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Design and Optimization of Ultrafiltration Membrane Setup for Wastewater Treatment and Reuse

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Design and Optimization of Ultrafiltration Membrane Setup for Wastewater Treatment and Reuse

Design och optimering av ultrafiltreringsmembraninställning för avloppsrening och återanvändning

Keywords: parameters, membrane, predict, optimize, quality, reduction.

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Abstract

With the advances in the membrane technology, there is an ongoing quest to determine the best optimal configuration for an adopted treatment as well as it's polishing to achieve cumulative sustainability for the treatment process. Henceforth, this thesis report is an evaluation to devise a membrane filtration process for investigating the possibility of treating pre-sedimented municipal wastewater with ceramic ultrafiltration, optimizing the membrane as a pre-treatment for reverse osmosis as an overall strategy for recovering wastewater.

Methods and various technological trends pertaining to membrane filtration of municipal wastewater were researched and documented, Interestingly the five most influential factors governing the membrane performance are identified: 1) Back pulsing Frequency 2) VRF 3) Run Time 4) Cross-Flow Rate 5) Trans Membrane Pressure (TMP). To get a thorough and holistic overview of parametric influence design of experiment (DOE) is devised to find the influence of above-given factors on outcoming responses as COD Reduction (%), Membrane Flux and Turbidity reduction (%).

16+3 DOE factorial tests are executed at Hammarby Sjöstadsverk, Joint Research Facility of IVL Swedish Environmental Research Institute & KTH Royal Institute of Technology on pilot plant WASLA incorporating an ATECH GmBh 20kDa, Type 7/6 Ultrafiltration membrane module where Factorial experiments resulted in a maximum value of flux of 274 LMH, 88.75% reduction of COD and 99.94% reduction of Turbidity.

Moreover, response values obtained from the Results of factorial experiments are fed in MODDE, generating a model using PLS Regression, The model summary presented predictivity and reproducibility trends w.r.t responses used. Furthermore, COD resulted in the worst fit followed by Turbidity, and the best fit was observed for Membrane Flux where model fit represented the ability to predict the respective parameter.

Optimization tool is utilised to simulate a case scenario where the Membrane flux response is maximized to a high value of 300 LMH and correspondingly 211.885 LMH value is recorded, Furthermore factor influence is identified to be TMP> VRF> Cross Flow >BP Frequency >Runtime.

Overall COD reductions are found out to be heavily influenced by the varying incoming feed therefore it is hard to analyze their interactions and predict their subsequent reduction behavior. Back pulsing overall was found out to be another non-influential factor colluding with results throughout the experimental duration with very little or no effect on the permeate water quality.

Sammanfattning

Med framstegen inom membrantekniken finns det en kontinuerlig strävan att fastställa bästa optimala konfigurationen för en antagen behandling samt att den är polerad för att uppnå kumulativ hållbarhet för behandlingsprocessen. Framgent är denna avhandlingsrapport en utvärdering för att utforma en membranfiltreringsprocess för att undersöka möjligheten att behandla försedimenterat kommunalt avloppsvatten med keramisk ultrafiltrering, optimera membranet som en förbehandling för omvänd osmos som en övergripande strategi för att återvinna avloppsvatten.

Metoder och olika teknologiska trender avseende membranfiltrering av kommunalt avloppsvatten undersöktes och dokumenterades. Intressant identifieras fem mest inflytelserika faktorer som styr membranprestanda: 1) Ryggpulserande frekvens 2) VRF 3) Körtid 4) Korsflödeshastighet 5) Trans Membrantryck (TMP). För att få en grundlig och holistisk överblick över parametrisk inflytande experimentet är utformat för att hitta påverkan av ovan givna faktorer på utgående svar som COD-Reduktion (%), Membranflöde och Turbiditetsreduktion (%).

16 + 3 DOE-faktortest utfördes vid Hammarby Sjöstadsverk, Joint Research Facility för IVL Swedish Environmental Research Institute & KTH Royal Institute of Technology på pilotanläggningen WASLA med en ATECH GmBh 20kDa, typ 7/6 Ultrafiltreringsmembranmodul där faktoriella experiment resulterade i en maximalt flödesvärde på 274 LMH, 88,75% reduktion av COD och 99,94% reduktion av turbiditet.

Dessutom matades svarvärden erhållna från resultat av faktoriella experiment i MODDE, vilket genererar en modell med PLS-regression, modellöversikt presenterad prediktivitet och reproducerbarhetstrender med användning av svar. Vidare, COD resulterade i den sämsta passningen följt av turbiditet och den bästa passningen observerades för Membraneflöde där modellpassning representerade förmågan att förutsäga respektive parameter.

Optimeringsverktyget användes för att simulera ett fallsscenario där membranflödesresponsen maximeras till ett högt värde av 300 LMH och motsvarande 211.885 LMH-värde registreras. Vidare identifieras faktorinflytande TMP> VRF> Crossflöde> BP Frekvens> Körtid.

Totalt har COD-reduktioner visat sig påverkas starkt av det varierande inkommande fodret, varför det är svårt att analysera deras interaktioner och förutsäga deras efterföljande reduktionsbeteende. Ryggpulsering visade sig totalt sett vara en annan icke-inflytelserik faktor som samverkar med resultat under hela försöksperioden med mycket liten eller ingen effekt på permeatvattenkvaliteten..

Acknowledgments

This Master of Science Thesis has been carried out as a part of the Master Programme – Environmental Engineering & Sustainable Infrastructure at the KTH Royal Institute of Technology, Stockholm, Sweden.

The master thesis was initiated and performed in close co-cooperation with IVL Swedish Environmental Research Institute whereas the experimentation part was performed at IVL & KTH's joint Research facility, Hammarby Sjöstadsverk, furthermore, the results and conclusion from the master's thesis would be further used to develop and help the Extended Project of Gotland's Water & Wastewater reuse.

I would like to express my gratitude to both of my supervisors, Dr. Elzbieta Plaza, Professor at KTH, and Mr. Fredrik Hedman, Project leader at IVL Swedish Environmental Research Institute. I appreciate their invaluable assistance and precious advice and guidance throughout the thesis work, Furthermore, I am grateful to them for allowing me to participate in an interesting research project.

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And finally, a big appreciation for my parents for giving me their wholehearted love, support, and encouragement during my studies in Sweden, I won't be here without you.

Abbreviations

AEB	Air Enhance Backwash
BOD	Biological Oxygen Demand
BPF	Back pulsing Frequency
CEB	Chemical Enhanced Backwash
CIP	Cleaning-In-Place
COD	Chemical Oxygen Demand
DOE	Design of Experiment
FO	Forward Osmosis
FNU	Formazin Nephelometric Unit
LSI	Langelier Saturation Index
LMH	Liter/m^2/hr.
MBR	Membrane Bioreactor
MF	Microfiltration
MWCO	Molecular Weight Cut Off
NF	Nanofiltration
RO	Reverse Osmosis
SDI	Silt Density Index
TMP	Trans Membrane Pressure
UF	Ultrafiltration
VRF	Volume Reduction Factor
WASLA	Pilot Plant
WWTP	Wastewater Treatment Plant
XF	Cross Flow
X-FLOW	Cross Flow

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1) Introduction

1.1) Background:

Sweden has been facing quite a bit of Ground Water level scarcity, a recent SGU " Geological Survey of Sweden" evaluation shown below in Fig.1) paints a horrific picture of the GWL across Sweden during the summer month of June, The Red areas show parts where the GWL is very much under the normal limit (Roden, 2017).

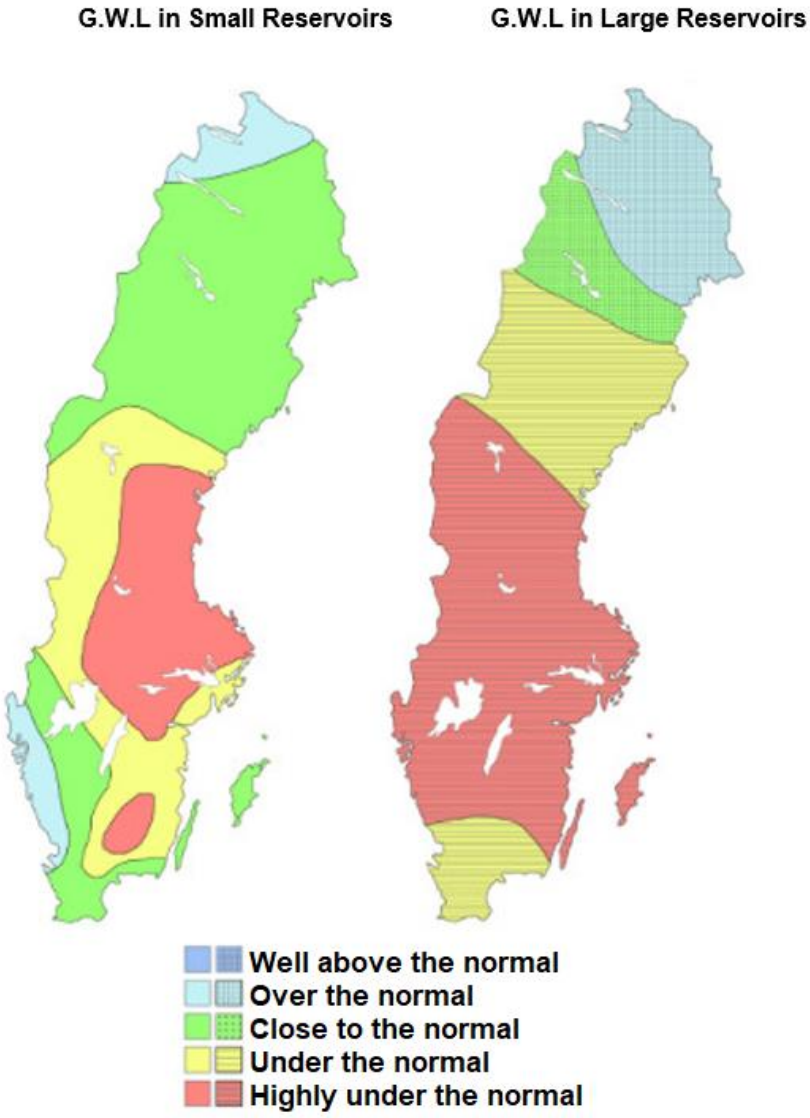

Figure.1) GWL of Sweden in June, modified from (Roden, 2017).

The same can be said for the island and remote parts of Sweden, according to a news report about two years ago mentions that GWL is so low in the Gotland and Öland that they might have been facing water scarcity during the summer months (Radio, 2016).

The main reason for the above could be increased migration and influx of tourists during the summer months. So, to combat these harsh conditions and provide water to everyone desalination plants are set up and another alternative being is making reuse of wastewater.

The thesis project is a part of an extended project which plans to provide a solution to the Gotland's Water shortage during the summer months though Municipal wastewater treatment using membrane technology.

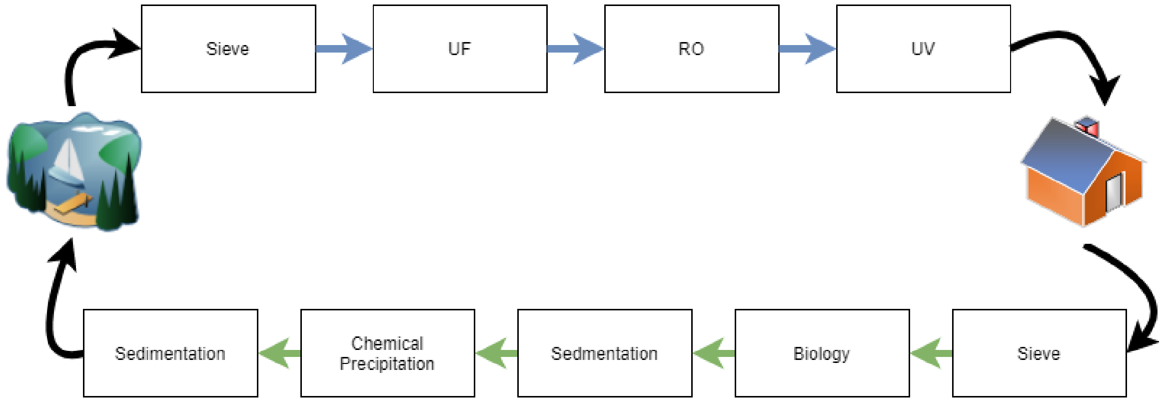

Figure.2) Schematic view of the typical water balance.

Here Figure.2) shows the typical process diagram of Water balance where water is extracted from a source and used for a purpose and then usually is returned to a recipient as wastewater. At Gotland, municipal water is produced through the desalination of seawater and discharged back to the sea after use from wastewater treatment plants.

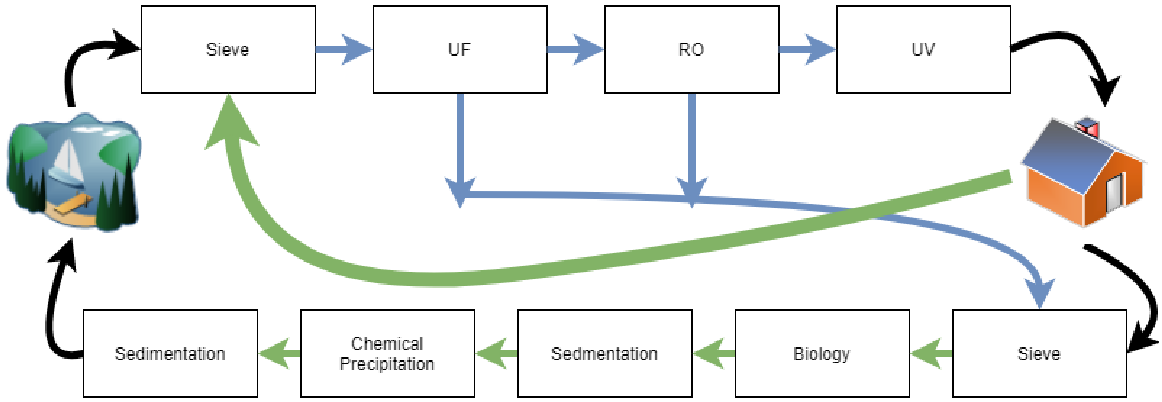

Figure.3) Redrawn water balance allowing the recovery of wastewater.

However, taking in the possibility to implement the idea of treatment of the wastewater before discharge into the Baltic sea, the salinity could be reduced about 15 times allowing a less energy-consuming desalination process. It involves the possibility of moving the treatment even before the traditional wastewater treatment plant and the energy-consuming and sensitive biology step could be excluded further and gains could be expected Figure.3) While making use of smaller and more concentrated waste streams might enable the use of more efficient wastewater treatment technology.

1.2) Aim & Objectives

The main aim of the thesis is to investigate the possibility of treating pre-sedimented municipal wastewater with ceramic ultrafiltration membranes as a pre-treatment for reverse osmosis as an overall strategy for recovering wastewater.

The objectives were specified after a thorough literature survey of the scientific knowledge base and practically used techniques for recovering municipal wastewater with membrane technology was: Investigating the influence of fouling is mainly evaluated through flux measurement, where a higher average flux would indicate less fouling. But also performing long term testing over 40 hours without cleaning in place would account for flux decrease.

Hence the objectives for the thesis work are as follows:

1. Evaluating the impact of back-pulsing for the series of factorial experiments on the pilot plant.
2. Assessment of expected permeate water quality based on evaluated responses.
3. Computer aided evaluation & prediction using the results concerning the given parameter's influence on permeate flux and efficiency for retaining particles and organic matter.
4. Generation of an optimal design configuration and design space for maximizing the membrane flux from the set of factorial experiments and recognize the most efficient setup of process parameters.

1.3) Limitations

The above work involved a great deal of physical and analytical work as laboratory experiments and their respective computer analyses on MODDE 12.04. It was ensured to maintain uniformity without any biases to obtain an accurate assessment of membrane performance barring some limitations; predominantly the unavoidable time limit, because of delays caused in membrane delivery from manufacturer and repair involved in the pilot plant. As a result, the repetition of experiments is not possible to check for experimental reliability and applicability. As, expanding and inclusion of several other Input and Output Parameters would be more reliable and valid, consequently improve the understanding of parameter interaction. Furthermore, the energy consumption and economic viability of the method involved were not discussed in the project as they were outside the scope of the project.

