \eta \cdot R_m^2} \right]^{-\frac{1}{2}}. \quad (6.5)$$

Equation 6.5 can be rewritten as

$$\left( \frac{J_0}{J(t)} \right)^2 - 1 = \left( \frac{2 \cdot \hat{R}_c \cdot \Phi_b \cdot \Delta P}{(\Phi_c - \Phi_b) \cdot \eta \cdot R_m^2} \right) t. \quad (6.6)$$

Combining Equations 6.2 and 6.6 gives

$$\left( \frac{J_0}{J(t)} \right)^2 - 1 = \left( \frac{2 \cdot b \cdot \Phi_b \cdot \Delta P}{R_m^2} \right) t. \quad (6.7)$$
The slope of the curve \( \left( \frac{J_0}{J(t)} \right)^2 - 1 \) versus \( t \), is equal to \( \left( \frac{2 \cdot b \cdot \Phi_b \cdot \Delta P}{R_m^2} \right) \).

The membrane resistance \( R_m \) was assumed to be the same as previously determined in Chapter 5. The fouling resistance was assumed to be negligible.

### 6.3 Results and Discussion

#### 6.3.1 Flexographic Ink

Table 6.1 and Table 6.2 lists the calculated values for “a” and “b” for flexographic ink suspensions of \( \Phi_b = 0.005 \) and 0.01, respectively.

**Table 6.1 - Values of “a” and “b” for flexographic ink suspension, \( \Phi_b = 0.005 \)**

<table>
<thead>
<tr>
<th>( \Phi_b ) (kPa)</th>
<th>( \Delta P ) (kPa)</th>
<th>Airflow (L/s)</th>
<th>( a ) (x 10^{22})</th>
<th>( b ) (x 10^{20})</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.005</td>
<td>-16.9</td>
<td>0.47</td>
<td>2.61 ± 0.07</td>
<td>11.51</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>2.23 ± 0.21</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.02 ± 0.17</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>1.55 ± 0.05</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>1.69 ± 0.18</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-33.8</td>
<td>0.47</td>
<td>4.03 ± 0.45</td>
<td>1.77</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>3.15 ± 0.21</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.73 ± 0.20</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>2.46 ± 0.15</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>2.15 ± 0.13</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-50.8</td>
<td>0.47</td>
<td>4.60 ± 0.02</td>
<td>1.41</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>4.00 ± 0.02</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>3.55 ± 0.03</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>3.46 ± 0.03</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>3.24 ± 0.08</td>
<td></td>
</tr>
</tbody>
</table>
As shown in Equation 6.1, "a" is inversely proportional to the shear stress, so when airflow increases it is expected that the value of "a" decreases. This behaviour is illustrated in Figure 6.1 where the values of "a" are plotted against the airflow values at different transmembrane pressures. For a more concentrated suspension, this behaviour was not so clear as shown in Figure 6.2. This was probably due to significant errors observed during the filtration at transmembrane pressure of 16.9 kPa.
Figure 6.1 - "d" versus airflow for flexographic ink suspension with solid volume fraction \( \Phi_b = 0.005 \) and different transmembrane pressures.

Figure 6.2 - "d" versus airflow for flexographic ink suspension with solid volume fraction \( \Phi_b = 0.01 \) and different transmembrane pressures.
Equation 6.1 also shows that the value of "a" is directly proportional to the specific cake resistance $\hat{R}_c$. Porter suggested that the cake specific resistance depends on the cake compressibility as showed previously in Equation 4.9 which is

$$\hat{R}_c = \alpha_0 \cdot (\Delta P)^s \cdot \rho_s \cdot \Phi_c,$$

(4.9)

where $\alpha_0$ is a constant related to size and shape of particles, $s$ the cake compressibility, and $\rho_s$ the mass density of solids comprising the cake.

Equation 4.9 also shows that the solid volume fraction of the cake $\Phi_c$ is directly proportional to the specific cake resistance. If the cake is highly compressible then the transmembrane pressure should have a significant effect in $\hat{R}_c$. Following Porter's Equation 4.9, an increase in the value of "a" is expected when increasing the transmembrane pressure. This phenomenon can be observed in Figure 6.3, where the value of "a" is plotted against the transmembrane pressure at different airflows for flexographic ink suspension with $\Phi_b = 0.005$.

![Flexographic Ink Suspension $\Phi_b = 0.005$](image)

Figure 6.3 - "a" versus transmembrane pressure for flexographic ink suspension with solid volume fraction $\Phi_b = 0.005$ and different airflows
Figure 6.4 shows a similar graph for flexographic ink suspensions with $\Phi_b = 0.01$. It is interesting to note that in most cases, in contrast to the behaviour previously observed in Figure 6.3, the value of “$a$” is unchanged as the transmembrane pressure increases, taking into account the error. From Figures 6.3 and 6.4 it can also be observed that the values of “$a$” are higher for lower values of airflow. This is consistent with the notion that “$a$” decreases as the shear stress increases.

![Graph showing the relationship between $a$ and transmembrane pressure for flexographic ink suspension with $\Phi_b = 0.01$.](image)

Figure 6.4 - “$a$” versus transmembrane pressure for flexographic ink suspension with solid volume fraction $\Phi_b = 0.01$ and different airflows

In Figure 6.5 the value of “$b$” versus transmembrane pressure is plotted for flexographic ink suspensions with solid volume fractions of $\Phi_b = 0.005$ and 0.01.
Figure 6.5 - "b" versus transmembrane pressure for flexographic ink suspensions

In Figure 6.5, the value of "b" decreases as the transmembrane pressure increases, and the decrease is more significant at lower transmembrane pressures. Assuming that "b" is directly proportional to the specific cake resistance $\hat{R}_c$, as presented in Equation 6.2, an opposite behaviour would be expected because an increase in the transmembrane pressure would lead to an increase in the specific cake resistance. However, a possible explanation for the behaviour observed in Figure 6.5 is that, at lower transmembrane pressures, the solid volume fraction of the cake $\Phi_c$ is close to $\Phi_b$, so $(\Phi_c - \Phi_b)$ approaches zero, and therefore, from Equation 6.2, "b" is large. As the transmembrane pressure increases, the solid volume fraction of the cake $\Phi_c$ increases, so "b" decreases.

Figure 6.5 also suggests that "b" decreases rapidly up to a certain value of transmembrane pressure, after which it decreases moderately. This can be explained by the fact that the solid volume fraction of the cake $\Phi_c$ reaches a maximum value, after which any increase in the transmembrane pressure no longer has a significant effect on $\Phi_c$. 
Tables 6.3 and 6.4 show the values of the average forward permeate fluxes during 360 seconds predicted by the model and the same values obtained during the experiments for flexographic ink suspensions of $\Phi_b = 0.005$ and 0.01, respectively.

