

**Flux enhancement in a spiral wrap
ultrafiltration element by using backpulsing**

by

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Declaration

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Abstract

The effect of backpulsing on the prevention of fouling of a 2.5 inch spiral wrap cross-flow ultrafiltration element was investigated experimentally. Backpulsing experiments with an organic (dextrin) solution and an inorganic (kaolin) suspension were performed using a polypropylene membrane with 100 000 molecular mass cut point. The dextrin feed concentration ranged from 250 to 750 mg/L, the kaolin feed concentration ranged from 100 to 300 mg/L, the feed flow rate ranged between 500 and 1500 L/h, and the feed pressure was fixed at 100 kPa. Backpulsing involves applying pressure pulses ranging from 100 to 150 kPa on the permeate space. The pulsing interval varied between 1 and 15 s and pulse duration varied between 0.1 and 0.5 s.

Experimental results showed that backpulsing was effective in reducing membrane fouling and improving membrane flux. With continual backpulsing the net flux was found to increase with increasing backpulsing pressure, increasing weakly with increasing cross-flow rate and decreasing strongly with increasing feed concentration. The best backpulsing parameters for the respective foulants were found to be the following: 0.2 s pulse duration, 3 s pulse interval and 150 kPa backpulse pressure for the dextrin solution, and 0.2 s pulse duration, 5 s pulse interval and 150 kPa backpulse pressure for the kaolin suspension. The best flux results achieved using backpulsing under these conditions were 3-fold and 1.5-fold higher than the fluxes obtained in the non pulsing case for the dextrin and kaolin feeds, respectively.

After the membrane had been exposed to fouling and then backpulsing, it was cleaned, using clean water with backpulsing. The flux values of the clean membrane, previously fouled with dextrin and kaolin were 62% and 71% of the original clean membrane fluxes, respectively.

The Taguchi method with L9 orthogonal array was used to identify the influential factors backpulsing that give maximum permeate flux. It was found that pulse pressure has the strongest effect on membrane flux. Pulse interval and pulse duration have negligible effects and, in comparison cross-flow rate has a weak effect on the membrane flux. It must be noted that these observations are only valid within the experimental boundaries, as identified during the preliminary investigation.

Opsomming

Die effek van teenpolsing op die aanvuiling van 'n 2.5-duim spiraal kruisvloei ultrafiltrasië element is eksperimenteel ondersoek. Teenpols eksperimente met 'n organiese (dekstraan) oplossing en 'n anorganiese (kaolien) suspensie is uitgevoer deur gebruik te maak van 'n polipropileenmembraan (100 000 molekulêre massa snypunt). Die konsentrasie van die dekstraanoplossing was tussen 250 en 750 mg/L en die konsentrasie van die kaolien oplossing was tussen 100 en 300 mg/L. Teenpolsing behels die aanwending van drukpols van tussen 100 en 150 kPa aan die kant van die produk (permeaat). Die polstussenposes het gewissel tussen 1 en 15 s en die duur van die polse tussen 0.1 en 0.5 s. Die vloeitempo was tussen 500 en 1500 L/h, en die toegepaste druk was 100 kPa.

Eksperimentele resultate het getoon dat terugpols effektief was vir die vermindering van membraanaanvuiling, en die verbetering van vloei deur die membraan. Met aanhoudende terugpolsing het die netto vloei toegeneem met toenemende terugpolsdruk. Daar was 'n effense toename met 'n toename in kruisvloeitempo en 'n sterk afname met toenemende voeroplossingskonsentrasie. Die beste terugpols parameters vir die twee verskillende aanvuilingsmateriale was soos volg: 0.2 s polsduur, 3 s polstussenpose en 150 kPa terugpolsdruk vir die dekstraanoplossing; en 0.2 s polsduur, 5 s polstussenpose en 150 kPa terugpolsdruk vir die kaolien-suspensie. Die beste resultate behaal vir vloei onder hierdie kondisies was 3-maal en 1.5-maal hoër as die vloei behaal sonder polsing, vir dekstraan en kaolien, onderskeidelik.

Nadat die membraan aan aanvuiling, gevolg deur terugpolsing, blootgestel is, is dit skoongemaak deur skoon water met terugpolsing te gebruik. Die vloei van die skoon membraan wat voorheen met dekstraan en kaolien aangevuil is was 62% en 71% van die oorspronklike vloei, onderskeidelik.

Die Taguchi metode met 'n L9 ortagonale reeks is gebruik om die belangrike terugpolsfaktore te bepaal wat 'n maksimum permeaatvloei tot gevolg gehad het. Die polsdruk het die grootste effek op die membraanvloei gehad. Polstussenpose en polsduur het 'n onbeduidende effek en die dwarsvloeitempo het 'n swak effek op membraanvloei gehad. Daar moet egter opgelet word dat hierdie waarnemings slegs van toepassing is binne die eksperimentele grense soos bepaal in die inleidende ondersoek van hierdie studie.

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List of Abbreviations

BP	Backpulsing
BSA	Bovine serum albumin
CFF	Cross-flow filtration
CP	Concentration polarization
CIP	Cleaning-in-place
DEF	Dead-end filtration
DOE	Design of experiment
ED	Electrodialysis
EDTA	Ethylenediaminetetracetic acid
MF	Microfiltration
MWCO	Molecular weight cut-off
NF	Nanofiltration
NOM	Natural organic matter
PA	Polyamide
PES	Polyethersulphone
PP	Polypropylene
PS	Polysulphone
RO	Reverse osmosis
SLS	Sodium lauryl sulphate
SWRO	Sea water reverse osmosis
TMB	Transmembrane pressure
UF	Ultrafiltration

List of Symbols

C_w	Concentration of solute at the membrane surface, eq. (2.2)	(g/L)
C_b	Concentration of solute in the bulk solution, eq. (2.2)	(g/L)
dp	Effective transmembrane pressure, eq. (2.1)	Pa
D	Diffusion coefficient, eq. (2.2)	(m ² /s)
J	Membrane flux, eq. (2.1)	L/(m ² .h)
J_v	Transmembrane flux, eq. (2.2)	L/(m ² .h)
J_o	Clean membrane flux	L/(m ² .h)
J_s	Steady-state fouled membrane flux with continual backpulsing	L/(m ² .h)
J_{so}	Steady-state fouled membrane flux without backpulsing	L/(m ² .h)
J_r	Recovered clean membrane flux after cleaning by backpulsing, using clean water	L/(m ² .h)
L	Liter	
n	The number of measurements taken in one test run, eq. (5.1)	
S/N	Signal-to-noise ratio, eq. (5.1)	
R_m	Membrane resistance, eq. (2.1)	(m ⁻¹)
R_f	Fouling layer resistance, eq. (2.1)	(m ⁻¹)
R_p	Concentration polarization resistance, eq. (2.1)	(m ⁻¹)
Y_i	The individually measured response value, eq. (5.1)	
μ	Viscosity of the solvent, eq. (2.1)	kg/(m.s)
Q_p	Net permeate flow, eq. (3.1)	L/h
t_f	The duration of forward filtration, eq. (4.1)	sec
t_b	The duration of reverse filtration, eq. (4.1)	sec

Chapter 1

Introduction and objectives

1.1 Introduction

Membrane filtration processes are widely used in many industrial separation applications and, in some situations, competes with conventional processes such as carbon adsorption, solvent extraction, distillation and ion-exchange [1]. The most common membrane processes are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). Although varying in transmembrane pressure difference (namely the driving force) and average pore diameter, each membrane serves as a selective barrier by permitting certain components of a mixture to pass through while rejecting the others. This results in two streams: permeate and retentate [2].

The major obstacles to the successful use of membrane separation processes are phenomena known as concentration polarization (CP) and fouling. CP results from the accumulation of rejected particles near or on the surface of the membrane due to convective and back-diffusion of the particles and solute molecules. As long as the particles or solute concentration at the membrane surface does not reach the saturation value the CP layer is mobile and does not offer a significant resistance to the permeate flow [3-5]. Fouling refers to the deposition of rejected particles on the membrane surface (external fouling) or the deposition and adsorption of small particles or macromolecules at the pore entrances or within the internal pore structure of the membrane (internal fouling) [6]. Fouling leads to loss of permeate flux, and a reduction in membrane selectivity, costing time and money to clean or replace membranes [7].

Various methods exist to decrease membrane fouling, including feed pretreatment [8, 9], surface modifications of membranes [10], hydrodynamic optimization of operating conditions, cleaning of membranes with chemical agents [11-14], periodic backwashing [15-20], and use of pulsatile flow [21-25]. Unfortunately all these techniques are inefficient in one way or another, so that periodic membrane cleaning is still unavoidable. The routine shutting down of filtration plants for chemical or mechanical cleaning, or both, is time-consuming and costly procedure. The chemicals used for cleaning may also reduce the lifetime and efficiency of the

membrane, and the disposal of the chemicals can also present a problem since they have to be disposed of in an environmentally friendly way.

A promising technique for fouling reduction that does not require the plant to be shut down and does not generate any waste fluids is backpulsing. Backpulsing involves reversing the permeate flow through the membrane from the permeate side to the feed side. Flow reversal occurs every few seconds or less when the pressure pulses (backpulses) are applied for short periods of time, much less than one second. This flow reversal disrupts the concentration polarization layer and dislodges deposited foulants from the membrane pores and the membrane surface, and the foulants are then swept away by the cross-flow (if present), leading to a reduction in membrane fouling and a considerable enhancement of the flux [26-30]. The principal parameters that are proposed to affect the pulsing are: pulse duration, pulse interval and backpulsing pressure. The pulsing shape was achieved as a square peak function (see Section 3.1.2).

Cross-flow filtration with backpulsing has been extensively studied by several groups for the cleaning of flat sheet membranes and capillary membranes, using various foulants. [31-33]. It has been reported to be a most effective method for the reduction of fouling and enhancing the permeate flux. There is an optimum combination of backpulsing parameters that reduces membrane fouling and maximizes the permeate flux. Very short pulse durations may not provide sufficient membrane cleaning, whereas long pulse durations can lead to unnecessary loss of permeate flux. In addition, for the shorter backpulse interval, less permeate flux is collected during forward filtration (loss of permeate during the backpulse), whereas significant fouling and flux decline occur during longer backpulse intervals [3, 27, 34].

1.2 Objectives

The objectives of this research were the following:

- Modify an existing spiral wrap cross-flow UF membrane pilot plant by adding a backpulsing unit, and place the entire system under control of Labview software.

- Apply continual backpulsing on the permeate space, as an in situ cleaning method for cross-flow UF filtration using different foulants: an organic foulant (dextrin) and an inorganic foulant (kaolin),
- Identify the optimum backpulsing parameters, e.g. pulse interval, pulse duration and backpulse pressure which should reduce the membrane fouling and maximize the permeate flux.
- Under the optimum conditions of backpulsing (as determined above), investigate the effects of operating conditions, e.g. feed flow rate and feed concentration, on the permeate flux.
- Carry out simple statistical analysis of data and modeling using the Taguchi approach for UF membrane experiments, for membranes fouled with dextrin.

Chapter 2

Background and literature review

2.1 History of membranes

A membrane is a barrier that separates two fluids and restricts the transport of one or more components of these fluids across the barrier [2]. A membrane can be homogeneous or heterogeneous, symmetric or asymmetric in structure, and solid or liquid [35].

The first recorded observation of a membrane separation was in 1748: Abbe Nollet [36, 37] discovered the effect of osmotic pressure when a pig's bladder was brought into contact with on one side a water-ethanol mixture and on the other side pure water. In 1908 Bechhold [38] produced collodion membranes with pore sizes below 0.01 micron. These membranes were initially used only in laboratory applications, but later became commercially available. The first commercial membranes were used for drinking water treatment at the end of World War II [39].

The first asymmetric RO membranes were produced by Sourirajan and Loeb in the early 1960s [39, 40]. Subsequently, large sums of research and development funding from the US Department of the Interior's Office of Saline Water (OSW) resulted in the commercialization of RO membranes. This also later led to the commercialization of UF and MF. The first synthetic membranes were made from cellulose acetate. Today membranes are made from a wide variety of chemically and thermally stable synthetic polymers, ceramics, metals and electrically-charged materials. Membrane modules are manufactured in different configurations: plate-and-frame, hollow fibers, spiral wound, and tubular membrane modules.

2.2 Membrane applications

Improvements and advances in membrane technology over the last four decades have seen their applications expand into many industrial sectors, such as chemical, petrochemical, mineral, pharmaceutical, electronics, beverages, beer/wine clarification, as well as wastewater purification and water desalination. Membrane separation processes compete with conventional processes such as carbon adsorption, solvent extraction, distillation, centrifugation, flocculation followed by multimedia filtration, and ion-exchange. Compared to conventional separation, membrane

processes offers several advantages, such as high quality products, the requirement for less chemical addition, and easier control of operation and maintenance. However, membrane fouling is still hampering the growth of industrial applications of membranes [2, 37].

2.3 Membrane separation processes

There are four general categories of cross-flow membrane filtration (see Figure 2-1). Depending on the size of the pores in the membrane, membrane separation is classified as Microfiltration (MF), Ultrafiltration (UF), Nanofiltration (NF) and Reverse Osmosis (RO).

2.3.1 Microfiltration

Microfiltration is a low pressure (typically 0.3 to 1.7 bar) membrane process for separating larger size solutes from aqueous solutions. The pore sizes of a MF membrane range from 0.1-10 microns. MF membranes are made from a number of organic and inorganic materials, for example:

- Polymeric membranes: polyamide (PA), polysulphone (PS), polyethersulphone (PES), polypropylene (PP), polycarbonate (PC).
- Ceramic membranes: alumina (Al_2O_3), zirconia.

MF is used primarily for separating macromolecules, large suspended particles, fungi and bacteria. It is finding increased application as a pretreatment method to other membrane processes, in pharmaceutical applications [41] and as a replacement for conventional clarification and filtration technologies [42].

2.3.2 Ultrafiltration

Ultrafiltration is a membrane process whose function lies between that of NF and MF. The pore size of a UF membrane ranges from 0.005 to 0.1 microns. This corresponds to a molecular weight cut-off of about 1,000 to 500,000 Dalton (molecular weight unit) [1]. Transmembrane pressures are typically 1-10 bars. UF separates out large organic molecules, colloids, bacteria and proteins, while all

dissolved salts and smaller molecules pass through the membrane. Most UF membranes used commercially these days are prepared from polymeric materials such as PS, PES, PP and polyethylene (PE).

UF has a variety of applications in the biological and pharmaceutical industries [41, 43]. It also has applications in food industries, such as in cheese making and whey protein fractionation in the dairy industry [44], sugar refining [45], and in the production of fruit juice and other beverages [46]. UF is also used to recover valuable materials and remove impurities in the electro-coat painting industry [47], water treatment industry [47], and pulp and paper industry [48].

UF has been accepted as an alternative to conventional pretreatment for brackish surface water and sea water reverse osmosis (SWRO) systems [49]. The use of UF systems as RO pretreatment has some significant advantages over RO systems designed to include conventional pretreatment:

- UF membrane systems take up less than 50% of the area of a conventional pretreatment system, which results in reduced construction costs. This means that a UF membrane system may be more favorable in cases where space is limited, or where the costs of civil works are high.
- UF membranes system are easier to operate than some conventional filtration processes.
- The operating costs of a UF membrane system may be lower than those for conventional pretreatment systems.
- UF concentrated waste streams are easier to dispose of relative to those from chemically enhanced conventional pretreatment processes.
- UF filtrate quality is usually better than that of conventional pretreatment process. The colloidal fouling load to the RO is reduced, with a significantly lower Silt Density Index (SDI) and turbidity in the feed water.

2.3.3 Nanofiltration

Nanofiltration is a low to moderately high pressure process; it is a pressure-driven process applied in the area between the separation capabilities of RO membranes and

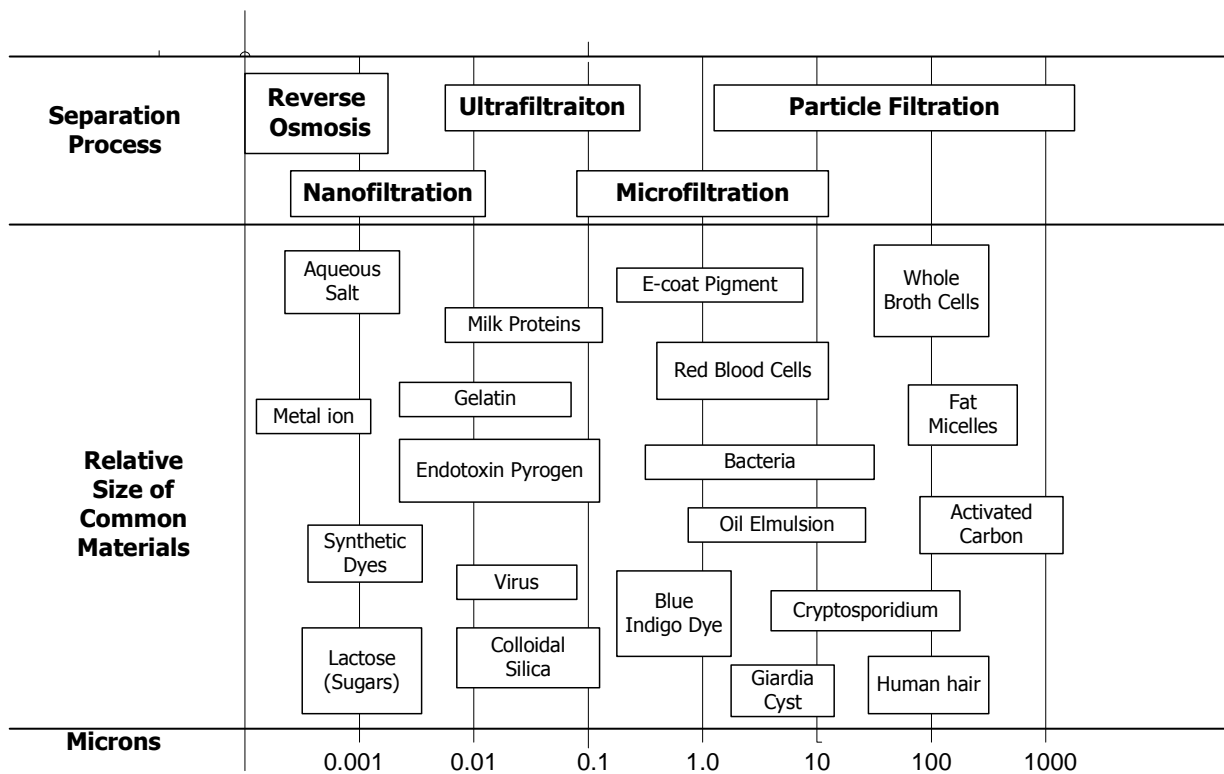


Figure 2-1: Reverse osmosis, nanofiltration, ultrafiltration, microfiltration, and conventional filtration are all related processes, differing principally in the average pore diameter of the membrane [50].

UF membranes. In NF the monovalent ions will pass more freely through the materials but divalent ions will be rejected. The pore size of NF membranes ranges between 0.01 and 0.0005 microns. Typically, NF membranes have sodium chloride rejections of between 20 and 80% and a molecular weight cut-off for dissolved organic solutes of 200-1000 Dalton. These properties are intermediate between those of RO membranes with a salt rejection of more than 90% and molecular weight cut off of less than 50 Dalton and UF membranes with a salt rejection of less than 5%.

NF membranes are commonly used as water softening membranes because they can very effectively remove most hard water components, i.e., carbonates and sulphates of calcium and magnesium. Depending upon the membrane, water chemistry, and operating conditions, NF membranes can remove more than 90% of feed water's hardness ions. NF membranes also remove large colour molecules. Other applications of NF membranes include caustic and acid recovery, concentration of dilute solutions, and desalting of cheese whey [2].

2.3.4 Reverse osmosis

Reverse osmosis is a membrane separation process capable of separating a solvent from a solution by forcing the solvent through a semi-permeable membrane by applying a pressure greater than the osmotic pressure of the solution. RO membranes remove nearly all dissolved salts, inorganic molecules, particulate matter including bacteria, viruses and organic molecules with a molecular weight greater than 150 Daltons. RO membranes can reject > 99% of dissolved salts. They essentially pass only water and molecules in the range of < 0.0005 μm . RO is used in the desalination of seawater or brackish water for drinking purposes, wastewater recovery, biomedical separations, and the removal of dissolved salts from high TDS effluents (e.g. mine water).

RO membranes are prepared from polymeric materials such as cellulose tri-acetate and aromatic polyamides, and from combinations of different materials when composite membranes are manufactured. Depending on their structures, RO membranes can be classified as either asymmetric or composite [35].

An asymmetric membrane has a very thin dense skin with a thickness of 0.1-0.5 μm . A porous sub-layer with a thickness of 50-150 μm supports the thin dense skin layer. The thin skin facing the feed solution acts as the selective layer, allowing water passage but rejecting dissolved salts. In asymmetric membranes, the selective top skin layer and the porous support layer are made of the same polymer material. In composite RO membranes, the selective top skin layer and the porous support layer are made of different polymeric materials. Composite membranes are typically manufactured by casting the skin layer, for instance polyamide, on top of a polysulphone ultrafiltration membrane [35, 51].

The following table summarizes the various membrane treatment processes, their applications(s), as well as comparable conventional treatment methods:

Membrane separation technology	Substances removed	Comparable conventional water treatment methods
Microfiltration	Bacteria and large colloids; separation of precipitates and coagulates	Ozonation-ultraviolet radiation, chlorination, sand filters, bioreactors and coagulation-settling tanks
Ultrafiltration	All of the above, plus viruses, high-molecular weight proteins, organics and pyrogen	Sand filters, bioreactors and activated carbon
Nanofiltration	All of the above, plus divalent ions, larger monovalent ions, colour and odor	Lime-soda softening and ion exchange
Reverse osmosis	All of the above, plus monovalent ions	Distillation, evaporation, ion exchange

2.4 Membrane modules

Although the membrane is the most important component in a membrane filtration process, membranes need to be economically manufactured and efficiently packed to provide accessible large areas before they can be used in filtration processes on a large scale. These packages are called membrane modules. Membrane modules are designed to avoid any leakage between the feed and permeate compartments and to ensure that at the membrane surface there is sufficient feed circulation to minimize concentration polarization and particle deposits. There are four main types of membrane modules used in industrial applications: plate-and-frame, hollow fiber, tubular and spiral wrap.

2.4.1 Plate-and-frame membrane modules

Flat sheet membrane modules were one of the earliest types of membrane modules. Figure 2-2 shows a schematic of a typical plate-and-frame membrane module. In this module the membrane layer is cast onto a sheet of non-woven backing, which is then cut to the appropriate shape to install in the modules. The modules are built up from membranes, feed spacer plates and product spacers which are layered together between two end plates. The feed flows in at the one end of the module and the retentate is collected at the other end of the module. The permeate flux is separated from the feed stream as illustrated in Figure 2-2. Flat sheet modules are easy to disassemble for cleaning and replacement of defective membranes. Flat sheet modules are currently only used in electrodialyses (ED) and evaporation systems and in a limited number of RO and UF applications with highly fouling feeds [47, 52].

2.4.2 Hollow fiber membrane modules

The hollow fiber is one of the best membrane configurations, as there is no additional supporting layer. Figure 2-3 shows a schematic of a typical hollow fiber membrane module. Hollow fibers generally have an inside diameter of 1 mm or less and outside diameters ranging from 2 to 2.5 mm. The feed is supplied to either the inside or outside of the fiber, and the permeate passes through the fiber wall to the other side of the fiber. When the dense top skin layer lies on the inside of the hollow fiber this type of operation is called “inside-out” filtration. When the skin layer lies on the

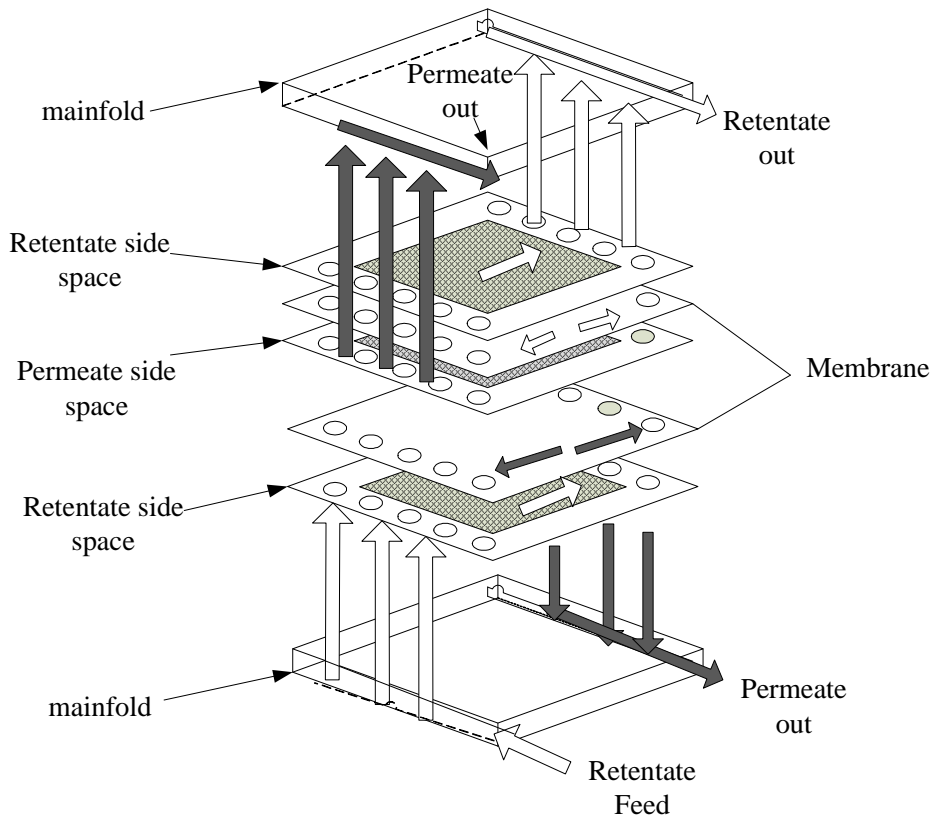


Figure 2-2: Schematic of a plate-and-frame module [47].