2) Literature Review

2.1) Municipal Wastewater

Wastewater is any form of water affected by human usage, Typically the term Municipal wastewater is used in conjunction with used water which is a result of all the manmade domestic, industrial, commercial and local activities in the respective municipality, village, town or city.

Usually, it is made up of water but most of its constituents depend on the source of origin of the wastewater, however, in general, it's made up of various physical, chemical and biological impurities again varying according to the type of origin source, type of use phase, local environmental characteristics etcetera.

Its Transportation methods include either through a sanitary sewage system or a Combined Sewage system that transfers both sewage water, grey water along with Stormwater runoff. Hence if the method of transportation is the combined one, encounter many Water volumes as compared to the Sanitary Sewage system alone.

Hence overall Wastewater is water affected by human usage composed of various organic and inorganic matter.

2.2) History (Swedish Perspective):

During the early 20th century there was a practice of little or no treatment applied to Urban Wastewater as it was directly disposed of into the various water bodies, but soon the lakes, rivers, and coastal areas began to get affected directly or indirectly as a result (Naturvårdsverket, 2019).

Soon Water pollution was taken into a consideration as a major source of municipal concern and soon Water Treatment plants were implemented to get a hold of increasing pollution values, However, a major boom in WWTP development and purification processes came around 1960s as the Eutrophication was leading to becoming an important concern for Swedes as it wasn't only affecting the environment but leading to disruption of natural Archipelago system and nature of Baltic Sea.

Hence it led to the establishment of (Swedish EPA) Swedish Environmental Protection Agency Naturvårdsverket in the year 1967 and it led the basis of the formation of the Environmental Protection Act (Naturvårdsverket, 2019).

2.3) Typical Municipal Wastewater Treatment Technologies:

Wastewater Treatment technologies vary on varying types of treatment technologies and what is the purpose of using them to achieve the required quality. Typically, water quality can be assessed on three main factors i.e. Physical, Chemical, and Microbial/Biological, the similar criteria are applicable for the Wastewater Quality assessment as well, however, the regulations of effluent quality among both vary starkly, some of the processes used for wastewater reclamation are displayed through Figure.4) below. Broadly Each category can be subdivided into the concentration of the following evaluating parameters:

Table.1) Water Quality Evaluation Parameters (Murcott, 2007)

Physical	Chemical	Microbial
Appearance/Turbidity	Organic / Inorganic	Bacteria
Odor/Smell	Naturally Occurring /Anthropogenic	Viruses
Taste	Radioactive	Protozoans

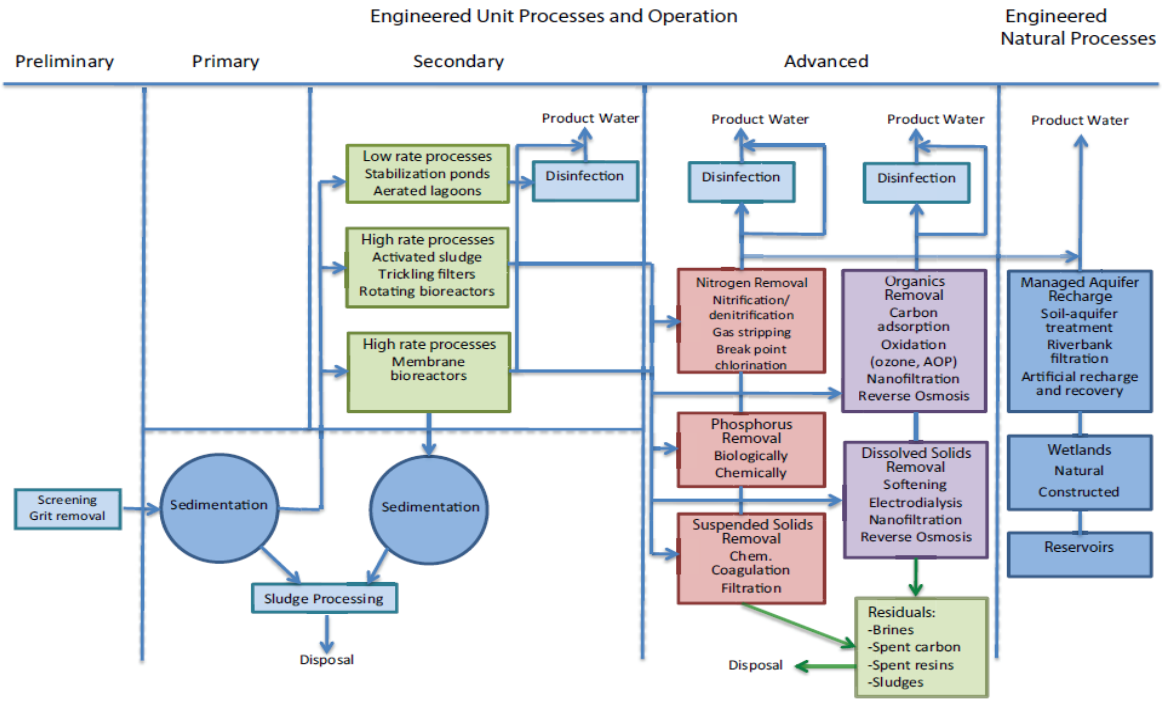

Figure.4) Common Treatment processes used for Water Reclamation & Reuse; Not all processes are employed as the deciding factor responsible for the product quality achieved (Water reuse, 2012).

Following up is a detailed historical development concerning various Wastewater technological improvements and their respective locations.

Table.2) Significant Events Timeline for Water & Wastewater Recovery & Reuse, (Asano et al.,2007).

Period	Location	Event
1962	La Soukra, Tunisia	Irrigation with reclaimed water for citrus plants and groundwater recharge to reduce saltwater intrusion into coastal groundwater.
1965	Israel	Use of secondary effluent for crop irrigation.
1968	Windhoek, Namibia	Research on direct potable reuse and subsequent irrigation.
1969	Wagga Wagga, Australia	Landscape irrigation of sporting fields, lawns, and cemeteries.
1977	Tel Aviv, Israel	Dan Region Project – Groundwater recharge via basins.
1984	Tokyo, Japan	Toilet flushing water for commercial buildings in the Shinjuku district using reclaimed water from the Ochiai WWTP.
1988	Brighton, UK	Inauguration of the specialist group on wastewater reclamation, recycling, and reuse at the 14th Biennial Conference of International Water Association, HQ-London, UK.
1989	Girona, Spain	Golf course irrigation using the reclaimed water from the Consorci de la Costa Brava wastewater treatment facility.
1999	Adelaide, South Australia	Virginia Pipeline Project, the largest water reclamation project in Australia - irrigating vegetable crops using reclaimed water from the Bolivar WWTP (120,000m^3/d).
2002	Singapore	NEWater- reclaimed water that has undergone significant purification using MF, RO, and UV disinfection.

Shown above through Table.2) gives us a timeline of significant research led until 2002. Also, through Figure.5) shown below we can come across the idea and usage of MF/UF in conjunction with RO and subsequent UV disinfection of Wastewater to use as a raw water source for Singapore's Water Supply.

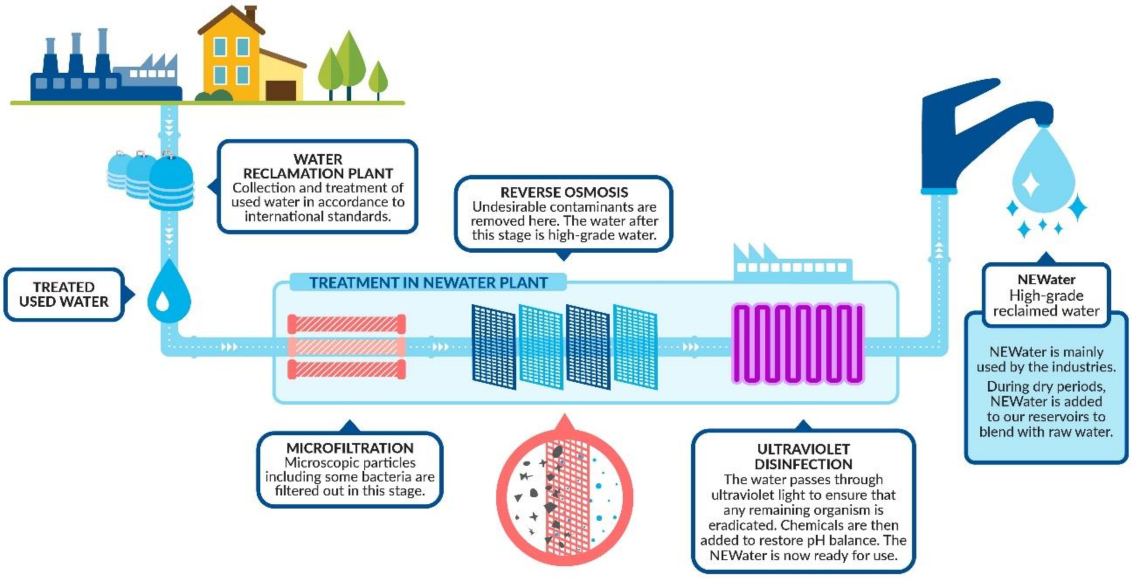

Figure.5) NEWater Process Scheme (NEWater, 2002).

The implementable idea has been researched and worked on by various researchers and academicians across the world however since NEWater was the inception of it hence, as a result, it was to be investigated a little more.

The main use of NEWater is industrial or air-cooling purposes as the water is being ultra clean thus accounting for its majority as Non-Potable usage since the water quality requirements of such fabrication plants are highly stringent than potable drinking water (NEWater, 2002).

However, it is sometimes practiced as a source for potable drinking water during dry periods when the NEWater is added in the water reservoirs to blend with the raw water and treated at the waterworks to ensure it stands up to the levels of drinking water (NEWater, 2002).

It's mentioned that the entire process scheme is working to produce at least 10000m^3/d by the year 2000, which is scaled up as of 2019.

So it's essential to understand the power of this technology and process scheme with the certain limitations such as initial investment and periodic maintenance and replacement of the membranes, despite all of these disadvantages it could be employable in places where Acute Water shortage is being faced similarly as discussed in above in the Introduction of the Gotland's Water Problem during summer months.

In Sweden, the wastewater is typically treated through the following treatment stages or a mix-up of the following:

- Membrane Treatment
- Biological Treatment
- Chemical Treatment
- Advanced Biological -Chemical (3 Stage Treatment)

The latest treatment technology utilized in Sweden is Membrane Bioreactor with its most ambitious plan to build the biggest MBR treatment plant at Henriksdal WWTP (The MBR Site, 2015).

MBR (Membrane Bioreactor):

This technology comprises of traditional physical and biological process interaction and the wastewater treatment process can be categorized by a suspended growth of biomass along with a Microfiltration /Ultrafiltration membrane system (Judd, 2011).

The Biological Unit is used for the biodegradation of waste compounds while the membrane module is responsible for the physical separation of treated water from Mixed Liquor, however, what makes this process advantageous over traditional ASP is that the use of membranes which have a pore diameter of 0.01 and 0.1 um which is entirely capable of removal of particulates and bacteria out of the Biological Processes thus removing the precursor process or using a Clarifier and Settling /Sedimentation.

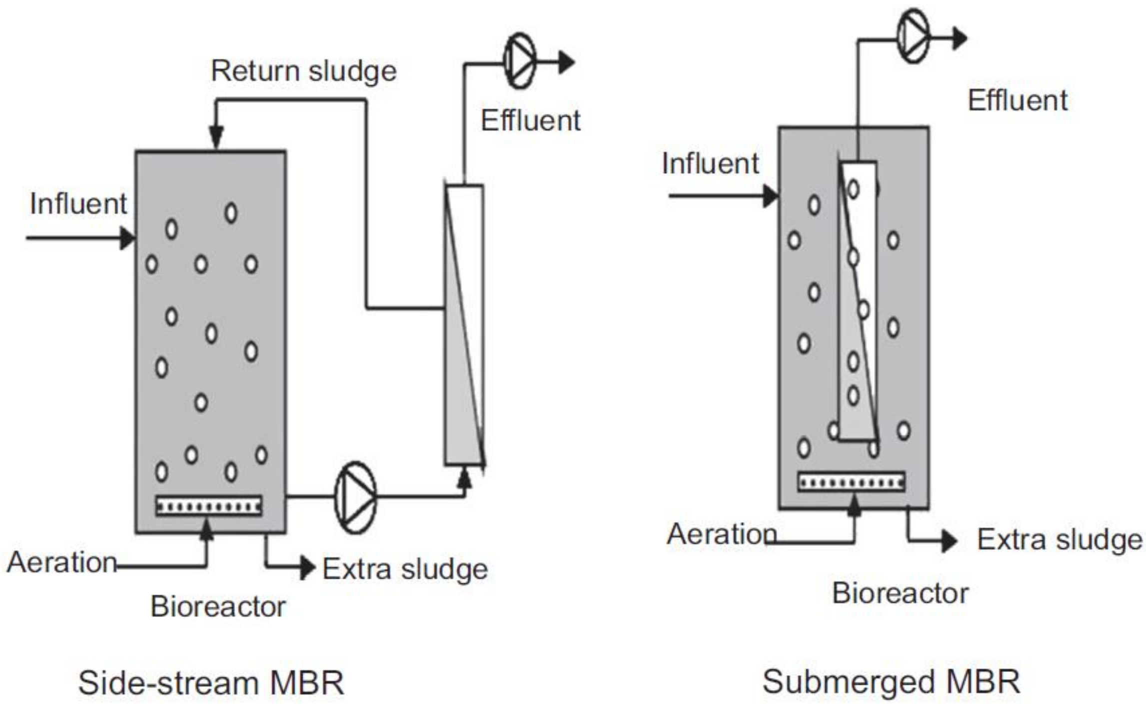

Figure.6) Configurations of side stream & immersed MBR's, modified from Jiang (2007).

Submerged MBR’s (sMBR’s) involves a separate membrane module along with Biological chamber and option of return sludge rerouted to the bioreactor however some modern configurations even involve the introduction of airflow into the membrane which intensifies the turbulence on the feed side of membrane module and therefore accounts for the reduction in operation costs and provides a better fouling control (Jiang, 2007).

While Submerged MBR's membrane module is directly submerged into the Bioreactor it selves and suction or vacuum pump are required to create a Transmembrane Pressure for Permeate Production, also this leads to no further recirculation being required since cross-flow is already in place due to aeration of Bioreactor This concept was developed by Yamamoto, Hiasa, Mahmood, and Matsuo (1989) to reduce energy consumptions w.r.t sMBR's. Given below Table.3) compares the various parameters while Figure.6) shown above illustrates both respective configurations of MBR’s.

Table.3) Comparison of sMBR & iMBR, Jiang (2007, p. 11).

Parameters	Side- Stream	Submerged
Complexity	Complicated	Simple
Flexibility	Flexible	Less Flexible
Robustness	Robust	Less Robust
Flux	High(40-100L/m^2 h)	Low(10-30L/m^2 h)
Fouling reducing methods	• Cross-flow • Airlift • Backwashing • Chemical Cleaning	• Air bubble agitation • Backwashing(not always possible) • Chemical Cleaning
Membrane packing density	Low	High
Energy consumption with filtration	High (2-10kW h/m^3)	Low (0.2-0.4kW h/m^3)

2.4) Membrane Technology:

A typical membrane can be defined as a thin layer of semi-permeable or selectively permeable material that is used for substance separation, when a driving force is applied across the membrane, as a result, the inlet stream is divided into two parts Permeate & Retentate. Here Retentate is the unfiltered solvent that couldn't cross the membrane while permeate is the filtered solvent. In general membrane filtration process can be applied across multiple domains, however, they are used for removal of bacteria, microorganisms, particulates, and natural organic material, which impart color, tastes, and odors to water and react with disinfectants to form disinfection byproducts.

Thus, overall Membrane treatment could be defined as a culmination of all Physical-Chemical and Biological Treatment processes in the Water & Wastewater Treatment Process.

The Advantages of employing membrane filtration technologies compared to conventional filtration are smaller space requirements, reduced labor requirement, better process automation and more effective pathogen removal(especially protozoan and bacteria) moreover the effluent generated is of uniform quality concerning suspended matter and pathogens thus the effluent turbidities are well below 1 NTU (Asano et al.,2007).

Nevertheless, no process is perfect and membrane filtration technology does have its fair share of disadvantages such as potential higher infrastructure costs, limited lifespan of membrane modules, and potential for irreversible membrane fouling resulting in a loss of optimal membrane productivity (Asano et al.,2007).