Table 6.3 - Average flux for flexographic ink suspensions with solid volume fraction of 0.005 during first 360 seconds of filtration

<table>
<thead>
<tr>
<th>$\Phi_b$ (kPa)</th>
<th>Airflow (L/s)</th>
<th>Model (L/m$^2$h)</th>
<th>Experiment (L/m$^2$h)</th>
<th>Difference (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.005</td>
<td>0.47</td>
<td>26.1</td>
<td>29.4</td>
<td>+11.2</td>
</tr>
<tr>
<td></td>
<td>0.94</td>
<td>27.5</td>
<td>31.1</td>
<td>+11.6</td>
</tr>
<tr>
<td></td>
<td>1.42</td>
<td>28.4</td>
<td>31.4</td>
<td>+9.6</td>
</tr>
<tr>
<td></td>
<td>1.89</td>
<td>30.3</td>
<td>32.4</td>
<td>+6.5</td>
</tr>
<tr>
<td></td>
<td>2.36</td>
<td>29.4</td>
<td>32.6</td>
<td>+9.8</td>
</tr>
<tr>
<td>-16.9</td>
<td>0.47</td>
<td>42.2</td>
<td>42.1</td>
<td>-0.2</td>
</tr>
<tr>
<td></td>
<td>0.94</td>
<td>44.2</td>
<td>44.4</td>
<td>+0.5</td>
</tr>
<tr>
<td></td>
<td>1.42</td>
<td>45.5</td>
<td>46.2</td>
<td>+1.5</td>
</tr>
<tr>
<td></td>
<td>1.89</td>
<td>46.5</td>
<td>46.7</td>
<td>+0.4</td>
</tr>
<tr>
<td></td>
<td>2.36</td>
<td>48.0</td>
<td>49.1</td>
<td>+2.2</td>
</tr>
<tr>
<td>-33.8</td>
<td>0.47</td>
<td>56.2</td>
<td>51.5</td>
<td>-8.4</td>
</tr>
<tr>
<td></td>
<td>0.94</td>
<td>57.4</td>
<td>53.9</td>
<td>-6.1</td>
</tr>
<tr>
<td></td>
<td>1.42</td>
<td>58.5</td>
<td>55.8</td>
<td>-4.6</td>
</tr>
<tr>
<td></td>
<td>1.89</td>
<td>58.7</td>
<td>56.4</td>
<td>-3.9</td>
</tr>
<tr>
<td></td>
<td>2.36</td>
<td>59.4</td>
<td>56.2</td>
<td>-5.4</td>
</tr>
</tbody>
</table>

| -50.8         | 0.47          | 56.2             | 51.5                   | -8.4           |
|               | 0.94          | 57.4             | 53.9                   | -6.1           |
|               | 1.42          | 58.5             | 55.8                   | -4.6           |
|               | 1.89          | 58.7             | 56.4                   | -3.9           |
|               | 2.36          | 59.4             | 56.2                   | -5.4           |
The difference of the average forward permeate flux between experimental and the model results vary from +21.5% to -8.7%. It is interesting to note that the model tends to over-predict fluxes at lower transmembrane pressures and under-predict at higher transmembrane pressures. However, these results show that the model correlates well with the experimental results. Figures 6.6 and 6.7 show the model and experimental flux versus airflow for flexographic ink at different transmembrane pressures with a solid volume fraction $\Phi_b = 0.005$ and 0.01, respectively.
Figure 6.6 – Flux versus airflow — experimental results and model prediction — flexographic ink with solid volume fraction of 0.005

Figure 6.7 – Flux versus airflow — experimental results and model prediction — flexographic ink with solid volume fraction of 0.01

A typical graph showing a comparison of the experimental results and the model prediction for flux versus time is shown in Figure 6.8. Other graphs are presented in Appendix 4.
Figure 6.8 – Flux vs. time for flexographic ink, $\Delta P = -33.8$ kPa, airflow of 1.42 L/s, and $\phi_b = 0.005$

6.3.2 Calcined Alumina

Table 6.5 and Table 6.6 gives the calculated values for "a" and "b" for calcined alumina suspensions of $\phi_b = 0.02$ and 0.03, respectively.

In Figures 6.9 and 6.10 the value of "a" versus airflow is plotted for transmembrane pressures of -16.9, -27.1 and -33.8 kPa for calcined alumina suspensions. As expected, a decrease in the value of "a" was observed when airflow was increased.
Table 6.5 - Values of “a” and “b” for calcined alumina suspension

<table>
<thead>
<tr>
<th>$\phi_b$</th>
<th>$\Delta P$ (kPa)</th>
<th>Airflow (L/s)</th>
<th>$a \times 10^{22}$</th>
<th>$b \times 10^{20}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.02</td>
<td>-16.93</td>
<td>0.94</td>
<td>2.42 ± 0.15</td>
<td>1.47</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>1.66 ± 0.13</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-27.10</td>
<td>0.94</td>
<td>2.94 ± 0.20</td>
<td>0.40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.05 ± 0.21</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-33.86</td>
<td>0.94</td>
<td>3.48 ± 0.26</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.24 ± 0.08</td>
<td></td>
</tr>
<tr>
<td>0.03</td>
<td>-16.93</td>
<td>0.94</td>
<td>2.54 ± 0.17</td>
<td>1.16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.27 ± 0.48</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-27.10</td>
<td>0.94</td>
<td>2.47 ± 0.18</td>
<td>0.51</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.22 ± 0.17</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-33.86</td>
<td>0.94</td>
<td>2.42 ± 0.17</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.30 ± 0.29</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6.9 - “a” versus airflow for calcined alumina suspension with solid volume fraction $\phi_b = 0.02$ and different transmembrane pressures
Figure 6.10 - "α" versus airflow for calcined alumina suspension with solid volume fraction $\Phi_b = 0.03$ and different transmembrane pressures.

Figures 6.11 and 6.12 plot the value of "α" versus the transmembrane pressure for airflows of 0.94 and 1.42 L/s for calcined alumina suspensions.

Figure 6.11 - "α" versus transmembrane pressure for calcined alumina suspension with solid volume fraction $\Phi_b = 0.02$ and airflows of 0.94 and 1.42 L/s.
Figure 6.12 - "a" versus transmembrane pressure for calcined alumina suspension with solid volume fraction $\Phi_b = 0.03$ and airflows of 0.94 and 1.42 L/s

Again it can be note that for more diluted suspensions the value of "a" increases as the transmembrane pressure increases as shown in Figure 6.11. In more concentrated suspensions, as shown in Figure 6.12, "a" remains constant with increasing pressure as was observed for flexographic ink suspensions (see Figure 6.4).

Figure 6.13 shows the value of "b" versus transmembrane pressure for calcined alumina suspensions with solid volume fraction of $\Phi_b = 0.02$ and 0.03. The same behavior of the value of "b" for flexographic ink suspensions was again observed. It is interesting to note, however, that the value of "b" for calcined alumina is approximately one order of magnitude smaller than the ones for flexographic ink suspensions. This is probably due to the different characteristics of the suspensions such as particle size.
Table 6.6 shows the value of the average forward permeate flux for the first 360 seconds of filtration predicted by the model and the same values obtained during the experiments for calcined alumina.