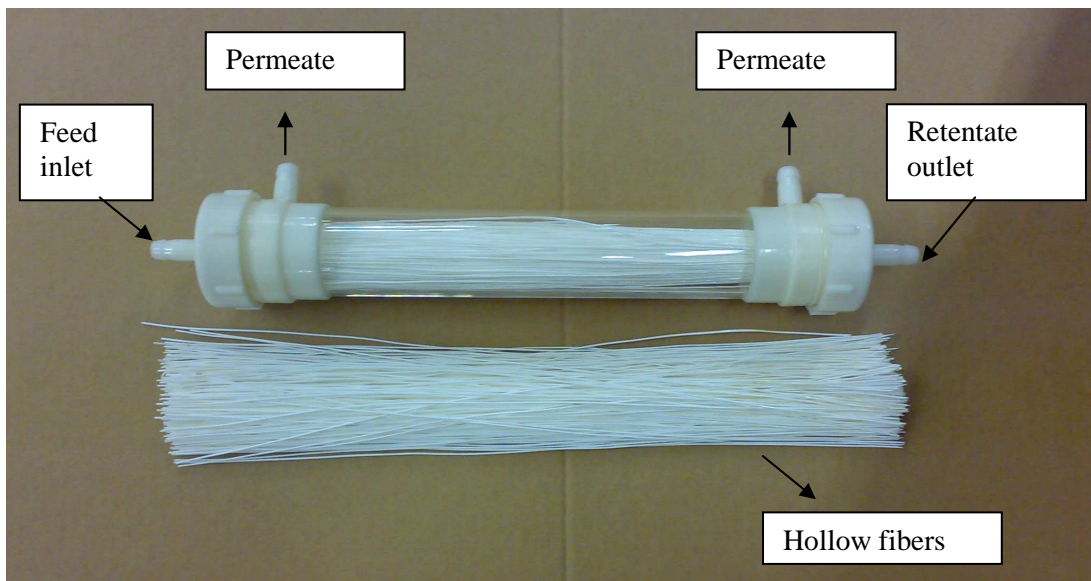


Figure 2-3: Schematic of a hollow fiber membrane module [47].

outside of the fiber then the operation is called "outside-in" filtration.

To create a membrane module, hundred or thousands of hollow fibers are mounted in a cylindrical housing (typically 4 to 12 inches in diameter). For the inside-out configuration the feed and permeate are sealed off from each other with a potting resin, which also forms a tube plate at the ends of the bundle. After the resin has hardened the bundle is cut, ensuring that the open ends of the hollow fiber are exposed. For the outside-in configuration the bundle is often arranged in a U-shape and the fibers are only sealed at one end. The hollow fiber module is characterized by a very large membrane surface area. Hollow fiber membranes are used in many industrial applications, and in the treatment of municipal drinking water [47, 52].

2.4.3 Tubular membrane modules

A tubular membrane module is the simplest configuration. Figure 2-4 shows a schematic of a typical tubular membrane module. The tubular membrane model is prepared by direct casting on a porous stainless steel or fabric tube. Tubular modules vary in tube diameter from 1-2.5 cm. In the tubular membrane model the feed flows through the tubes and the permeate moves outward, perpendicularly, through the membranes and the supportive tubes, similar to the "inside-out" filtration in the case of hollow fiber modules.

Tubular membrane modules are easy to clean and do not need significant pretreatment of the feed when used in MF and UF. Tubular membranes have a much larger diameter compared to hollow fibers. Thus, the membrane packing density will be less for the tubular membranes [47, 52].

2.4.4 Spiral wrap membrane modules

The spiral wound membrane element configuration is one of the most widely used in industrial applications due to the high membrane packing density and relatively lower capital cost, compared to other membrane configurations. Figure 2-5 shows a schematic of a typical spiral wrap module. In the spiral wrap modules two flat sheet membranes are separated by a permeate spacer. The resulting envelope is sealed on

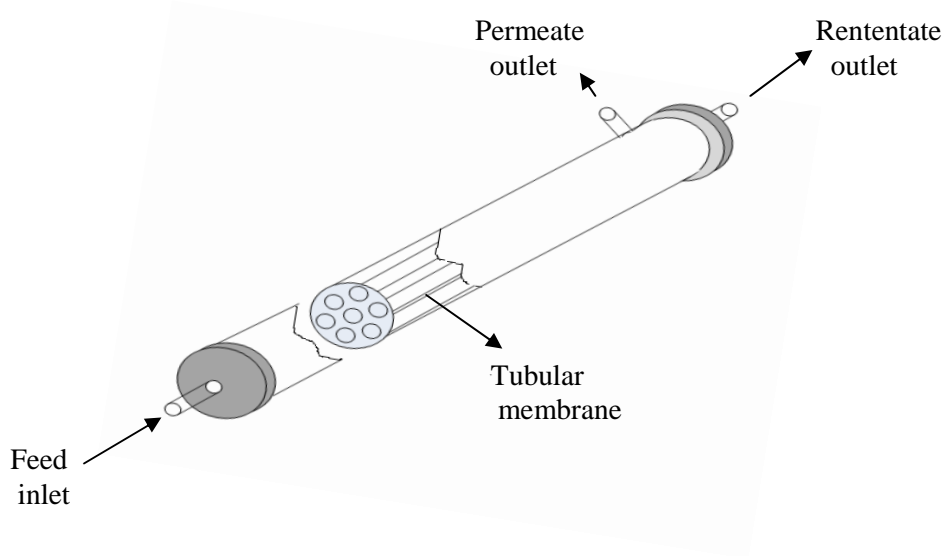


Figure 2-4: Schematic of a tubular membrane module [47].

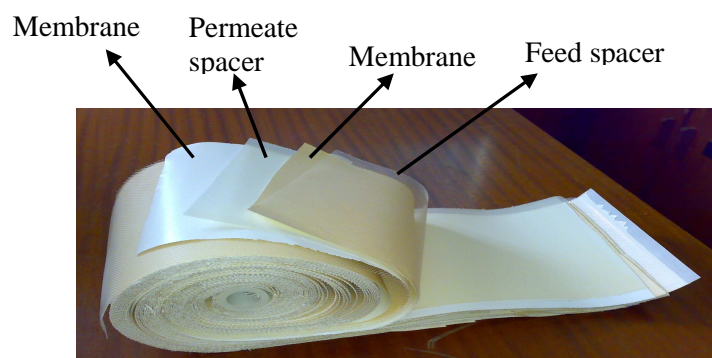


Figure 2-5: Schematic of a spiral wound membrane module [47].

three edges with suitable glue. The open end membrane envelope is attached to a central tube that collects the permeate. A feed spacer is inserted between each pair of envelopes. The envelopes and the feed spacers are then wrapped around the central tube to form the module.

The spiral wrap module can have a diameter of 300 mm and a length up to 1.5 m. Spiral wrap modules are compact and the pressure drop is lower than for tubular or plate-and-frame modules [47, 52].

2.4.5 Cross-flow filtration and dead-end filtration

Membrane filtration can be operated in dead-end filtration (DEF) or cross-flow filtration (CFF) (see Figure 2-6). During DEF operation all the feed solution passes through the membrane and out of the module on the permeate side. As the permeate is collected the rejected particles and macromolecules build-up on the membrane surface. This increased growth of the cake layer causes a rapid decline in the permeate flux through the membrane. As a result, the DEF process must be stopped periodically in order to clean the filter by removal of the particles or to replace the filter medium. DEF is used mainly for feed streams with a low fouling potential; with high fouling potential feed streams rapid flux decline and possible blockage of membranes would occur. Over the past three decades CFF has been increasingly used as an attractive alternative to DEF to help limit the amount of fouling occurring.

In CFF the feed solution flows parallel or axially to the membrane surface. Unlike DEF, the cake layer in CFF does not build-up indefinitely, but rather remains relatively thin as the high shear created on the membrane surface by the feed solution flowing tangential to the membrane surface sweeps the deposited particles toward the module exit. The cross-flow configuration is effective for controlling concentration polarization and the cake build up on the membrane. Because of this, higher fluxes may be maintained over prolonged time periods as opposed in the case of DEF [52, 53].

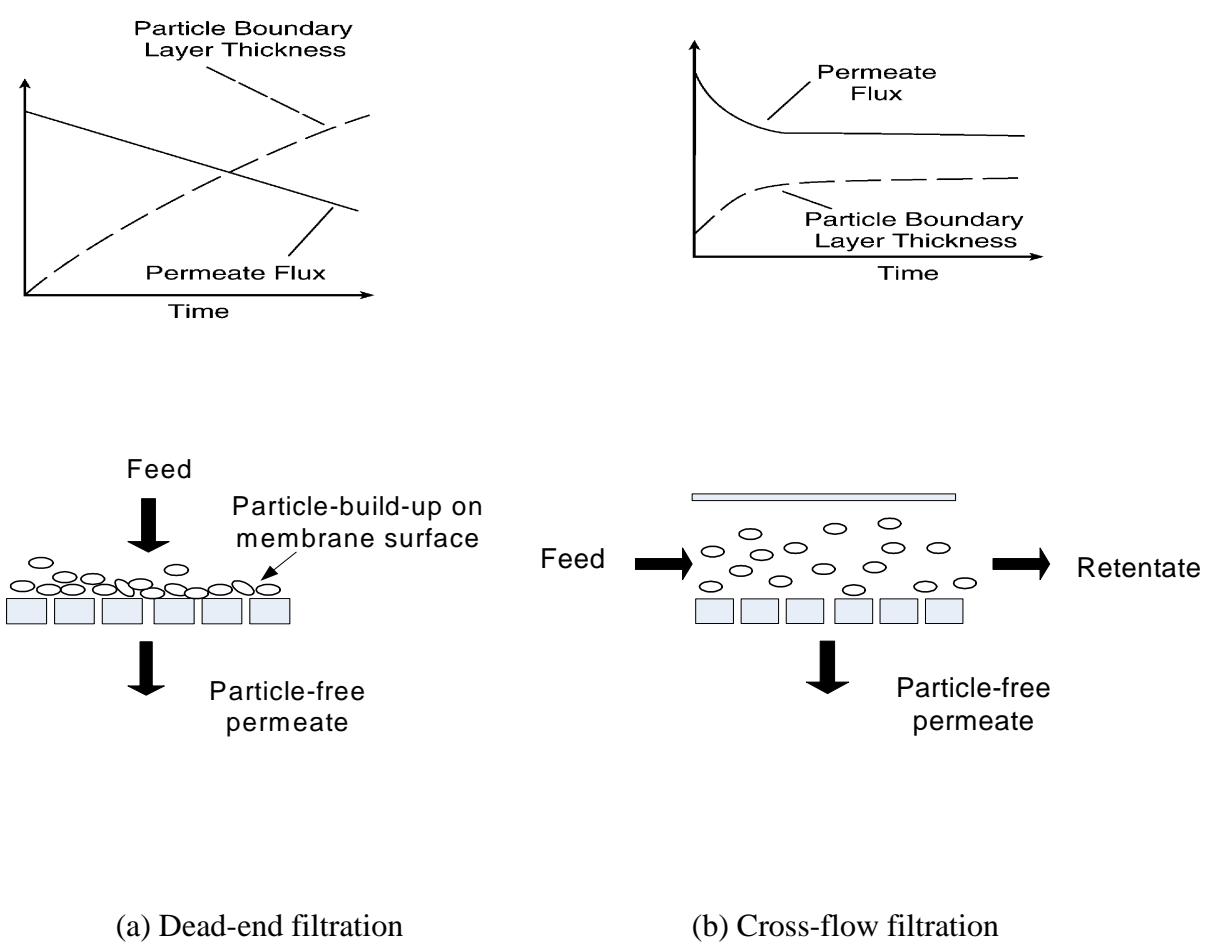


Figure 2-6: Schematic of the cross-flow and dead-end filtration processes [52].

2.5 Concentration polarization and membrane fouling

2.5.1 Introduction

One of the major problems associated with the operation of membrane processes is the decrease in the flux with time, due to concentration polarization (CP) and membrane fouling. The flux decline, particularly during MF and UF is often very severe; the permeate flux is often less than 5% that of pure water after a given period of time [54]. The typical decrease in flux with time shows an initial rapid decline followed by a long and gradual flux decline. Traditionally, the initial flux decline is attributed to CP (a rapid build up of solute particle concentration near the membrane surface) and pore blocking, while the long term decline is attributed to various modes of membrane fouling, including adsorption, chemical interactions and cake formation [54, 55].

One much used CP model considers a number of resistances in series. Therefore, during the transfer of components from the bulk of the solution to the permeate, the resistance is due to the following:

- resistance due to the membrane (R_m)
- resistance due to the fouling layer (R_f)
- resistance due to the polarization layer (R_p)

Therefore the flux of a membrane (J) can be expressed as

$$J = dP / \mu (R_m + R_f + R_p) \quad (2.1)$$

where μ is the viscosity of the solvent and dP the transmembrane pressure.

2.5.2 Concentration polarization

Concentration polarization is a phenomenon that occurs near or on the surface of a membrane due to the enhancement in the concentration profile of solutes in the liquid phase adjacent to the membrane surface. Hence the convective transport of the solute to the membrane surface is greater than the diffusive and convective transport away from the membrane [55, 56].

When the CP on the membrane surface reaches a maximum value, the CP layer aggravates all forms of surface fouling phenomena, including scale formation, by low solubility mineral salts, cake formation by colloids, gel formation by organics, and biofilm formation by bacteria [6, 47]. CP, through these secondary processes, causes a decline in the permeate flux through the membrane and changes the selectivity of the membrane process. CP is considered to be a reversible phenomenon that disappears as soon as the operating pressure is released [3].

CP effects can be described mathematically by a film model [57], which assumes that, even in turbulent flow, a laminar boundary layer is obtained adjacent to the membrane surface. During the UF filtration processes the solute concentration continues to increase until steady-state is attained, at which point the convective transport toward the membrane is balanced by back diffusion away from the membrane. Therefore, a constant concentration profile of the rejected material is obtained in the laminar boundary layer, and the concentration at the membrane surface is always higher than that in the bulk solution. The concentration profile and the overall mass transport in the laminar boundary layer at the membrane surface are shown schematically in Figure 2-7. Here, C_w and C_b are the solute concentration at the membrane surface and in the bulk solution, Y_b is the boundary layer thickness, and J_v is the transmembrane flux. The ratio of C_w/C_b is generally referred to as the CP modulus. It is determined by the overall mass transport in the laminar boundary layer in the steady-state from a simple mass balance according to eq.(2.2) [57]:

$$\frac{C_w}{C_b} = \frac{\exp(J_v Y_b / D)}{R + (1 - R) \exp(J_v Y_b / D)} \quad (2.2)$$

where D is the diffusion coefficient of the solute in the feed solution in the boundary layer and R is the membrane rejection.

According to eq. (2.2), CP is mainly determined by the permeate rate, the diffusion coefficient of the solute, and the thickness of the boundary layer.

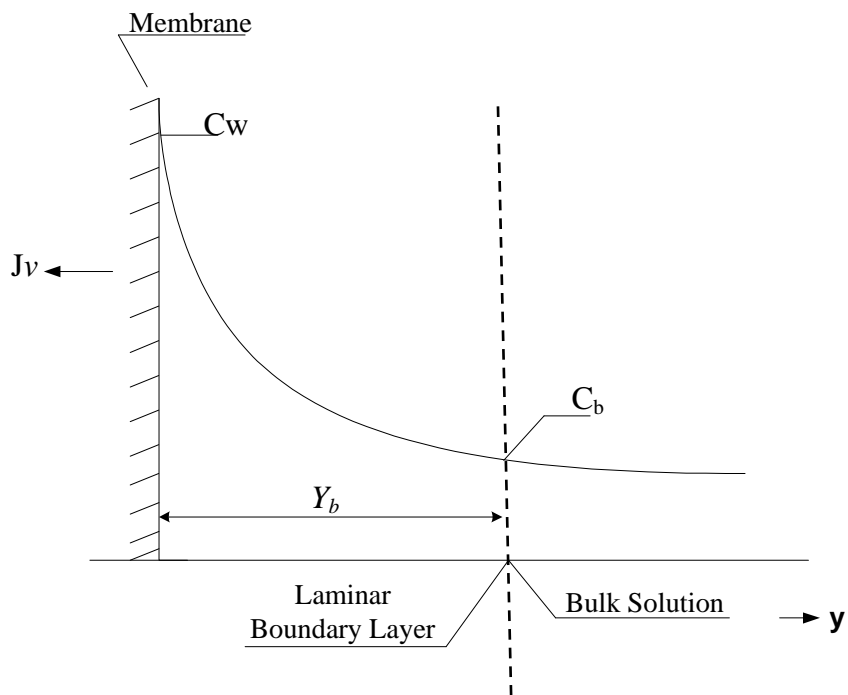


Figure 2-7: The concentration polarization profile in the boundary layer in the steady state during ultrafiltration [55].

2.5.3 Membrane fouling

Fouling occurs in all membrane filtration processes (RO, NF, UF and MF). Membrane fouling refers to the deposition of rejected particles, colloids, macromolecules, salts, etc. on the membrane surface or inside the membrane in the pores; and causes a flux decline and reduced the membrane performance [58-65].

The fouling rates are influenced by the nature of the solute, concentration of the solute and membrane type. The deposition on the membrane surface depends on the force acting on the particle and its size. At the membrane surface the foulants may become attached to the membrane by processes such as adsorption, deposition and pore blocking [2, 58, 66]. Figure 2-8 depicts the four major mechanistic models that are typically used to describe membrane fouling:

- Internal pore blocking, whereby material not rejected at the pore entrance is adsorbed or trapped on the pore wall or in the membrane support
- Pore bridging, which is partial obstruction of the pore entrance
- Complete pore blocking, whereby the pore entrance is sealed
- Cake formation, when particles accumulate on the surface of the membrane.

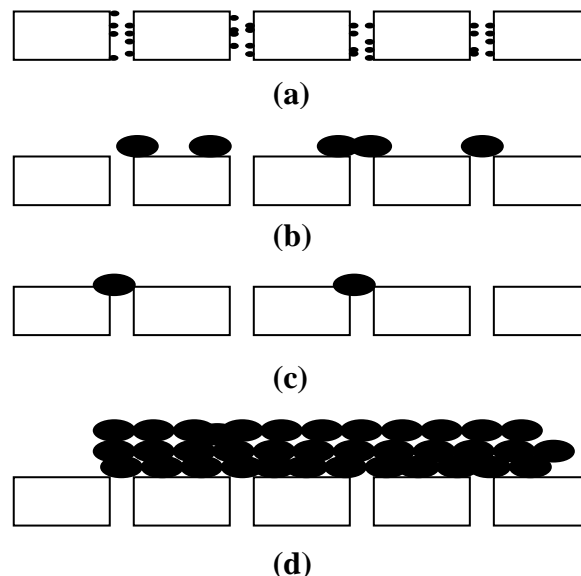


Figure 2-8: Mechanisms of membrane fouling: (a) internal pore blocking; (b) partial pore blocking; (c) complete pore blocking; (d) cake layer [2].

The main causes of pore blocking are high pressure and high feed concentration. Generally pore blocking is irreversible fouling. When irreversible fouling occurs the membrane module needs to be replaced or the separation process must be completely shut down for physical or chemical cleaning of the system.

Various types of fouling can be distinguished depending on the material deposited. Scaling, colloid, biological and organic fouling are briefly described below.

2.5.3.1 Inorganic fouling/scaling

Inorganic fouling or scaling is caused by the accumulation of inorganic precipitates such as metal hydroxides in the feed water on the membrane surface. Precipitates are formed when the concentration of ions in the feed exceeds their saturation concentration. Scaling is a major concern in RO and NF. Scaling fouling can be controlled by acidifying the feed, making use of commercial anti-scalants or by using an ion-exchange water softener [58].

2.5.3.2 Particulate/colloid fouling

Particulate or colloid fouling can be defined as the deposition of particulate material, e.g. suspended solids, colloids and microorganisms, on the membrane surface. Rivers or lakes, which have a high concentration of suspended solids and colloids, are prone to cause particulate fouling. SDI is the most commonly used parameter to predict particulate fouling. A $SDI > 3$ means that particulate fouling is likely to be a problem, and frequent, regular cleaning will be needed [58, 67-69].

2.5.3.3 Biological/microbial fouling

Microbial fouling is a result of the formation of biofilms on membrane surfaces. Once the microbial matter (bacterial/algal/fungal) attaches to the membrane it starts to multiply and produce biopolymers. The severity of microbial fouling is largely related to the characteristics of the feed water. Some membranes are very susceptible to bacterial attack. Periodic treatment of the feed water with bactericide usually controls biological fouling [58, 67-69].

2.5.3.4 Organic fouling

Organic fouling occurs widely in membrane filtration with source waters containing relatively high quantities of natural organic matter (NOM). Surface water (lakes, rivers) typically contains more NOM than ground water. Organic fouling is defined as the chemical or physical adsorption of organic compounds onto the membrane, which is usually followed by the formation of a cake or gel layer at the membrane surface. Filtration or carbon adsorption is used to control organic fouling, by removing the organic material from the feed [58, 67-69].

2.6 Strategies to reduce membrane fouling

In recent years many studies have been carried out in efforts to understand the underlying factors that limit the performance of cross-flow membrane processes and to find a solution to the flux decline of membranes that is caused by CP and membrane fouling. Many techniques have been used to decrease membrane fouling and disrupt the CP layer in cross-flow MF and UF membranes. These include the following:

2.6.1 Pretreatment of feed water

Pretreatment is typically applied to the feed water prior to its entering the membrane system in order to minimize the membrane fouling, extend membrane life and improve the membrane performance. Figure 2-9 illustrates conventional pretreatment systems of water. Conventional pretreatment includes coagulation, sedimentation, filtration using sand and/or multimedia filters, lime softening and activated-carbon adsorption. In these filters particles found in the raw source water are agglomerated and flocculated by chemicals such as ferric chloride, alum and polymers. Multimedia filtration can trap and remove suspended solids from water that pass through the media. Biological fouling can be controlled by sodium bisulphate addition and chlorination. Organic fouling is controlled by pre-filtration through granulated activated carbon. Scaling is controlled by reducing the recovery or by adding chemicals (e.g. acid and scale inhibitors). Coagulation, flocculation followed by settling and/or filtration is a very effective pretreatment method for removing colloidal and suspended matter.

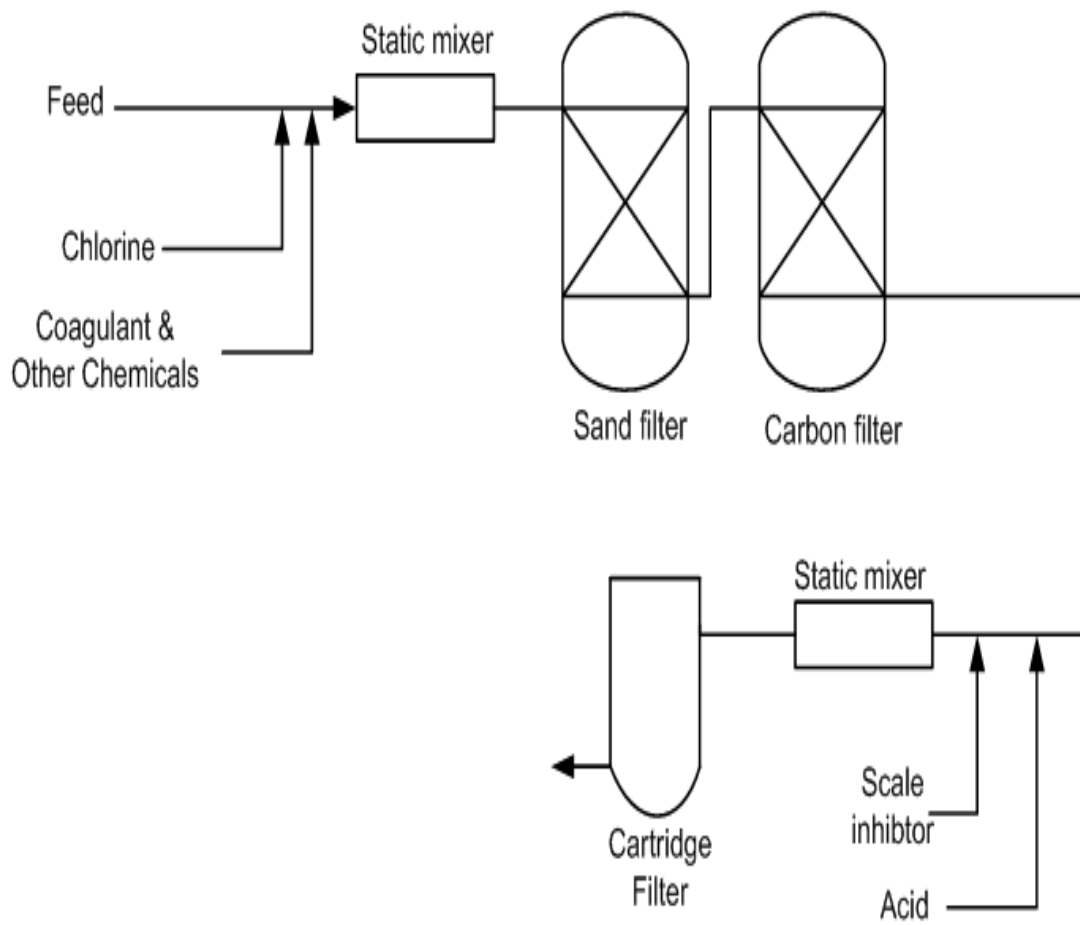


Figure 2-9: Schematic of a conventional water pretreatment systems where the feed turbidity is low [70].