2.4.1) Classification of Membrane Processes:

Microfiltration (M.F):

Microfiltration is a type of physical filtration process where a contaminated fluid is passed through a special pore-sized membrane of the size of about 0.03 to 10 microns (1 micron=0.0001 millimeter) to separate microorganisms and suspended particles from the feed. (Mrwa.com, 2005).

Ultrafiltration (U.F):

It is a variety of membrane filtration where forces resulting from pressure or concentration gradients lead to a physical separation through a semipermeable membrane. Suspended solids and solutes of high molecular weight are retained in the retentate, while solvent and low molecular weight solutes pass through the membrane in the permeate (filtrate). It is one of the most common separation processes utilized in industry and research for purifying and concentrating macromolecular (10^3 - 10^6 Da) solutions, especially protein solutions (Wang, Lei, and Olennikov, 2016).

Nanofiltration (N.F):

A relatively recent membrane filtration process targeting low total dissolved solids (pre-treated) water such as surface water and fresh groundwater, to soften (polyvalent cation removal) and removal of disinfection by-product precursors such as natural organic matter and synthetic organic matter (Basile, Cassano and Rastogi, 2015).

Reverse Osmosis (R.O):

This process involves the use of hydraulic pressure to overcome the osmotic pressure against the membrane to purify (pre-treated) liquids without incorporating the phase change (Basile, Cassano, and Rastogi, 2015). It has since the inception of the process become an integral part of the purification process industrywide to produce high-quality water. Table.4) shown below displays a comparison between the R.O and other membrane processes.

Table.4) Comparison of Membrane Technologies, Adapted from (Singh, 2006)

Process	Pore Size	Driving Force	Transport Mechanism
Microfiltration	0.05-10 um	Pressure, 1-2 bar	Sieving
Ultrafiltration	0.001-0.05 um	Pressure, 2-5 bar	Sieving
Nanofiltration	<2.0 nm	Pressure, 5-15 bar	Capillary Flow
Reverse Osmosis	<1.0 nm	Pressure, 15-100 bar	Capillary Flow

Molecular Weight Cut-Off (MWCO):

It is one of the most important tools for describing /characterizing UF membranes, defined as the molecular weight at which typically 90 percent of the macromolecular solute is rejected by the membrane. However MWCO classification also has some limitations such an as UF membranes pore size distribution is not ‘sharp' also the UF membranes are usually hydrophobic therefore they tend to absorb proteins on the surface and inside the membrane pores hence resulting in higher retention which can be a result of a) reduced pore size, b) secondary film formation on the membrane surface (Singh, 2006).

Forward Osmosis: Similar to the Reverse Osmosis in the scenario that it is indeed an osmotic process except here the same osmotic pressure is used to drive and separate water from the rest of the dissolved solutes across a semi-permeable membrane instead of an external hydraulic pressure. Thus, this process makes use of the natural osmotic pressure differential across the membrane by employing various buffer solutions creating ideal concertation gradients to facilitate the separation. (Basile, Cassano, and Rastogi, 2015).

2.4.2) Membrane Module Arrangements:

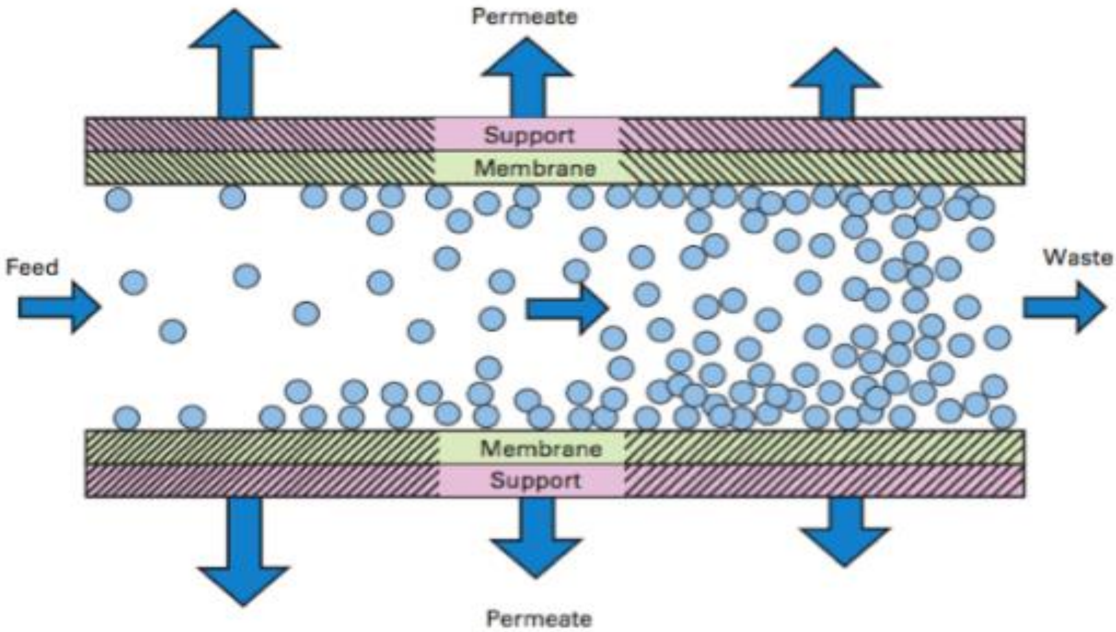

Figure.7) Tubular Membrane Filtration Technology (Porexfiltration.com, 2019)

Tubular Module: This design makes use of membranes typically cast on the inside of either plastic or porous paper components with diameters ranging from 5 – 25 mm and lengths from 0.6 - 6.4 m (Porexfiltration.com, 2019). Illustrated above though figure.7) Multiple tubes are housed in a PVC or steel shell. The feed of the module is passed through the tubes, accommodating the radial transfer of permeate to the shell side. Shown above through Fig.3) the working procedure of tubular membrane which involves tangential crossflow and area mainly used to process the difficult feed streams such as high dissolved solids, suspended solids, etc., (Synderfiltration.com, 2019). This design allows for easy cleaning however the main drawbacks result in low permeability, high volume hold-up within the membrane, and low packing density.

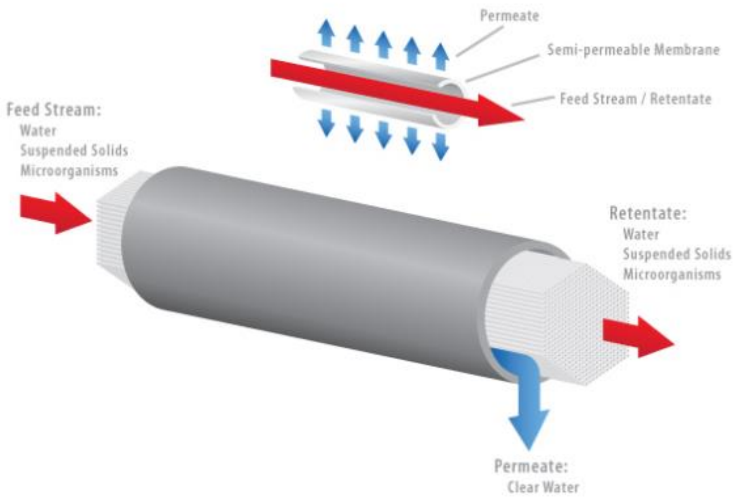

Figure.8) Hollow Fiber Membrane Module (Synderfiltration.com, 2019)

Hollow-Fiber Module: This design is pretty much like the tubular membrane arrangement with a sheet and tube arrangement; however, the single module can contain up to 50 to thousands of hollow fibers and therefore make them self-sufficient and supporting. The diameter of each and fiber is of the order 0.2 - 3mm with feed going across the tubes while permeate collection happens radially outside as shown in the figure.8) above, however, since most of the makeup of membranes is due to single fiber its main drawbacks include fiber breakage and irreversible fouling amongst the fibers (Synderfiltration.com, 2019).

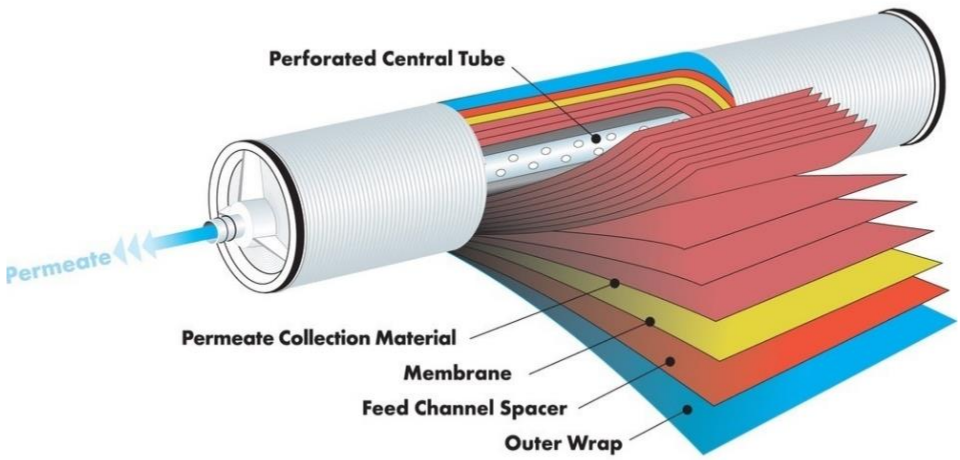

Figure.9) Spiral Wound Membrane Module (MICRODYN-NADIR, 2019)

Spiral Wound Module: It's Makeup consist of multiple membranes, feeds spacers, permeate spacers, and a permeate tube where feed travels through the flow channels tangentially across the length of the element as exhibited through Figure.9) above. Filtrate which is smaller than the molecular weight cut-off will then pass across the membrane surface into the permeate spacer, where it is carried down the permeate spacer towards the permeate tube. The remainder of the feed then becomes concentrated at the end of the element body. Hence it offers high fouling as to tubular module and can't offer mechanical cleaning unlike tubular module (Synderfiltration.com, 2019).

Flat Sheet Module: This type of membrane module consists of multiple flat sheets housed together in plate and frame or cassette devices. There exists some separation between individual membranes for the flow of feed which of order 0.5 - 2.0mm. As illustrated in Figure.10) below the membranes are supported with the help of plats and porous spacers on the permeate side and they are clubbed together in a sandwich to form a module or a cartridge. The membrane surface area per unit volume may vary from 300 to 500 m2 /m3 depending upon the separation between the membrane sheets. While the biggest disadvantage of this arrangement module is that it doesn't allow backflushing as the membrane is having only a single support side (Doran, 2013).

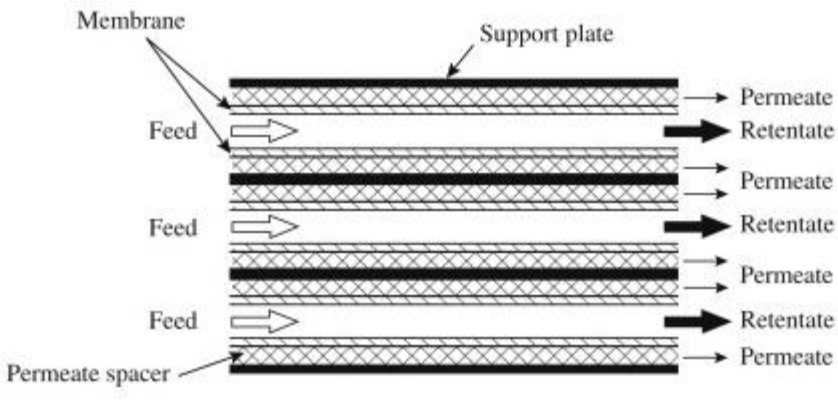

Figure.10) Flat Sheet Membrane filtration in the plate and filter module (Doran, 2013).

2.4.3) Mode of Operation

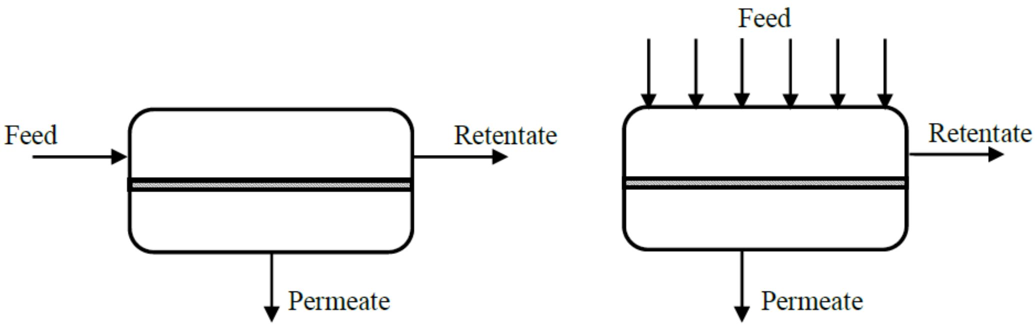

Figure.11) General membrane Mode of Operation with Crossflow(left) and dead-end flow (right) sides respectively (Voittonen, 2018).

Dead End: Unlike established cross-flow membrane processes – the feed is inserted or applied perpendicularly to the filter element and it is ensured that the entirety of feed passes through the filter element while retentate solids forming a cake layer on the element surface as illustrated above in Figure.11).

However, in the case of charged dead-end microfilter cartridges, the separation is based on two mechanisms: pore blockage/surface retention due to size, and surface/pore adsorption due to electrostatic interaction (Singh, 2006). While void of the charge, the pore size rating is nominal, offering (80–90% retention) whereas, in the presence of a charge, the pore size rating is absolute, thus offering better filtration and reduced cake formation.

Crossflow: It is a type of filtration process which involves a tangential flow of feed water across a membrane surface as illustrated above in Figure.11), Also it is typically utilized in wastewater treatment using filtration. The turbulence created across the membrane surface provides optimal flux performance and prolongs filter functionally.

With the aid of the Tubular pinch effect, it is said to help and is scientifically proven to be better than the dead-end flow filtration method. Cross filtration working principle is introducing feed water under pressure across the membrane surface, thus avoiding direct feedwater flow onto the filter. During filtration, any material smaller than the crossflow membrane pore passes through the membrane, while larger suspended particulates remain in the retentate stream (Porexfiltration.com, 2019).

2.5) Membrane Materials

Material selection is an important factor in an effect to optimize overall control and obtain the best possible results as membrane performance is inherently dependent on the membrane material, a) Feed composition b) Separation Goals c) Operating Parameters (Synderfiltration.com, 2019). Since it's of utmost importance that membrane material should have enough throughput of the permeate while also bearing a high selectivity (Voittonen, 2018). The physical interfacial properties such as interfacial tension and adsorption are considered the most important physical property as it involves the interaction of solid membrane material with liquid and gas phase, as the degree of swelling in a polymeric membrane in an organic solvent or change in crystallinity at higher temperatures (Singh, 2006). As a rule of thumb, the membrane materials can be classified into three main categories Organic, Inorganic & Biological, however broadly and as per industrial standards and mentioned by (Ladewig, Nadhim and Al-Shaeli, 2017) biological membranes are not apt to be used for industrial treatment procedures for particular in applications which require high temperatures as they tend to be unstable because of their poor thermomechanical stability. Henceforth, only the following two main categories of membrane materials would be discussed further namely Organic/Polymeric and Inorganic /Ceramic Membranes.

2.5.1) Polymer vs Ceramic

Polymeric membranes provide a range of Properties for separation and modification that can even improve membrane selectivity. Usually, they are desirable as they seem to be chemically and thermally stable, also during manufacturing or particular combination that is preferred where materials with higher melting point and high crystallinity are preferable, however (Singh, 2006) claims that the main reason for the drop in permeate flux as the result of irreversible adsorption of particles such as protein to the membrane surface (fouling) (Bergstedtaiche, 2011) and have a low shell life about 1 year in case of hydrophilic polymeric membranes and about 3-4 years of hydrophobic membranes on the offset most of the polymeric membranes typically offer variability in operation and have low investments costs (Singh, 2006). Ceramic membranes are micropores and serve as a viable alternative for polymeric membranes due to their superior thermal, chemical stability, and robust design. The ceramic membranes employ micropore sieves where separation takes place based on the size and speed of particles through a convoluted and twisted pore path. Overall, the Ceramic membrane has an upper hand on the polymeric membrane due to various reasons discussed above in the text and presented through the Table.5) below.