The difference of the average forward permeate flux between experimental and the model results vary from +15.3% to -18.3%. In general, these results show that the model gives good permeate flux prediction.
Table 6.6 – Average flux for calcined alumina suspensions with solid volume fractions of 0.02 and 0.03 during first 360 seconds of filtration

<table>
<thead>
<tr>
<th>( \Phi_b )</th>
<th>( \Delta P ) (kPa)</th>
<th>Airflow (L/s)</th>
<th>Model (L/m²h)</th>
<th>Experiment (L/m²h)</th>
<th>Difference (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.02</td>
<td>-16.9</td>
<td>0.94</td>
<td>21.5</td>
<td>22.6</td>
<td>+4.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>23.9</td>
<td>28.2</td>
<td>+15.3</td>
</tr>
<tr>
<td></td>
<td>-27.1</td>
<td>0.94</td>
<td>38.4</td>
<td>31.5</td>
<td>-18.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>39.7</td>
<td>33.8</td>
<td>-14.9</td>
</tr>
<tr>
<td></td>
<td>-33.8</td>
<td>0.94</td>
<td>38.9</td>
<td>35.4</td>
<td>-8.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>40.9</td>
<td>41.1</td>
<td>+0.5</td>
</tr>
<tr>
<td>0.03</td>
<td>-16.9</td>
<td>0.94</td>
<td>18.2</td>
<td>18.6</td>
<td>+2.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>18.7</td>
<td>19.4</td>
<td>+3.6</td>
</tr>
<tr>
<td></td>
<td>-27.1</td>
<td>0.94</td>
<td>29.7</td>
<td>25.6</td>
<td>-13.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>30.1</td>
<td>26.8</td>
<td>-11.0</td>
</tr>
<tr>
<td></td>
<td>-33.8</td>
<td>0.94</td>
<td>34.6</td>
<td>30.4</td>
<td>-12.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>34.8</td>
<td>31.2</td>
<td>-10.3</td>
</tr>
</tbody>
</table>

The difference of the average forward permeate flux between experimental and the model results vary from +15.3% to -18.0%. In general, these results show that the model shows good correlation with the experimental flux data. Figures 6.14 and 6.15 show the model and experimental flux versus airflow for calcined alumina suspensions at different transmembrane pressures with a solid volume fraction \( \Phi_b = 0.02 \) and 0.03, respectively.
Figure 6.14 - Flux versus airflow - experimental results and model prediction - calcined alumina suspension with solid volume fraction of 0.02

Figure 6.15 - Flux versus airflow - experimental results and model prediction - calcined alumina suspension with solid volume fraction of 0.03

A typical graph of flux versus time showing a comparison of the experimental results and the model prediction for alumina suspension is shown in Figure 6.16. Other graphs are presented in Appendix 4.
Figure 6.16 – Flux versus time for calcined alumina, $\Delta P = -33.8$ kPa, airflow of 1.42 L/s and $\phi_b = 0.03$

### 6.3.3 Yeast Suspensions

Table 6.7 and Table 6.8 gives the calculated values for “a” and “b” for yeast suspensions of $\phi_b = 0.01$ and 0.02, respectively.
Table 6.7 - Values of “α” and “β” for yeast suspension, Φ₀ = 0.01

<table>
<thead>
<tr>
<th>Φ₀</th>
<th>ΔP  (kPa)</th>
<th>Airflow (L/s)</th>
<th>a ( \times 10^{22} )</th>
<th>b ( \times 10^{20} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>-16.93</td>
<td>0.47</td>
<td>2.47 ± 0.02</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>2.33 ± 0.30</td>
<td>1.66</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>2.47 ± 0.07</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>2.14 ± 0.09</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>1.55 ± 0.2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-33.8</td>
<td>0.47</td>
<td>1.67 ± 0.13</td>
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<td>0.94</td>
<td>1.71 ± 0.04</td>
<td>0.97</td>
</tr>
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<td></td>
<td></td>
<td>1.42</td>
<td>1.42 ± 0.01</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>1.34 ± 0.01</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>1.33 ± 0.04</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-50.8</td>
<td>0.47</td>
<td>3.69 ± 0.18</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>1.17 ± 0.02</td>
<td>1.04</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>3.00 ± 0.37</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>1.96 ± 0.02</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>2.13 ± 0.08</td>
<td></td>
</tr>
</tbody>
</table>
Table 6.8 - Values of “a” and “b” for yeast suspension, \( \Phi_b = 0.02 \)

<table>
<thead>
<tr>
<th>( \Phi_b )</th>
<th>( \Delta P ) (kPa)</th>
<th>Airflow (L/s)</th>
<th>( a ) ( \times 10^{22} )</th>
<th>( b ) ( \times 10^{20} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.02</td>
<td>-33.8</td>
<td>0.47</td>
<td>6.72 ± 0.63</td>
<td>1.76</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>5.14 ± 0.32</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>6.82 ± 1.75</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>5.66 ± 0.59</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>4.94 ± 0.42</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-50.8</td>
<td>0.47</td>
<td>5.22 ± 0.06</td>
<td>2.28</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>5.51 ± 0.25</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>4.66 ± 0.40</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>5.01 ± 0.21</td>
<td></td>
</tr>
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<td></td>
<td></td>
<td>2.36</td>
<td>5.06 ± 0.09</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-67.7</td>
<td>0.47</td>
<td>7.82 ± 0.85</td>
<td>3.09</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>6.86 ± 0.48</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>5.89 ± 0.24</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>5.89 ± 0.08</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>5.28 ± 0.32</td>
<td></td>
</tr>
</tbody>
</table>

Figures 6.17 and 6.18 show the value of “a” versus airflow at different transmembrane pressure for yeast suspensions. In general, as in the previous applications, “a” tends to decrease as the airflow increases. Again, for the more concentrated suspensions, significant errors were observed during filtration at low transmembrane pressures.
Figure 6.17 - "$\alpha$" versus airflow for yeast suspension with solid volume fraction $\Phi_b = 0.01$ and different transmembrane pressures

Figure 6.18 - "$\alpha$" versus airflow for yeast suspension with solid volume fraction $\Phi_b = 0.02$ and different transmembrane pressures
Figures 6.19 and 6.20 show the graphs "a" versus transmembrane pressures for yeast suspensions with different airflows. Interestingly, for both concentrations it was observed that, within error, "a" is unchanged as the transmembrane pressure increased.

![Graph 6.19](image)

Figure 6.19 - "a" versus transmembrane pressure for yeast suspension with solid volume fraction $\Phi_b = 0.01$ and different airflows.

![Graph 6.20](image)

Figure 6.20 - "a" versus transmembrane pressure for yeast suspension with solid volume fraction $\Phi_b = 0.02$ and different airflows.

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In Figure 6.21 the value of “$b$” versus transmembrane pressure are plotted for yeast suspensions with solid volume fractions of $\phi_b = 0.01$ and $\phi_b = 0.02$. The values of “$b$” for yeast suspensions were found to be in the same order of magnitude of the calcined alumina suspensions, and at least one order of magnitude lower than the values of “$b$” of flexographic ink suspensions.