The selection of pretreatment methods is based on the feed water quality, membrane material, module configuration, recovery, and the desired effluent quality [70]. The pretreatment process may consist of all or some of the following treatment steps:

- Removal of large particles using a coarse strainer
- Clarification (e.g. settling, MF, UF) with or without flocculation
- Water disinfection with chlorine
- Clarification and hardness reduction using lime treatment
- Reduction of alkalinity by pH adjustment
- Multimedia filtration
- Reduction of free chlorine using sodium bisulphate or activated carbon filters

2.6.2 Pulsatile flow (flow destabilization)

One method to reduce membrane fouling and improve the flux is pulsatile flow, or flow destabilizing. Oscillations and unsteady flow can introduce pulsations into the feed space [21]. Finnigan and Howell [23] investigated the effect of pulsatile flow on protein UF fluxes in a baffled tubular membrane system. They observed that the permeate flux improved up to three-fold by the incorporation of periodically spaced doughnut-shape baffles within the tubes. Howell et al. [24] reported that the filtration performance for yeast cell harvesting was greatly improved by using an oscillatory flow mixing technique, in both a tubular and a flat sheet membrane system. Gupta and coworkers [25] investigated the effects of the frequency and amplitude of the pulsating flow on the flux when filtering apple juice using ceramic MF membranes. They reported a flux improvement up to 140% when using a pulsed feed flow at 1 Hz. Gupta et al. [22] found a permeate flux enhancement of more than 50% during helically baffled cross-flow MF.

2.6.3 Gas sparging

Gas sparging refers to the creation of a gas-liquid two-phase flow, at the membrane surface, by the injection of gas bubbles into the feed stream. Air sparging has been shown to reduce CP and fouling in membrane filtration. Cui [71] showed that air-sparging could reduce the CP layer and increase the flux by up to 270% during MF of yeast suspensions. Cui and Wright [72, 73] used a tubular membrane and dextran

and bovine serum albumin (BSA), in cross-flow UF experiments. They studied the effect of gas sparging on permeate flux and membrane rejection. The system was tested over a range of operational parameters (transmembrane pressure, liquid cross-flow velocity, gas sparging rate and feed concentration orientation) [72]. Cui and Wright [73] They showed that significant improvements could be achieved at low gas flow rates. Flux increases of up to 320% were achieved with gas sparging on the feed side, compared to the case of single liquid phase cross-flow UF. Laborie et al. [74] reported that the permeate flux increased by about 110% when using air sparging during the UF of clay suspensions using hollow fiber membranes.

2.6.4 Ultrasound

Ultrasound is the waves passage through a medium at a frequency above 18 KHz. Ultrasound has been widely used as a method for cleaning materials because of the cavitation phenomenon [75]. Several researchers have investigated the use of ultrasound to reduce membrane fouling and enhance the permeate flux. Kobayashi and coworkers [76-78] used an ultrasonic bath to reduce membrane fouling. They found that ultrasound is effective in reducing the membrane fouling as it led to increased flux and improved membrane filtration performance. Zhu and Liu [79] found that ultrasound could increase the membrane performance by up to 200%. Jianxin et al. [80] used ultrasound together with flushing to clean a nylon MF membrane. Cleaning using ultrasound together with flushing can clean fouled membranes and completely restore the original membrane morphology. Jianxin et al. [81] used three methods to clean fouled membrane: forward flushing, ultrasonic cleaning, and ultrasound together with forward flushing. They found that the ultrasonic procedure can effectively not only detect deposition and growth of a fouling layer on the membrane in real-time but also monitor the progress of membrane cleaning and evaluate the cleaning effectiveness of the three cleaning methods.

2.6.5 Chemical cleaning

Periodic chemical cleaning is still the most effective way to restore the initial flux of a membrane and maintain the selectivity performance of the membrane system. The frequency and type of cleaning is determined by the feed water quality [37].

The cleaning in place (CIP) method is most often used for membrane cleaning. There are many different cleaning chemicals that can be used to remove membrane fouling and restore the membrane flux. The types most commonly used are acids, alkalis, chelatants, detergents and sterilizers [11, 12].

Caustic solution is typically used to clean membranes fouled by organic and microbial foulants [37]. Metal chelating agents, such as ethylenediaminetetraacetic acid (ETDA), can also be used to clean membranes fouled by organic foulants [13]. Acid cleaning agents, such as hydrochloric or citric acids, are used primarily for removing common scaling compounds [14]. A number of factors affect the cleaning efficiency of chemical cleaning: concentration of cleaning chemicals, temperature and length of the cleaning period [12].

2.6.6 Reverse filtration (backpulsing/backflushing)

Another technique that is used to reduce fouling is backflushing and backpulsing [30, 32]. The term backflushing refers to low-frequency permeate flow reversal, typically once every 2-10 min, while backpulsing involves reversing the permeate flow through the membrane from the permeate side to the feed side for short periods of time, typically less than one second (s), at high frequency (typically once every few seconds). In both cases the flow reversal dislodges and lifts the deposited foulants, which are then swept away by the cross-flow.

A schematic of the backpulsing process for cross-flow filtration is shown in Figure 2-10. There are several parameters associated with backpulsing, Backpulse duration is defined as the amount of time the filtration system operates under negative TMP, pulse amplitude is defined as the absolute value of maximum TMP during backpulsing, and backpulse interval is the duration of time between two consecutive pulses.

Cross-flow filtering with backflushing and backpulsing has been extensively studied by several groups for various membrane/foulant systems. Both have been reported to be a most effective method for the reduction of fouling and enhancing the net permeate flux.

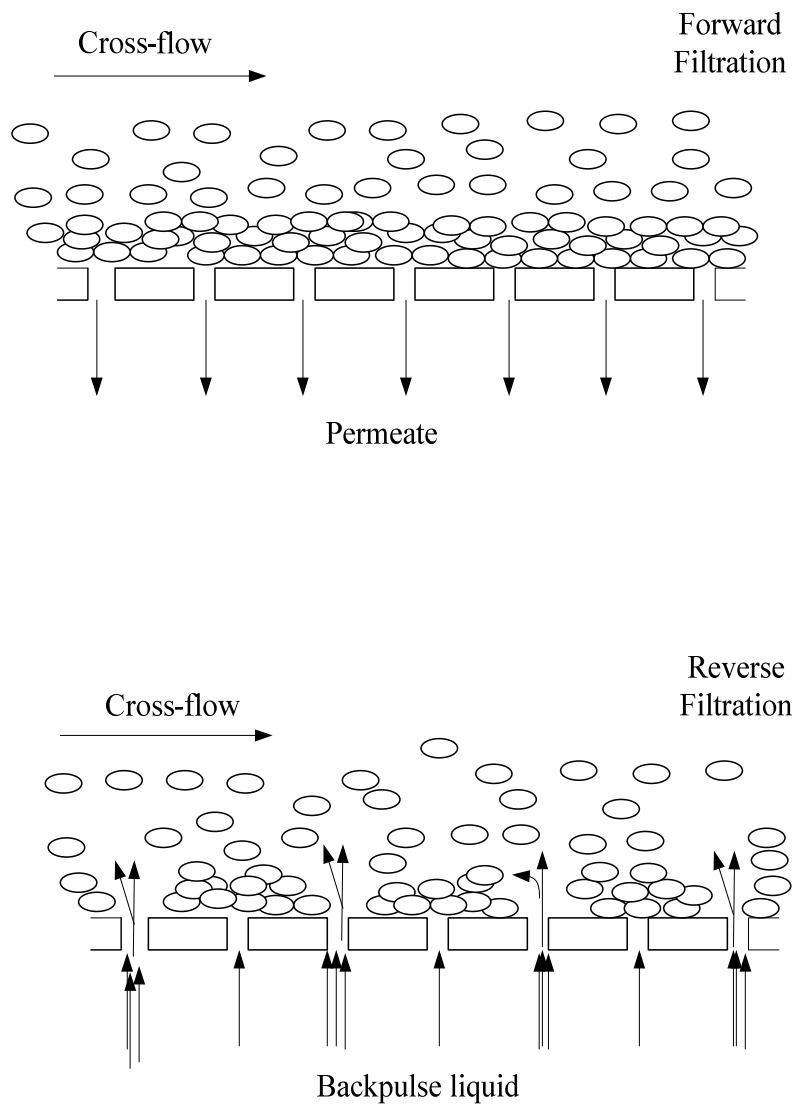


Figure 2-10: Schematic of forward and reverse cross-flow filtration during backpulsing operating.

Kroner et al. [82] used backflushing during the filtering of *E. coli* bacteria, using a polycarbonate membrane with a cut-off of 20 k Daltons. They observed a 50% enhancement in the net flux with backflushing (for 5 s every 5 min). Matsumoto et al. [83, 84] achieved up to a ten-fold flux increase with backflushing by reversing the transmembrane pressure for 5 s every 3 min, for yeast suspensions. Nipkow et al. [85] obtained an increase of about 42% in the permeate flux with backflushing of a MF cell-recycle pilot scale system, used for the continuous cultivation of *Clostridium thermosulfurogens*.

Kim and Chang [86] used periodic backflushing for separating haemoglobin (MW 62,500) and dextran (MW 10,000) through hollow fiber membrane with a molecular weight cut-off of 30 kD. For a backflushing duration of 11.25 s, the optimum frequency of backflushing to give maximum permeability was about 0.2 min⁻¹. Vigneswaran and coworkers [87] studied the cross-flow MF of feed water from a wastewater treatment plant. Membrane performance was significantly improved after periodic backflushing. The optimum conditions of the backflushing were 1 min backflushing frequency and 1 s pulse duration. Nakatsuka et al. [88] investigated the UF of surface water using hollow fiber membranes combined with backflushing. They concluded that the backflushing pressure should be more than twice the forward filtration pressure in order to maintain a constant and high flux. Kennedy et al. [20] reported results on the intermittent cross-flow of a hollow fiber UF system and found that the efficiency of backflushing was more dependent on the backwashing time than on the pressure. They also reported that 100% of the flux could be restored when backflushing was preceded by cross-flushing, while 95% of the flux could be restored with backflushing alone.

Srjaroonrat et al. [89] reported that CP and fouling can be controlled by periodic backflushing during filtration of oil/water emulsions using ceramic membranes. They found that the flux increased when backflushing was applied, and the optimum forward and reverse filtration times were 1 and 0.75 min, respectively. Bhave et al. [90] found that high flux could be sustained at a backpulse interval of 1 min. In the absence of backpulsing the flux decreased rapidly in the first 15 min of filtration.

Mugnier et al. [91] also reported that the backflushing process was very effective for reducing membrane fouling. They obtained 100% improvement of permeate flux during zeolite filtration by cross-flow MF with backflushing.

Jones et al. [92] found that an optimum backpulse amplitude of 10 kPa and optimum frequency of 0.01 Hz maximized the permeate flux for cross-flow microfiltration of solutions of kaolin clay containing hydrated aluminum silicate. Rodgers and Sparks [93] performed a study to determine the effect of negative transmembrane pressure pulsing on solute rejection for an albumin (MW 69,000) and gamma-globulin (MW 159,000) mixture in UF through 100 KD nominal pore size cellulosic membrane. The solute flux was found to be two orders of magnitude higher than that without pulsing; however, the observed retention of albumin was reduced from about 99% to 63%. Rodgers and Sparks [33] also studied the effect of transmembrane pressure pulsing on the CP boundary layer. Solutions of 1% bovine serum albumin (MW 69,000) at pH 7.4 in 0.15 NaCl buffered solution were filtered in a cross-flow module through cellulosic membranes. The operating pressures varied from 75 to 140 kPa, while the backpulsing pressure was 5 to 30 kPa above the respective operating pressures. The frequency of backpulsing ranged from 0 to 5 Hz. They observed that the flux enhancement did not change with an increase in the negative pressure amplitude after a certain minimal value. Rodgers and Miller [94] determined the effect of backpulsing on transient steric hindrance for BSA separation by UF in an unstirred batch cell. They reported an increase in the sieving coefficients for BSA when backpulsing was used in conjunction with fresh membranes.

Nikolov et al. [95] investigated the effect of the backpulsing pressure on the performance of a tubular UF membrane. They reported that a synchronized backpulse frequency of 5 Hz gave a permeate flux that was nearly three-fold higher than in non-pulsed cases. Wenten [28] described the use of the backpulsing process to maintain high fluxes and increase protein transmission in beer filtration. He found that with a backpulse duration of 0.1 s, the protein transmission increased from 68% to 100% using a cross-flow MF membrane together with backpulsing. Redkar and Davis [32] varied the durations of the forward and reverse portions of the backpulse

cycle. A flux enhancement of 30-fold over the long-term flux without backpulsing was obtained under optimal conditions of a 0.5 s backpulse every 5 s. Parnham and Davis [31] observed an increase in the net flux when the forward and backpulse pressures were increased: more than ten-fold increases in the net flux were found and the protein transmission was improved from 60 to 100% under optimum operating conditions of backpulsing (2.5 Hz pulse frequency and 0.09 s pulse duration). Sondhi et al. [96] observed increasing permeate flux up to five-fold and a 100% flux recovery with backpulsing (0.5 s every 30 s), when filtering a chromiumhydroxide suspension using cross-flow MF. Levesley and Hoare [97] used of rapid backpulsing for the recovery of a soluble enzyme, yeast alcohol dehydrogenase, from a suspension of homogenised bakers' yeast cells using microfiltration membrane. The backpulsing conditions were fixed at a reverse transmembrane pressure pulse of 0.1 s, applied every 1 s. An increase in solute transmission of up to five times was obtained, although the permeate flux was not improved by backpulsing.

2.7 Summary

As discussed above, a variety of methods has been used to reduce membrane fouling for different membranes/foulants systems. However, specific methods should be selected for specific applications, since each method has its advantages and disadvantages or limitations. Moreover the routine shutting down of filtration plants for chemical or mechanical cleaning, or both, is a slow and costly procedure. In this study, in order to prevent or minimize membrane fouling and thus maintain high permeate flux during filtration operation, continual backpulsing on the permeate space is used as an in situ cleaning method to improve the efficiency of a 2.5 inch spiral wrap UF membrane. The effects of different pulse intervals, durations and pressures, cross-flow rates and feed concentrations were investigated using organic and inorganic foulants to determine the best backpulsing conditions that give maximum permeate flux in spiral wrap UF element.

Chapter 3

Experimental

3.1 Experimental set-up

3.1.1 Cross-flow UF experimental apparatus without backpulse

A basic cross-flow UF plant without a backpulse unit and Labview control was built by Hydrophil (Pty) Ltd- SA, for the Polymer Science Institute, Stellenbosch University. A schematic of the experimental set-up is given in Figure 3-1. The clean water tank (R1) and feed tank (R2) were connected to the three-way valve (V1). The feed was pumped by a pump (Leader Pump, Model Ecojet R100, Italy) through the line (L1) to the cross-flow filtration element. A spiral wrap polypropylene UF membrane element with 100 kDa MWCO was used in these experiments (UF-pHt, Alfa Laval Company, Denmark). The characteristics of this element are given in Table 3-1.

Table3-1: Characteristics of the GR40PP UF membrane module element (UF-pHt, Alfa Laval Company, Denmark)

Membrane characteristics	Value	Unit
Membrane surface area	0.6	m ²
Flux on clean water	250-260	L/(m ² .h)
Feed spacer thickness	0.048	inch
MWCO	100,000	Dalton
Temperature limit	0-75	°C
pH range	2-10	-
Pressure limit for module	1-10	bar
Element length	17	inch
Outer diameter of element	2.5	inch

The permeate through the line (L2) and the retentate through the line (L3) are eventually returned to the feed tank to prevent changes in the feed concentration. In the absence of the backpulsing two magnetic flow sensors (Burkert fluid control system, Type 8045, SA) were used to measure the flow rates: the feed flow (FIT1) and the permeate flow (FIT2) (flow rate range 100-5000 L/h). A low flow rate sensor (Burkert fluid control system, Type 8071, SA) was used to measure the

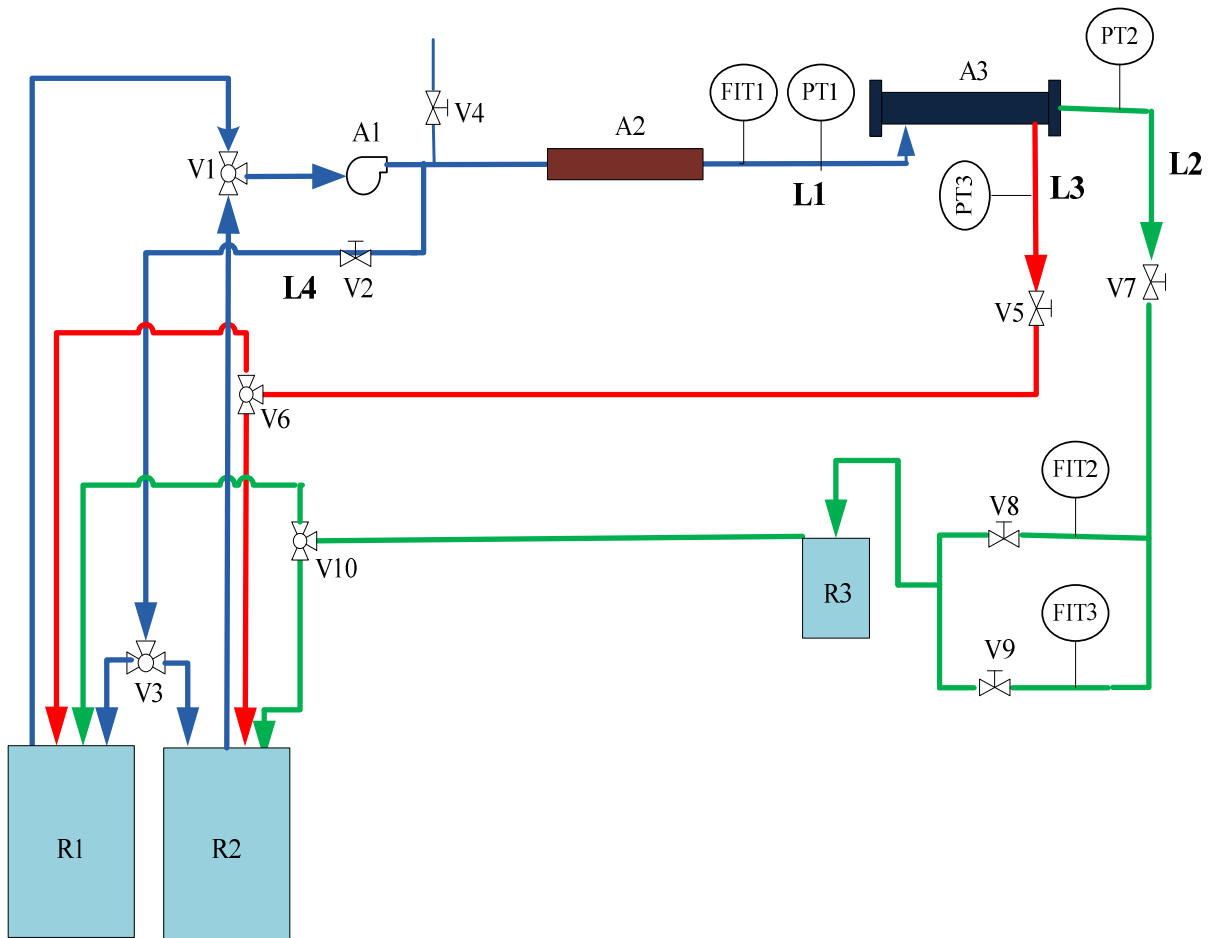


Figure 3-1: Cross-flow UF experimental apparatus without backpulse.

Legend:

- A1: Feed pump
- A2: Damper
- A3: Cross-flow module
- L1: Feed line
- L2: Permeate line
- L3: Retentate line
- L4: Bypass flow line
- V1/V3/V6/V10: 3-way valve
- V2/V4/V5/V7/V8/V9: Proportional valve
- PT1/PT2/PT3: Pressure transmitter
- FIT1/FIT2/FIT3: Flow indicator transmitter
- R1: Clean water tank
- R2: Fouling solution tank
- R3: Permeate tank

permeate flow (FIT3) for a low flow rate range (2-100 L/h). The pressures on the feed side (PT1), the permeate side (PT2) and the retentate side (PT3) were recorded using pressure transmitters (0-600 kPa; Model S-10, WIKA Instruments, Milnerton, SA).

3.1.2 Cross-flow UF experimental apparatus with backpulse

The cross-flow UF plant shown in Figure 3-1, controlled with the Labview programme, was then modified as shown in Figure 3-2. A damper (A2) was placed before the element in order to protect the feed pump from oscillation flow. Two temperature sensors (TT1) and (TT2) were placed in the clean water tank (R1) and feed tank (R2). The digital readouts were recorded in the PC. The temperature in the tanks was maintained using temperature control circuits (A7) and (A8).

A backpulse unit was designed and attached on the permeate side of the filtration element through the solenoid valve (VS1). The pressure for the pulses was obtained using a centrifugal pump (Provincial Pumps, Model LOWARA CEAM 70/3, SA), together with a pressurized tank (R5) and a manual pressure relief valve (V11). A pressure gauge (PI4) measured the pressure on the feed side of the fast acting solenoid valve VS1. The permeate liquid was used as feed for the pump in the pulsing unit.

The backpulsing was achieved using two fast acting solenoid valves: one normally closed (VS1) placed after the pressurized tank (R5) and the other normally open (VS2) placed in the permeate space. The two solenoid valves have a minimum switching time of 25 milliseconds and were operated at 24 volt power supply. The switching of the solenoid valves is controlled by the analogue output of a computer, which gives a variable pulse interval (1 to 15 s) and pulse duration (0.1 to 0.5 s), and was connected to control terminals of a solid-state relay that was connected to a 24 volt power supply. Note that during forward (normal) filtration the solenoid valve VS2 is open and solenoid valve VS1 is closed. During backpulsing (reverse filtration) solenoid valve VS1 is open and the solenoid valve VS2 is closed.

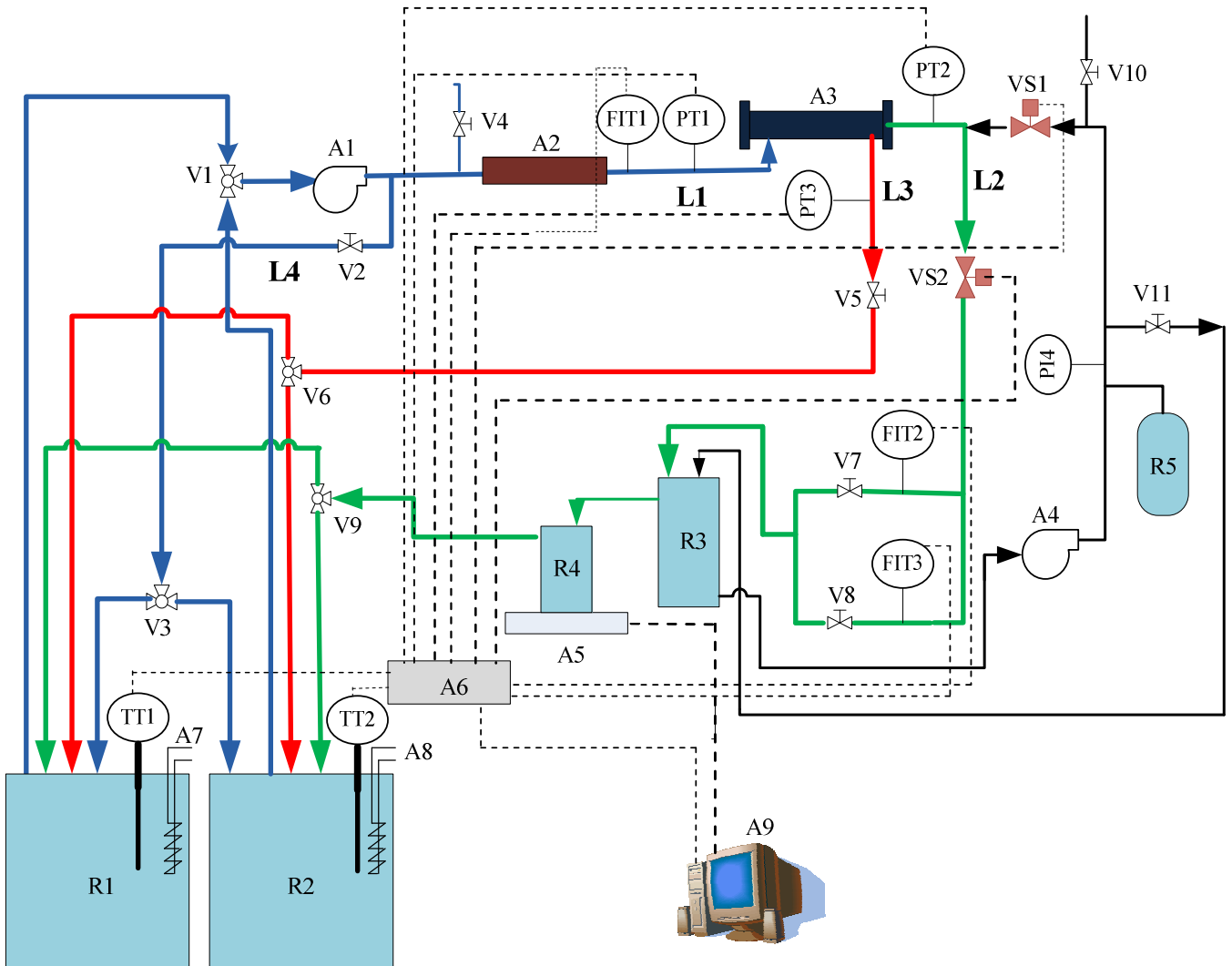


Figure 3-2: Cross-flow UF experimental apparatus with backpulse.