Table.5) Comparison of Ceramic vs Polymeric Membranes (Bergstedtaiche, 2011)

Ceramic membranes	Polymeric Membranes
Superior mechanical strength	Subject to mechanical damage
One "piece" per element	Bundles of hundreds of hollow fibres
Good chemical resistance	The polymer can be attacked
High capital costs	Lower cost/GPM capacity
Little operational experience	Ubiquitous

2.5.2) Modification of Membrane Surface

Modification of the membrane surface to alter or improve the antifouling properties of the membrane.

An effective strategy is minimizing of the attractive interactions between the membrane surface and the components of the feed by chemical modificatioon of the membrane surface, as most of the pressure-driven filtration processes treating aqueous feed streams and foulants are generally hydrophobic, which can be worked upon and surface modification strategies could be employed to focus on changing the hydrophobicity along with surface roughness with interfacial polymerization,(Upadhyaya, Qian, and Ranil Wickramasinghe, 2018).

(Arza and Kucera, 2016) Mentions some of the most impacting parameters favoring adhesion and biofilm formation are:

a) Hydrophobicity: More hydrophobicity leads to more adhesion.

b) Surface roughness: Rougher surface accounting for higher adhesion.

c) Surface charge: the more neutral the charge, the more adhesion of bacteria (which are negatively charged)

It is also to be noted that most of the Techniques to modify membrane properties, such as roughness, charge, or hydrophilicity, include coating the membrane and using a membrane made of a different polymer to minimize bacterial adhesion. While chemical properties include the addition of Antimicrobial nanoparticles, such as silver, titanium dioxide, and carbon nanotubes, incorporated into membranes can help to limit adhesion.

2.6) Typical Problems in Membrane Technology for Wastewater Treatment:

Membrane scaling: Scaling is mostly due to deposition of colloidal materials in proximity with membrane surface which might result in fouling as it results in the introduction of a large number of foulants in the membrane system thus (Singh, 2006) mentions that reduction in TSS as a pretreatment could reduce in membrane scaling because of colloids.

Concentration Polarization: Reduction in the permeate flow because of deposition/buildup of retained molecules across the membrane which happens because the feed mixture is composed of several many things and the rate of permeation is different for each component, This leads to a formation of layer nearby membrane surface resulting in depletion of the solution's permeate while the concentration of non-permeate increases across membrane causing the development of a concertation gradient across the membrane and reduces the flux through the membrane (Separationprocesses.com, 2019).

Membrane Fouling: Reduction in the membrane's performance due to deposition of the unwanted solute particles in the membrane which can result in loss of permeate flow across the membrane. Fouling is a culmination of physical and chemical processes happening when the feed water interacts with the membrane surface. The flux across the membrane surface which has undergone fouling can be represented through the mathematical equation below (Field, 2010):

$$\boldsymbol{J = \frac{\Delta p - \Delta \pi}{\eta (R_m + R_f)}} \qquad \text{Eq.1)}$$

Where J, membrane flux, and other symbols can be referred to the Table.6) below

Table.6) Equation Terms and their Symbols

Symbol	Stands for
$\boldsymbol{J}$	Membrane Flux
$\boldsymbol{\Delta p}$	Transmembrane Pressure
$\boldsymbol{\Delta \pi}$	Osmotic Pressure
$\boldsymbol{\eta}$	Viscosity
$\boldsymbol{R_m}$	Resistance of Fouling
$\boldsymbol{R_f}$	Resistance of Membrane

Membrane Fouling process in an inorganic ceramic can be presented as a culmination of 4 main process stages as mentioned by (Wei, 2015):

a) in the beginning stage oil droplets with a diameter smaller than the membrane pore size directly enter permeate liquid after crossing the membrane (10 seconds-few minutes.)
b)When passing through the membrane pore oil drops are adsorbed on the inner surface as a result of the electrostatic force of attraction, meanwhile, oil drops of similar size with membrane pore size blocks the whole membrane pore causing a reduction of effective cross-section area and flux begins decreasing by a great amount.
c)The saturation of oil drops adsorption causes oil drops to adsorb or attach on the surface between pores.
d)Enrichment of oil drops between the surface of pores results in linking of the oil drops together to form an oil layer covering the entirety of membrane causing sever blocking
Types of Fouling:
Generally Fouling can be classified into two ways either internal or external, respectively, the difference between the two being sites where the deposition of the particles occurs.

Internal fouling occurs when the deposit occurs inside the pores of the membranes, resulting in total clogging or complete blocking of the membrane pores. While, External fouling occurs when the deposit happens on the external surface of the membrane, thereby leading to an accumulation of particles that form a cake layer on the membrane (Voittonen, 2018).

The size of the foulants is the ultimate deciding factor as to which type of fouling would be caused and is typically the main issue for the process involved, typically smaller foulants enter the pores while larger ones are deposited on the external surface. In Ultrafiltration, external fouling is the dominant and accounts for roughly 80% of the resistance. Depending on the nature of the foulants and membrane, different types of fouling mechanics are possible due to a different chemical or physical interactions between the foulant and membrane (Voittonen, 2018).

Some authors claim the following are the culprits in causing fouling: (Voittonen, 2018) (Singh, 2006).

- *Organic Molecules adsorption:* Organic fouling occurring due to the adsorption of organic compounds (Humic & Fulvic acids represent about 80% of the Total organic carbon have low amounts of organic levels measure in TOC (Total Organic Carbon).
- *Particulate deposition:* Caused by colloidal particles such as silica, clay, iron, and aluminum.
- *Microbial adhesion:* Causing Biofouling as a result of the formation of a film of microbes on the membrane surface causing shrinking and shortening of the effective pore size. (Aliasghari Aghdam et al., 2015) describes the various fouling mechanisms with the help of Figure.12) shown below

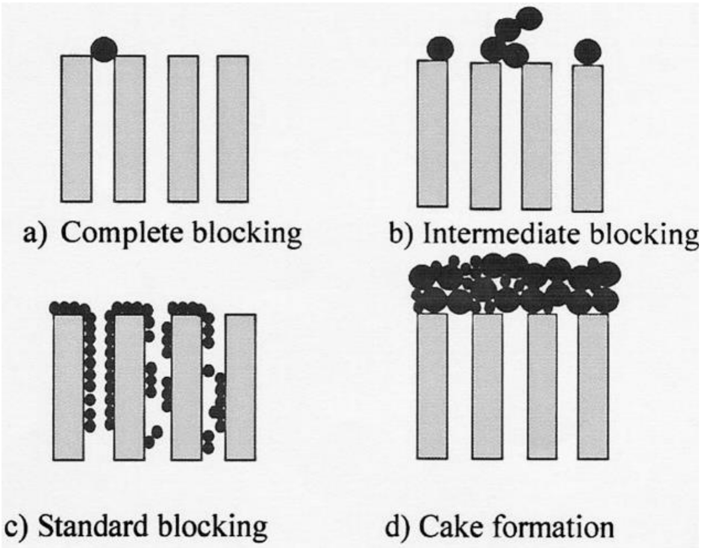

Figure.12) Various Fouling Mechanisms adopted from (Aliasghari Aghdam et al., 2015)

In particular for RO & NF systems since they are less rugged than MF & UF membranes they need to be taken specific care regarding the water quality of feed hence as a rule of thumb to minimize scaling and fouling for the same the water feed input to RO & NF must be kept less than 1.0 NTU, SDI less than 4.0, LSI less than zero (Singh, 2006).

2.7) Membrane Cleaning:

This section mentions the cleaning Processes & Recommendations on cleaning the membrane.

Sometimes membrane fouling can be responsible for the drop in membrane flux by about 80 %, however, the time of it happening can vary from minutes to several days depending on various factors, regardless of the factors it is a must-do thing to exercise control or in an order to execute effectively and optimize flux (Singh, 2006). A recommendation states started cleaning the ceramic membrane once the flux drops below 30L/m^2/hr. = 3.9 dm^3/hr (Wei, 2015). This can also be considered as the absolute limit of performance, however, if membrane cleaning is not initiated it can lead to further degradation of the membrane.

The two conventional methods for cleaning the membranes are as follows:

- *Physical Cleaning:* High-pressure water washing aided with mechanical scrubbing and backwashing, therefore this type of cleaning aims to remove the fouling which occurs on the external surface of the membrane.
- *Chemical Cleaning:* Membrane Treatment with dilute alkali and acid combination or whatever is needed, enzyme, surfactant, complexing agent, and oxidant are usually used for membrane cleaning.

Back pulsing/Backwashing:

However, both the terms Backwashing and back pulsing are used in conjunction thus both of them mean the same thing, it is an important procedure for MF and UF membrane filters as for conventional media filters to remove solids accumulated at the membrane surface.

Conventionally Back pulsing procedures are somewhat site and manufacturer dependent, but the typical characteristics of membrane back pulse are:

- Each membrane unit is back pulsed individually in a sequence so that not all membrane units are back pulsed simultaneously,

- The back pulse consists of a flow reversal through the membrane for a short period i.e. 1/2 to 5 mins.

- As back pulsing also results in loss of operation time and production collection during the same, it must be ensured that it is only practiced in need, at kept to a bare minimum.

- Typical Time between respective back pulse is about 15 to 60 minutes and is triggered by either exceeding a threshold transmembrane pressure (TMP), falling below a target flux, reaching a total volume of water production through the membrane since the last back pulse or reaching a set operating time.

The goal of the back pulse is to clean the membrane to its original TMP and flux performance. However, over time the TMP will increase at a given flux. Thus, chemical cleaning is implemented periodically to help restore the membrane towards its best practical TMP condition. Additionally, some manufacturers have developed their back pulse methods using chemicals such as acids, bases, surfactants, or proprietary chemicals to achieve better TMP's and delay the need for chemical cleaning. In such systems, there may indeed be a need for careful attention to cross-connection control and membrane rinsing.

Transmembrane Pressure (TMP): Calculated by the formulae given below.

$$\Delta p = \frac{P1 - P2}{2} - Pp \qquad \text{Eq.2)}$$

- **Δp = Trans-membrane pressure.**
- **P1 = Pressure at the entry of the module.**
- **P2 = Pressure at the exit of the module.**
- **P_p = Pressure of permeate.**

Relaxation:

It is the process scheme which involves ceasing of the standard operation in regular intervals to stop permeate flux thus pausing the filtration mechanism to achieve reduced fouling and thus increasing the time required for back to back chemical cleaning.

Some studies recommend that using relaxation and back pulsing in a combination results in optimum cleaning configuration (Kang, Lee & Kim, 2003) (Vallero, Lettinga & Lens, 2005).

Cleaning in Place:

(CIP) is a non-traditional method used for cleaning the interior surfaces of pipes, vessels, process equipment, filters, and fittings, without disassembly. Depending on various processes and working principles, a typical CIP design principle is adopted amongst one of the below:

- deliver highly turbulent, the high flow-rate solution to effect good cleaning (applies to pipe circuits and some filled equipment).
- give solution as a low-energy spray to fully wet the surface (applies to lightly soiled vessels where a static spray ball may be used).
- deliver a high energy impinging spray (applies to highly soiled or large diameter vessels where a dynamic spray device may be used).

Elevated temperature and chemical detergents are often employed to enhance cleaning effectiveness. Therefore, the CIP was employed in such a way to clean the equipment while operating Chemical Enhanced backwash (CEB) simultaneously. Table.7) shown below lists various target contaminants Chemicals used for Cleaning tests via CIP:

Table.7) Chemical Cleaning Agents (Membrane filtration guidance manual, 2005)

Category	**Chemicals Commonly Used**	**Typical target Contaminant(s)**
Acid	• Citric Acid ($C_6H_8O_7$) • Hydrochloric Acid (HCl)	Inorganic Scale
Base	• Caustic (NaOH)	Organics
Oxidants	• Sodium Hypochlorite • Chlorine (Cl_2) Gas • Hydrogen Peroxide(H_2O_2)	Organics; Biofilm
Surfactants	• Various	Organics; Inert particles

3) Methodology

The Methodology of this thesis project is simple can be divided into different sections, firstly a Literature survey done earlier was used to design Parameters, which are further used to investigate the best possible solution to exercise fouling control and flux optimization without compromising the permeate water quality. Further on with utilization of current wastewater trends for ultrafiltration pertaining ceramic membranes a series of Factorial experiments were performed on the initial influencing factors collecting the main responses; finally, the data collected from the factorial experiments were fed into the MODDE Analytical software where DOE was used to extract the best case and look at the experiments statistically. Each section is further described below.

3.1) Parameter Design

The literature study for the project was aimed to find out the current trends and innovations with membrane technologies used for water and wastewater purification and reuse.

Factors and responses were framed out based on the literature review, their relative importance, and relatability to the focus of interest and were fed into the software generating an experimental worksheet shown below in Table.8). The parameters were investigated by running a series of factorial experiments at the pilot plant (WASLA) at IVL Environmental Research Institute's Research Facility at Hammarby Sjöstadsverket, Henriksdal.

Factors are the parameters which are found out to be the most influential according to the requirements, while responses are the system information extracted as a result of the experiment. DOE is based upon an interaction-based process model where input and results are processed through the system and influence and model predictions are generated. Total Factorial experiments are 19 (16+3) also inclusive of 3 center point experiments that have an alike factor configuration to evaluate and compare result responsiveness.

3.2) Factor Design and Setting

The factors used in this report are shown in Table.7) and further explained below.

Table.8) Factorial Experiments Design Factors

Factors

Name	Abbr.	Units	Type	Settings	Transform	Precision
BP Frequency	BPF	minutes	Quantitative	0,08333 to 1	None	0,0229
VRF	VRF		Quantitative	2 to 10	None	0,2
TMP	TMP	bars	Quantitative	1,5 to 7	None	0,138
Run Time	Run	hours	Quantitative	2 to 18	None	0,4
Cross Flow	XF	m/s	Quantitative	4 to 6	None	0,05

Back Pulsing Frequency (BP-F): Self-designed factor which takes in account of the frequency when a Back pulsing is arranged amidst the permeate collection operation, the values range from 0.08333 to 1 where 0.08333 is 5/60 standing for back pulsing every 5 minutes onwards per 60 minutes of operation and 1 is 60/60 hence the back pulsing would be done every 60 minutes of 60 minutes of permeate collection.

Volume reduction factor: (VRF) is used to estimate the maximum water recovery rate. VRF can be defined as the ratio between total feed volume and concentrate volume and indicates how many times feed water was concentrated. The values of VRF ranged from 2 to 10 in the Design of Experiments.

Total feed volume VRF = Concentrate volume, the highest acceptable VRF is limited by the flux value and permeate quality.

For Volume Reduction Factor implementation typical flow rate from permeate flowmeter FT02 _MV. The permeate value is then used to adjust the Retentate pump.

The values in dm3/h are converted into ml/min, so for implementation of the value of the VRF = 2, the flow rate of the Retentate pump is entered in ml /min thus dividing the total flow rate into two parts.

While for a value of VRF = 10, the permeate flow rate is divided into 10 parts, and then for each one part of the total flow is taken out through the retention pump.

Run Time: total duration of the experimental run Range of Values include 2 to 18, The 2-hour experiments were performed on the same day it selves while 10 and 18 hour long experiments were run overnight and data of the experimental run was collected despite the Pilot running for a time longer than the experimental run for sake of reliability and respecting working hours of the Research facility.

TMP: Transmembrane Pressure controlled via the experimental console available on the pilot WASLA, ranges include from 1.5 to 7 with three values included 1.5, 4.25, and 7 Bar Respectively.

Cross Flow: This parameter is defined as the rate of cross-flow allowed across the membrane and changes the value of velocity across the membrane channel can be varied with the help of valve on the Cross Flow pump with values ranging from lowest 25 l/min to a maximum of 65 l/min, however for respecting the optimum values the respective membrane fit in the membrane module the allowable cross flow limit is determined according to the permitted velocity across the membrane module by the membrane manufacturer specified below in the Table.9) taking the values were further converted into l/min with the help of the table supplied by the manufacturer and further explained in Appendix 2.

Table.9) Working parameters/Factors found out from experimentation on the existing membrane.

Parameter	Lowest operating value	Average operating value	Highest operating value	Recommended values for ATech (UF)
X-Flow (m/s)	4	5	6	2.9 – 6 m /s
BP Frequency (mins)	5	32	60	X
VRF	2	6	10	X
TMP (Bars)	1.5	4.25	7	1.5 - 3 bar

3.3) Response Design

The responses used in this report are shown in Table.10) and further explained below:

Table.10) Factorial Experiment's Design Responses.