![Graph showing $b$ versus transmembrane pressure for yeast suspensions](image)

Figure 6.21 - “$b$” versus transmembrane pressure for yeast suspensions

Tables 6.9 and 6.10 show the values of the average forward permeate fluxes during 360 seconds predicted by the model and the same values obtained during the experiments for yeast suspensions of $\phi_b = 0.01$ and $\phi_b = 0.02$, respectively.
Table 6.9 - Average flux for yeast suspensions with solid volume fraction of 0.01 during first 360 seconds

<table>
<thead>
<tr>
<th>$\phi_0$</th>
<th>$\Delta P$ (kPa)</th>
<th>Airflow (L/s)</th>
<th>Model (L/m²h)</th>
<th>Experiment (L/m²h)</th>
<th>Difference (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>-16.9</td>
<td>0.47 22.7</td>
<td>21.1</td>
<td>21.1</td>
<td>+7.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94 23.0</td>
<td>21.3</td>
<td>21.3</td>
<td>+7.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42 22.7</td>
<td>21.0</td>
<td>21.0</td>
<td>+7.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89 23.4</td>
<td>22.1</td>
<td>22.1</td>
<td>+5.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36 25.2</td>
<td>24.6</td>
<td>24.6</td>
<td>+2.4</td>
</tr>
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<td></td>
<td>-33.8</td>
<td>0.47 41.9</td>
<td>41.7</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>0.94 41.7</td>
<td>39.4</td>
<td>39.4</td>
<td>+5.5</td>
</tr>
<tr>
<td></td>
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<td>1.42 43.4</td>
<td>42.9</td>
<td>42.9</td>
<td>+1.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89 43.9</td>
<td>43.4</td>
<td>43.4</td>
<td>+1.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36 44.0</td>
<td>43.1</td>
<td>43.1</td>
<td>+2.0</td>
</tr>
<tr>
<td></td>
<td>-50.8</td>
<td>0.47 47.1</td>
<td>39.3</td>
<td>39.3</td>
<td>+16.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94 59.3</td>
<td>48.2</td>
<td>48.2</td>
<td>+18.7</td>
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<tr>
<td></td>
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<td>1.42 48.6</td>
<td>46.2</td>
<td>46.2</td>
<td>+4.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89 52.5</td>
<td>49.8</td>
<td>49.8</td>
<td>+5.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36 51.6</td>
<td>48.0</td>
<td>48.0</td>
<td>+7.0</td>
</tr>
</tbody>
</table>
Table 6.10 - Average flux for yeast suspensions with solid volume fraction of 0.02 during first 360 seconds

<table>
<thead>
<tr>
<th>$\phi_b$</th>
<th>$\Delta P$ (kPa)</th>
<th>Airflow (L/s)</th>
<th>Model (L/m²h)</th>
<th>Experiment (L/m²h)</th>
<th>Difference (%)</th>
</tr>
</thead>
<tbody>
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<td>22.5</td>
<td>17.2</td>
<td>+23.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>23.5</td>
<td>18.8</td>
<td>+20.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>22.5</td>
<td>16.8</td>
<td>+25.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>23.1</td>
<td>18.2</td>
<td>+21.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>23.6</td>
<td>18.8</td>
<td>+20.3</td>
</tr>
<tr>
<td></td>
<td>-50.8</td>
<td>0.47</td>
<td>28.0</td>
<td>24.8</td>
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<td></td>
<td></td>
<td>0.94</td>
<td>27.7</td>
<td>24.2</td>
<td>+12.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>28.7</td>
<td>25.5</td>
<td>+11.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.89</td>
<td>28.3</td>
<td>25.4</td>
<td>+10.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
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<td>24.4</td>
<td>+13.5</td>
</tr>
<tr>
<td></td>
<td>-67.7</td>
<td>0.47</td>
<td>28.5</td>
<td>27.4</td>
<td>+3.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.94</td>
<td>29.2</td>
<td>28.1</td>
<td>+3.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.42</td>
<td>30.2</td>
<td>29.3</td>
<td>+3.0</td>
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<td></td>
<td></td>
<td>1.89</td>
<td>30.2</td>
<td>29.3</td>
<td>+3.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.36</td>
<td>31.0</td>
<td>30.1</td>
<td>+2.9</td>
</tr>
</tbody>
</table>

The difference of the average forward permeate flux between experimental and the model results vary from +0.5\% to +25.3\%. It is interesting to note that the model tends to over-predict fluxes for all tests. Yet, these results show that the model gives good correlation with the experimental data. Figures 6.22 and 6.23 show the model and experimental data flux versus airflow for calcined alumina suspensions at different transmembrane pressures with a solid volume fraction $\phi_b = 0.01$ and 0.02, respectively.
Figure 6.22 - Flux versus airflow - experimental results and model prediction - yeast suspension with solid volume fraction of 0.01

Figure 6.23 - Flux versus airflow - experimental results and model prediction - yeast suspension with solid volume fraction of 0.02

A typical graph of flux versus time showing a comparison of the experimental results and the model prediction for yeast suspension is shown in Figure 6.24. Other graphs are presented in Appendix 4.
Figure 6.24 – Flux versus time for yeast, $\Delta P = -33.8$ kPa, airflow of 1.42 L/s and $\Phi_b = 0.01$

6.4 Summary

The objective of this chapter was to validate quantitatively the model by substituting the suspension and operating variables into the model equation and comparing with the experimentally determined filtration data. Six suspensions were tested: The experimental validation of the model was carried out by substituting the experimentally determined suspension and operating variables into the model. Some variables were grouped to form two lumped parameters “$a$” and “$b$”.

The parameter “$a$” was determined experimentally from the pseudo steady-state flux, when the value of time $t$ is high, and the membrane resistance is so small compared to the cake resistance that it could be neglected. The parameter “$b$” was determined applying the dead-end filtration theory. The slope of $\left(\frac{J_0}{J(t)}\right)^2 - 1$ versus $t$ is equal to $\left(\frac{2 \cdot b \cdot \Phi_b \cdot \Delta P}{R_m^2}\right)$. The behavior of these lumped parameters “$a$” and “$b$” was analyzed in respect to airflow and transmembrane pressure.

The validation of the model was accomplished, and the results showed an overall agreement between model prediction and experimental observations.
The objective of the thesis was to develop a model for flux prediction in a unique membrane system for use in the pulp and paper industry. The membrane system, patented by Zenon Environmental Inc., consists of a submerged hollow-fibre microfiltration unit, and uses air to create turbulence, which, in turn, produces shear forces at the membrane surface. The shear forces help to keep the membrane surface clean thus improving flux.

The first step to accomplish the objective was to undertake a preliminary investigation in order to select potential applications of microfiltration in the pulp and paper industry. After conducting a literature survey, it was concluded that the treatment of some effluents originating from pulp and paper processes would have good potential. Four different applications were selected for further laboratory screening tests. These tests provided important information that enabled an analysis of the technical feasibility of each application. Moreover, the tests also provided important information about the behaviour of the system with respect to flux for each tested application, as described in Chapter 4.

A summary of the observations in the preliminary tests is presented below:

(i) Transmembrane pressure had an effect on flux, i.e., flux increased as transmembrane pressure increased. It was observed, however, that normally the transmembrane pressure had a greater effect on flux during the filtration of the dilute suspensions as compared to the more concentrated ones. It was also observed that the flux increased significantly up to a certain value of transmembrane pressure, after which it increased only moderately.