Legend:

- A1: Feed pump
- A2: Damper
- A3: Cross-flow module
- A4: Backpulse pump
- A5: Electronic balance
- A6: Terminal data acquisition
- A7/A8: Temperature control circuit
- A9: PC for data acquisition by Labview programs
- L1: Feed line
- L2: Permeate line
- L3: Retentate line
- L4: Bypass flow line
- V1/V3/V6/V10: 3-way valve
- V2/V4/V5/V7/V8/V9: Proportional valve
- VS1/V5/VS2: Solenoid valve
- PT1/PT2/PT3: Pressure transmitter
- PI4: Pressure indicator
- FIT1/FIT2/FIT3: Flow transmitter
- R1: Clean water tank
- R2: Fouling solution tank
- R3: Permeate tank
- R4: Overflow permeate tank
- R5: Pressurized tank
- TT1/TT2: Temperature transmitter

All sensor signals were transferred to the PC via a terminal data acquisition device (model # SCB-68, National Instrumental, SA). The main operating variables and pulse shape generated during filtration operation are monitoring by a computer program running under Labview software. As can be seen in Figure 3.3, the graphic interface Labview window on the PC shows the data logging time of pulse intervals, pulse durations and operating parameters (i.e. feed, retentate and permeate pressure, feed flow rate, temperature of feed and permeate). The interface panel of Labview (see Figure 3.3) also shows two charts. The top one shows the amplitude as a function of time. It shows that the pulse duration can be defined from where the pressure starts to rise to the sharp drop. This chart also shows two curves: the upper curve (white) is the primary pulse in the permeate space, while the lower curve (red) is the secondary pulse, as observed at the input of the feed space. The second chart, at the bottom of the PC screen, shows the curves of operating parameters as a function of filtration time.

3.1.3 Flux measurement during backpulsing

When the backpulsing was applied the permeate flux could not be measured using flow meters (FIT2) and (FIT3) due to the fluctuating reading and pulsing liquid. Therefore both of permeate flow and liquid pulse were collected in the permeate tank (R3). At the top of the permeate tank a weir was built and connected with an overflow pipe to break the waves that were generated on the surface of the permeate tank during backpulsing. The overflow (net permeate flow) was drained into a small tank (R4) on the electronic balance (CBK32, Adam Equipment, SA). The electronic balance was connected to the PC using a RS232 port, which converted the output signal into a flow rate, and which was recorded in the PC every minute. The net permeate flux was then calculated (using eq.3.1) by dividing this flow rate by the membrane surface area. All the data collected were recorded in the PC into a Microsoft Excel 2000 spreadsheet.

$$J = \frac{Q_p}{A} \quad (3.1)$$

where: J is the net permeate flux $L/(m^2.h)$, Q_p is the net permeate flow (L/h) and A is the membrane surface area of the membrane used in this study ($0.6 m^2$).



Figure 3-3: Front panel of a Labview system with controls and indicators.

3.2 Membrane preparation

The spiral wrap PP membrane was obtained from the supplier in dry form and had to be cleaned and disinfected before use according to the following procedure:

- Clean hot water (50-55 °C) was circulated through the membrane for 5 min at 100 kPa feed pressure and 1000 L/h feed flow rate.
- Caustic was added slowly to the feed water to achieve a pH of 10.5-11.
- The feed solution was recirculated through the element for 30 min.
- The alkaline feed solution was flush out using clean water, until a neutral pH was achieved.

After completion of the cleaning of the element, the clean membrane flux was measured, it was found to be between 250 and 260 L/(m².h).

3.3 Feed solutions

3.3.1 Organic solution (Dextrin)

Dextrin from corn (CAS # 9004-53-9) was obtained from Sigma-Aldrich SA. Dextrin solutions were prepared in RO water in concentrations of 250, 500 and 750 mg/L. Each solution was freshly prepared immediately prior to each experimental run.

3.3.2 Inorganic suspension (Kaolin)

Inorganic suspensions of kaolin were prepared by mixing different quantities of kaolin (Serina Trading, SA) with RO water to obtain final concentrations of 100, 300 and 500 mg/L. A kaolin suspension was continuously mixed to prevent settling of the kaolin in the feed tank (R2). The physical and chemical properties of kaolin are given in Table C-1.

3.4 Cross-flow UF experimental procedures

In all these cross-flow UF experiments a 2.5 inch spiral wrap UF element (100,000 MWCO) was used. The feed pressure was set at 100 kPa and the temperature was maintained at 27±0.5 °C in the R1 and R2 tanks hence normalization of flux was not required. The feed flow rate could be adjusted to a desired value by using valves V2 and V5. Experiments were carried out using pulse intervals of between 1 and 15 s, pulse duration of between 0.1 and 0.5 s, and backpulse pressure pulses of between 100 and 150 kPa. All pressures references reported in this thesis are related to gauge

pressures). Both permeate and retentate were recycled back to the feed tank to maintain the concentration of the feed solution. Most of the experiments were repeated twice to ensure the reproducibility of the experimental results.

Each experiment commenced with RO water being circulated through the system for 30 min to obtain the clean membrane flux. Backpulsing was then started and the difference in the flux due to water flowing in the reverse direction (loss of flux during backpulsing) noted over the next 30 min. The feed was then changed from clean water to foulant solution by using the three-way valve (V1), with continual backpulsing. The decline in permeate flux due to the membrane fouling was observed and the steady-state flux was measured over a 3 h filtration period. Then the feed was changed back to clean water, with the backpulsing still on (backpulse cleaning) for 30 min, to remove the fouling layer and to wash the concentration polarization layer out of the filter. At this point the retentate was not recycled back to the feed in order to avoid contamination of the clean water in the tank (R1). The backpulsing was then switched off and the change in permeate flux was determined over the next 30 min. After each run CIP was carried out to restore the initial clean membrane flux.

3.5 Chemical cleaning of the membrane element

Backpulsing is a good method for fouling prevention and/or reduction but it cannot completely remove the fouling. Therefore chemical cleaning also needs to be done to restore the initial membrane flux.

The chemical cleaning agents used in this study were calcium hypochlorite (CaCl_2O_2) as disinfectant, ethylenediaminetetracetic acid (EDTA) as metal chelating agent, and sodium lauryl sulphate (SLS) as an anionic surfactant. These cleaning agents are the most common compounds in commercial cleaning products used for organic-fouled membranes [98, 99]. The CaCl_2O_2 , EDTA and SLS were purchased from Protea Chemicals (Cape Town, SA) and used with no further purification. Solutions were prepared in the feed tank (R1) using RO water to which 0.1% EDTA, 0.1% SLS and 0.01% CaCl_2O_2 were added.

The procedure for the CIP cycle involved the steps listed below:

- Drain the system completely before CIP is carried out.

- Circulate the cleaning solution through the system at low feed pressure (50 kPa) and a cross-flow rate of 1000 L/h.
- Soak the membrane for 1 h in order to swell/dissolve foulants on the membrane surface.
- Circulate the cleaning solution through the system at a feed pressure of 50 kPa and feed flow rate 1000 L/h, while backpulsing with permeate (0.1 pulse duration, 3 s pulse interval and 100 kPa pulse pressure) for 1 h.
- Rinse the system with RO water from the feed tank (R1) to remove all traces of the cleaning solution and measure the clean membrane flux. (In all the experiments it was found to be between 250 and 260 L/(m².h)).
- Repeat the CIP if the restoration of flux was not satisfactory.

Chapter 4

Results and discussion

4.1 Introduction

In this chapter, the effects of varying pulse intervals, pulse durations, backpulse pressures, cross-flow rates and feed concentrations were investigated using organic and inorganic foulants, to determine the best backpulse conditions that give the maximum permeate flux in a spiral wrap UF element.

4.2 Organic foulant (dextrin)

4.2.1 Experiments without backpulsing

Figure 4-1 shows the change in the net permeate flux with time, without backpulsing, for a 500 mg/L dextrin feed solution at a feed pressure of 100 kPa and a flow rate 1000 L/h. In this experiment the RO water was circulated through the system for 30 min and the clean membrane flux measured. Then the feed was changed to the dextrin solution using the three-way valve (V1) and a very rapid flux decline was observed, from an initial value of about 260 L/(m².h) to about 110 L/(m².h), within the first 5 min. this was followed by a gradual decline until a steady-state flux value, which was between 25 and 27 L/(m².h), was reached after 90 min of fouling operation. The initial rapid decline in the permeate flux is a result of the rapid deposition of dextrin particles on the membrane surface which blocked or constricted the membrane pores.

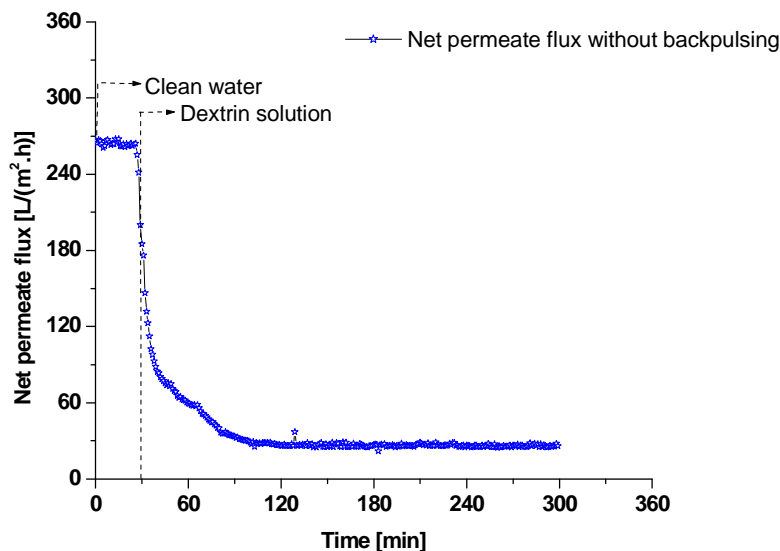


Figure 4-1: Net permeate flux through a polypropylene spiral wrap membrane module as a function of time. (Feed pressure 100 kPa, cross-flow rate 1000 L/h, temperature 27±0.5 °C , and dextrin feed solution 500 mg/L.)

4.2.2 Effect of pulse durations and intervals on the permeate flux

All experiments were run using 500 mg/L dextrin, with the backpulse duration varying from 0.1 to 0.5 s and the backpulse interval varying from 1 to 15 s. In all cases the backpulse pressure was 150 kPa. The feed pressure was fixed at 100 kPa and the feed flow rate at 1000 L/h.

Each experiment commenced with a flow of clean water for 30 min and then measuring the clean membrane flux. The backpulsing was then switched on and the difference in the flux due to water flowing in the reverse direction noted over the next 30 min. Figure 4-2 depicts the permeate flux during the repeated cycles of forward and reverse filtration. Cyclic operation is employed in which a period of forward filtration of duration t_f is followed by a period of reverse filtration (backpulsing) of duration t_b . The flux loss during backpulsing was calculated according to the following relationship:

$$\text{Flux loss due to backpulsing} = \left(\frac{t_b}{t_b + t_f} \right) \times J_0 \quad (4.1)$$

where J_0 is the clean membrane flux .

The flux losses at pulse duration 0.2 s and pulse interval 3 s can be calculated using eq. (4.1):

$$t_b = 0.2 \text{ s} , t_f = 2.8 \text{ s} \text{ and } J_0 = 259 \text{ L}/(\text{m}^2.\text{h})$$

The flux loss is 17.3 L/(m².h), which is agreement with an experimental value (see Table A-1). The calculation of the flux loss at different pulse intervals and pulse durations is reported in appendix A.

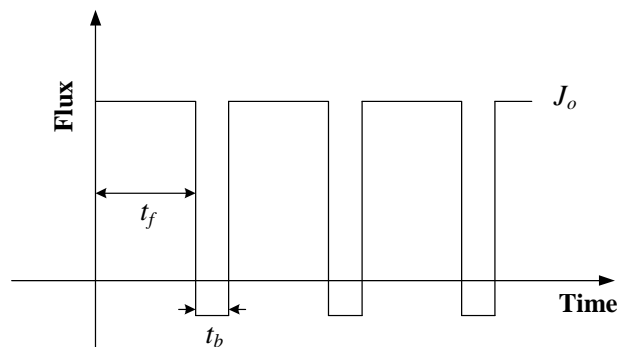


Figure 4- 2: Schematic of backpulsing and permeate flux during repeated cycles of forward and reverse filtration.

Figures 4-3 and 4-4 show the influence of the backpulse duration and backpulse interval on the permeate flux. In all cases the net permeate flux declined rapidly after commencement of fouling and then gradually declined over time due to growth of fouling layer. This initial rapid decline in the permeate flux is believed to be a combination of irreversible fouling (pore blocking, pore adsorption) and initial reversible fouling (fouling layer deposition) on the membrane surface. After about 180 min of fouling operation an apparent steady-state flux was reached.

It will later be shown that backpulsing appears to be effective in limiting the long-term fouling that occurs due to the build-up of the fouling layer on a membrane surface, this behavior is similar to what was reported by Rodgers and Miller [100]. They found that when filtering a binary protein mixture, backpulsing was not effective in completely eliminating the initial fouling that was due to pore adsorption and a fouling layer on the membrane surface.

The steady-state fouled membrane flux with continual backpulsing at different pulse intervals and durations, and the percentage of the flux improvement, are shown in Table 4-1. The percentage of the flux improvement due to the backpulsing was calculated according to the following relationship:

$$\% \text{ Flux improvement} = \frac{J_s - J_{so}}{J_o} \times 100 \quad (4.2)$$

where J_s is the steady-state fouled membrane flux with continual backpulsing (at 300 min), J_{so} is the steady-state fouled membrane flux without backpulsing and J_o is the clean membrane flux (directly after a CIP).

Table 4-1: Steady-state fouled membrane flux with continual backpulsing and percentage flux improvement at different pulse intervals and durations

Pulse duration (s)	J_s L/(m ² .h)					Flux improvement %				
	Pulse interval (s)					Pulse interval (s)				
	1	3	5	10	15	1	3	5	10	15
0.1	90.3	93.2	80.6	-	-	25.5	26.7	21.8	-	-
0.2	86.7	101.4	87.	84.3	79.0	24.2	30.0	24.3	23.3	21.2
0.3	-	94.3	88.6	88.0	71.4	-	27.1	24.7	24.8	18.1
0.5	-	-	62.7	75.2	79.2	-	-	14.7	19.6	21.3

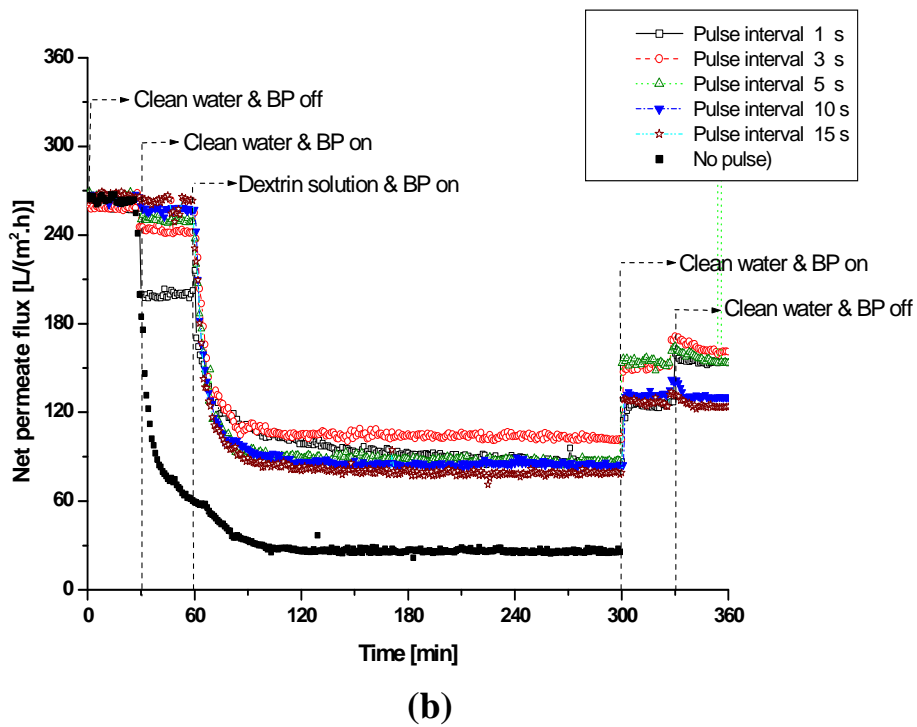
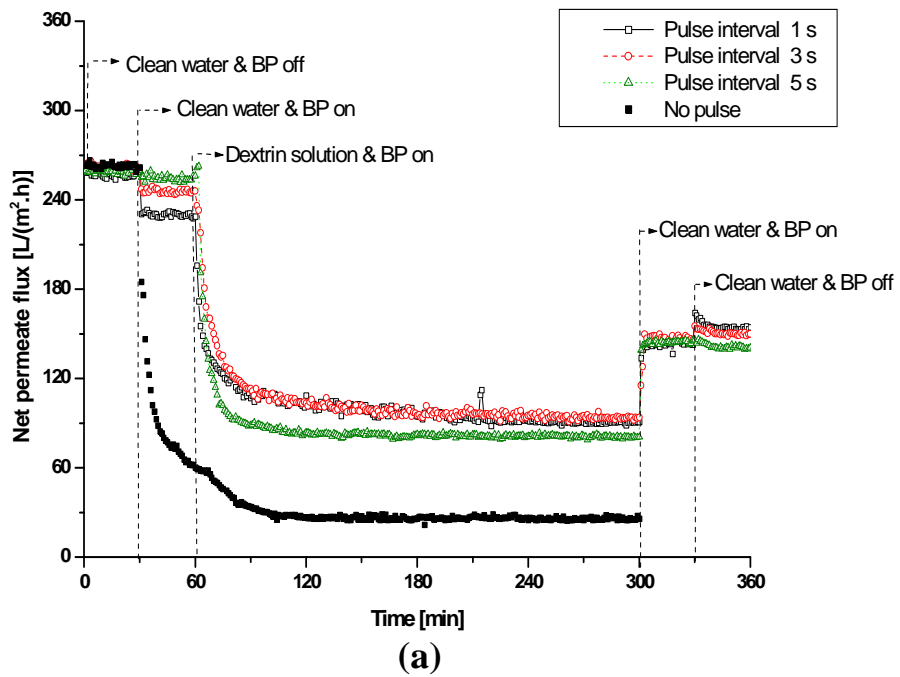


Figure 4-3: Net permeate flux as a function of time for different pulse intervals: (a) pulse duration 0.1 s and (b) pulse duration 0.2 s (Backpulse pressure 150 kPa, feed pressure 100 kPa, temperature 27 ± 0.5 °C, cross-flow rate 1000 L/h and dextrin feed solution of 500 mg/L.)

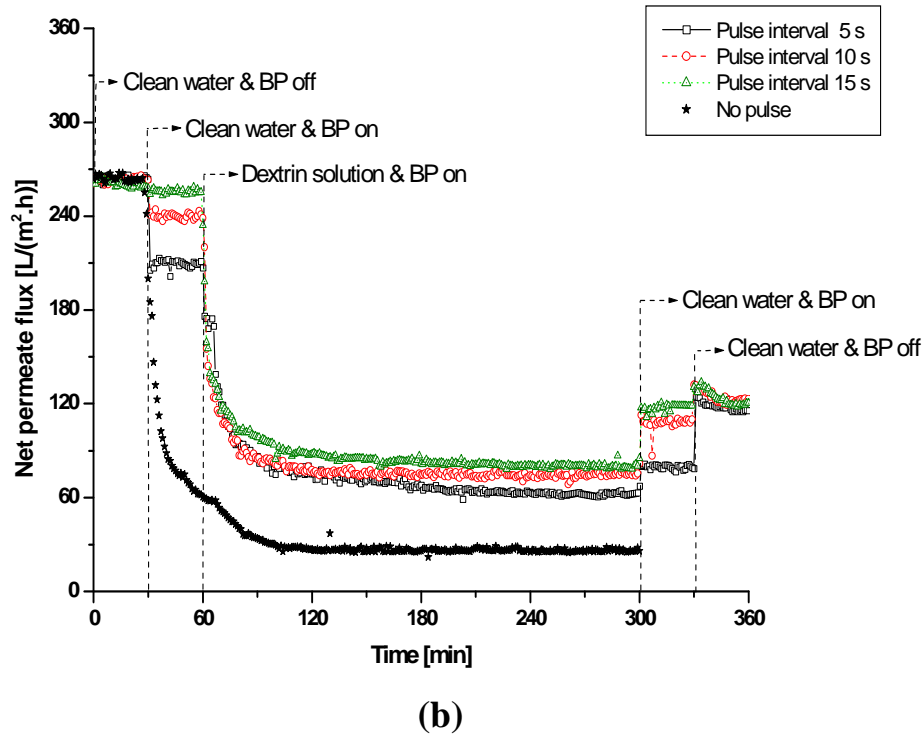
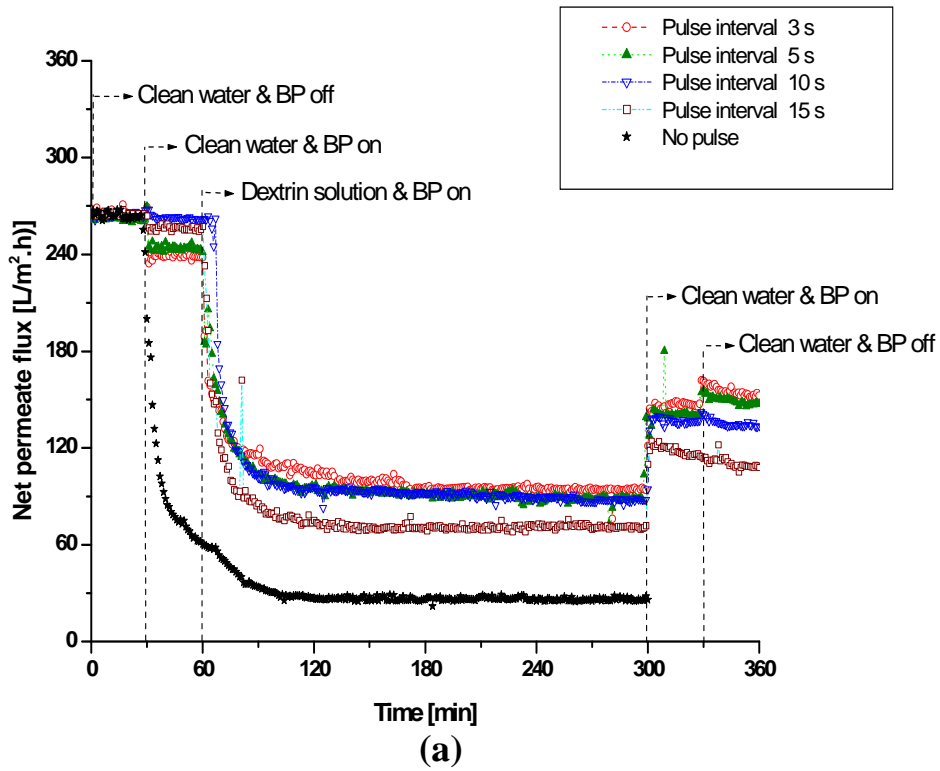


Figure 4-4: Net permeate flux as a function of time for different pulse intervals: (a) pulse duration 0.3 s and (b) pulse duration 0.5 s (Backpulse pressure 150 kPa, feed pressure 100 kPa, temperature 27 ± 0.5 °C, cross-flow rate 1000 L/h and dextrin feed solution of 500 mg/L.)

The results in Figure 4-5 clearly show that when backpulsing is used, whatever the backpulsing conditions, the net permeate flux increases compared to in the non pulsing case. It was also observed in these experiments that the steady-state fouled membrane flux with continual backpulsing increases with an increase in the pulse interval and an increase in the pulse duration, then reaches a maximum value, and then decreases with a further increase in the pulse interval and an increase in the pulse duration. This is because for the shorter pulse interval (i.e. 1 s) and longer pulse duration (i.e. 0.5 s), less permeate flux is collected during forward filtration (loss of permeate flux during the backpulsing), while a longer pulse interval (i.e. 15 s) and shorter pulse duration (i.e. 0.1 s) gives a chance for a reversible fouling layer to build up on the membrane surface, which results in a decrease in the permeate flux.

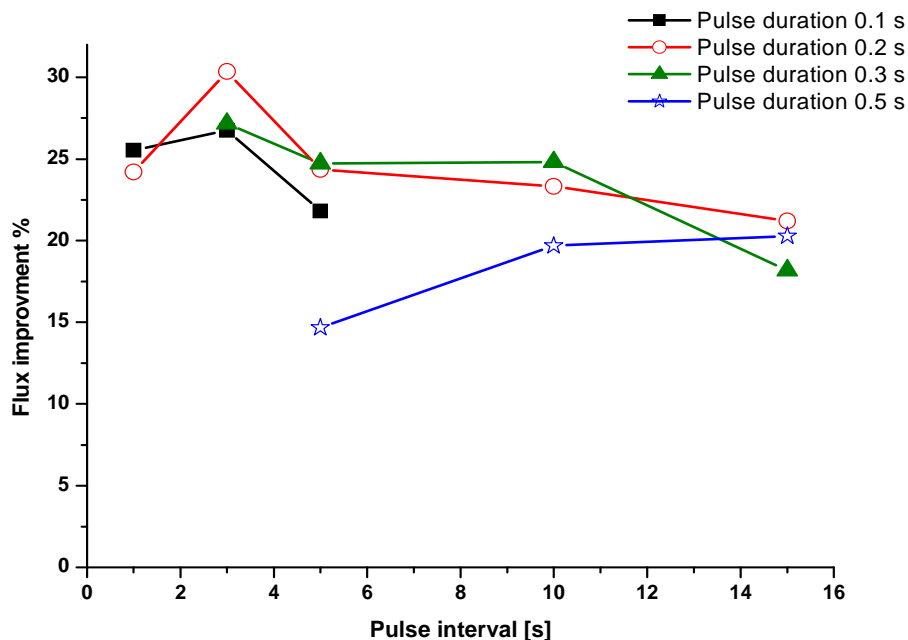


Figure 4-5: Effect of backpulsing on the steady-state fouled membrane flux at different pulse intervals and pulse durations.