	Name	Abbr.	Units	Transform	Type	Min	Target	Max
1	COD	COD	mg/l	None	Regular	0	99	100
2	Membrane Flux	Flux	LMH	None	Regular	0	300	400
3	Turbidity	Tur	F.N.U	None	Regular	0	99	100

COD Reduction (%): Chemical Oxygen Demand values were used as an estimation of organic matter and for the possible biological activity of microbes in the permeate and feedwater checked. The dissolved organic matter with a Molecular weight less than 20kDa would pass the membrane while larger particles would be retained.

Turbidity Reduction (%): Turbidity is the cloudiness or haziness of a fluid caused by large numbers of individual particles that are generally invisible to the naked eye, similar to smoke in the air. The measurement of turbidity is a key test of water quality.

Fluids can contain suspended solid matter consisting of particles of many different sizes. While some suspended material will be large enough and heavy enough to settle rapidly to the bottom of the container if a liquid sample is left to stand (the settable solids), very small particles will settle only very slowly or not at all if the sample is regularly agitated or the particles are colloidal. These small solid particles cause the liquid to appear turbid.

Formazin Nephelometric Units are referred to as FNU also means that the instrument is measuring scattered light from the sample at a 90O angle from the incident light.

FNU is typically used when referencing the ISO 7027 (European) turbidity method, also generally FNU = NTU (Support.hach.com, 2019).

Membrane Flux: Flux is generally given in l/m2/hr. (LMH); however, the pilot measured the flowrate in dm3/hr. Therefore, to obtain the membrane flux the permeate flow rate must be divided by the membrane area 0.13m2.

SDI: Silt Density Index was used to estimate the quality of the permeate and to estimate the treatability in a further step by Reverse osmosis.

NOTE: However, startup-problem were experienced with the SDI-equipment available at the research facility made it hard to carry out the procedure which involved permeate transfer to the IVL office at Valhallavägen for each experiment, as a result: SDI is excluded from the included response list.

3.4) Design of Experiments:

DOE begins after entering the above-mentioned factors and responses in the software MODDE 12.04 and which is used to generate the no. of Experimental Runs, time, and other constraints. It was preferred to go with the default and first recommendation of experimental Design which included 16+3 Runs with an interactive model which gives us a good enough representation of the information extractable from the experiments further shown below highlighted as Figure.13).

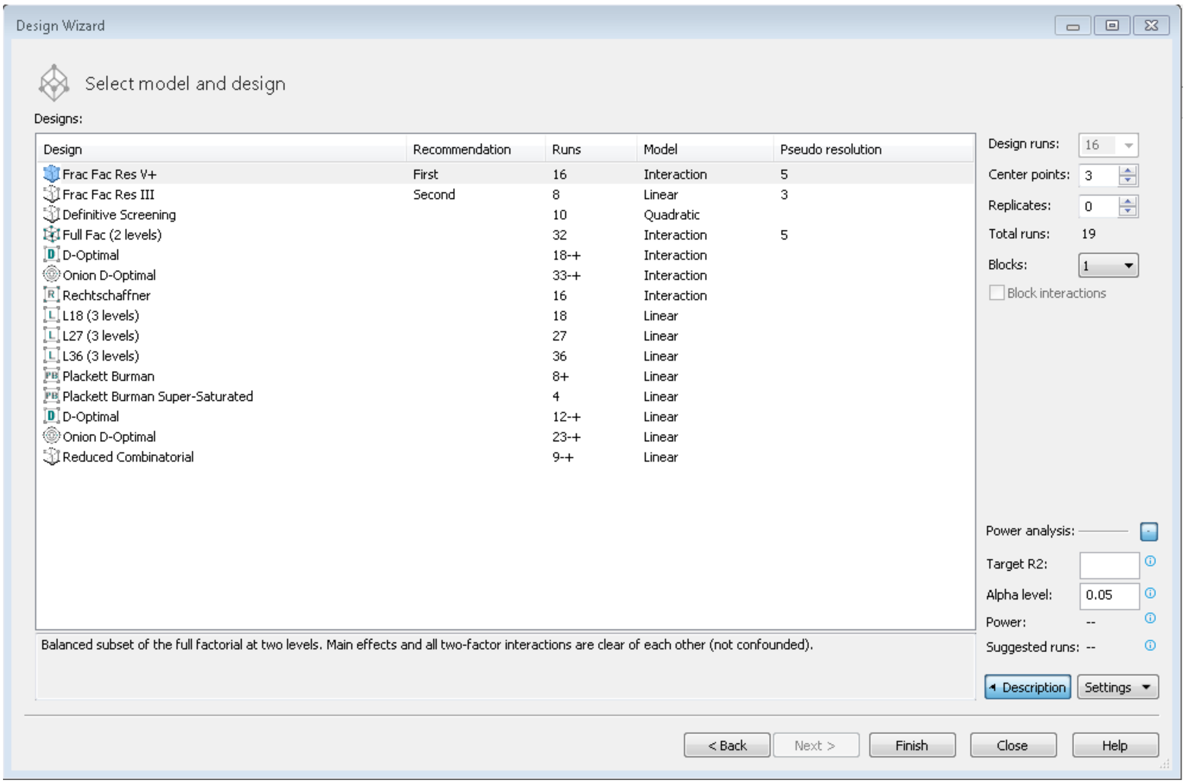

Figure.13) Design Wizard in MODDE 12.04 which shows the DOE chosen for the experiment along with model and settings.

After the selection of the recommended option a DOE worksheet is obtained which describes the preset configuration of values of Factorial Experiments to be done to maintain uniformity, though for the sake of simplicity and easiness in performing the factorial experiments the worksheet shown below in Table.11) is arranged in ascending order according to the Run order of the experiments.

Table.11) Final Worksheet in MODDE 12.04 showing experiments model with 19 factorial experiments sorted according to the run order.

Worksheet

Exp No	Exp Name	Run Order	Incl/Excl	BP Frequency	VRF	TMP	Run Time	Cross Flow	COD	Membrane Flux	Silt Density Index	Turbidity
16	N16	1	Incl	1	10	7	18	6				
2	N2	2	Incl	1	2	1,5	2	4				
3	N3	3	Incl	0,08333	10	1,5	2	4				
4	N4	4	Incl	1	10	1,5	2	6				
5	N5	5	Incl	0,08333	2	7	2	4				
17	N17	6	Incl	0,541665	6	4,25	10	5				
8	N8	7	Incl	1	10	7	2	4				
19	N19	8	Incl	0,541665	6	4,25	10	5				
7	N7	9	Incl	0,08333	10	7	2	6				
11	N11	10	Incl	0,08333	10	1,5	18	6				
12	N12	11	Incl	1	10	1,5	18	4				
1	N1	12	Incl	0,08333	2	1,5	2	6				
9	N9	13	Incl	0,08333	2	1,5	18	4				
13	N13	14	Incl	0,08333	2	7	18	6				
6	N6	15	Incl	1	2	7	2	6				
10	N10	16	Incl	1	2	1,5	18	6				
14	N14	17	Incl	1	2	7	18	4				
18	N18	18	Incl	0,541665	6	4,25	10	5				
15	N15	19	Incl	0,08333	10	7	18	4				

3.5) Experimental Setup & Procedure

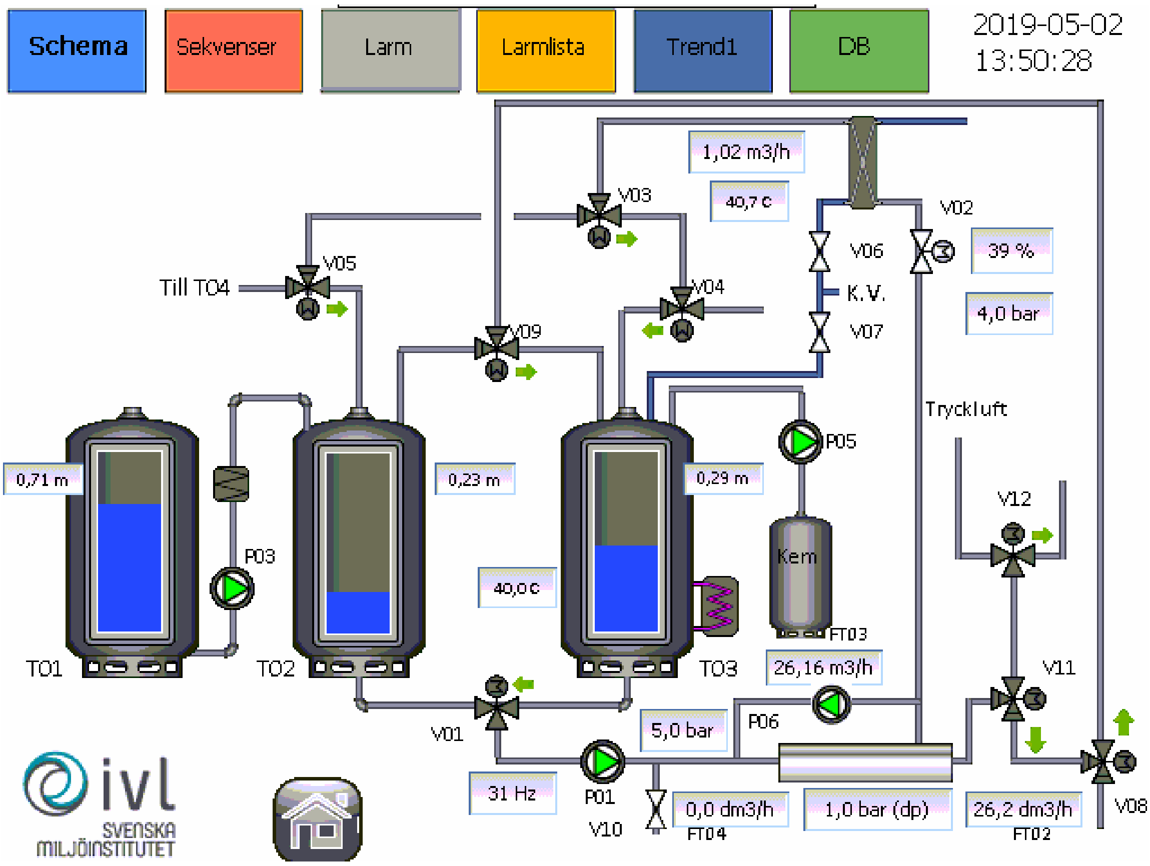

Figure.14) Process scheme for the factorial test with ATECH type 7/6 membrane showing a CIP wash.

Pilot Setup: The Pilot plant shown above in Figure.14) was used to perform the 16+3 factorial experiments devised by DOE. The plant was fitted with ATech GmBh Al_2O_3 coated, type 7/6, MWCO 20 kDa, ceramic membrane see Table.12) below. The notations PP_{OX}, TT_{OX}, FT_{OX} mentioned in the Figure.12) above stands for pressure, temperature, and flow meters respectively, Inlet is pretreated wastewater while Coldwater is used for washing rinsing and cooling for evaluating membrane's performance. The process starts with taking feedwater up directly from Stockholm Vatten och Avfall's Henriksdal WWTP facility which is pumped up to IVL Hammarby Sjöstadsverket research facility's Sedimentation tank of 5.4m^3 capacity, with a continuous treatment capability of the inlet water and remaining sludge.

Further, the feed from the sedimentation tank is transferred to To_1 after going across a 100-micron filter. Tank To_1 here acts as a buffer tank between the sedimentation tank and T_{O2} to regulate the amount of water added to T_{O2} and thus ensuring no overflow occurs from sedimentation occurs with the help of fluid level sensors placed in all tanks to make the whole process automated, simple yet secure.

Table.12) Membrane properties of the ATECH Al_2O_3 Ceramic Membrane provided by the Manufacturer.

Atech Al_2O_3 membrane:	**7 duct design, type 7/6**
Support Material:	α-alumina oxide
Membrane Material:	Ultra-Filtration: Al_2O_3
Overall Length:	1.000 / 1.200 mm (Standard)
Outer Diameter:	25.4 mm +- 0.5 mm
The diameter of the Duct:	approx. 6 mm
Number of Channels:	7
Filter Area: approx.	$0.13m^2$
pH:	0-14 with 80-90^o C.
UF Molecular Weight Cut Off:	20 kD
Mean Pore Diameter:	0.02µm

Cleaning Operation: Once the operation is started, water is circulated with the eliminating a flow of $1m^3/h$ at $FT0_1$ while the rest of the cross flow is controlled with the help of Cross flow pump which also is varying between 1.9 to 3.3 m^3/h –converted to 31.66 -55 l/min, while permeate exits the system and is collected with the opening of Valve V_{08}. Each test has various permeate flux which is measured across FT02 with varying pressures and test duration, Feed pressure was regulated with PP_{01} varying between 2 - 9 bars and the operating time ranging from a minimum of 2 hours to maximum of 18 hours. The flow and TMP were measured and monitored with FT_{01}, PP_{01}, PP_{02}, TT_{01}. Once a test is completed it is ensured that the membrane module is completely free of feedwater and maintaining uniformity before another test could be run the whole tank T02 was automatically empties by pumping the water to the drain via V_{05}, sometimes this alone won't be enough to completely empty T_{02} and membrane module hence the remaining water was emptied by opening V_{11}, V_{10} and V_{12} along with rinsing T_{02} with clean water. In the meantime, during the cleaning operation, the Back pulsing is happening as well according to the respective time config as supplied Every 5, 32, and 60 minutes, Figure.13) below shows the process of back pulsing happening at a frequency of every 5 minutes across the pilot plant console.

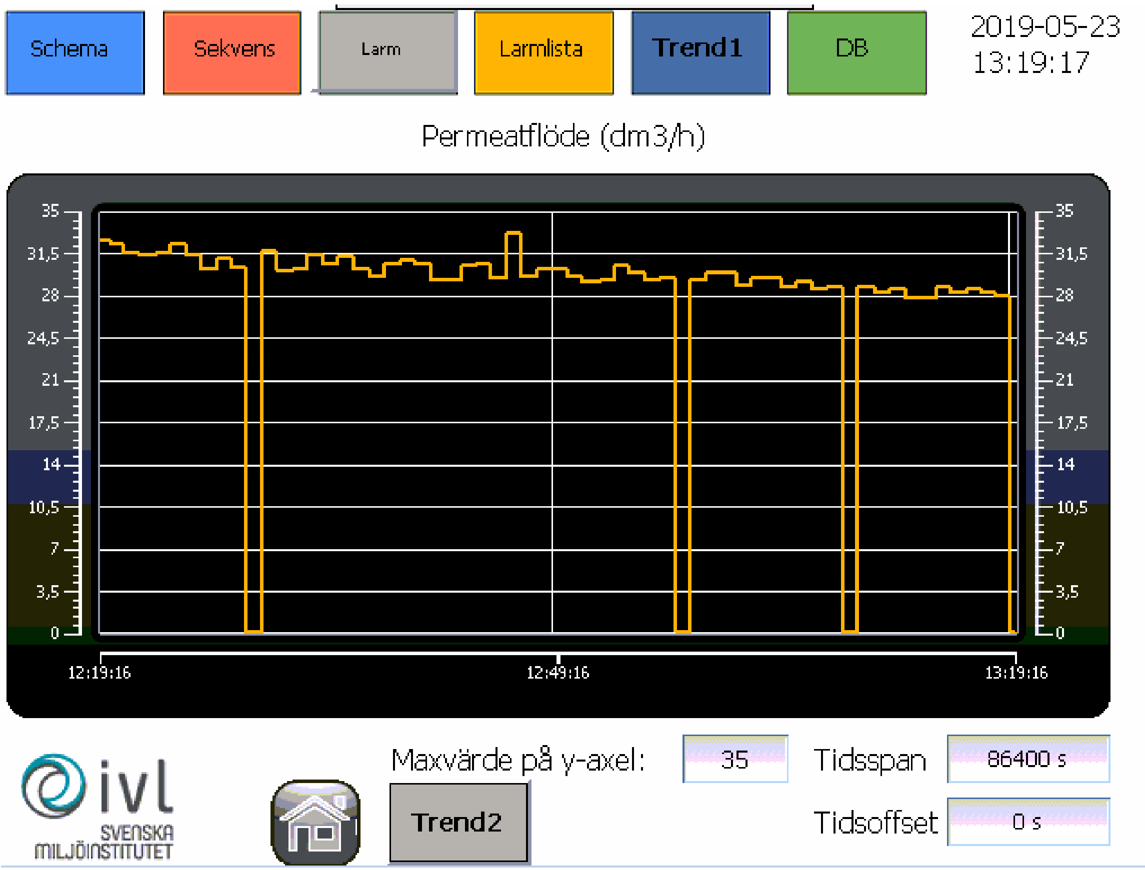

Figure.15) Back pulsing operation during the membrane's cleaning operation.