(ii) An increase in airflow also increased flux. It was observed that in some applications there was a limit after which the increase of airflow would no longer have a significant effect on flux. During the membrane bio-reactor filtration in Application 4, it was observed that airflow did not have any significant effect on flux. Originally it was speculated that this phenomenon was due to the fact that the biological sludge would have a different behaviour from other types of suspensions because of its rheological characteristics. However, other reasons could also explain this
phenomenon. For example, the range of airflow values tested may have reached the limit after which no significant effect of airflow is observed.

(iii) Concentration of the suspension had an important effect on flux. As expected, flux was always considerably lower for the more concentrated suspensions.

(iv) The cleaning of the membranes was usually easy to accomplish and the clean water flux was restored. However, in the case of the BCTMP effluent, it was difficult to clean the membranes using the standard cleaning procedures, and the clean water flux was not restored. This indicated that the characteristics of the suspension can have a significant effect on the interaction of the solutes and the membrane, causing severe flux decline due to internal fouling.

Microfiltration of old newsprint de-inking effluent containing flexographic ink was considered to have good industrial applicability, and thus microfiltration was selected for modeling. Considering the observations from the preliminary studies, and after conducting a literature survey on the existing models for flux prediction in microfiltration and ultrafiltration systems, a novel model was proposed.

The existing models for microfiltration have focused on concentration polarization, and on the different mechanisms of back-transport of particles from the membrane surface. The mechanisms of back-transport of particles from the membrane surface can be divided in two categories: diffusion and hydrodynamics. Some of the models proposed in the literature are primarily based on diffusion mechanisms accounting for the back-transport of solute away from the membrane surface. Others assume that in systems where hydrodynamic shear forces take place at the membrane surface, the back-transport of particles is caused mainly by these forces, and diffusion can be neglected. Because of the characteristics of the system in which high-shear stresses are produced by air bubbles at the membrane surface, the model assumed that non-diffusive transport phenomena are the main mechanisms for the back-transport of particles from the membrane surface to the bulk solution during filtration. Thus the back-transport of particles caused by diffusion was neglected.

The industry often uses reverse filtration as a mechanism to keep the membrane clean, and thus improving the overall flux. The transient flux, i.e., the flux before reaching the quasi-steady-state, is an important characteristic of the model when reverse filtration
(backflushing) is considered. In a previous work, published by Redkar (1994), the behaviour of flux decline in cross-flow filtration was approximated by modeling it as in the dead-end filtration. Based on the experimental observations made in this work with the submerged hollow-fiber system, this approximation is valid only for the first few seconds of filtration after which the flux decline is significantly lower when air is applied to the system.

The experimental observations led to a modification of the model with respect to the particle mass balance at the membrane surface of the dead-end filtration theory. A new transient flux expression was developed which includes a term to express the transport of solute away from the membrane surface due to shear stress. This term was based on an expression proposed by Schulz and Ripperger (1989).

The following assumptions were made in the derivation of the model:

(i) the fluid properties are constant with respect to time
(ii) the shear stress is constant with respect to time
(iii) the specific cake resistance is constant with respect to time
(iv) the solute concentration in the permeate is negligible
(v) back diffusion of particles is negligible

Although several other important characteristics related to the suspension such as particle size, particle size distribution, and particle shape are not explicit in the model, their effects appear indirectly in the constants.

The model was originally developed for a specific membrane configuration to be used in pulp and paper applications. However, the time-dependent expression for flux, as shown in Equation 4.6, may be applied to any type of high-shear membrane microfiltration system, and different types of suspensions. In this work, only newtonian fluid was considered. For non-newtonian fluids in which viscosity is dependent on the shear rate, i.e., viscosity varies with varying shear rate, the velocity gradient \( dU/dy \) in Equation 4.6, can be replaced by an expression that defines the particular fluid behaviour.
A limitation of the model is that the decrease in flux due to irreversible fouling (internal fouling) is not considered. Although an empirical solution for this problem has been addressed in Chapter 4, a more substantial study is required if a complete solution is desired.

It is important to consider that the microfiltration configuration used in this work is unique, and the effects produced by air bubbles on the membrane surface are difficult to estimate. As described in Chapter 4, the model does take into account the effect of shear stress at the membrane surface, but it considers it as a single parameter. Two questions arise from these premises: (i) how shear stress is affected by airflow? (ii) what are the indirect effects of airflow on the hydrodynamics of the system during filtration?

This thesis did not intend to give an answer for the above questions, and so future work is required. However, a possible relationship between shear stress and airflow may be estimated by the empirical correlation of superficial gas velocity, as discussed in Chapter 4.

The second question addressing the effect of airflow on the hydrodynamics of the system is also important and brings about other questions. For instance, is the effect of airflow on permeate flux the same for different membrane fibre length? Is it the same for different membrane fibre tension or for different membrane fibre density? Again, the present work did not try to answer these questions. However, it is recommended that a thorough study of these parameters is undertaken in order to better understand the system, and propose specific modifications that could improve the model.

This work did not determine the optimum conditions, such as the forward and reverse filtration times and transmembrane pressures, where a maximum value of the average flux per time is obtained. Nevertheless, these conditions can be estimated by using the model. In practice, it is crucial to consider the mechanical limitations of the equipment such as the speed of control valves. This would limit, for instance, the minimum possible reverse filtration time. It is also important to consider the system efficiency, e.g., how small the reverse transmembrane pressure can be to effectively remove the cake. These values need to be determined experimentally.
The model was first verified numerically by computer simulations. These numerical simulations conformed to the anticipated behaviour of its parameters, as described in Chapter 5. The experimental validation of the model was accomplished using a lab-scale module, which had the same membrane fibre length as is used in industrial-scale modules. Although this has caused some difficulties in physically managing the system, especially during the membrane cleaning procedure, the results obtained from testing are presumably comparable to what is expected from an industrial-scale system.

It would be difficult to validate the model using the ONP effluent, because of the complexity of this type of suspension. Therefore, three synthetic materials, flexographic ink, calcined alumina and yeast, were selected for testing, and the required variables were carefully determined. Flexographic ink was chosen because of its connection to the selected potential industrial application. Calcined alumina and yeast were chosen because of their uniform particle size distribution, and the latter also because of its fouling characteristics as a biological material.

A difficulty found when calcined alumina and yeast suspensions were tested was that the particles tended to settle at the bottom of the tank, especially when low airflow values were tested. In order to minimize this problem, the suspension was vigorously stirred between each experimental run.

Membranes were cleaned after each run and reused, i.e., the same membranes were used in all tests. This resulted in permanent progressive fouling of the membranes. Therefore, membrane resistance increased over time. For example, for the first tests with new membranes, performed with calcined alumina, the forward membrane resistance was 4.59 x 10^{11} \text{ m}^{-2}, while prior to the yeast tests it was 9.37 x 10^{11} \text{ m}^{-2}.