As can be seen from Figures 4-3 and 4-4, the maximum value of a steady-state fouled membrane flux (at 300 min) was 102 L/(m².h) (40% of the clean membrane flux). This was obtained when the pulse duration 0.2 s was applied at a backpulse interval of 3 s. This represents a three-fold improvement over the steady state flux with no backpulsing.

The membrane was then cleaned by changing the feed solution from dextrin solution to clean water and applying continual backpulsing during the period between 300 and 330 min. The backpulse was switched off at this point. The flux declined slightly and then reached the steady-state (because of some of the residual foulant in the system and in the element). Table 4-2 shows the recovered clean membrane flux (after cleaning the membrane with BP and clean water). The percentage recovery of the clean membrane flux can be determined by the following equation:

$$\% J_r = \frac{J_r}{J_o} \times 100 \quad (4.3)$$

where J_o is the clean membrane flux (directly after a CIP) and J_r is the recovered clean membrane flux after cleaning the membrane with backpulsing and clean water. Figure 4-6 shows that the backpulsing is effective for cleaning the membrane. In the best case the permeate flux increased up to 161 L/(m².h) (63% of the clean membrane flux).

Table 4-2: Recovered clean membrane flux after changing the feed from dextrin solution to clean water and applying continual backpulsing during the period between 300 to 300 min

Pulse duration (s)	J_r L/(m ² .h)					J_r %				
	Pulse interval (s)					Pulse interval (s)				
	1	3	5	10	15	1	3	5	10	15
0.1	154.3	150.8	140.4	-	-	60.1	59.4	55.1	-	-
0.2	153.5	161.2	152.3	130.4	123.7	60.2	63.2	59.7	51.2	49.7
0.3	-	152.1	147.9	133.6	108.5	-	59.6	58.0	52.1	43.1
0.5	-	-	115.6	123.4	120.5	-	-	45.1	48.2	47.0

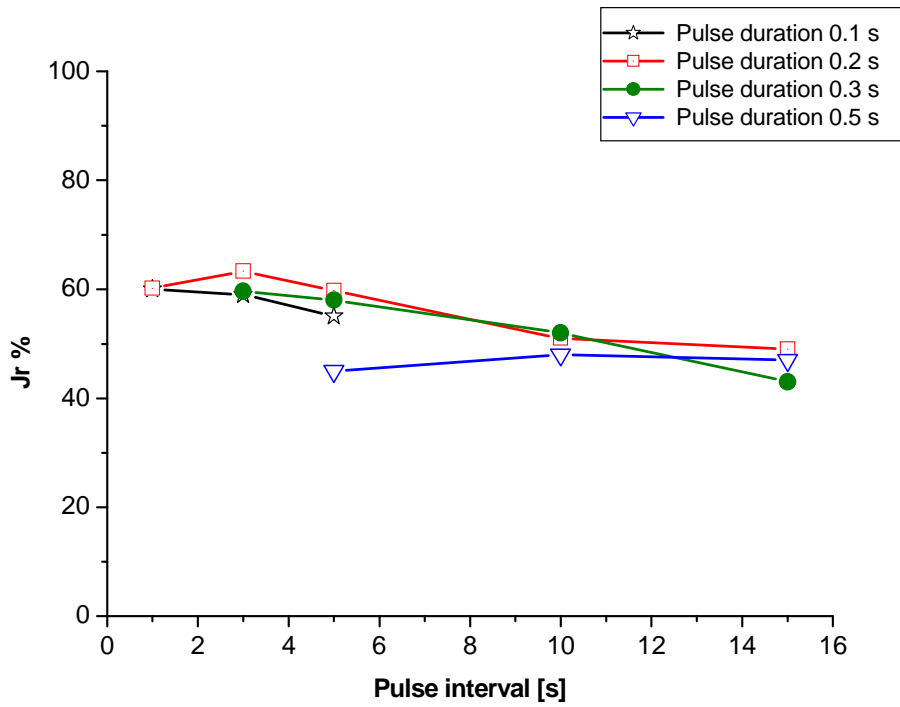


Figure 4-6: Recovered clean membrane flux after changing the feed from dextrin solution to clean water and applying continual backpulsing during the period between 300 to 300 min.

4.2.3 Effect of backpulse pressure on the permeate flux

Backpulse pressure is one of parameters that has a significant influence on foulant removal and enhancement of the net permeate flux during plant operation. Experiments were performed using a dextrin concentration of 500 mg/L at 1000 L/h feed flow rate. The feed pressure was fixed at 100 kPa. The backpulsing was applied at 0.2 s pulse duration and 3 s pulse interval, using three different backpulse pressures (100, 125 and 150 kPa). Transmembrane pressure (TMP) is defined as the difference between the average feed-side pressure and the average permeate-side pressure. The observed peak in the TMP values for these backpulse pressures were about 0, 25 and 50 kPa above the pressure in the feed space, respectively. Hence reverse flow occurs and is observed as a transient negative TMP due to the peak pulse pressure.

The effect of backpulse pressures on net permeate flux is shown in Figure 4-7. The steady-state fouled membrane flux (at 300 min) increased significantly with increasing backpulse pressure. However, significant improvements over the results for backpulse pressures of 100 and 125 kPa in the net permeate flux were seen at a backpulse pressure of 150 kPa, where the steady-state fouled membrane flux was about 102 L/(m².h), approximately four-fold higher than when no backpulsing was applied. When the backpulse pressure of 100 kPa (the lowest backpulse pressure used in this study) was used, the steady-state permeate flux obtained was only 65 L/(m².h) (25% of the clean membrane flux). At this pressure no backflow occurred and the membrane was only vibrated, which can shake or peel the fouling layer on the membrane surface.

Figure 4-7 also shows the results of cleaning of the membrane with clean water and backpulsing during the period between 300 and 330 min. The backpulse was then switched off and the net permeate fluxes (at 360 min) were about 63%, 52% and 45% of the clean membrane flux, obtained at backpulse pressures of 150, 125 and 100 kPa, respectively. These results clearly show that a high backpulse pressure is more effective for cleaning the membrane and reducing membrane fouling than a low backpulse pressure.

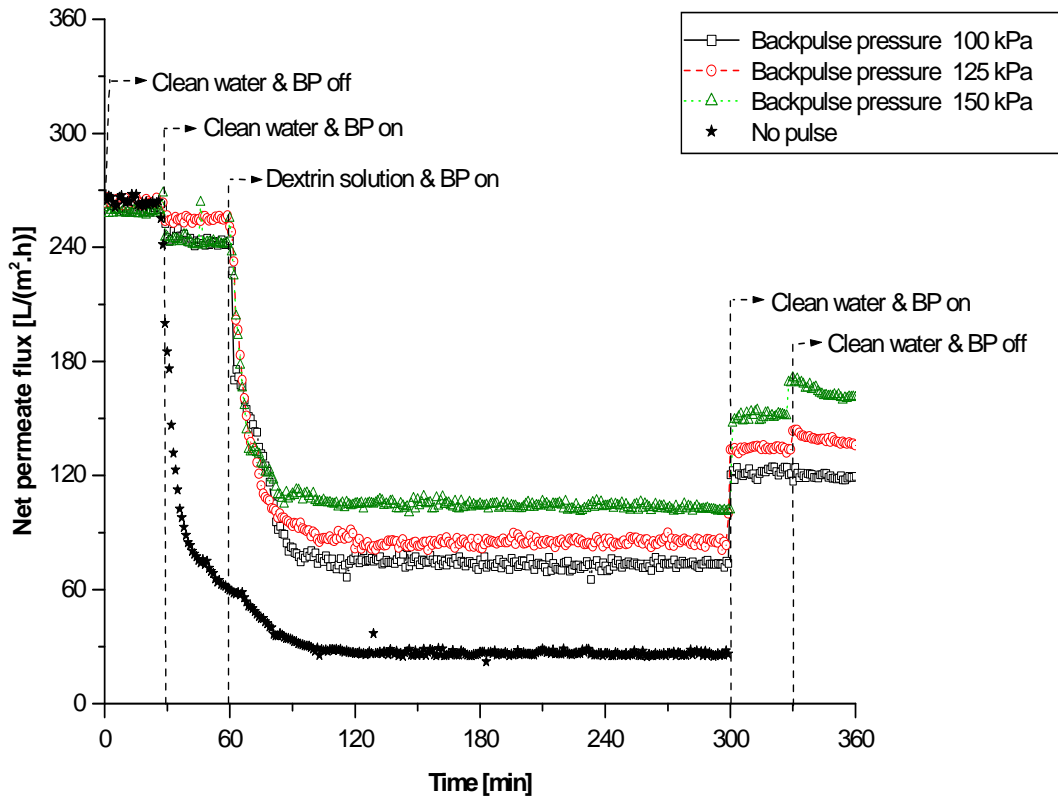


Figure 4-7: Net flux as a function of filtration time with backpulsing at different backpulse pressures. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, 3 s pulse interval, 0.2 s pulse duration, 1000 L/h cross-flow rate, dextrin feed solution 500 mg/L.)

4.2.4 Effect of cross-flow rate on the permeate flux

It has been shown in the previous sections that pulse duration, pulse interval and backpulse pressure all affected the net permeate flux. The feed flow rates and thus the cross-flow velocity also affect the membrane fouling, and therefore the net permeate flux through the membrane.

Feed flow rates of 500, 1000 and 1500 L/h were investigated (using the operating parameters: 100 kPa feed pressure and 500 mg/L dextrin feed concentration). The backpulsing was applied by fixing the pulse duration, pulse interval and backpulse pressure at 0.2 s, 3 s and 150 kPa, respectively. The effects of cross-flow rates on net permeate flux in the presence and absence of backpulsing is shown in Figure 4-8. Note that the higher the cross-flow rate applied to the membrane the higher the permeate flux observed in both cases, with and without backpulsing. This can be explained by the high flow rate generating high shear rates at the membrane surface, which act to reduce both fouling formation and the concentration polarization layer on the membrane surface [101]. Furthermore in the backpulsing case the steady-state fouled membrane flux (at 300 min) increases up to a value of about 112 L/(m².h) at 1500 L/h feed flow rate. This is due to the effect of the increased cross-flow combined with the effect of backpulsing. Note that high cross-flow rates not only act to sweep foulant away after it has been lifted off the membrane surface by backpulsing, but may also help to remove foulants from the membrane surface.

Figure 4-8 also shows the results of cleaning of the membrane at different feed flow rates by using clean water with backpulsing for 30 min (period between 300 and 330 min), and then switching off the backpulsing for the next 30 min. It was found that the net permeate fluxes (at 360 min) at the three feed flows were similar, they were about 58%, 63% and 59% of the clean membrane fluxes for feed flow rates of 500, 1000 and 1500 L/h, respectively.

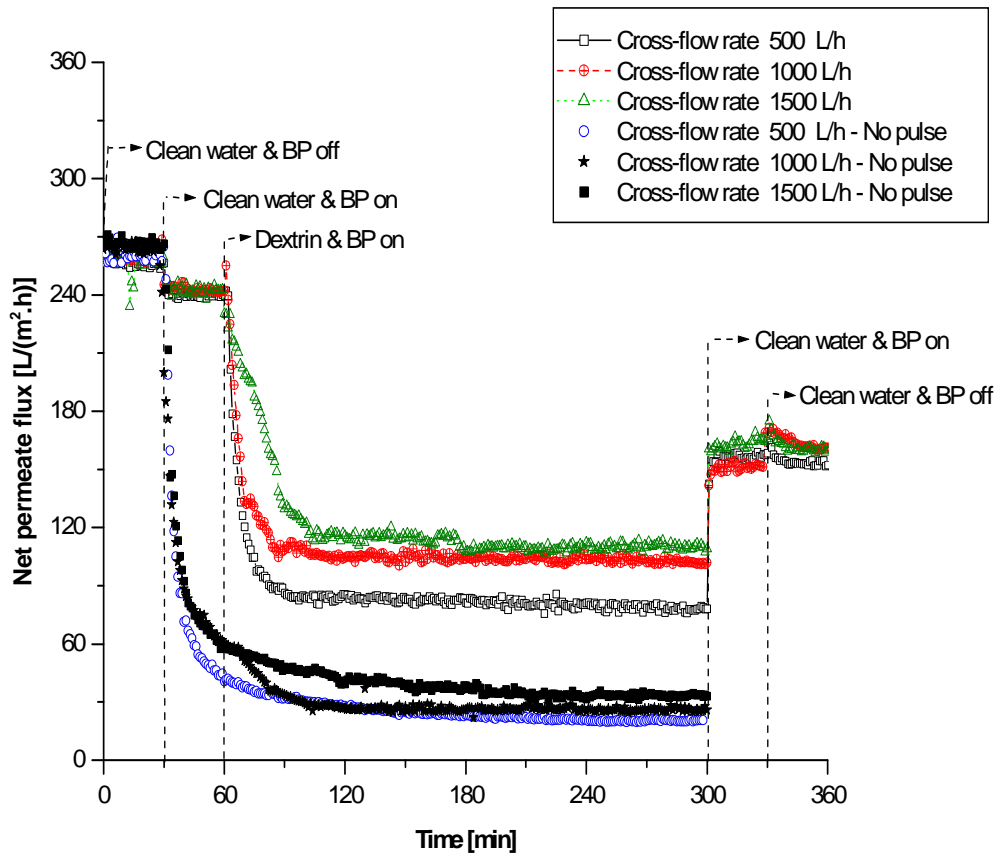


Figure 4-8: Net flux as a function of filtration time with backpulsing at various cross-flow rates. (Feed pressure 100 kPa, backpulse pressure 150 kPa, temperature 27 ± 0.5 °C, 3 s pulse interval, 0.2 s pulse duration, dextrin feed solution 500 mg/L.)

4.2.5 Effect of dextrin concentration on the permeate flux

The effect of different dextrin feed concentrations, 250, 500 and 750 mg/L, on the permeate flux was also investigated, with and without backpulsing. In all cases the experiments were performed using the following operating conditions: 100 kPa feed pressure, 1000 L/h feed flow rate, and backpulsing applied at the best conditions, namely 0.2 s pulse duration, 3 s pulse interval and 150 kPa backpulse pressure. The effects of different feed concentrations on the net permeate flux with and without backpulsing are shown in Figure 4-9. In both cases, with and without backpulsing, the higher feed concentration resulted in a lower net permeate flux. This result is to be expected, as the higher dextrin concentration almost certainly forms a thicker fouling layer, which offers a higher resistance to the permeate flow, resulting in a reduction of the flux through the membrane. Whereas at lower dextrin concentration the fouling layer forms a thinner, which offers a lower resistance to the permeate flow. As can be seen in Figure 4-9, the steady-state fouled membrane flux (at 300 min) of all dextrin solutions increased considerably in the presence of backpulsing, the net permeate flux increased by 2.5-fold 3-fold and 3.5-fold over the non pulsing case, for the 250, 500 and 750 mg/L dextrin solutions, respectively.

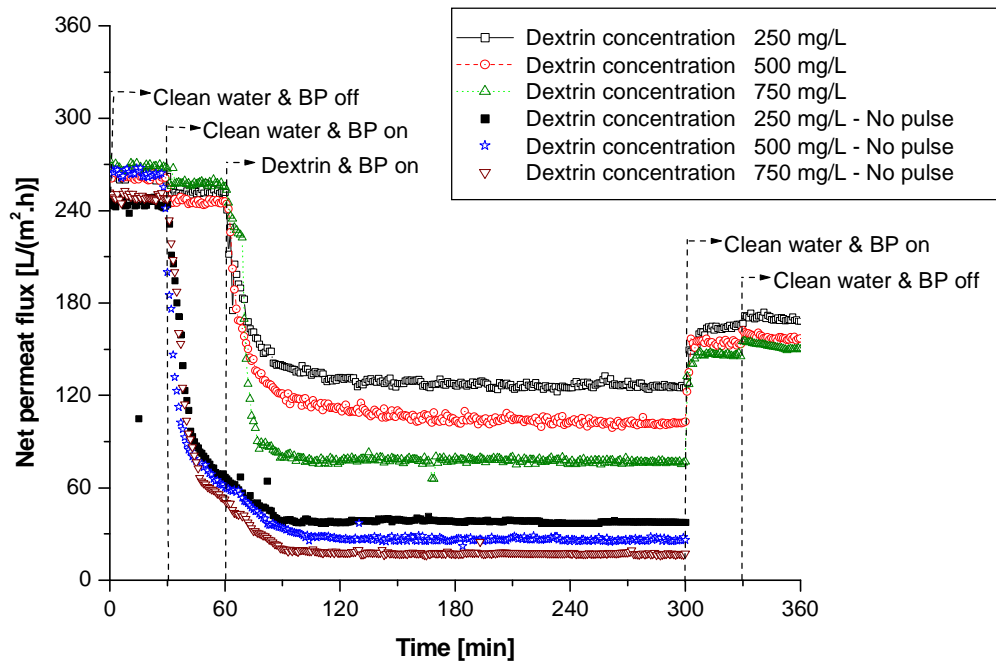


Figure 4-9: Net flux as a function of filtration time with and without backpulsing using different feed concentration. (Feed pressure 150 kPa, temperature $27\pm 0.5^{\circ}\text{C}$, 3 s pulse interval, 0.2 s pulse duration and 1000 L/h cross-flow rate).

Figure 4-9 also shows the result of cleaning of the membrane, done by changing the dextrin solution to clean water, using backpulsing for the period of time from 300 to 330 min, and then switching the backpulsing off for the next 30 min. It was found that the net permeate flux was at about 68%, 63% and 60% of the clean membrane flux in the cases of feed concentrations of 250, 500 and 750 mg/L, respectively.

4.3 Inorganic fouling (kaolin)

4.3.1 Effect of pulse durations and intervals on the permeate flux

Figures 4-10 to 4-12 show the experimental data of net permeate flux versus time for experiments performed with and without backpulsing. All the experiments were performed at a fixed feed pressure of 100 kPa and 1000 L/h feed flow rate, using 300 mg/L of kaolin in RO water as an inorganic foulant.

The backpulsing experiments were carried out at a fixed backpulse pressure of 150 kPa, with pulse durations ranging from 0.1 to 0.3 s and pulse intervals ranging from 1 to 10 s. The flux losses due to backpulsing at different pulse interval and for different pulse durations were calculated using eq. (4.1). (See Appendix A.)

Each of the results shown in Figures 4-10 to 4-12 is characterized by a moderately fast decline in the net permeate flux after commencement of fouling, then reaching a near steady-state flux after about 150 min. This behavior has been observed and confirmed by several researchers [65, 102]. This initial decline in permeate flux results primarily from the fast deposition of kaolin on the membrane surface, and the subsequent building of a fouling cake layer. These results were not too similar to the previous results (see Figures 4-3 and 4-4), because with dextrin foulant the steady-state flux was reached more rapidly than in the case of the kaolin suspension.

The net steady-state fouled membrane flux with continual backpulsing at different pulse intervals and durations, and the percentage flux change are shown in Table 4-3. The percentage of the flux change due the backpulsing was calculated by using eq. (4.2).

Table 4-3: Steady-state fouled membrane flux with continual backpulse and percentage flux change at different pulse intervals and durations

Pulse duration (s)	J_s L/(m ² .h)				Flux improvement %			
	Pulse interval (s)				Pulse interval (s)			
	1	3	5	10	1	3	5	10
0.1	123.2	126.3	113.4	-	25.2	27.1	21.9	-
0.2	-	126.6	131.9	124.3	-	27.2	29.2	26.2
0.3	-	117.2	120.8	108.4	-	24.3	25.8	20.9

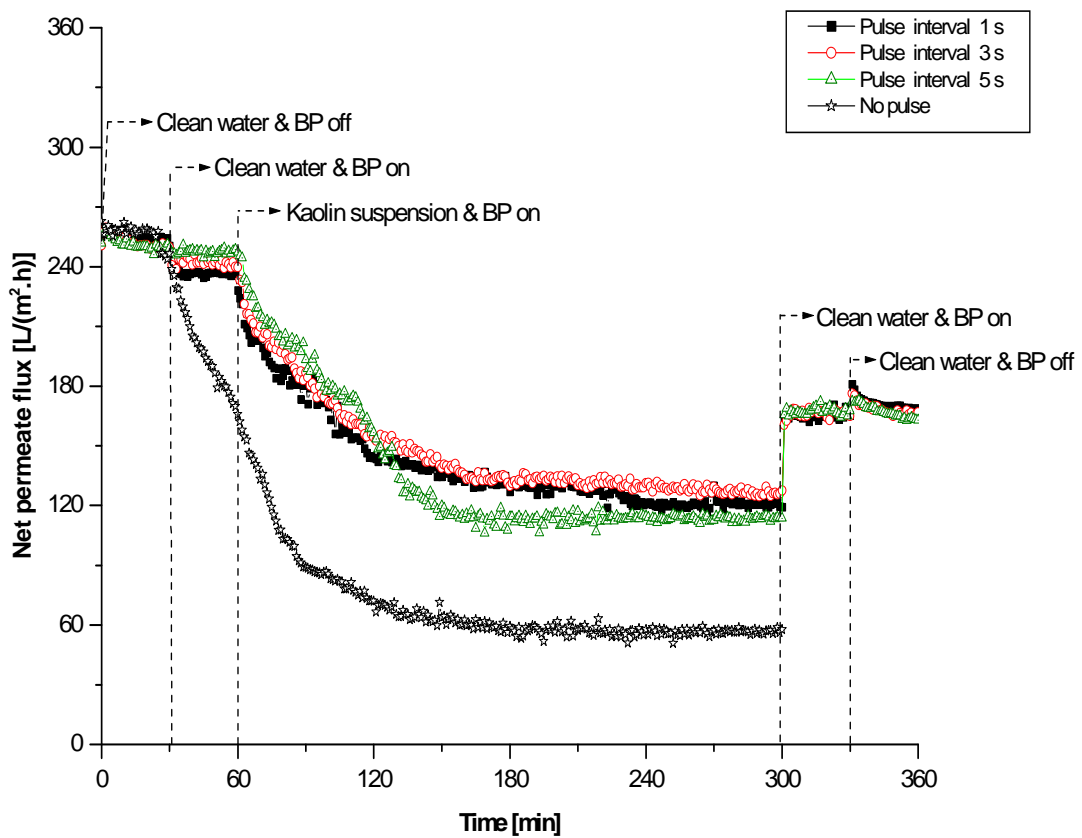


Figure 4-10: Net permeate flux as a function of time for pulse duration 0.1 s at different pulse intervals. (backpulse pressure 150 kPa, feed pressure 100 kPa, temperature 27±0.5 °C, and cross-flow rate 1000 L/h, kaolin feed suspension 300 mg/L.)

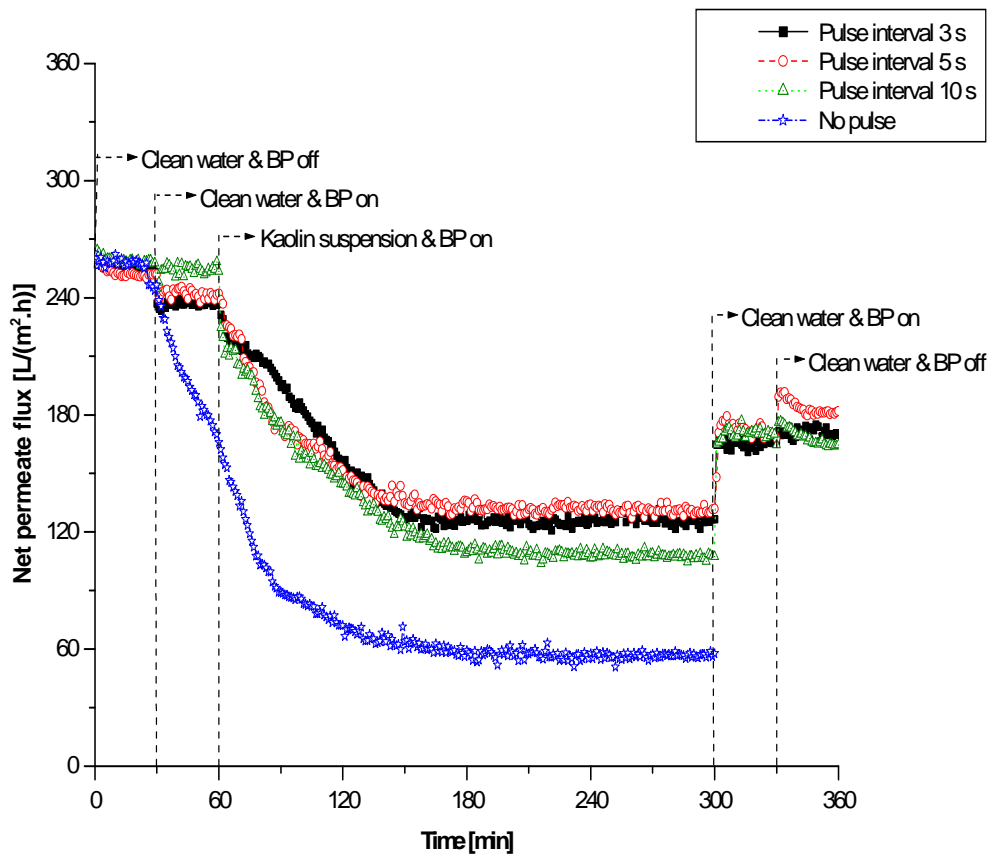


Figure 4-11: Net permeate flux as a function of time for pulse duration 0.2 s at different pulse intervals. (backpulse pressure 150 kPa, feed pressure 100 kPa, temperature 27 ± 0.5 °C, and cross-flow rate 1000 L/h, kaolin feed suspension 300 mg/L.)