CIP: CIP is implemented and the whole system was washed after each test cycle of 40 hours with Ultrasil 25 manufacture by Ecolab, by filling T03 with 0.3m about 80 liters of water with about 0.4 liters of Cleaning chemical. Ultrasil 25 consists of various phosphates and chlorides, as well as other enzymes, it was added to T03 in liquid form. The whole solution was then heated to 40-500C as shown in Figure.15) above. Thus, CIP was initiated where the whole solution was circulated form T03 across the membrane module and then back to T03. The wash is performed with a flow of 1m3 measured across FT01 with a standard TMP of 4.5 Bar and lasted for about 30 minutes. Once the wash is finished the whole solution is emptied via V04 and the membrane module was also emptied manually via V12. Clean water was then added to T03 and circulated across to rinse they system repeatedly for about 12 minutes at 250 C and for measuring the flux across the membrane module via FT02, as illustrated through the figure.13) above. Multiple recommendations from the Literature review suggested (see section 2.7) that pressure in the wash should be kept on higher ranges to ensure a smooth and thorough cleaning.

3.6) Sampling Procedure and Data Recording

COD: To calculate the COD values from the feed and permeate two different Photometric Test Kits were used respectively:

1)Xylem - WTW C3/25 COD Chemical Oxygen Demand O_2 kit: Used until experiments number 6 according to Run Order.

Photometric test kit for measurement of COD Chemical oxygen demand O2 Available range:10 - 150 mg/l COD.

2)Xylem - WTW C4/25 COD Chemical Oxygen Demand O_2 Kit: Used for Experiments No.7 and afterward according to Run Order.

Photometric test kit for measurement of COD Chemical oxygen demand O2 Available ranges:25 - 1500 mg/l COD.

Sample preparation: Since experiments until no.6) were sampled and analyzed with C3/25 test kit which has a significantly lower detection range thus the experimental Samples for Feed & Permeate were diluted using distilled water in a ratio of 1:2 since the cuvettes require 3ml of the sample, however for the rest of the experiments from no.) 7 - 19 no dilution of the sample was needed or done.

Sampling Procedure (Both Test Kits have the same sampling procedure:)

- Draw the samples from Feed Sedimentation Tank, Permeate Collection Tank in a reusable plastic container from after flux stabilization.
- Preheat the Thermoreactor to preset 148^{o}C for 02:00 hours.
- Take 3 ml of Sample with help of Pipette and pour the sample in the test cuvette placed on the rack inside the Cooling Chamber.
- After closing the Cuvette lids and shake all the sample Cuvette vigorously.
- Place the Sample Cuvettes in the Thermoreactor and start the countdown.
- Collect the samples after the countdown reaches 00:00 hr. and Thermoreactor starts beeping.
- Place the Cuvettes on a rack inside the cooling Chamber and wait for the cuvettes to cool down for 30 minutes.
- Take out the Cuvette Samples and read the sample readings using a spectrophotometer.

SDI:

The collected permeate from the ultrafiltration was filtered through a 0.45 µm filter at 2.1 bar and about 20 degrees.

The time for filtering 500 ml at time 0, 5, 10, and 15 minutes was measured when possible and SDI calculated using the following equation:

$$\mathbf{SDI = 100 * (1 - \frac{T_i}{T_f}) / T_t} \quad \text{Eq.3)}$$

- **T_i = Initial time in seconds required to obtain a sample.**
- **T_f = Time required to obtain sample after 15 minutes.**
- **T_t = Total time in minutes.**

Turbidity: Turbidity of the samples was measured using HACH's 2100Q Portable Turbidimeter, the sampling operation is simple involving a collection of samples in small glass bottles and aligning them against the set markings of turbidimeter and after closing the lid, READ button is used and results are displayed and can even be stored in the Turbidimeter as well.

Membrane Flux: Permeate Flux for the respective experimental run was determined through the basis of the readings from Flow sensor FT_02 from the WASLA Pilot Plant. Experimental Run sheet was generated using SQL server 5.0 to recover data from the various sensors including FT_02 and was further arranged in an excel sheet.

An average of all the readings through the experimental run excel sheet was taken to calculate the mean membrane flux in across the experimental run time.

3.7) Model Fitting:

This section discusses the statistical techniques used for model Building and analysis in MODDE 12.04.

Multiple linear regression (MLR) is a statistical technique that exploits several explanatory variables to predict the outcome of a response variable. The objective of MLR is to model the linear relationship between the independent (factors) variables and dependent (response) variables such as every value of the independent variable x is associative of the value of dependent variable y (Kenton, 2019).

Partial least squares regression (PLS regression) is a statistical method used to form a meaningful relationship between two sets of variables X & Y across a multivariate dataset. It forms a linear regression relationship between the predicted variables and the observable variables to a new space instead of finding hyperplanes of maximum variance between the response and independent variables as is done in PCR (Principal Component Regression). As both the X and Y data are projected to new spaces, the PLS family of methods is also known as bilinear factor models.

MLR, PLS, PCR, and several other methods are all Inverse Least Squares (ILS), based models. Given a set of predictors, **X** (m samples by n variables), and a variable to be predicted, **y** (m/1), they find **c**, such that the estimate of **y, ý = Xc**, from **c = X+y**. The difference between the methods is that they all employ various ways to estimate **X+**, the pseudoinverse of X (Switch from MLR to PLS? 2019).

Hence after the data from experiments was taken, it was fit into the model employing PLS cause of its simple model interaction and correspondingly better fit concerning the interactive model.

4.) Results & Discussion

4.1) Wastewater Factorial Test

The following were the results obtained from the factorial experiments for more details visit Table.12) below. The Flowrate from permeate was taken to be the average readings and then further divided by the surface area of membrane 0.13 m^2 to get the membrane flux since the main evaluatory responses are COD Reduction, Membrane flux, and Turbidity Reduction. The response analysis is done by carefully reading and understanding the main results obtained from individual responses are plotted and explained below:

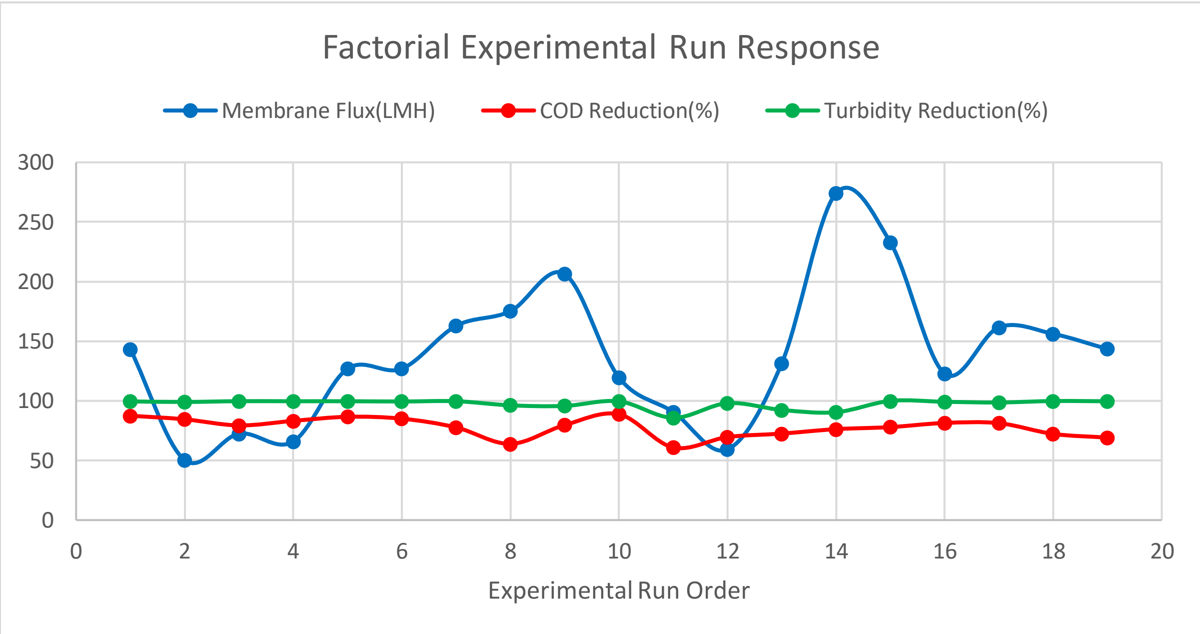

Figure.16) Graph Showing the Factorial Experiments Responses over w.r.t Run order

The responses were plotted on the Y-axis against an Experimental run order of a total of 16+3 experiments on the X-axis and the graph that came out to be is shown above in Figure.16) while Table.13) & Table.14) show detailed results of the Factorial Experiments respectively.

General trends on membrane flux are shown in Blue color, across all experiments, the minimum observable flux is about 50LMH while the highest recorded flux is >250LMH accounting for a more than 500% difference between maximum & minimum values.

The Membrane flux of the experiments, in the beginning, is 143.3077 $L/m^2/h$ and peaks about 274 $L/m^2/h$ with averaging about 140.646 $L/m^2/h$. COD reduction trends are shown above in Red color follows a typical downward trend until experiment 8 and then it experiences a slight rebound with a peak reduction observed at experiment 10 and then tracing it back for the rest of the experimental duration. While Turbidity reductions shown in Green color remains uniform around 90 – 100% throughout the experimental order except for one experiment no.11 which can be counted as an anomaly and will be discussed further.

Table.13) Results obtained with recorded Responses.

Experiment Run order w. Date	Turb. Feed (F.N.U)	Turb. P F.N.U	Turb. (F-P)	COD Feed (mg/l)	COD Permeate (mg/l)	COD (F-P)	Avg. Flowrate (dm^3/h)	Flux ($L/m^2/hr$)	Turb. (F-P) %	COD (F-P) %
1) 25/04/19	492	2.37	489.63	650.	81.00	569.00	18.63	143.30	99.51	87.53
2) 2/05/19	997	9.30	987.70	620.	96.00	524.00	6.55	50.38	99.06	84.51
3) 07/05/19	323	0.90	322.10	612.	126.00	486.00	9.42	72.46	99.72	79.41
4) 07/05/19	393	1.33	391.67	679	114.00	565.00	8.56	65.84	99.66	83.21
5) 08/05/19	442	1.42	440.58	807	108.00	699.00	16.50	126.92	99.67	86.61
6) 14/05/19	509	2.70	506.30	763	114.00	649.00	16.50	126.92	99.46	85.05
7) 20/05/19	542	1.72	540.28	930	207.00	723.00	21.17	162.84	99.68	77.74
8) 22/05/19	443	16.7	426.30	637	229.66	407.57	22.77	175.15	96.23	63.95
9) 23/05/19	442	18.8	423.20	763	155.00	608.00	26.83	206.38	95.74	79.68
10) 28/05/19	1070	4.36	1065.64	1419.33	159.66	1259.67	15.55	119.61	99.59	**88.75**
11) 24/06/19	352.	50.0	302.00	667.66	261.00	406.66	11.79	90.69	85.79	60.90
12) 27/06/19	348	6.88	341.12	790	240.00	550.00	7.70	59.23	98.02	69.62
13) 01/07/19	547	41.8	505.20	983	270.00	713.00	17.09	131.46	92.35	72.53
14)02/07/19	388	37.0	351.00	880.66	209.00	671.66	35.63	**274.07**	90.46	76.26
15)03/07/19	762	0.45	761.55	985	217.00	768.00	30.28	232.92	**99.94**	77.96
16)/08/07/19	861	6.73	854.27	1075	199.33	875.67	15.97	122.84	99.21	81.45
17)09/07/19	668	9.21	658.79	1085.66	203.33	882.33	21.02	161.69	98.62	81.27
18)10/07/19	666	1.01	664.99	887.66	246.00	641.66	20.30	156.15	99.84	72.28
19)11/07/19	441	1.50	439.50	751.33	231.66	519.67	18.65	143.46	99.65	69.16

Table.14) Experimental Worksheet with all Parameter values.

Ex p No	Exp Name	Run Order	Incl/Excl	BP Frequency	VRF	TMP (Bar)	Run Time(hr)	XF (m/s)	COD Red.(%)	Membrane Flux(LMH)	Turbidity Red. (%)
16	N16	1	Incl	1	10	7	18	6	87.53	143.3	99.51
2	N2	2	Incl	1	2	1.5	2	4	84.51	50.38	99.06
3	N3	3	Incl	0.083	10	1.5	2	4	79.41	72.46	99.72
4	N4	4	Incl	1	10	1.5	2	6	83.21	65.84	99.66
5	N5	5	Incl	0.083	2	7	2	4	86.61	126.92	99.67
17	N17	6	Incl	0.54	6	4.25	10	5	85.05	126.92	99.46
8	N8	7	Incl	1	10	7	2	4	77.74	162.84	99.68
19	N19	8	Incl	0.54	6	4.25	10	5	63.95	175.15	96.230
7	N7	9	Incl	0.08	10	7	2	6	79.68	206.38	95.74
11	N11	10	Incl	0.08	10	1.5	18	6	88.75	119.61	99.59
12	**N12**	**11**	**Excl**	**1**	**10**	**1.5**	**18**	**4**	**60.90**	**90.69**	**85.79**
1	N1	12	Incl	0.083	2	1.5	2	6	69.62	59.23	98.02
9	N9	13	Incl	0.083	2	1.5	18	4	72.53	131.46	92.35
13	N13	14	Incl	0.083	2	7	8	6	76.26	274.07	90.46
6	N6	15	Incl	1	2	7	2	6	77.96	232.92	99.94
10	N10	16	Incl	1	2	1.5	18	6	81.45	122.84	99.21
14	N14	17	Incl	1	2	7	18	4	81.27	161.69	98.62
18	N18	18	Incl	0.54	6	4.25	10	5	72.28	156.15	99.84
15	N15	19	Incl	0.083	10	7	18	4	69.16	143.46	99.65

One noticeable observation came out of experiment no.11) gave pretty unique results where almost all of the three responses of were outliers if compared with the rest of the experiments as it showcased a record low values for COD and Turbidity Reduction %, standing at 60.90% and 85.79% respectively while the third response Membrane Flux was also showcased a value of 90.69 l/m^2 /h lying amongst lowest 20 percent values of all other Membrane Flux responses.

On further analysis, it was found that it could be related to that the experiment was performed after the equipment failure and leakages, which took almost one month to rectify. Once everything has been repaired the Cleaning operation wasn't able to regain its original efficiency. On the other hand, it could also be possible that because of this breakdown and as a result of discontinuity in experiments the membrane could have suffered some serious fouling. Therefore Experiment no.11) was excluded from the model design making total no. of runs to be 15 + 3 (18) shown above in Table.1) above.

Secondly, since uniformity across all the experiments was maintained apart from this brief gap of discontinuity of experiments and varying values of the Turbidity Feed with more than 50% variance on multiple instances could be led to believe that it's most probably because of the aesthetically changing water quality of feedwater which can’t be made to keep constant and uniform.

Also, the mean values from experimental Runs for Membrane flux, COD, and Turbidity reduction were found out to be 18.4967 dm^3/hr or 142.28 LMH, 78.72 % & 98.13 % respectively and further statistics from the factorial experiments are displayed across Table.15) shown below.

Also, one thing to be noted was the last experiment was run for more than 18 hours as the experiment started on Friday and the pilot was inaccessible to be shut down on Weekends. Hence the experiment was run for about a total of 40 hours and the results were almost similar as plotted against 18 hours timeline, The Flux wasn’t decreasing over time and the average flux over the entire duration was found out to be about 140 LMH which leads us to a conclusion that longer runs experimental runs don’t have a significant negative effect over flux decrease. i.e; back pulsing isn’t found out to be an insignificant factor in improving the flux decline over time as it’s found out to be a uniform decrease over longer runs.

And Longer runs could be beneficial for the practicality of less maintenance however it must important to get hold of the absolute limit of performance in the experimental duration and the expected significant decrease in quality of permeate or flux decrease over time.

Table.15) PLS Model’s Worksheet Statistics with Evalautory responses.