The qualitative validation of the model was carried out and, in general, the model realistically represented the experimental filtration behaviour. Although different materials, with different physical and rheological characteristics were tested, generally the flux behaviour conformed to the model prediction. For every tested suspension, flux increased as transmembrane pressure increased. However, the rate of increase was different for each application. For example, transmembrane pressure had a higher effect on flux during ink
suspension filtration than during calcined alumina suspension. Lojkine (1992) suggests that increases in transmembrane pressure are counteracted by increases in resistance due to deposition of material on the membrane surface and the degree and rate of membrane fouling. Normally, for more concentrated suspensions, the effect of transmembrane pressure was less significant. Thus, factors such as particle size, particle shape and compressibility, and suspension concentration play an important role in the effect of transmembrane pressure on flux.

During the filtration of ink and yeast suspensions a significant increase in flux was observed when airflow was increased from 0 to 0.94 L/s, after which the effect of air was very small. The rate of increase on flux due to airflow was more significant during flexo-ink suspensions. This is in agreement with the studies of Tarleton and Wakeman (1994) on the influence of cross-flow velocity on flux for different materials: they observed that the effect of cross-flow velocity of flux was more significant when finer materials were tested. Moreover, in the system configuration used for this work other factors may have an indirect effect on flux. For example, the concentration of solids in the suspension may affect the bubbling regime in the tank, thus changing the hydrodynamics of the system.

In order to validate the model quantitatively, it was necessary to group some parameters that are difficult to determine into two lumped parameters, which were easier to determine. The results showed an overall agreement between model prediction and experimental observation.

In order to facilitate the use of the model by the industry, a computer program was developed in Microsoft Visual Basic™ as presented in Appendix 5.

Finally, it is important to point out that this project was undertaken in collaboration with Zenon Environmental Inc. I believe this research helped Zenon to better understand the potential uses of the microfiltration system in the pulp and paper industry. The model studies not only assisted Zenon to better understand their microfiltration configuration, but it also may be a useful tool for future system design.
Membrane microfiltration is a potential separation technology for pulp and paper industry, particularly to treat effluents and to facilitate their reuse. From the preliminary laboratory tests, the following conclusions were drawn:

(i) Application 1: Old Newsprint (ONP) De-inking Effluent Containing Flexographic Ink
The treatment of this effluent using microfiltration proved to be technically feasible, and it has good potential for industrial application.

(ii) Application 2: Effluent from Paper Coating Operation
The removal of suspended solids by microfiltration led to good quality permeates. The filterability of these effluents was poor, especially for the "coating break" effluent. The recovery of the concentrate at a consistency of 16.5% is possible. However, considering the flux limitation of this stream, Application 2 was not considered appropriate for this type of membrane configuration.

(iii) Application 3: Effluent from Bleached Chemi-thermo-mechanical Pulp (BCTMP) Mill
The filterability of this effluent was very poor, and the membrane did not completely recover its water flux capacity after cleaning. The reverse osmosis experiment has not been carried out, and an analysis of mill integration is difficult without these results. However, the flux limitation encountered in the microfiltration experiments suggests that this hollow-fibre configuration is not appropriate to treat this effluent.

(iv) Application 4: Membrane Bio-Reactor Treatment of OCC Recycling Effluent
The effluent from the OCC mill has high levels of COD (3500 mg/L) and BOD₅ (2000 mg/L). An overall removal rate of 95% of COD and 99% of BOD₅ was achieved after stabilization of the system. The permeate fluxes obtained during the filtration tests were high. The recycling of the treated effluent to the mill process is viable.
From the results of the screening tests, two applications using microfiltration were identified and have good potential industrial applicability. They were:

(i) Microfiltration treatment of ONP effluent containing flexographic ink;
(ii) Membrane bio-reactor treatment of OCC effluent.

This part of the work contributed new information about the general use of membranes in the pulp and paper industry and about the effectiveness of the membrane system tested.

The second part of the work, a novel semi-empirical model for flux prediction in high-shear microfiltration systems was developed. The model was validated numerically by computer simulations and experimentally using synthetic suspensions. Numerical simulations of this new model conform to the anticipated behaviour of its parameters. The model predicted that:

(i) Assuming the membrane and fouling resistance negligible compared to the cake resistance, flux was directly proportional to the square root of the transmembrane pressure $\Delta P$ for incompressible cakes ($s \rightarrow 0$). Otherwise, for highly compressible cake ($s \rightarrow 1$), the transmembrane pressure drop $\Delta P$ would have an insignificant effect on flux.

(ii) Flux decreased with an increase in permeate viscosity $\eta_p$. In the low viscosity region, small changes in viscosity caused significant changes in flux, while in the high viscosity region, the changes were smaller.

(iii) Increases in the solid volume fraction in the suspension $\phi_s$ resulted in decreases of the flux, and the effect of transmembrane pressure $\Delta P$ on flux was more significant for lower values of $\phi_s$.

(iv) Increases in solid volume fraction in the cake $\phi_c$ resulted in decreases of the flux. The model also predicted that flux tended to reach steady-state faster as the solid volume fraction in the cake $\phi_c$ increased.

(v) Flux increased as shear stress $\tau_w$ increased. A rapid increase in flux was observed until a certain value of the applied shear stress, after which it increased moderately.

(vi) The effect of transmembrane pressure $\Delta P$ on flux was more significant when higher shear stresses were applied.
The validation of the model was accomplished, and the results showed an overall agreement between model prediction and experimental observations. Generally an increase in permeate flux was observed when airflow was increased, due to an increase in the shear stress at the membrane surface. As predicted by the model, a rapid increase occurred until a certain value of applied airflow, after which only small changes were observed. For all applications, backflushing had a significant effect on restoring the initial membrane flux. During three cycles of filtration of every suspension, the initial flux was almost completely restored, and the average permeate flux per cycle did not vary significantly.
9. RECOMMENDATIONS

(i) Test other potential applications of microfiltration in the pulp and paper industry, including options where suspended solid removal pre-treatment is required.

(ii) Verify the model using industrial suspensions.

(iii) Extend the model to consider long-term fouling.

(iv) Increase understanding of the hydrodynamics of the system, in order to determine:

- the optimum rate of airflow needed;
- the effect of membrane fiber density (related to the number of fibers per unit of area) on membrane performance;
- the effect of membrane tension on the performance of the system.

(v) Determine the optimum value for the forward filtration time, as well as the forward filtration transmembrane pressure.

(vi) Determine the optimum value for the reverse filtration time, as well as the reverse filtration transmembrane pressure.
REFERENCES


152


Ferry, J.D., Chemical Reviews, 19 (3): 373 (1936).


Green, G., Belfort, G., Desalination, 35:129 (1980).


Matthiasson, E., Sivik, B., Desalination, 35(1,2,3): 59 (1980).


Olsen, O., Desalination, 35(1,2,3): 291 (1980).


Appendix 1

Particle size distribution for flexographic ink, calcined alumina, and yeast
Result Derived Diameters Report

Sample Details
Run Number: 2
Record Number: 6
Sample File: SILVA1
Sample Path: D:SIZERS\DATA1
Sample Name:

System Details
Range Lens: 300RF mm
Presentation: 3TJD
Analysis Model: Polydisperse
Modifications: Active

Sample Details
Beam Length: 2.40 mm
[Particle R.I. = (2.5935, 1.0000); Dispersant R.I. = 1.3300]
Killed Result Channels: < 0.05 um; > 5.69 um.