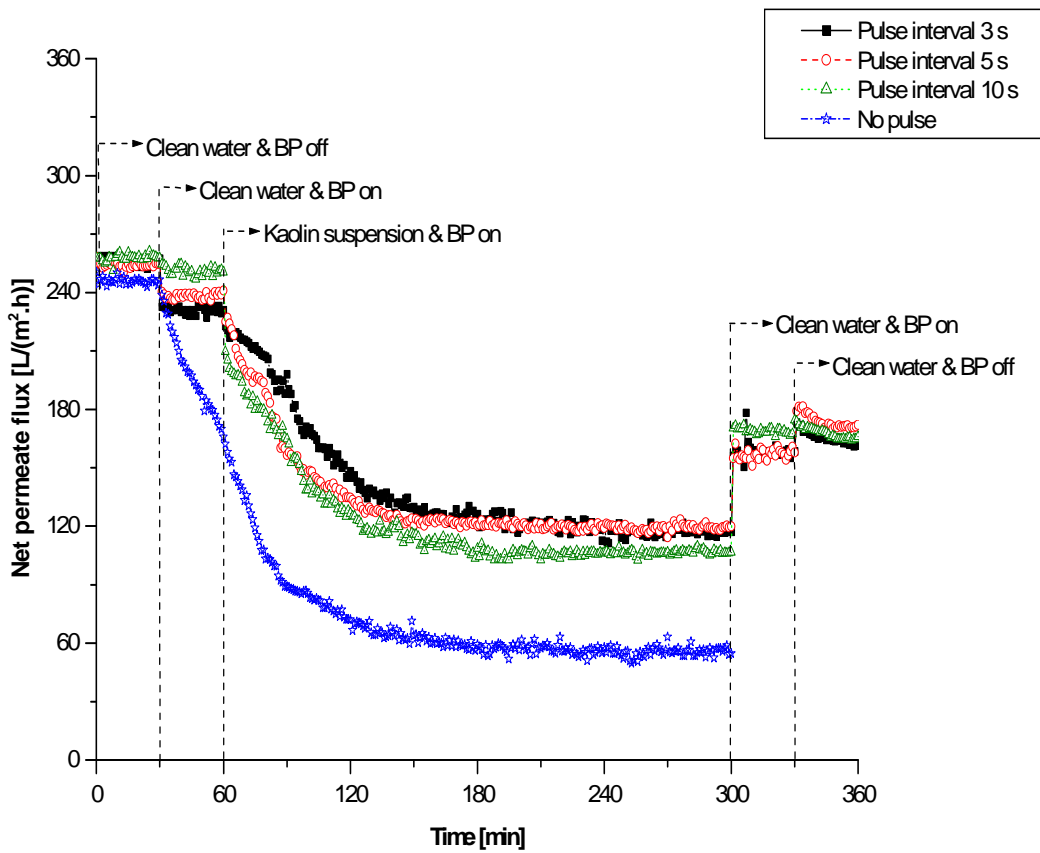


Figure 4-12: Net permeate flux as a function of time for pulse duration 0.3 s at different pulse intervals. (backpulse pressure 150 kPa, feed pressure 100 kPa, temperature 27 ± 0.5 °C, and cross-flow rate 1000 L/h, kaolin feed suspension 300 mg/L.)

Figure 4-13, shows that the backpulsing technique is highly effective in enhancing permeate flux and reducing membrane fouling for all the backpulsing conditions used in this study, but there was an optimum for the backpulsing conditions. For the shorter backpulse interval, less permeate flux is collected during forward filtration (loss of permeate during the backpulsing), whereas significant fouling and flux decline occurs during longer backpulse intervals. Furthermore, longer backpulse durations are not preferable due to unnecessary permeate loss. Very short backpulse durations are also undesirable because the backpulse is too short to remove the foulant layer effectively.

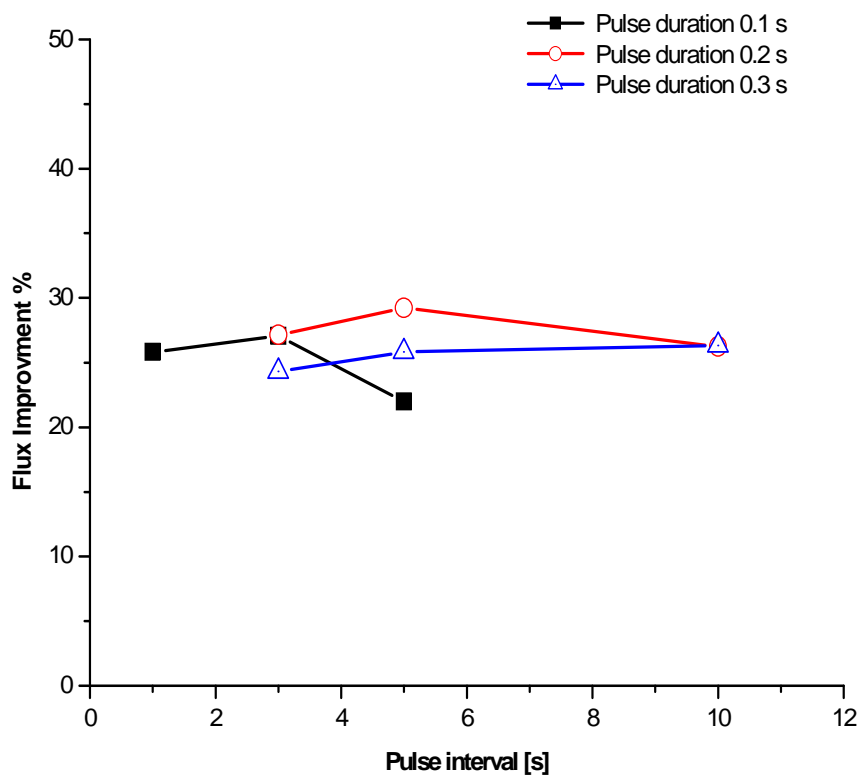


Figure 4-13: Effect of backpulsing on steady-state fouled membrane flux at different pulse intervals and pulse durations.

As shown in Figures 4-10 to 4-12, the maximum value of the steady-state fouled membrane flux with continual backpulsing was 135 L/(m².h) (at 300 min), achieved at a pulse interval of 5 s and pulse duration of 0.2 s. This is 1.5-fold greater than without backpulsing.

The membrane was then cleaned by changing the feed solution from kaolin suspension to clean water and using continual backpulsing was applied during the period between 300 and 330 min. Table 4-4 tabulates the clean membrane flux (after cleaning with backpulsing using clean water) and the percentage of clean membrane flux recovery (determined using eq. 4.3).

Table 4-4: Recovered clean membrane flux after changing the feed from dextrin solution to clean water and continual backpulsing during the period between 300 to 300 min

Pulse duration (s)	J_r L/(m ² .h)				J_r %			
	Pulse interval (s)				Pulse interval (s)			
	1	3	5	10	1	3	5	10
0.1	168.8	166.8	162.9	-	66.1	65.1	64.6	-
0.2	-	169.9	181.9	165.6	-	67.2	71.1	65.1
0.3	-	162.4	171.8	165.9	-	64.2	67.3	65.0

Figure 4-14, shows that the backpulsing is effective for cleaning the membrane. In the best case the net permeate flux (at 360 min) increased up to 71% of the clean membrane flux after, using the pulse conditions 5 s pulse interval and 0.2 s pulse duration.

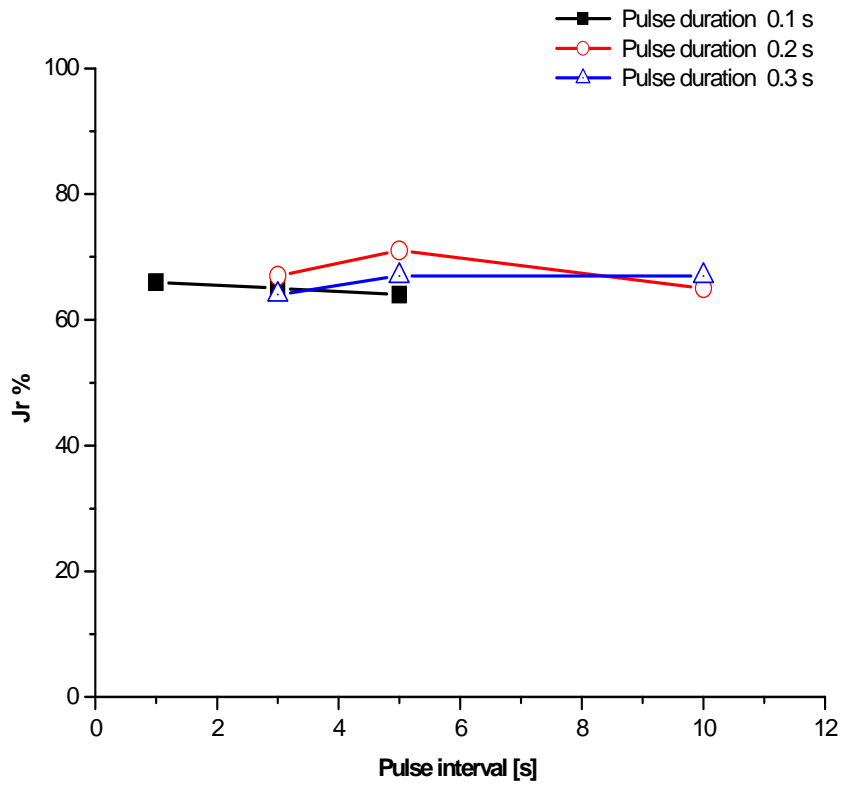


Figure 4-14: Percentage of clean membrane flux recovery after changing the feed from dextrin solution to clean water and continual backpulsing during the period between 300 to 300 min.

4.3.2 Effect of backpulse pressure on the permeate flux

Experiments carried out to investigate the effect of backpulse pressure using a kaolin concentration of 300 mg/L, a feed pressure of 100 kPa and a feed flow rate of 1000 L/h. Backpulsing pressures of 100, 125 and 150 kPa were investigated at a fixed pulse duration of 0.2 s and pulse interval of 5 s. The observed peak TMP values were 0, 25 and 50 kPa above the pressure in the feed space for backpulse pressure 100, 125 and 150 kPa respectively. The effect of backpulse pressure on the permeate flux is shown in Figure 4-15. The backpulse pressure has a significant influence on the permeate flux. The net permeate flux decreased with decreasing backpulse pressure. Note that at a backpulse pressure of 100 kPa (net peak pressure value = 0), although there was no reverse flow of permeate, the membrane was vibrated, which peeled the fouling layer off the membrane.

The maximum value of the steady-state fouled membrane flux (at 300 min) of 135 L/(m².h) was observed at a backpulse pressure 150 kPa, 0.2 s pulse duration and 5 s pulse interval. This is a 1.5-fold increase in the long-term flux, compared to that without backpulsing.

The results also show the effect of different backpulse pressures when cleaning the membrane by replacing the kaolin suspension with clean water and backpulsing during the period between 300 and 330 min. The backpulse was then switched off and the net permeate fluxes at 360 min were about 71%, 67% and 58% of the clean membrane flux, obtained at backpulse pressures of 150, 125 and 100 kPa, respectively. These results clearly show that the backpulse pressure has significant influence on the extent of flux restoration, as was the case with dextrin (Section 4.2.3).

4.3.3 Effect of cross-flow rate on the permeate flux

The cross-flow rates of 500, 1000 and 1500 L/h were investigated at operation conditions of 100 kPa feed pressure, 300 mg/L kaolin concentration. Backpulsing was applied at 150 kPa backpulse pressure, 0.2 s pulse duration and 5 s pulse interval. The effect of the cross-flow rate on the permeate flux with backpulsing is shown in Figure 4-16.

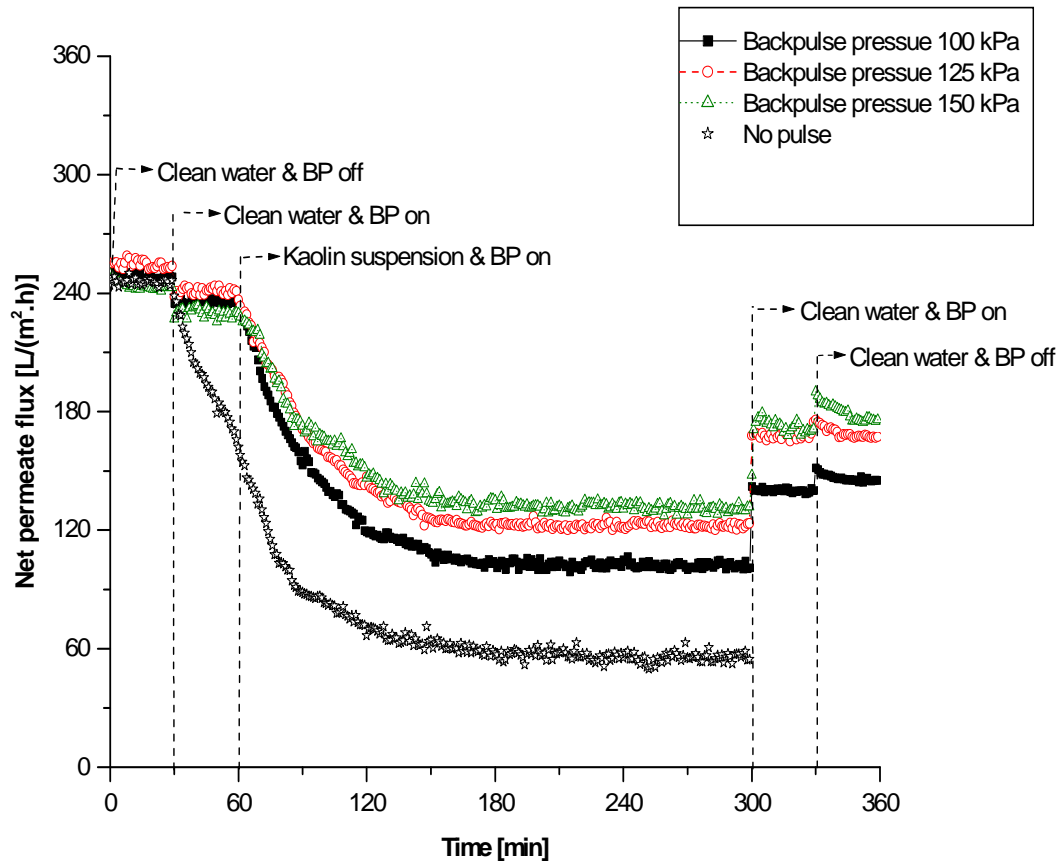


Figure 4-15: Net permeate flux as a function of time of filtration with backpulsing at different pressure pulses. (pulse interval 5 s, pulse duration 0.2 s, feed pressure 100 kPa, temperature 27 ± 0.5 °C, 1000 L/h cross-flow rate, kaolin feed suspension 300 mg/L.)

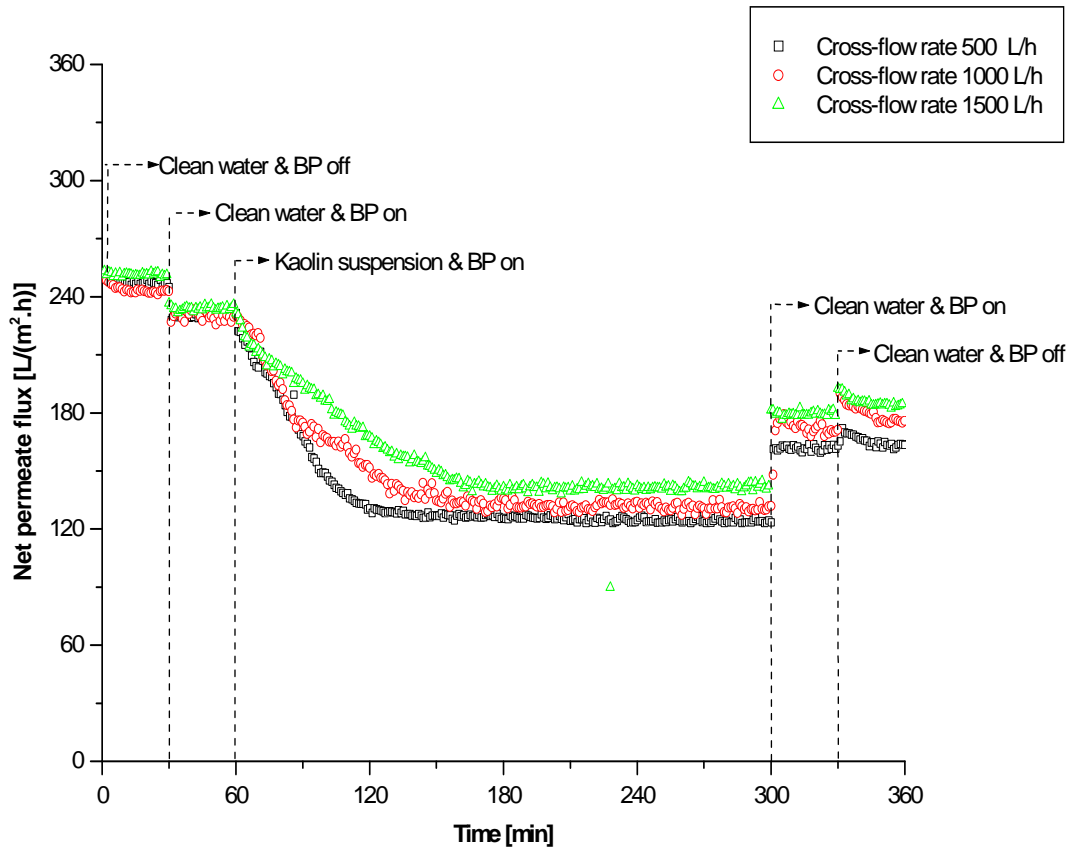


Figure 4-16: Net permeate flux as a function of filtration time with backpulsing at various cross-flow rates. (Feed pressure 100 kPa, backpulse pressure 150 kPa, temperature 27 ± 0.5 °C, pulse interval 5 s, pulse duration 0.2 s, kaolin feed suspension 300 mg/L.)

The higher cross-flow rate is more effective in reducing membrane fouling and enhancing flux. This can be explained by the high cross-flow rate generating a higher shear rate at the membrane surface. The maximum value of the steady-state fouled membrane flux of 143 L/(m².h) which was 57% of the clean membrane flux, was observed at cross-flow rate of 1500 L/h. At the low cross-flow rate of 500 L/h the permeate flux was found to be only 123 L/(m².h) (49% of the clean membrane flux). This was expected, as decreasing the cross-flow rate will reduce the shear rate at the membrane surface, which leads to increased cake layer formation and lower permeate flux.

Figure 4-16 also shows the effect of the three cross-flow rates on membrane cleaning using clean water with backpulsing, during the period of time from 300 to 330 min, (after which the backpulsing was switched off). The permeate flux (at 360 min) could be maintained at 64%, 71% and 75% of the clean membrane flux for cross-flow rates of 500, 1000 and 1500 L/h, respectively.

4.3.4 Effect of feed concentration on the permeate flux

The effect of three different concentrations of kaolin suspensions, namely 100, 300 and 500 mg/L, on the permeate flux were investigated, with and without backpulsing. In each case the experiments were run under the following operating conditions: 100 kPa feed pressure and 1000 L/h cross flow rate. The backpulsing was applied at 150 kPa backpulse pressure, 5 s pulse interval and 0.2 s pulse duration.

The effect of feed concentration on the permeate flux, with and without backpulsing, is shown in Figure 4-17. In both cases, with and without backpulsing, the higher feed concentration resulted in a faster decline of the permeate flux. This result is to be expected, as at low kaolin concentration the cake layer is thin and its removal does not result in a large reduction of the permeate flux, whereas at the higher kaolin concentrations a thick cake layer forms, and this leads to an increase the membrane fouling, resulting a greater decline in the permeate flux.

As can be seen in Figure 4-17, the steady-state fouled membrane flux (at 300 min) of all kaolin suspensions increased considerably in the presence of backpulsing.

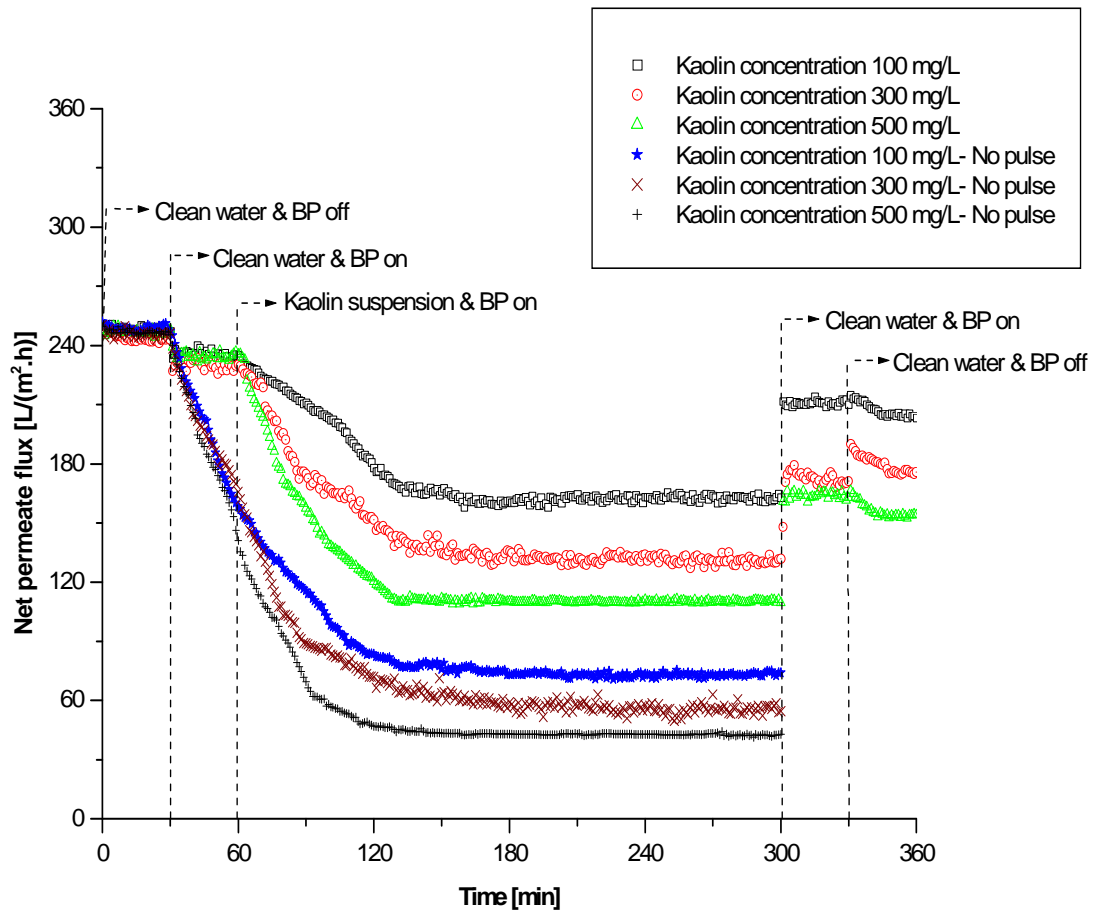


Figure 4-17: Net permeate flux as a function of filtration time with backpulsing using different kaolin feed concentrations. (Feed pressure 100 kPa, backpulse pressure 150 kPa, pulse interval 5 s, pulse duration 0.2 s, temperature 27 ± 0.5 °C and 1000 L/h cross-flow rate.)

The net permeate flux was increased 1.3-fold, 1.5-fold and 1.6-fold over the non pulsing case, for the 100, 300 and 500 mg/L kaolin suspensions, respectively.

Figure 4-17 also shows results of membrane cleaning by changing the kaolin suspension to clean water, using backpulsing (at 300 to 330 min), and then switching off the backpulsing. It was found that the net permeate flux can be maintained at about 82%, 71%, and 64% of the clean membrane flux in the cases of kaolin feed concentrations of 100, 300 and 500 mg/L, respectively.

Chapter 5

Data analysis and identification of critical parameters affecting the membrane flux

5.1 Experimental design

Several methods are available for the design of experiments to optimize the processing parameters. They include simple single-factor by single-factor approaches, the full factorial and fractional-factorial approaches and Taguchi experimental designs [103,104]. A full factorial design of experiments will include all possible combinations of the factors involved in a study, resulting in a very large number of trial runs that are time consuming and costly.

A statistical approach was developed by Taguchi to reduce costs, improve quality, and achieve robustness [105,106]. The Taguchi method is a technique that can substantially reduce the number of experimental runs. It can also be used to analyze the significance of each control factor. In the Taguchi method, responses are measured at selected combinations of the control factor levels. Each combination of control factor levels is called a run and each measures an observation. In essence, the Taguchi method uses the signal-to-noise (S/N) ratio to analyze the experimental data and find the optimal factor combination [105].

Based on results of preliminary investigation performed in Chapter 4, this chapter focuses on evaluating the main effects of the backpulse factors and feed flow rate on membrane flux, while all other process factors (i.e. temperature, feed pressure, foulant concentration and membrane design) were kept constant. Four factors were selected and varied at three levels. In Table 5-1, the factors A, B, C, and D are denoted: the pulse interval, the pulse duration, the backpulse pressure and feed flow rate, respectively. Table 5-1 also shows the values for the three levels of experimental settings. Based on results of the preliminary investigation (see Section 4.2.1), the following levels were selected:

- 1- The pulse duration was varied between 0.1 and 0.3 s.
- 2- The pulse interval was varied between 1 and 5 s.
- 3- The backpulse pressure was varied between 100 and 150 kPa.
- 4- The feed flow rate was varied between 500 and 1500 L/h.

In this work, the L9 orthogonal array with four factors and three levels for each factor, where only nine experiments are required instead of 81 experiments, was selected. An orthogonal array has the balancing property that, for each pair of

columns, all factor level combinations occur an equal number of times. In a L9 orthogonal array there are nine factor level combinations for each pair of columns, and each combination occurs once. Table 5-2 shows the standard L9 orthogonal array of the nine experiments to be used. Each experiment is based on a combination of level values. Each three-level factor has two degrees of freedom (DOF) and the DOF equals the number of levels minus one. Therefore, the DOF required for four factors, each at three levels, is eight ($8 = 4 \times (3-1)$).