Response	*COD Reduction %*	*Membrane Flux(LMH)*	*TurbidityReduction(%)*
Worksheet runs	19	19	19
N	18	18	18
Min	63.95	50.38	90.46
Max	88.75	274.07	99.94
Mean	78.72	140.64	98.13
Q(25%)	72.53	119.61	98.02
Q(75%)	84.51	162.84	99.67
Median	79.54	137.38	99.49
Std. dev.	6.98	59.04	2.74
Min/Max	0.72	0.18	0.90
Std. dev./Mean	0.08	0.41	0.027
Skewness	-0.50	0.47	-2.011
Skewness test	-0.94	0.88	-3.75
Kurtosis	-0.46	0.34	3.34

4.2) Model Fit Summary

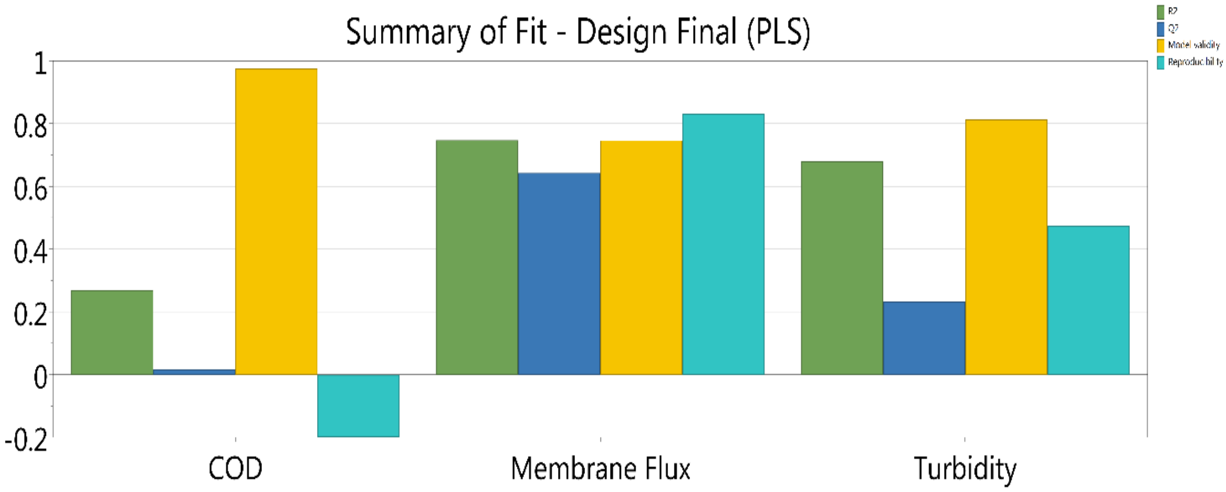

Figure.17) Model Fit Summary (PLS) MODDE 12.04.

The model was fitted using Partial Least Square Regression as summary Statistics were presented in 4 parameters R2, Q2, Model Validity, and Reproducibility, where ideally 1 or 100% is perfect.

In Regression models such as PLS, Coefficient of determination is referred to as R2, while Q2 stands for Cross Validated Redundancy, There are no optimal values for which the model up-holds true however what these values seem to give us an understanding of how acceptable and the accurate fit is the dataset to the model respectively.

The range of R2 & Q2 is between 0 and 1, though for R2, the higher its value, the higher is its predictive accuracy. According to Chin (1998) and Henseler et al. (2009), R2 value greater than 0.67 indicate a high predictive accuracy, a range of 0.33 - 0.67 indicated a moderated effect, R2 between 0.19 and 0.33 indicate low effect, while the R2 value below 0.19 is considered unacceptable (the exogenous variables unable to explain the endogenous dependent variable), Specifically, a Q2 value larger than zero for a particular reflective endogenous construct indicates the path model's predictive relevance for this particular construct (F. Hair Jr et al., 2014). It should, however, be noted that while comparing the Q2 value to zero is indicative of whether an endogenous construct can be predicted, it does not say anything about the quality of the prediction (Rigdon, 2014).

However, according to (User guide to MODDE, 2017), Q2 shows an estimate of the future prediction precision and should be greater than 0.1 for the significant model while greater than 0.5 results in a good Model, They also mention that Q2 is the best and most sensitive indicator in model summary Results.

While Model Validity is a test for diverse test problems wherein low values may be indicating of statistically significant model problems while higher values are generally reflective of a good model with less statistically problems involves, although (User guide to MODDE, 2017) also mentions an exception in case of models having a value of Q2 > 0.9 might produce an extremely low model validity as a result of higher sensitivity or extremely good replicates.

Therefore the Model fitted is illustrated above through Figure.17) returned the statistics w.r.t the three inherent Responses and based on analytical information by looking through the summary histogram it's easy to say that model fits the best in case of Membrane flux Response then Turbidity reduction while showing the least Q2 predictive values in response w.r.t COD reduction, For more detailed statistics regarding model summary go through table no.16) depicted below.

However, going through Table.16) which provides the numerical values for the Figure.17) As shown above, the model validity in all the three responses came out to be >0.5 hence model fit is valid and reproducible w.r.t Membrane flux and Turbidity reduction responses.

One another important note that can be drawn from the experiment's respective reproducibility histogram is that except COD all other experiments have reproducible results since COD reduction has a value of - 0.2.

For COD Model one of the lowest Q2 value is observed which is reflective of the predictive ability w.r.t COD reduction to be rendered ineffective and therefore it symbolizes cannot predict COD reduction effectively, however by going through the trends shown in the table.10) COD reduction between 60 - 90% can be expected. One more limitation mentioned in the COD Reduction response is due to change in the measuring Cuvettes form Xylem C3 to C4 for experiments no.6 to experiments no.7) and afterward respectively might have resulted in an erratic COD response outputs as mentioned in section 3.1.2) Responses under COD. Since membrane removes most (>90%) molecules larger than 20 kDa but dissolved molecules smaller than that are likely to pass. The COD is a blunt measurement and the amount of smaller organic molecules in the feed could vary independently of COD.

Table.16) (PLS)Model Summary Statistics.

	R2	***R2 Adj.***	***Q2***	***SDY***	***RSD***	***N***	***Model Validity***	***Reproducibility***
COD	0.27	-0.034	0.01	6.98	7.10	18	0.97	-0.2
N = 18	Cond. no. =	1.24						
Comp. = 2								
Membrane Flux	0.74	0.66	0.64	59.04	33.96	18	0.74	0.83
N = 18	Cond. no. =	1.23						
Comp. = 2								
Turbidity	0.68	0.32	0.23	2.74	2.26	18	0.81	0.47
N = 18	Cond. no. =	1.819						
Comp. = 2								

4.3) Parameter Interaction & Predictions:

All figures shown below are simulated at a BP frequency of 0.08333/ 5minutes, VRF = 10, and Run time of 10 hours.

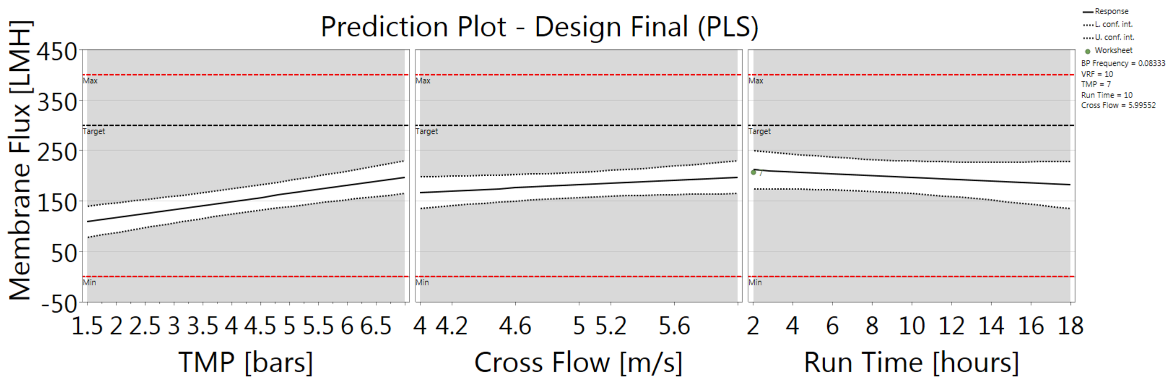

Figure.18) Prediction Plot (PLS) Observable Flux w.r.t TMP, Xflow & run time.

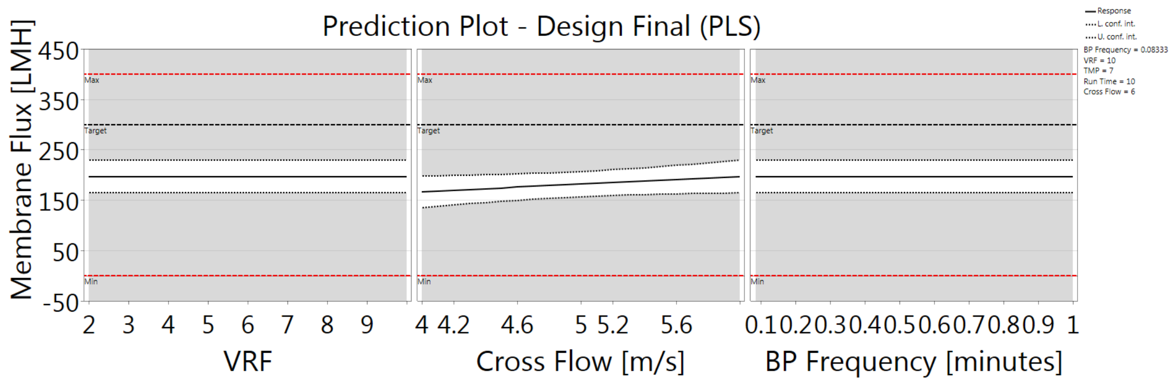

Figure.19) Prediction Plot (PLS) Observable Flux w.r.t VRF, Xflow & BP Frequency.

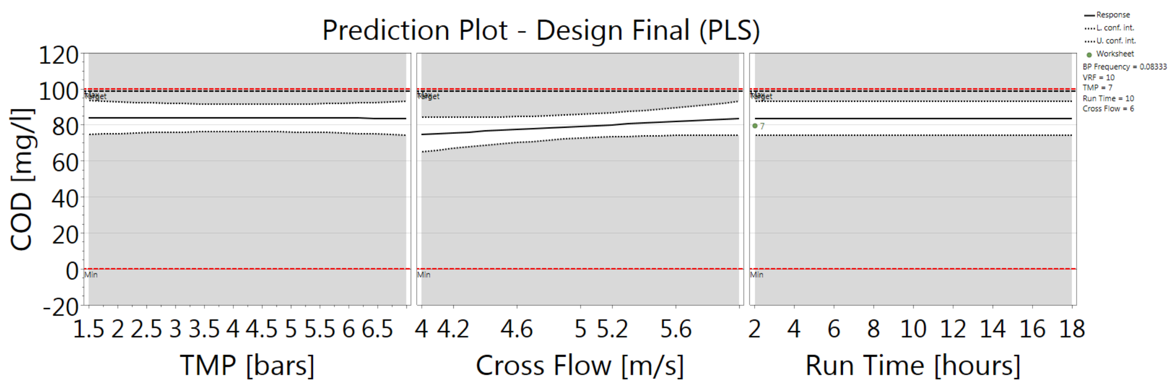

Figure.20) Prediction Plot (PLS) COD Reduction w.r.t TMP, Xflow & Run time.

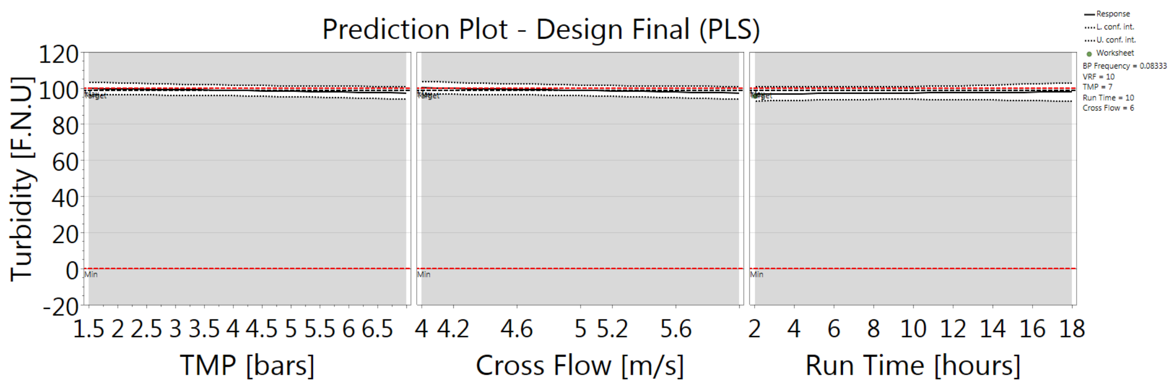

Figure.21) Prediction Plot (PLS) COD w.r.t TMP, Xflow & Run time.

Given above are the prediction plots for further experiments based on analysis from the results coming out of the factorial experiments.

Most of them show then trendlines of Factors and Response interactions: Most Notable interactions being a variation of membrane Flux across TMP and Cross Flow, Figure.18) directly establishes that Membrane flux is directly proportional to TMP where the graph has a slope of 21.42 LMH/bar, in case of Flux against it follows a similar trend although the slope seems less steep. Though in case of the trend against Run time altogether a different scenario is observed which portrays flux decrease over run time, the slope is – 2.22 LMH /hour.

Since one of the aims of the thesis project was to optimize flux as it is considered as the main response and is investigated in detail and Figure.19) shows us the prediction plot between Membrane Flux vs Crossflow, VRF & Back Pulsing Frequency respectively. Looking upon both the curves across VRF and BP Frequency the slope of both curves seems to be similar with no noticeable visual change, although it is to be noted that this consideration also consists of other factors to be constant or specified which are fixed as B.P Frequency = 0.08333, VRF = 10, TMP = 7 Bar, Run Time = 10 Hours, XF = 6 m/s which is a common consideration valid for all the Figures.16, 17, 18, and 19. Therefore strong reasoning could be made that curves without any considerable slope might be an inaccurate representation considering the other factor's influence.

Also, an almost similar case could be made of Predicted COD reduction vs TMP and Runtime are shown across Figure.20). On the other hand, the COD reduction prediction curve w.r.t Crossflow shows a directly proportional relationship where the curve shows a positive slope of 5mg/l reduction per 1m/s increase of crossflow.

Figure.21) shows the Turbidity Reduction interactions with TMP Cross Flow and Run Time, and for all of the curves paint a similar story with a very low yet noticeable negative slope but its proximity to the target limit of the graph makes it hard to quantify despite that one can draw this conclusion that Turbidity Reduction is decreasing although it is very little with an increase across TMP and Crossflow while it is increasing as the duration of experiments increase.

Since one of the project's objective involved a prediction of future experiments after evaluation of interactions amidst Factors and Responses, to test the same roughly 9 arbitrary Factor values were entered in the Prediction model of MODDE and following results were obtained as displayed below through Table.17) which predicts the outcome response values of the following experiments with the respective change in factor values and estimates the results with a predictive accuracy of +/- 15% with a display of lower and upper limits for every response respectively, glancing across the Responses the COD reductions of hover in the range of 70-80%. while flux varies significantly with change in VRF, Crossflow, and TMP to be most impacting and for Turbidity reduction, a minimum of 95% to a maximum of 99.77 % reductions are observed.

Table.17) Set up of Predicted Experiments with randomized Factor values and their predictive response.

BP Freq.	VRF	TMP	Run Time	XF	COD F-P (%)	Low	Up	Flux (LMH)	Low	Up	Tur. F-P (%)	Low	Up
0.083	10	5	10	5	79.09	73.41	84.783	149.97	131.986	167.9	98.7	96.1	101.3
1	10	1.5	10	5	78.82	71.44	86.20	93.91	67.0256	120.7	98.5	94.62	102.55
0.6	2	1.5	10	4	79.71	70.10	89.33	78.93	44.1437	113.7	96.33	93.09	99.58
0.6	2	7	10	6	75.19	65.83	84.54	196.98	164.75	229.2	96.23	93.04	99.43
1	2	7	2	6	75.19	65.83	84.54	211.88	173.592	250.1	97.41	93.40	101.42
1	2	7	18	4	80.96	71.25	90.67	152.13	108.902	195.3	99.7	95.70	103.85
0.5	2	6	2	6	75.26	66.91	83.60	187.82	154.368	221.2	95.9	92.97	98.83
0.5	10	6	18	6	83.80	75.46	92.15	174.11	134.071	214.1	98.1	94.33	101.98
0.33	4	4.5	18	6	77.50	71.56	83.44	162.14	128.166	196.1	96.8	93.31	100.45

4.4)Response Plots:

All figures shown below are simulated at a BP frequency of 0.08333/ 5 minutes, VRF = 10 and Run time of 10 hours.