Result Statistics
Concentration = 0.0009 %Vol
Density = 1.080 g / cub. cm
Specific S.A. = 37.5467 sq. m / g

A.S.T.M Derived Diameters (um)

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<tr>
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<tr>
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Distribution Moments

| Volume | 0.26 |
| Surface | 0.15 |
| Length | 0.10 |
| Number | 0.08 |

Distribution Percentiles (um) -- Volume

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<th>Percentile</th>
<th>Size</th>
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<td>0.37</td>
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<td>20.0 %</td>
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Distribution Modal Sizes (um)

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<th>Volume %</th>
<th>Size (um)</th>
<th>Volume %</th>
<th>Size (um)</th>
<th>Volume %</th>
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<td>100.00</td>
<td>5.99</td>
<td>100.00</td>
</tr>
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</table>

**Density**

- Polydispersity: ±0.05 um
- Conc. = 0.0009 %Vol
- Beam: 2.40 mm
- Sampler: None
- Measurements: 3/JO
- Density = 1.030 g/cm³
- Distribution: Volume
- Vdum D(v, 0.1) = 0.07 um
- D(v, 0.5) = 0.19 um
- D(v, 0.9) = 0.52 um
- Span = 2.37E+00
- Analysis: Polydispers

**Killed Result Channels:**

- Size Under % (um) = 0.05 um
- Size Under % (um) = 5.69 um

**S.S.A. = 37.5467 m²/g**

**Run No:** 2
**Rec. No:** 6
**Beam:** 240 mm
**Simpler:** None
**Analysis:** Polydispers

**Malvern Instruments Ltd.**
Malvern, UK
Tel.: [44] (0)1684-892456 Fax.: [44] (0)1684-892789
Sample ID: Baker Yeast
Sample File: SILVA
Sample Path: D:ISIZERS\DATA
Sample Notes: 

Run Number: 2
Record Number: 6

System Details
Beam Length: 2.40 mm
Sampler: None
Obscuration: 15.7 %
Residual: 0.725 %

Analysis Model: Polydisperse
Modifications: None

Distribution Type: Volume
Concentration = 0.0097 %Vol
Density = 1.080 g / cub. cm
Span = 1.253E+00
Specific S.A. = 1.2390 sq. m / g

A.S.T.M Derived Diameters (um)

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Distribution Moments

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Distribution Percentiles (um) — Volume

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<tr>
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</tr>
<tr>
<td>50.0 %</td>
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Distribution Modal Sizes (um)

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<tr>
<td>1</td>
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</tbody>
</table>

Mahem instruments Ltd.
Mahem, UK
Tel:+(44) (0)1684-892456 Fax:+(44) (0)1684-892789

Page 3
12 Aug 98 13:27
### Result: Histogram Table

- **ID:** Baker Yeast
- **File:** SILVA
- **Path:** D:\SIZERS\DATA1
- **Range:** 300RF mm
- **Presentation:** 30HD
- **Measurements:** None
- **Run No.:** 2
- **Rec. No.:** 6
- **Beam:** 2.40 mm
- **Sampler:** None
- **Analysis:** Polydisperse
- **Density:** 1.080 g/cm³
- **D(4, 3) =** 5.70 um
- **D(4, 0) =** 5.26 um
- **Uniformity:** 3.850E-01
- **Source:** Malvern Instruments Ltd.

<table>
<thead>
<tr>
<th>Size (um)</th>
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<th>Size (um)</th>
<th>Volume Under %</th>
<th>Size (um)</th>
<th>Volume Under %</th>
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- **S.S.A. =** 1.2390 m²/g
- **D(3, 2) =** 4.46 um
- **D(3, 0.9) =** 9.30 um

- **Obs.:** 15.7 %
- **Residual: 0.725 %

**Malvern Instruments Ltd.**
**Malvern Instruments Ltd.**
**16 Aug 98 13:27**
Sample ID: Alumina
Sample File: SILVA
Sample Path: D:\SIZERS\DATA
Sample Note: Surfactant added (IGEPAL)

Run Number: 4
Record Number: 8

Measured: Sat Jul 25 1998 3:17PM
Analysed: Sat Jul 25 1998 3:17PM
Result Source: Analysed

System Details
- Range Lens: 300RF mm
- Presentation: 3GHD
- Analysis Model: Polydisperse
- Modifications: None

Sampler: None
Obscuration: 11.2%
Residual: 1.688%

Distribution Type: Volume
Concentration = 0.0013 % Vol
Density = 1.080 g / cub. cm
Span = 1.03E+01
Uniformity = 3.097E+01

A.S.T.M Derived Diameters (um)

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<td>0.18</td>
<td>0.14</td>
</tr>
<tr>
<td>D[2, N]</td>
<td>0.18</td>
<td>0.14</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>D[1, N]</td>
<td>0.12</td>
<td>0.084</td>
<td>6.2426</td>
<td></td>
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</tbody>
</table>

Distribution Moments
- Mean
  Volume: 18.99
  Surface: 0.38
  Length: 0.18
  Number: 0.12

- Standard Deviation
  Volume: 80.081
  Surface: 2.647
  Length: 0.188
  Number: 0.084

- Skewness
  Volume: 4.5814
  Surface: 137.0441
  Length: 166.9811
  Number: 6.2426

- Kurtosis
  Volume: 20.5091
  Surface: 20814.6191
  Length: 361317.3750
  Number: 2887.6846

Distribution Percentiles (um) – Volume

<table>
<thead>
<tr>
<th>Percentile</th>
<th>Size</th>
<th>Percentile</th>
<th>Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.0 %</td>
<td>0.16</td>
<td>80.0 %</td>
<td>1.40</td>
</tr>
<tr>
<td>20.0 %</td>
<td>0.24</td>
<td>90.0 %</td>
<td>6.41</td>
</tr>
<tr>
<td>50.0 %</td>
<td>0.60</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Distribution Modal Sizes (um)

<table>
<thead>
<tr>
<th>Mode</th>
<th>Size</th>
<th>Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.92</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>353.42</td>
<td>3</td>
</tr>
</tbody>
</table>

Result Derived Diameters Report

Malvern Instruments Ltd.
Malvern, UK
Tel:+44 (0)1684-882456 Fax:+44 (0)1684-882789