Although it is accepted that interactive effects between the factors may influence the outcome of results, the Taguchi method (as applied here) would not be able to verify such effects. As much, the aim of this limited statistical analysis was merely to highlight the relative prominence of the four important factors, thus contextualizing the observations made in Chapter 4.

Table 5-1: Design factors and their levels used in the Taguchi method

Design factor	Symbol	Low level (1)	Mid level (2)	High level (3)
Pulse interval	A	1 s	3 s	5 s
Pulse duration	B	0.1 s	0.2 s	0.3 s
Backpulse pressure	C	100 kPa	125 kPa	150 kPa
Feed flow rate	D	500 L/h	1000 L/h	1500 L/h

Table 5-2: Standard L9 orthogonal array used for the Taguchi method in this study

Run	Control factors			
	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

5.2 Results and discussion

The results of the nine final runs, using Dextrin solution with different combinations of the four factors, are shown in Appendix B. Two responses are observed for these experiments:

- The steady-state fouled membrane flux with continuous backpulsing (J_s), which was measured after 300 min of filtration operation.
- Recovered clean membrane flux after cleaning the membrane using RO water while backpulsing (J_r), measured after 360 min of filtration operation.

The measured responses are presented in Table 5-3.

Table 5-3: Experimental results of Taguchi orthogonal array L9

Run	Factors				Responses	
	A (s)	B (s)	C (kPa)	D (L/h)	J_s L/(m ² .h)	J_r L/(m ² .h)
1	1	0.1	100	500	56	118
2	1	0.2	125	1000	85	155
3	1	0.3	150	1500	83	159
4	3	0.1	125	1500	88	150
5	3	0.2	150	500	96	158
6	3	0.3	100	1000	64	125
7	5	0.1	150	1000	82	149
8	5	0.2	100	1500	58	120
9	5	0.3	125	500	81	146

5.2.1 The signal-to-noise (S/N) ratio analysis

Taguchi Methods use the signal-to-noise (S/N) ratio to analyse the test run results, because the S/N ratio represents both the average (mean) and variation (scatter) of the experimental results. In order to evaluate the influence of each of the selected factors on the response, the S/N ratio for each factor had to be calculated. In this study the S/N ratio was chosen according to the criterion *the-larger-the-better*, in order to

maximize the responses. The S/N ratio for *the-larger-the-better* target for two responses was calculated as follows:

$$S/N_{LTB} = -10 \cdot \log\left[\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2}\right] \quad (5.1)$$

where S/N_{LTB} is the-larger-the-better signal-to-noise ratio, y_i is the individually measured response value (experiment result) and n is the number of measurements taken in one test run.

The S/N_{LTB} ratios for the two responses J_s and J_r are shown in Table 5-4. The effect of each process factor on the S/N_{LTB} ratio at different levels for each response can be separated because the experimental design is orthogonal. The averages of the S/N_{LTB} ratios at different levels of the process parameters for J_s and J_r are summarized in Table 5-5.

Figure 5-1 shows the average S/N_{LTB} ratio graph for the steady-state fouled membrane flux with continuous backpulsing (J_s) and the recovered clean membrane flux after cleaning with RO water and backpulsing (J_r). It can be noticed from this figure that the pulse pressure (C) is the most important factor affecting the responses: the maximum value of response is at the highest level of pulse pressure (C3). This result is to be expected, as the higher pulse pressure is the most effective factor in removing foulant. The minimum value of the response is at the lowest level of pulse pressure (C1) 100 kPa. At this pressure the membrane is only vibrated and membrane cleaning is not effective. It can also be seen in Figure 5-1 that the pulse interval (A) and pulse duration (B) have a lower relevant effect within the experimental limits, while the feed flow rate (D) shows the lowest effect among the four factors considered here.

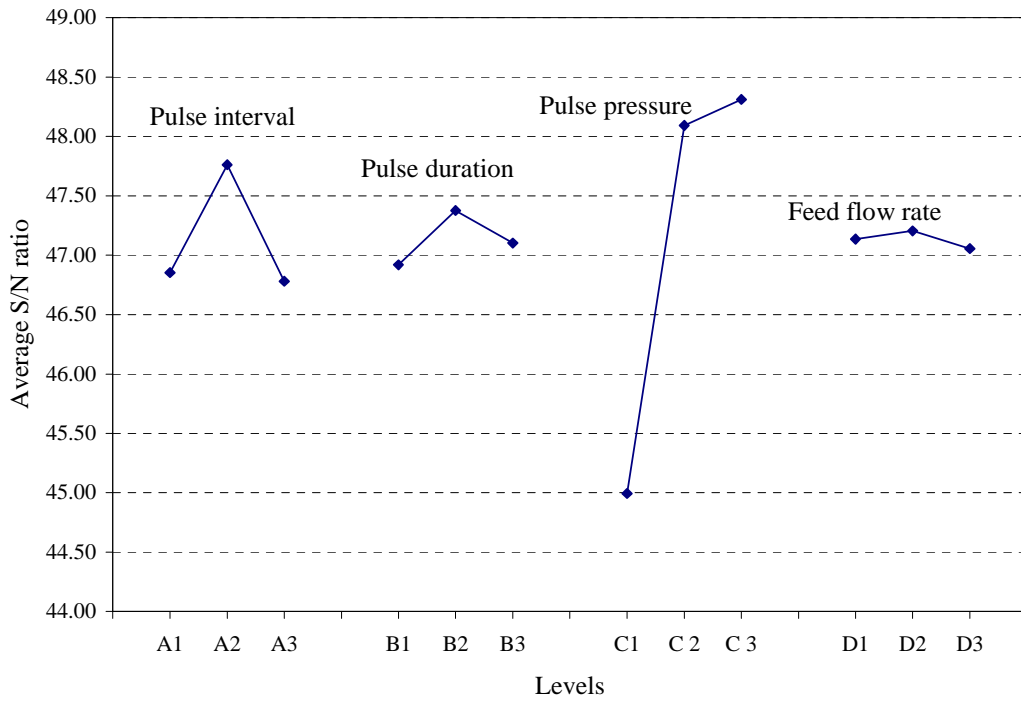
For the low level of the pulse interval (A1), less permeate flux is collected during forward filtration (loss of permeate during backpulsing), whereas fouling and flux decline occurs at the high level of pulse interval (A3). Furthermore, the high level of pulse duration (B3) is not preferable due to unnecessary permeate loss. The low level of pulse duration (B1) is too short and not sufficient to remove the foulant layer effectively.

Table 5-4: S/N_{LTB} ratio for responses J_s and J_r

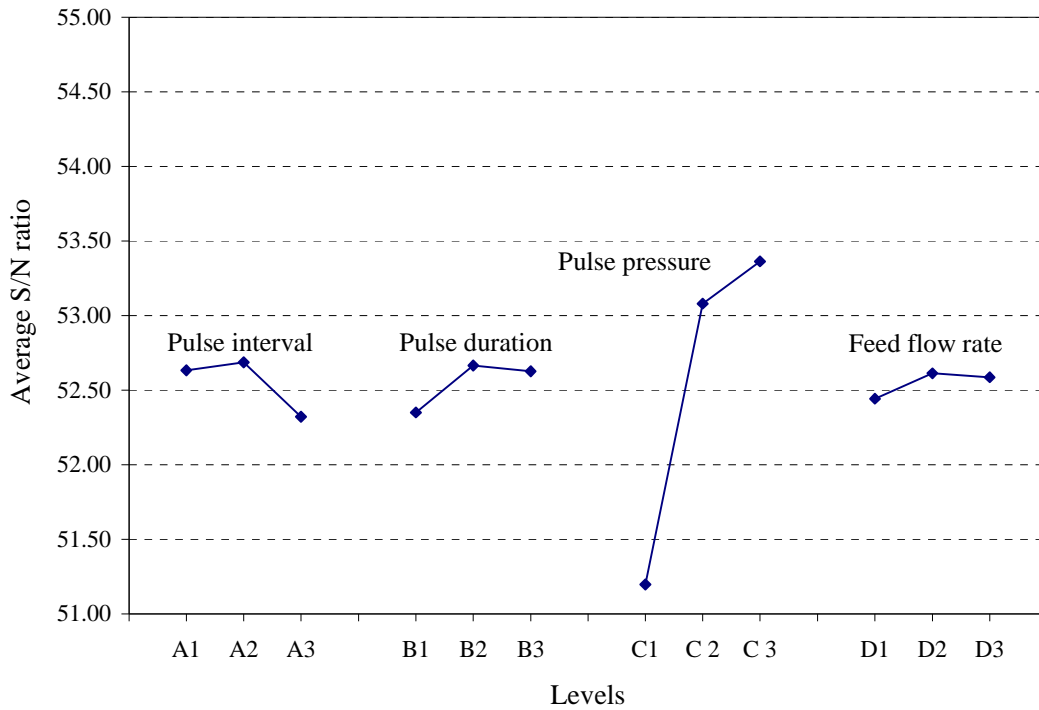
Exp. No.	S/N_{LTB} ratio	
	For J_s	For J_r
1	44.5	50.9
2	48.1	53.4
3	47.9	53.8
4	48.4	53.1
5	49.2	53.52
6	45.7	51.48
7	47.8	53.01
8	44.8	51.13
9	47.7	52.83

Table 5-5: Average S/N_{LTB} ratio for each level of the process variables

Process variables	J_s			J_r		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
Pulse interval (A)	46.85	47.76	46.78	52.63	52.69	52.32
Pulse duration (B)	46.92	47.38	47.10	52.35	52.66	52.63
Pulse pressure (C)	44.99	48.09	48.31	51.20	53.08	53.36
Feed flow rate (D)	47.14	47.21	47.06	52.44	52.61	52.59



(a)



(b)

Figure 5-1: Average S/N_{LTB} ratio graphs for: (a) steady-state fouled membrane flux with continual backpulsing (J_s), and (b) recovered clean membrane flux after cleaning the membrane with RO water and backpulsing (J_r).

5.2.2 Regression model

Regression analysis using a spreadsheet program (Microsoft Excel, 2003) was applied for data analysis and to develop simple regression models for both the fouled membrane flux (J_s) and recovered clean membrane flux (J_r).

By applying linear regression, the following linear model equations were generated:

$$J_s = 9.25 - 0.25 A + 3.33 B + 0.55 C \quad (5.2)$$

$$J_r = 53.97 - 1.42 A + 21.67 B + 0.69 C \quad (5.3)$$

Note that D was omitted from these equations, since its coefficient turned out to be very close to zero (see Tables 5-6 and 5-7 below). However, this does not imply that feed flow rate has a less significant effect on flux than the other three variables.

Table 5-6 Estimates of the regression coefficients for (J_s) response.

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	9.25	25.69	0.36	0.74	-62.08	80.58
Pulse interval (A)	-0.25	2.14	-0.12	0.91	-6.20	5.70
Pulse duration (B)	3.33	42.87	0.08	0.94	-115.68	122.35
Pulse pressure (C)	0.55	0.17	3.23	0.03	0.08	1.03
Feed flow (D)	0.00	0.01	-0.16	0.88	-0.03	0.02

Table 5-7 Estimates of the regression coefficients for (J_r) response.

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	53.97	22.52	2.40	0.07	-8.55	116.50
Pulse interval (A)	-1.42	1.88	-0.75	0.49	-6.63	3.80
Pulse duration (B)	21.67	37.58	0.58	0.60	-82.66	126.00
Pulse pressure (C)	0.69	0.15	4.57	0.01	0.27	1.10
Feed flow (D)	0.00	0.01	0.31	0.77	-0.02	0.02

Tables 5-6 and 5-7 show the estimates of the regression coefficients and other important statistical values, such as the *P-value* for the individual coefficients and the confidence intervals. If the *P-value* is very small (less than 0.05) then the individual terms in the model have a significant effect on the response. Based on this, it is clear that the main effect factor is pulse pressure (C), while the effects of pulse interval

(A) and pulse duration (B) on membrane flux are insignificant. Although the feed flow rate (D) has a more notable effect than pulse interval and pressure, it is still small compared to the effect of pulse pressure. It must immediately be stated here that these observations are only valid and of value if considered inside the experimental boundaries selected for pulse pressure, interval and duration.

Figures 5-2 and 5-3 show the relationship between the actual and predicted values of J_s and J_r models respectively. These figures indicate that these linear regression models are adequate, because the residuals in the prediction of each response are rather small.

5.3 Summary

In this Chapter the Taguchi method was applied to identify the influential factors backpulsing that give maximum permeate flux in spiral wrap UF element. An orthogonal array with four factors was selected. Statistical regression analysis of results indicates that the pulse pressure has the largest contribution to the total sum of squares and correspondingly has a major influence on the membrane flux. Pulse interval and pulse duration have negligible effects and, in comparison, cross-flow rate has a weak effect on the membrane flux. It must be noted that these observations are only valid within the experimental boundaries, as identified during the preliminary investigation.

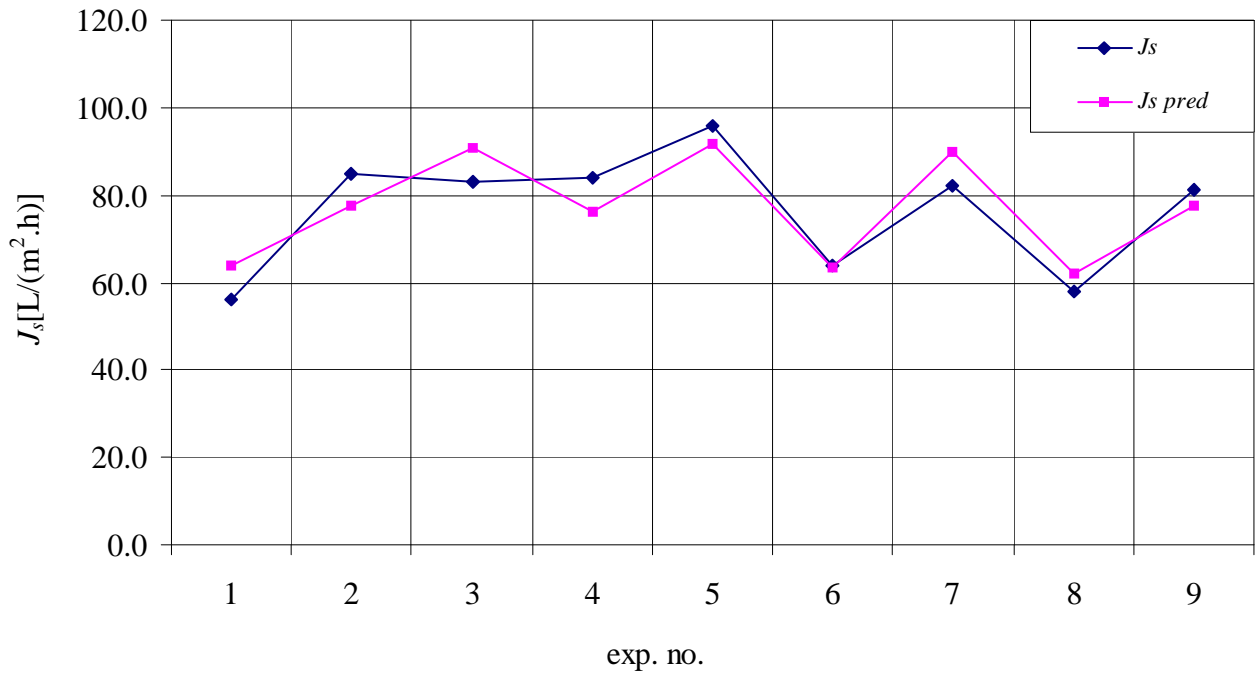


Figure 5-2: Effect of actual process parameters on response J_s against the predicted.

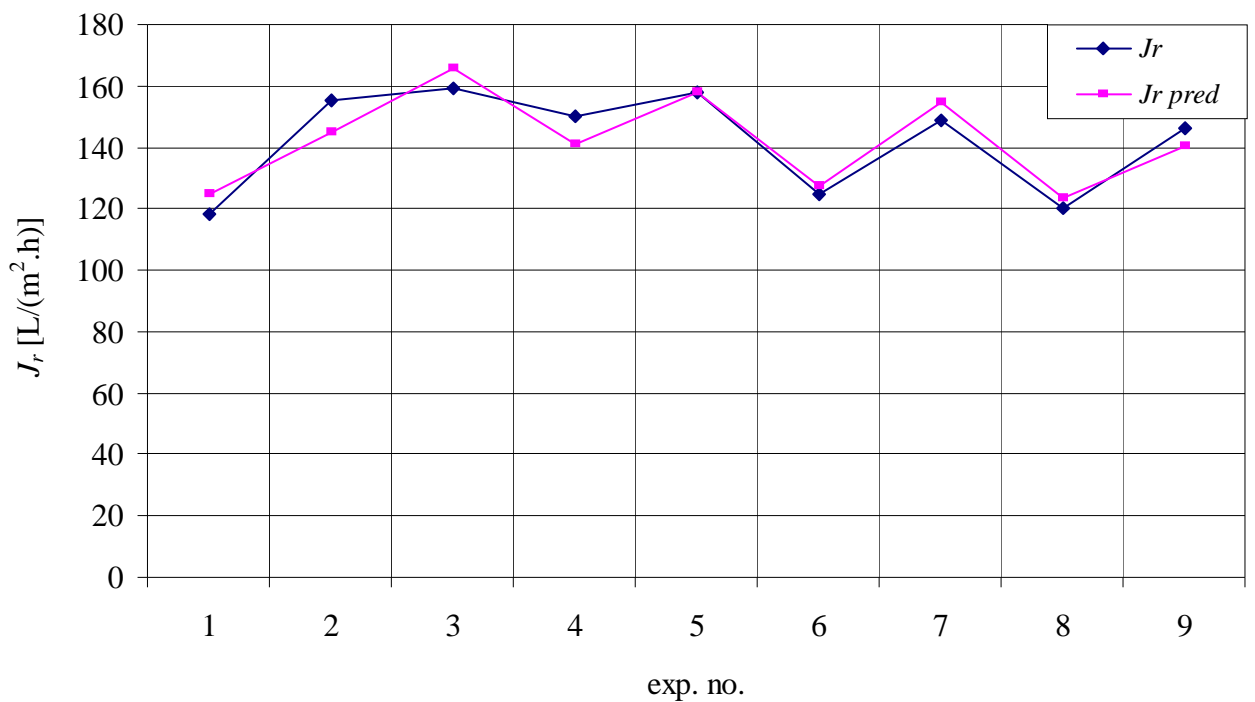


Figure 5-3: Effect of actual process parameters on response J_r against the predicted.

Chapter 6

Conclusions and recommendations

6.1 Conclusions

- A spiral wrap UF plant was modified to include a backpulsing unit. Control of the pulse shape was eventually achieved as a square peak function. The pulse shape and the UF plant were successfully controlled, and monitored by a Labview program. It was now possible to determine the effects of the backpulse interval, duration and pressure, feed flow rate and feed concentration on the permeate flux, using organic and inorganic foulants.
- It was found that in both cases of fouling with organic (dextrin) and inorganic (kaolin) foulants, backpulsing was not effective in completely removing the initial fouling due to both pore adsorption and a fouling layer on the membrane surface. Backpulsing appeared to be effective only in preventing the long-term fouling that occurs due to the build-up of a fouling layer on the membrane surface.

However, the use of continuous backpulsing proved to be highly effective in reducing membrane fouling and enhancing permeate flux under all the backpulsing conditions used in this study. Optimum backpulse conditions were identified. For the shorter backpulse intervals (i.e. 1 s) less permeate flux was collected during forward filtration (loss of permeate flux during the backpulsing), whereas significant fouling and flux decline occurred during longer backpulse intervals (i.e. 15 s). Furthermore, longer backpulse durations (i.e. 0.5 s) were not preferable due to unnecessary permeate loss. Very short backpulse durations (i.e. 0.1 s) were also undesirable because they were too short to remove the foulant layer effectively.

- The optimum backpulsing duration and interval were determined for each foulant. These were as follows: 0.2 s pulse duration and 3 s pulse interval for dextrin solutions, and 0.2 s pulse duration and 5 s pulse interval for kaolin suspensions. The net permeate fluxes achieved with backpulsing under these conditions were as high as 3-fold and 1.5-fold greater than the saturation flux values recorded during the non pulsing case for dextrin and kaolin, respectively.

The backpulse pressure was found to have a significant influence on the permeate flux. The permeate flux increased significantly with increasing backpulse pressure. A significant improvement in the permeate flux was observed when a backpulse pressure of 150 kPa was used, compared to 100 kPa.

- The operating parameters such as feed flow rate and feed concentration were investigated with and without backpulsing. It was found the higher the cross-flow rate applied to the membrane the higher the saturation permeate flux observed in both cases, with and without backpulsing. The concentration of the feed was found to have a significant influence on the permeate flux. The higher feed concentration resulted in a lower permeate flux in both cases, with and without backpulsing.
- Flux recovery after operation, applying backpulsing with clean water, was found to be quite effective. The permeate flux could typically to be recovered from 40% to 63% and from 54% to 72% of the original clean membrane flux, for the feed solutions of dextrin and kaolin respectively.
- Statistical analysis of data revealed that the pulse pressure had the strongest effect on the net membrane flux. Pulse interval and pulse duration have negligible effects and, in comparison, cross-flow rate has a weak effect on the membrane flux. It must be noted that these observations are only valid within the experimental boundaries, as identified during the preliminary investigation.

6.2 Recommendations

- Optimize the backpulsing in a spiral wrap plant for operation periods of several days, weeks or months.

- Investigate the effect of the backpulse technique on the performance of MF/UF membranes in different types of membrane modules (e.g. a capillary membrane module).
- Backpulsing has been identified as a promising approach to combating fouling in membranes. In principle, it can be used on-line. However, these conclusions have been based on small-scale laboratory studies, which have not taken the economic feasibility of the approach into account. Further studies should include assessment the of economic viability and technical feasibility of using backpulsing on a larger-scale to minimize fouling in membrane filtration plants.

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Appendix A
Flux loss due to backpulsing for cross-flow
UF element

A.1 Flux loss due to backpulsing for cross-flow UF element (Dextrin case)

Flux losing due to BP = Clean membrane flux without BP - Clean membrane flux with BP

Table A-1: Flux loss experimentally due to backpulsing (BP)

Pulse duration (s)	Avg. clean membrane flux without BP L/(m ² .h)					Avg. clean membrane flux with BP L/(m ² .h)					Flux losing due to BP L/(m ² .h)				
	Pulse interval (s)					Pulse interval (s)					Pulse interval (s)				
	1	3	5	10	15	1	3	5	10	15	1	3	5	10	15
0.1	257.1	262.0	258.9	-	-	231.2	253.1	254.1	-	-	25.8	8.9	4.8	-	-
0.2	259.7	259.2	265.6	265.6	266.8	203.4	242.9	256.2	259.6	262.7	56.3	16.2	9.4	6.1	4.1
0.3	-	265.0	262.3	264.9	261.4	-	241.4	244.8	256.6	256.7	-	23.6	17.5	8.4	4.9
0.5	-	-	264.1	262.8	260.6	-	-	233.9	250.5	252.6	-	-	30.2	12.3	7.9

Table A-2: Comparison of flux losing due to backpulsing between experimental values and calculated values

Pulse duration (s)	Pulse interval (1 s)			Pulse interval (3 s)			Pulse interval (5 s)			Pulse interval (10 s)			Pulse interval (15 s)		
	Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)		
	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %
0.1	25.7	25.8	0.4	8.7	8.9	2.2	5.2	4.8	6.9	-	-	-	-	-	-
0.2	51.8	56.3	8.7	17.3	16.2	5.9	10.6	9.4	11.4	5.3	6.0	11.9	3.6	4.1	13.8
0.3	-	-	-	26.5	23.6	10.8	15.7	17.5	10.9	7.9	8.3	4.8	5.2	4.9	6.2
0.5	-	-	-	-	-	-	26.4	30.2	14.3	13.1	12.3	6.5	8.7	7.9	9.2

A.2 Flux loss due to backpulsing for cross-flow UF element (Kaolin case)

Flux losing due to BP = Clean membrane flux without BP - Clean membrane flux with BP

Table A-3: Flux loss experimentally due to backpulsing

Pulse duration (s)	Avg. clean membrane flux without BP L/(m ² .h)				Avg. clean membrane flux with BP L/(m ² .h)				Flux losing due to BP L/(m ² .h)			
	Pulse interval (s)				Pulse interval (s)				Pulse interval (s)			
	1	3	5	10	1	3	5	10	1	3	5	10
0.1	255.6	252.3	251.3	-	232.1	241.1	245.7	-	23.6	9.1	5.6	-
0.2	-	255.7	252.9	259.4	-	237.2	242.2	254.1	-	18.5	10.8	5.4
0.3	-	255.9	254.1	257.7	-	231.2	238.3	250.9	-	24.7	15.8	6.7

Table A-4: Comparison of flux losing due to backpulsing between experimental values and calculated values

Pulse duration (s)	Pulse interval (1 s)			Pulse interval (3 s)			Pulse interval (5 s)			Pulse interval (10 s)		
	Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)			Flux loss due to BP L/(m ² .h)		
	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %	Calculated value	Experimental value	Error %
0.1	25.6	23.6	8.3	8.4	9.1	7.7	5.2	5.6	7.3	-	-	-
0.2	-	-	-	17.2	18.5	7.2	10.3	10.8	4.5	5.18	5.4	4.7
0.3	-	-	-	25.6	24.7	3.5	15.4	15.8	2.8	7.71	6.7	14.3

Appendix B

Experimental data used for Taguchi orthogonal array L9

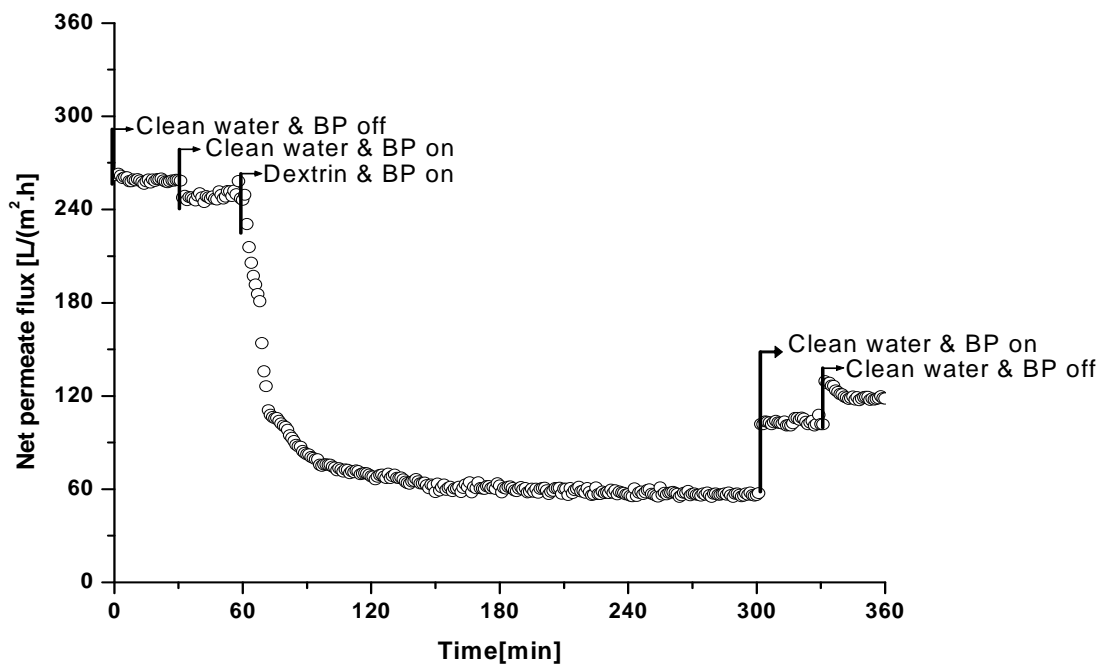


Figure B-1: Net permeate flux in run #1: 1 s pulse interval, 0.1 s pulse duration, 100 kPa pulse pressure, 500 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution (500 mg/L).