12 Aug 98 13:29
result: histogram table

id: alumina
file: silva
path: d:\sizers\data
range: 300rf mm
presentation: 3q4d
modification: none

beam: 2.40 mm
sample: none
analysis: polydisperse
obt: 11.2%
residual: 1.688%

calculated:

conc. = 0.0013 %vol
distribution: volume
\( d(v, 0.1) = 0.16 \) um
\( d(v, 0.5) = 0.60 \) um
\( d(v, 0.9) = 6.41 \) um
s.s.a. = 14.7633 m^2/g

span = 1.036e+01

<table>
<thead>
<tr>
<th>size (um)</th>
<th>volume under</th>
<th>volume under</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.055</td>
<td>0.12</td>
<td>0.635</td>
</tr>
<tr>
<td>0.061</td>
<td>0.28</td>
<td>0.700</td>
</tr>
<tr>
<td>0.067</td>
<td>0.58</td>
<td>0.772</td>
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<tr>
<td>0.074</td>
<td>1.00</td>
<td>0.851</td>
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<tr>
<td>0.082</td>
<td>1.55</td>
<td>0.938</td>
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<td>1.03</td>
</tr>
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<td>0.239</td>
<td>19.82</td>
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<tr>
<td>0.263</td>
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<tr>
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<td>25.77</td>
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</tr>
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<td>0.320</td>
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<tr>
<td>0.353</td>
<td>32.08</td>
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<tr>
<td>0.389</td>
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</tr>
<tr>
<td>0.429</td>
<td>38.46</td>
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</tr>
<tr>
<td>0.473</td>
<td>41.72</td>
<td>5.45</td>
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<tr>
<td>0.522</td>
<td>45.03</td>
<td>6.01</td>
</tr>
<tr>
<td>0.576</td>
<td>48.38</td>
<td>6.63</td>
</tr>
</tbody>
</table>

density = 1.080 g/cm^3
\( d(4, 3) = 18.99 \) um
\( d(0.3) = 0.38 \) um

uniformity = 3.097 e+01

measured: 7/25/98 3:17pm
analysed: 7/25/98 3:17pm
source: analysed

malvern instruments ltd.
malvern, uk
phone: (01684-892456) fax: (01684-892789)
Appendix 2

Graphs flux versus time at different transmembrane pressures

Flux versus time for calcined alumina at transmembrane pressures of -16.9, -27.1, and -33.9 kPa, airflow of 0.94 L/s, and suspension solid volume fraction $\Phi_b=0.02$

Flux versus time for calcined alumina at transmembrane pressures of -16.9, -27.1, and -33.9 kPa, airflow of 0.1.42 L/s, and suspension solid volume fraction $\Phi_b=0.02$
Flux versus time for calcined alumina at transmembrane pressures of -16.9, -27.1, and -33.9 kPa, airflow of 0.94 L/s, and suspension solid volume fraction $\phi_s=0.03$

Flux versus time for calcined alumina at transmembrane pressures of -16.9, -27.1, and -33.9 kPa, airflow of 1.42 L/s, and suspension solid volume fraction $\phi_s=0.03$
Flux versus time for flexographic ink at transmembrane pressures of -16.9, -33.8, and -50.8 kPa, airflow of 0.47 L/s, and suspension solid volume fraction $\phi_b=0.005$
Flux versus time for flexographic ink at transmembrane pressures of -16.9, -33.8, and -50.8 kPa, airflow of 0.47 L/s, and suspension solid volume fraction $\phi_b=0.01$
Flux versus time for yeast suspension at transmembrane pressures of -16.9, -33.8, and -50.8 kPa, airflow of 0.94 L/s, and suspension solid volume fraction $\Phi_b=0.01$
Appendix 3

Graphs flux versus time at different airflows

Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -16.9 kPa, and suspension solid volume fraction $\phi_b = 0.02$

Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -27.1 kPa, and suspension solid volume fraction $\phi_b = 0.02$
Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -33.8 kPa, and suspension solid volume fraction $\Phi_b = 0.02$

Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -16.9 kPa, and suspension solid volume fraction $\Phi_b = 0.03$
Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -27.1 kPa, and suspension solid volume fraction $\phi_b = 0.03$

Flux versus time for alumina with airflow of 0, 0.94 and 1.42 L/s, transmembrane pressure of -33.8 kPa, and suspension solid volume fraction $\phi_b = 0.03$
Flux versus time for flexographic ink with airflow of 0, 0.47 and 2.36 L/s, transmembrane pressure of -16.9 kPa, and suspension solid volume fraction $\phi_b = 0.005$.
Flux versus time for flexographic ink with airflow of 0, 0.47 and 2.36 L/s, transmembrane pressure of -50.8 kPa, and suspension solid volume fraction $\Phi_b = 0.005$

Flux versus time for flexographic ink with airflow of 0, 0.47 and 2.36 L/s, transmembrane pressure of -16.9 kPa, and suspension solid volume fraction $\Phi_b = 0.01$
Flux versus time for flexographic ink with airflow of 0, 0.47 and 2.36 L/s, transmembrane pressure of -33.8 kPa, and suspension solid volume fraction $\Phi = 0.01$

Flux versus time for flexographic ink with airflow of 0, 0.47 and 2.36 L/s, transmembrane pressure of -50.8 kPa, and suspension solid volume fraction $\Phi = 0.01$
Flux versus time for yeast suspension with airflow of 0, 0.94 and 2.36 L/s, transmembrane pressure of -16.9 kPa, and suspension solid volume fraction $\Phi_b = 0.01$

Flux versus time for yeast suspension with airflow of 0, 0.94 and 2.36 L/s, transmembrane pressure of -33.8 kPa, and suspension solid volume fraction $\Phi_b = 0.01$
Flux versus time for yeast suspension with airflow of 0, 0.94 and 2.36 L/s, transmembrane pressure of -50.8 kPa, and suspension solid volume fraction $\phi_s = 0.01$
Appendix 4

Graphs flux versus time: experimental results and model prediction

Flux versus time for flexographic ink, $\Delta P = -50.8$ kPa, airflow of 0.94 L/s and $\phi_b = 0.005$

Flux versus time for flexographic ink, $\Delta P = -33.8$ kPa, airflow of 0.94 L/s and $\phi_b = 0.005$
Flux versus time for flexographic ink, $\Delta P = -16.9$ kPa, airflow of 2.36 L/s and $\Phi_b = 0.005$

Flux versus time for flexographic ink, $\Delta P = -33.8$ kPa, airflow of 1.89 L/s and $\Phi_b = 0.01$
Flux versus time for flexographic ink, $\Delta P = -50.8$ kPa, airflow of 0.94 L/s and $\phi_b = 0.01$

Flux versus time for flexographic ink, $\Delta P = -16.9$ kPa, airflow of 1.42 L/s and $\phi_b = 0.01$
Flux versus time for yeast, $\Delta P = -16.9$ kPa, airflow of 1.89 L/s and $\Phi_b = 0.01$

Flux versus time for yeast, $\Delta P = -33.8$ kPa, airflow of 0.94 L/s and $\Phi_b = 0.01$
Flux versus time for yeast, $\Delta P = -50.8$ kPa, airflow of 2.36 L/s and $\Phi_0 = 0.01$.

Flux versus time for yeast, $\Delta P = -33.8$ kPa, airflow of 0.94 L/s and $\Phi_0 = 0.02$.
Flux versus time for yeast, $\Delta P = -50.8$ kPa, airflow of 1.89 L/s and $\phi_b = 0.02$

Flux versus time for calcined alumina, $\Delta P = -16.9$ kPa, airflow of 1.42 L/s and $\phi_b = 0.02$
Flux versus time for calcined alumina, $\Delta P = -33.8$ kPa, airflow of 0.94 L/s and $\phi_b = 0.03$

Flux versus time for calcined alumina, $\Delta P = -27.1$ kPa, airflow of 1.42 L/s and $\phi_b = 0.03$
Appendix 5

Computer program for flux prediction in high-shear microfiltration system