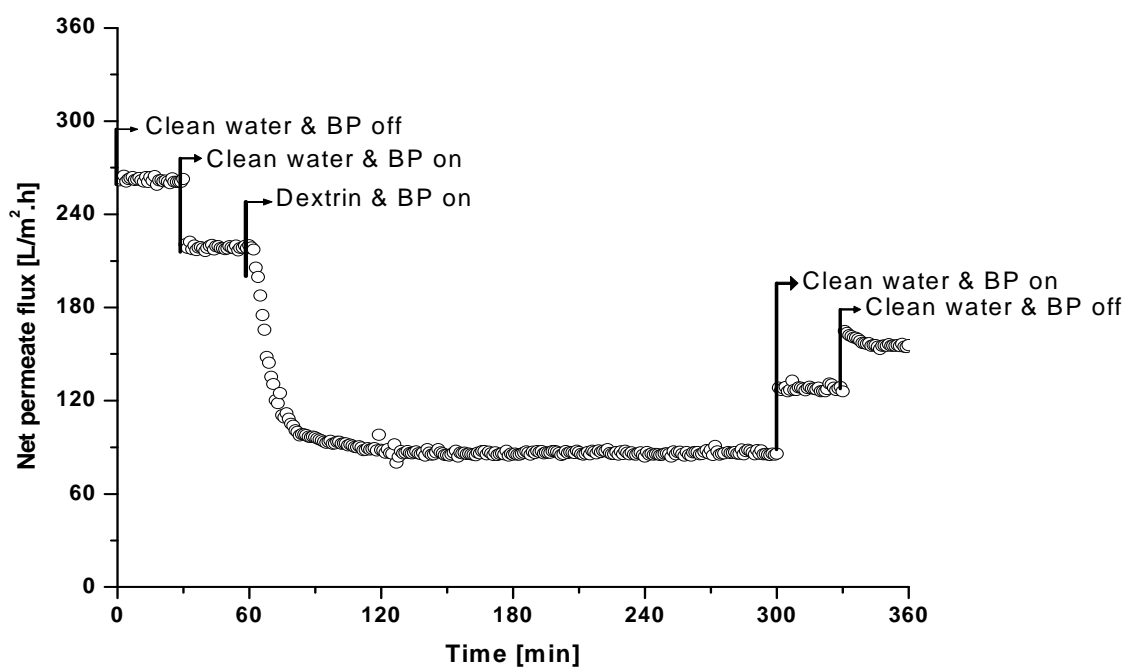


Figure B-2: Net permeate flux in run #2: 1 s pulse interval, 0.2 s pulse duration, 125 kPa pulse pressure, 1000 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

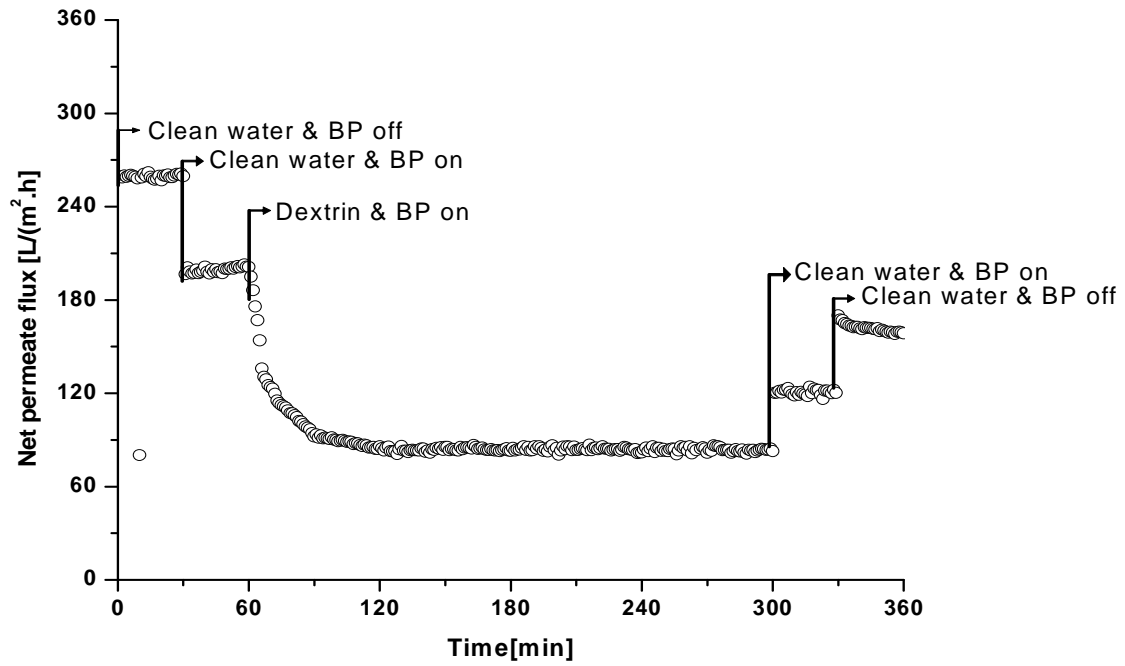


Figure B-3: Net permeate flux in run #3: 1 s pulse interval, 0.3 s pulse duration, 150 kPa pulse pressure, 1500 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

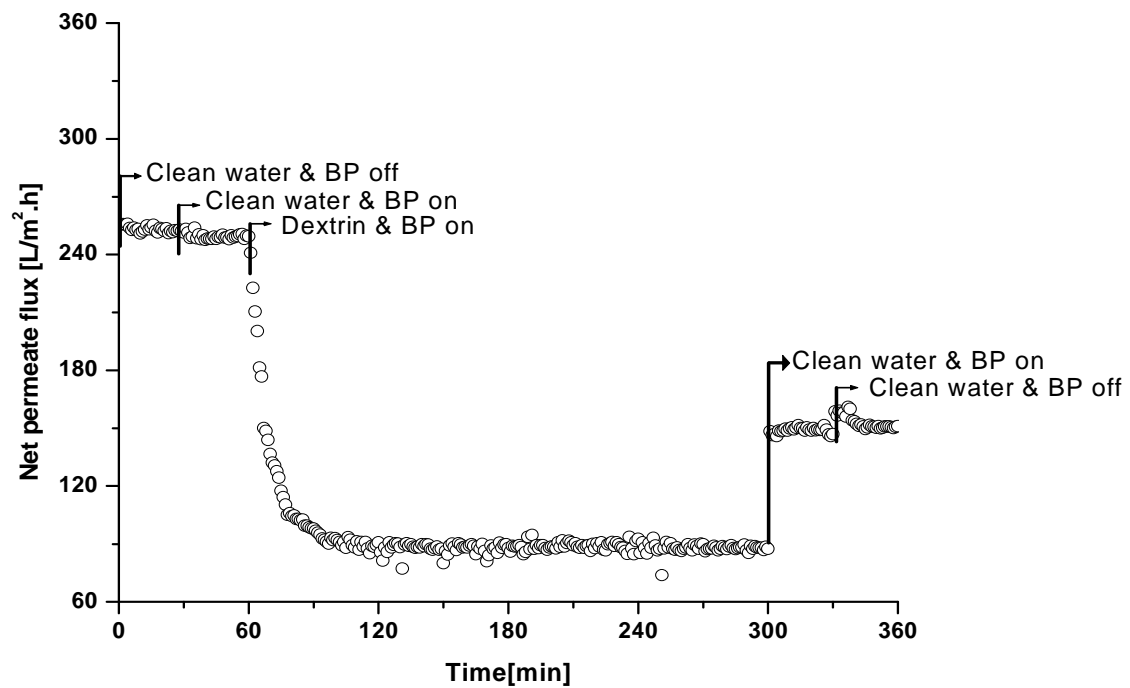


Figure B-4: Net permeate flux in run #4: 3 s pulse interval, 0.1 s pulse duration, 125 kPa pulse pressure, 1500 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

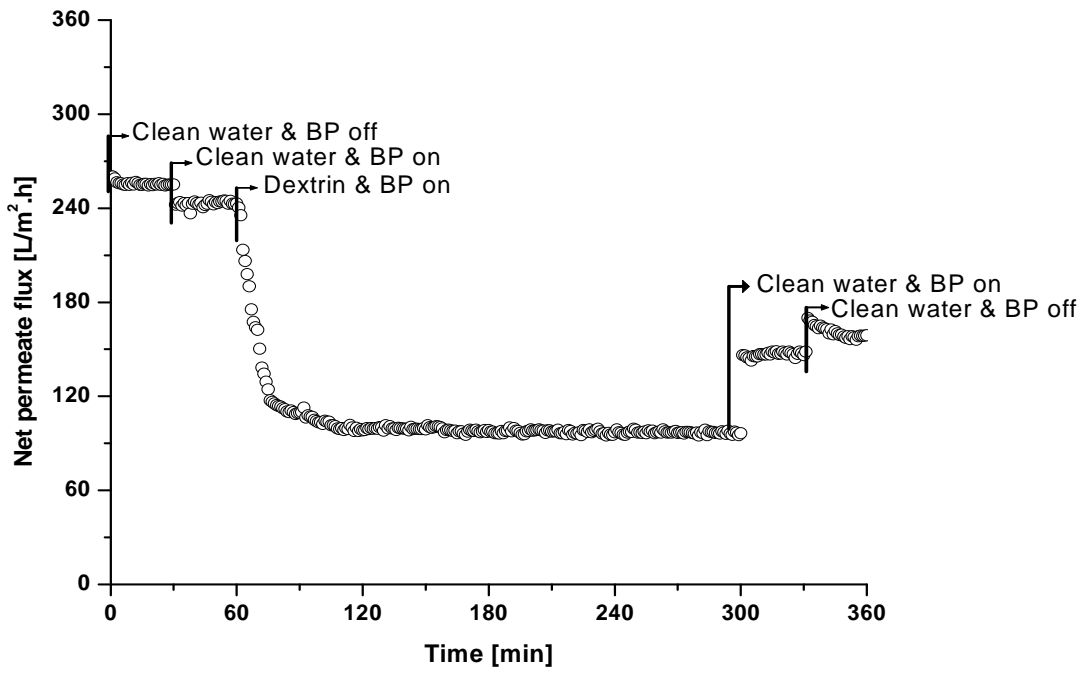


Figure B-5: Net permeate flux in run #5: 3 s pulse interval, 0.2 s pulse duration, 150 kPa pulse pressure, 500 L/h. (Feed pressure 100kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

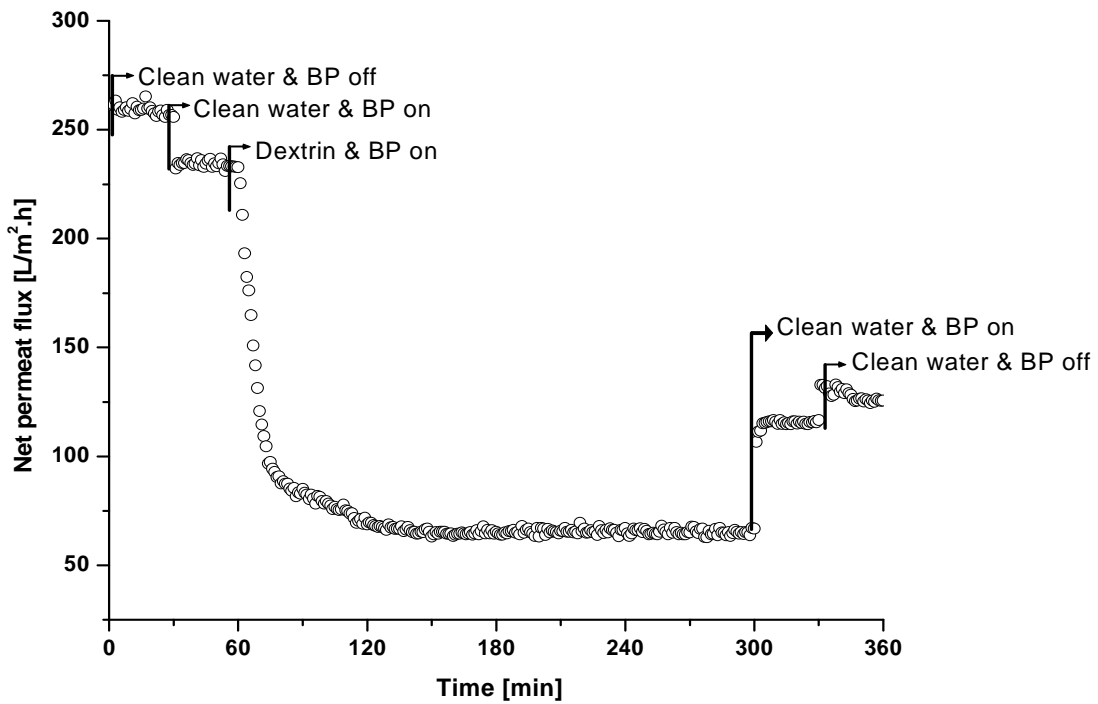


Figure B-6: Net permeate flux in run #6: 3 s pulse interval, 0.3 s pulse duration, 100 kPa pulse pressure, 1000 L/h. (Feed pressure 100kPa, temperature 27 ± 0.5 °C, dextrin feed solution (500 mg/L).

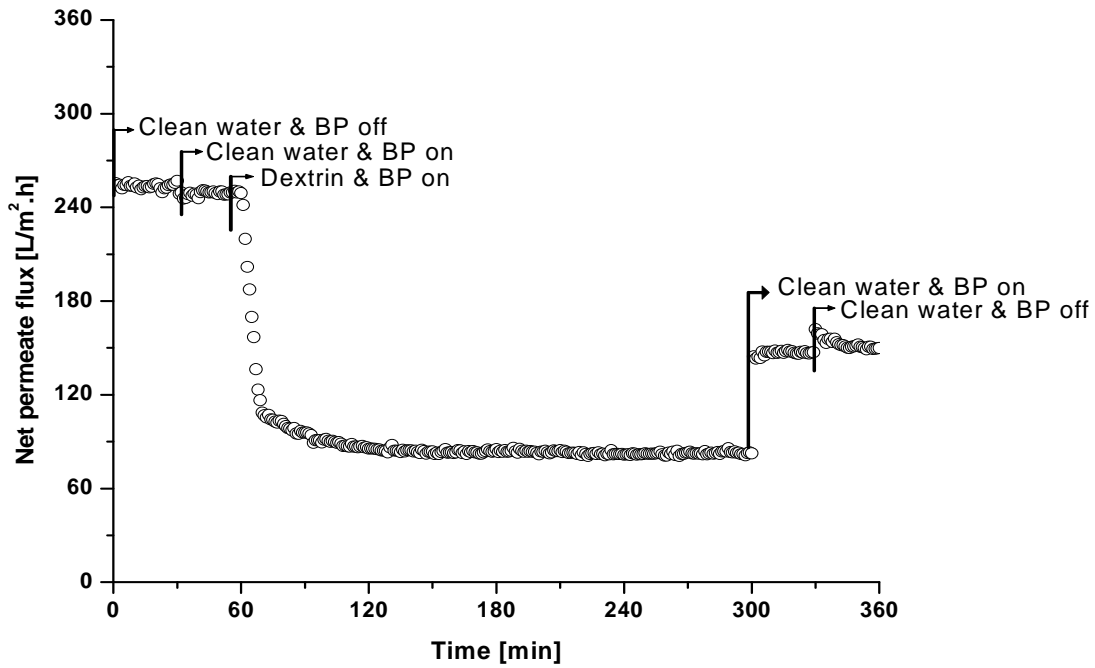


Figure B-7: Net permeate flux in run #7: 5 s pulse interval, 0.1 s pulse duration, 150 kPa pulse pressure, 1000 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

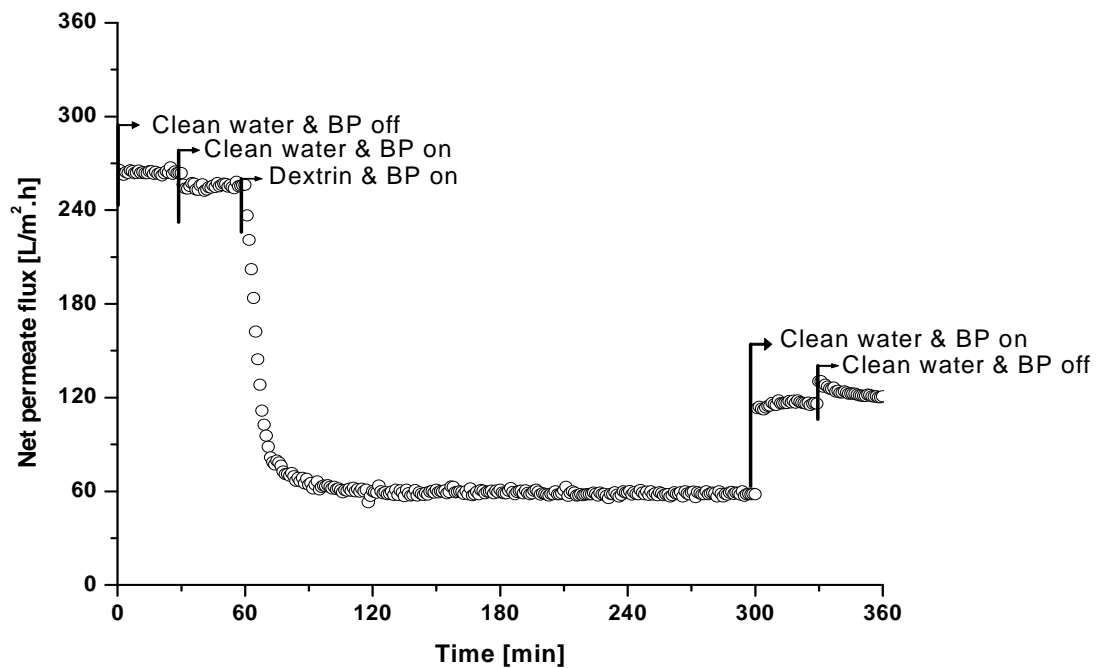


Figure B-8: Net permeate flux in run #8: 5 s pulse interval, 0.2 s pulse duration, 100 kPa pulse pressure, 1500 L/h. (Feed pressure 100 kPa, temperature 27 ± 0.5 °C, dextrin feed solution 500 mg/L).

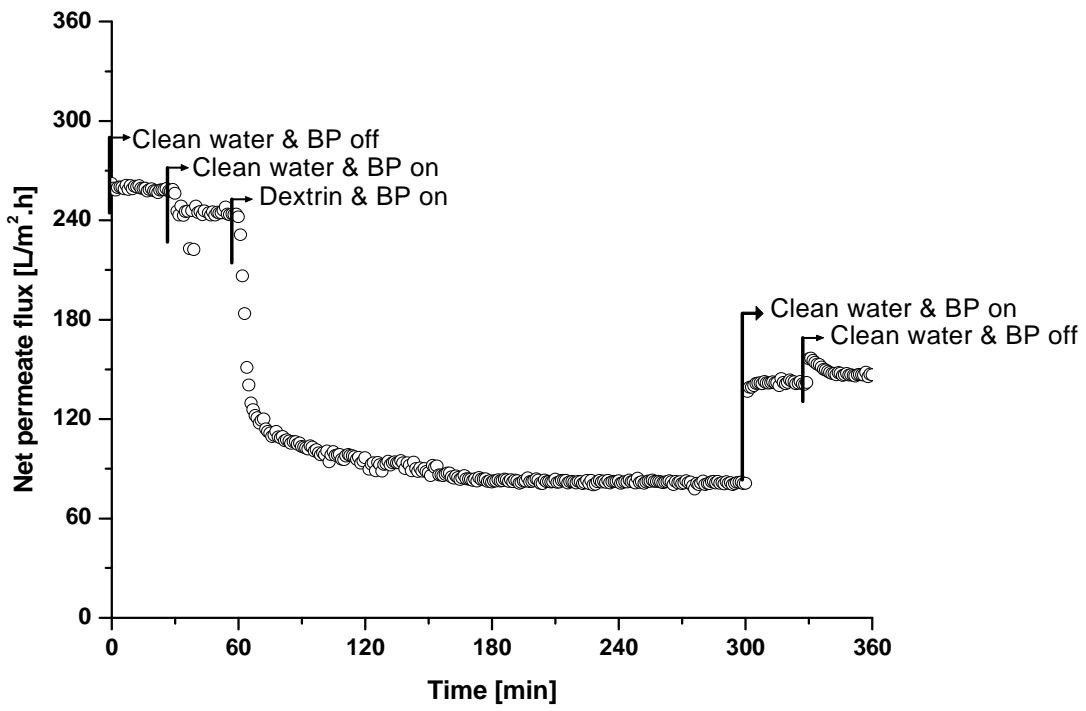


Figure B-9: Net permeate flux in run #9: 5 s pulse interval, 0.3 s pulse duration, 125 kPa pulse pressure, 500 L/h. (Feed pressure 100 kPa, temperature 27±0.5 °C, dextrin feed solution 500 mg/L).

Appendix C
Materials and equipment

C.1 Material properties

Table C-1 : Physical and chemical properties of kaolin

Physical properties		Typical value
Particle size distribution	< 10 micron	83%
	< 2 micron	60%
Mean particle size (D50)		1.5 micron
Residue (< 45 micron)		2.1%
Reflectance (Elrepho R457)		79% (off-white in colour)
Fired reflectance (Elrepho R457)		81%
Green shrinkage (%)		2.41
Fired shrinkage (%)		12.2
pH value		7-8
Specific gravity of Kaolin mineral		2.60
Mohs hardness		2.0-2.5
Moisture content		3%
Oil absorption (linseed oil)		45 ml/100g
Chemical analysis		Typical value
SiO ₂		46.12%
Al ₂ O ₃		37.86%
Fe ₂ O ₃		0.28%
TiO ₂		0.55%
CaO		0.16%
MgO		0.18%
Na ₂ O + K ₂ O		0.58%

Table C-2: Properties of dextrin

Type	Type I
Form	Powder
Total impurities	≤ 5% Reducing sugar
Colour	Off-white to yellow
Water insoluble	Not more than 10%
Alcohol soluble	Not more than 0.5%

C.2 Equipment

Equipment	Specifications
Feed pump (A1)	<ul style="list-style-type: none"> • Ecoject pump, Model R90 • Delivery: up to 24 m³/h. • Head: up to 38 m. • Power up to 0.6 kW
Backpulsing pump (A4)	<ul style="list-style-type: none"> • Stainless steel threaded centrifugal pump • Delivery: up to 31 m³/h. • Head: up to 62 m. • Maximum operating pressure: 8 bar. • Temperature of pumped liquid: -10 °C to 85 °C. • Single-phase 220-240 V. • Power up to 3 kW.
Solenoid valve (VS1,VS2)	<ul style="list-style-type: none"> • Type No: MK10 • Orifice: 10 mm • Port connection: G1/4 -G3/4. • Pressure: 0-40 bar.
Pressure transmitter (PT1,PT2,PT3)	<ul style="list-style-type: none"> • Model S-10 • Pressure: 0-10 Bar. • Signal output: 4-20 mA. • Power supply: 10-30 V DC (3-wires)
Flow indicator transmitter (FIT1, FIT2)	<ul style="list-style-type: none"> • Type 8045 • Stainless steel sensor • Measuring range: 0.2 to 10 m/s • Pressure range: 2-10 bar • Temperature : -10 to 110 °C • Power supply: 18-36 V DC filtered and regulated (3-wires)
Feed tank R1, R2	Plastic tank, 200 L
Permeate tank (R3)	Plastic tank, 50 L