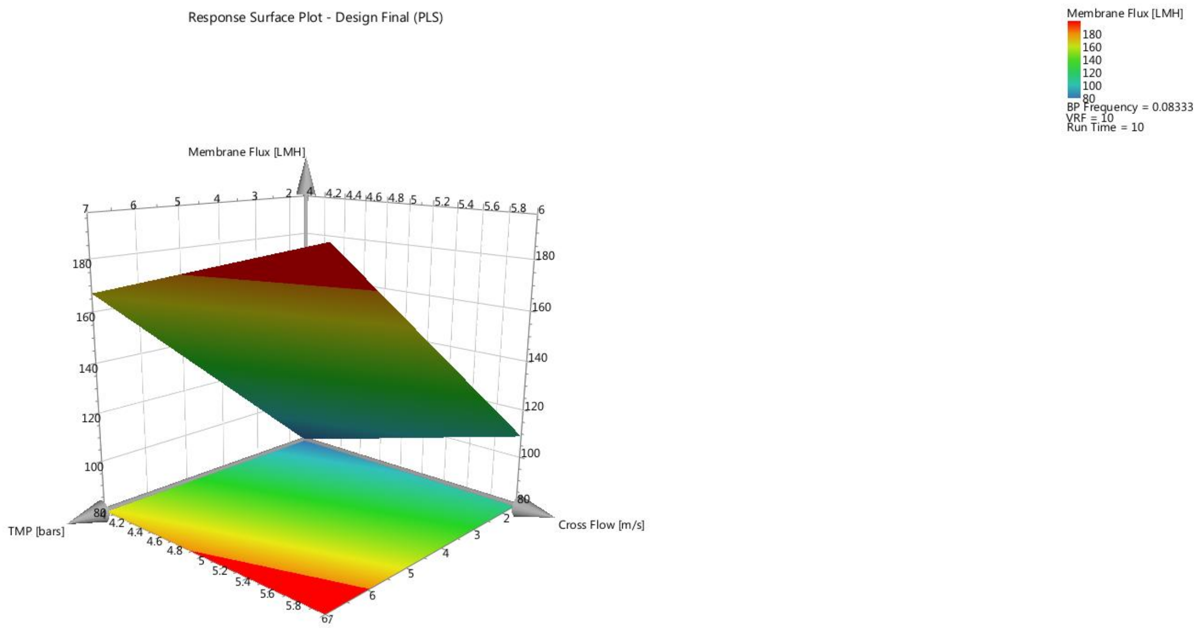

Figure.22) Response Surface Plot (PLS) Membrane Flux, TMP, and Cross flow on three respective axes.

Figure.22) displayed above portrays a Response surface plot which gives away the most comfortable area or zone of optimized membrane performance, If TMP is maintained to a region consisting with values ranging from 5 to 7 Bars while values of Crossflow are 6 m/s or higher it ultimately leads to an observable membrane flux of 190 LMH or higher, while this zone offers highest possible flux its area coverage is almost less than 15 percent of the whole array of possible configurations possible.120 -140 LMH is observable across a much higher area of more than 70 percent of the surface plot which is also easier to maintain the above for similar reasons.

While Figure.23) shown below similarly discusses the COD response plot as discussed above for Figure.22), however, the ideal zone of achieving best COD reduction is much narrower ranging in terms of TMP, however, Crossflow isn't that impactful of a parameter for the same thus making ideal zone's area about 10 percent of the whole area. Therefore if factor setting corresponding to that particular area is practiced would contribute for optimum COD reductions, however reflecting upon and accounting for earlier findings which suggest the prediction of COD reduction values are not extremely reliant, yet aligning with the earlier results it's safe to say that the model can predict an estimated COD reduction of 70-80 percent regardless of the optimum configuration of factors even taking in account of varying feed of Municipal wastewater.

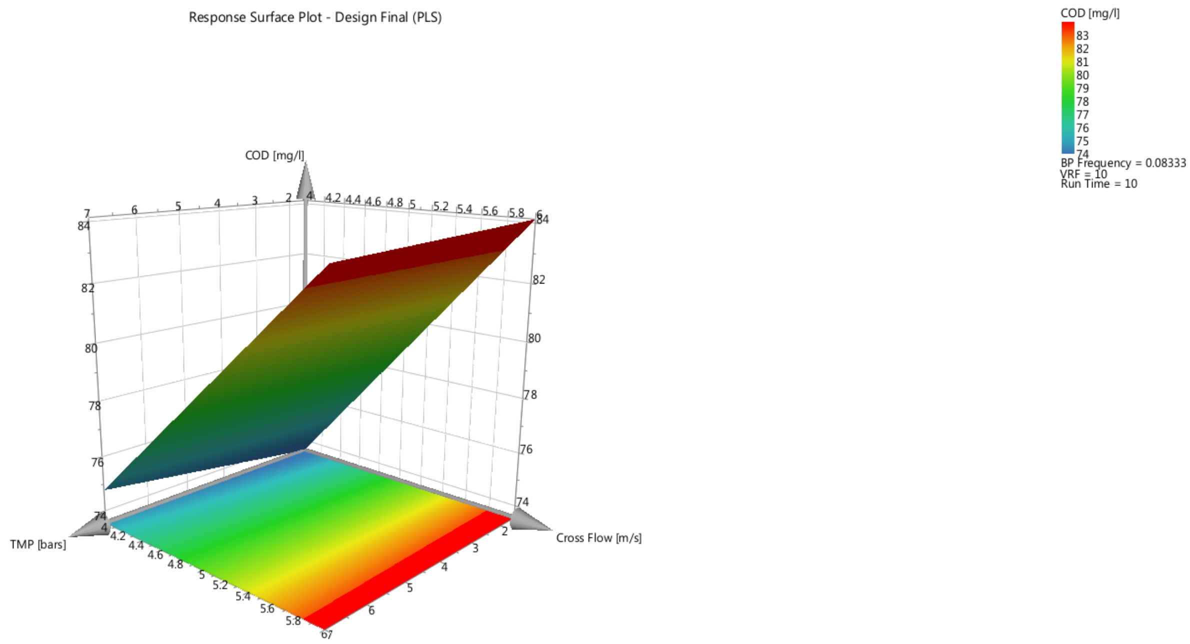

Figure.23) Response Surface Plot (PLS) COD, TMP, and Cross flow on three respective axes.

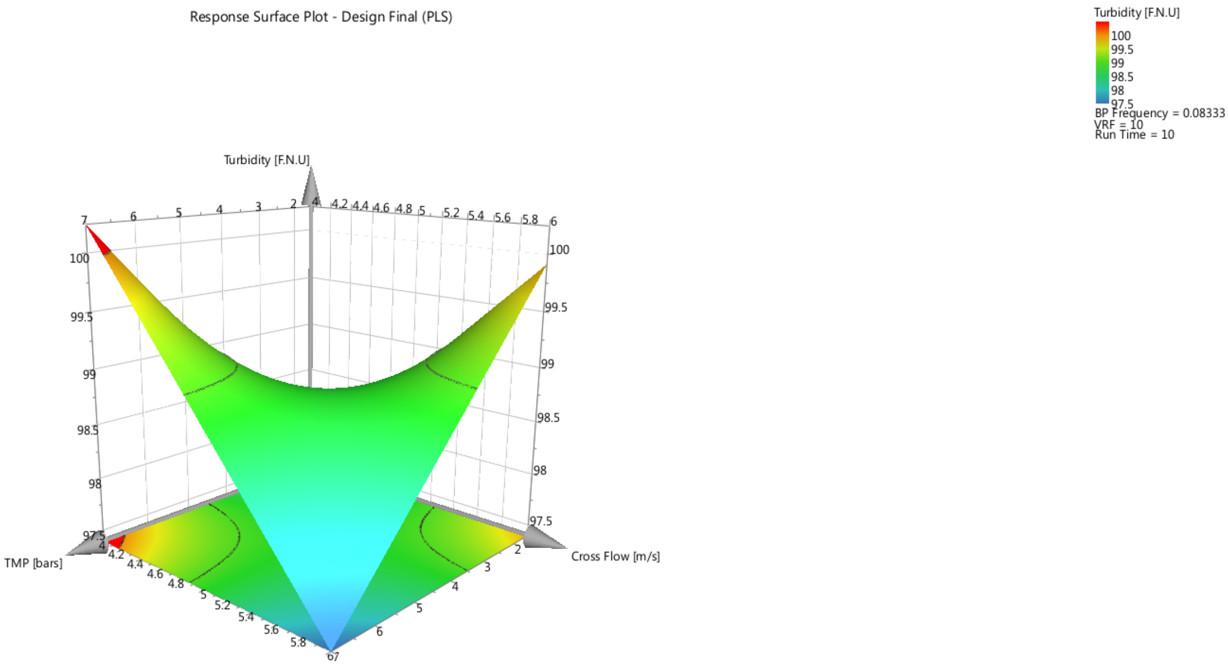

Figure.24) Response Surface Plot (PLS) Turbidity, TMP, and Cross flow on three respective axes.

Figure.24)Shows Response Plot of Turbidity which instead of single optimum zone displays us two areas on apex ends of the surface plot estimating peak reductions might be observed and it does differ contrastingly with the other mentioned graphs, however since the values of Turbidity Reduction are in proximity it's harder to set up a clear demarcation line of the best zones, yet lower TMP < 4.2bar and Higher Cross flow **ϵ**5.8 – 6 m/s factorial settings favor to make the best possible Turbidity reduction scenario while the other zone corresponds with low crossflow < 2 m/s and higher TMP > 6 bars lead towards the second-best Turbidity Reduction Scenario.

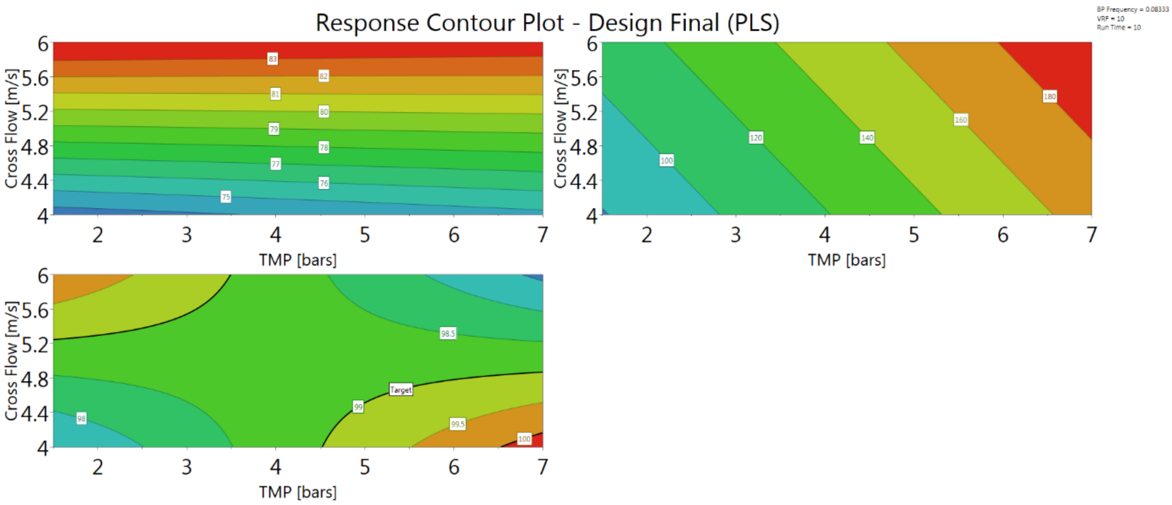

Figure.25) Response Contour Plot Showing preferable zones of the area with mentioned Responses 1) COD Reduction 2) Membrane Flux 3) Turbidity Reduction in a clockwise direction.

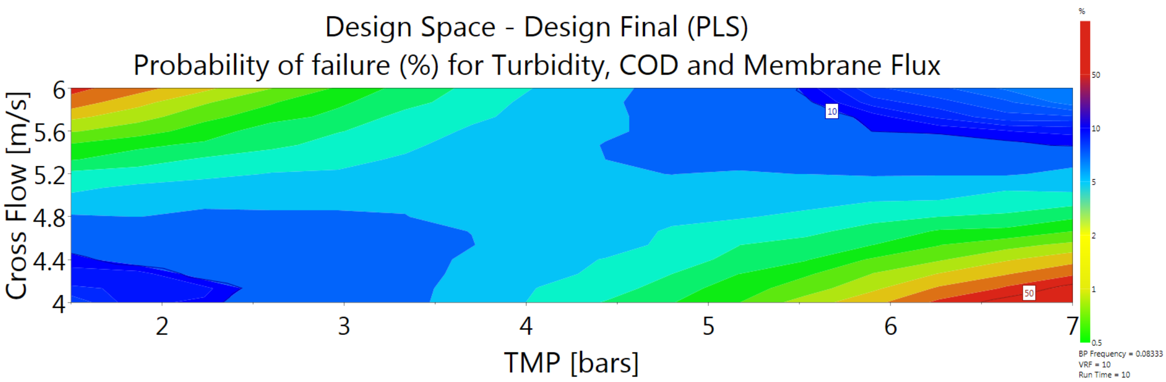

Figure.26) Design Space (PLS) Showing preferable zones of the area with mentioned Responses COD Reduction, Membrane Flux, and Turbidity Reduction.

Response Contour Plot showed above in Figure.25) illustrates the preferable zones, areas of optimum responses as for COD Reduction, the optimum zone only lies in conjunction with higher crossflow values preferably > 5.8m/s.

For Membrane flux both higher crossflow and higher TMP are preferable with a flux of 180 LMH and greater is achievable for Crossflow > 5.2 m/s and TMP >7 Bar.

In the case of Turbidity reduction, a clear-cut zone is demarcated by the contour Plot where the values of Turbidity reduction higher than 99 % are achievable.

Since discussed earlier as safety limit the experiments until a 10 percent probability of failure can be considered as do-able therefore the optimum Design Space of Factors is lined in until dark blue having multiple ranges, shown in Figure.26) however what is interesting is that the MODDE registers the failure zones having higher high TMP ϵ 6.5 - 7 Bar and Low Cross flow values of order 4 - 4.2m/s.

4.5) Optimization

As part of a project was aimed towards a final optimization of Membrane Flux which is considered as one of the most important responses w.r.t Water & Wastewater Treatment, so it was designed to optimize and evaluate the results. Since the Factorial tests resulted in a maximum achievable flux of 270 LMH the flux was set up to be maximized up to 300 LMH shown in Table.18) below and rest responses were targeted as usual and the Results that followed are presented across the Table.18) showed below, the software automatically selects the best experiment according to the least Probability of failure, However for experimental sakes, a 10 percent safety limit is allotted and can experiments which have a probability of failure won't be considered further, taking this into account this the results have an Experimental Success rate of > 50 % with the best possible flux scenario having a flux value of 211.885 LMH with 5.2 % probability of failure. The interactions influence of Factors in this for this particular Optimization was also considered and Factors were plotted in a form of a pie chart as Figure.27) which showed TMP as most impactful with 29.4 % closely followed by VRF with 27.5 % impact and Crossflow with 21 % while backpulsing Frequency with 14.5 % and Run time as least impacting Factor with a meagre 7.41%.

One trend came into notice was higher values of experimental Prob. of failure are directly are coherent higher Back Pulsing Frequency values which strongly suggests that backpulsing done more often is better w.r.t to Experimental Success, Also if compared against the statistics obtained from Factorial Experiments, better Flux and Turbidity Reduction are observed and shown in Table.15) of Section 4.1)Wastewater Factorial Test respectively.

Table.18) Optimization Criteria

Response	Criterion	Min	Target	Max	Pred. min	Pred. max	Graph
COD	Target	0	99	100	73.5263	84.1283	
Membrane Flux	Maximize	0	300		49.6158	211.885	
Turbidity	Target	0	99	100	92.089	100.433	

Table.19) Optimized Responses Worksheet according to the Maximized Flux criterion.

BP Frequency	VRF	TMP	Run Time	Cross Flow	COD	Membrane Flux	Turbidity	Iterations	Prob. of failure
0.166	5.12	6.44	16.39	4.67	78.46	159.94	96.85	32	1.5%
0.217	3.77	6.37	9.85	5.91	77.22	185.83	95.58	31	0.29%
0.114	8.38	5.79	5.2	5.29	79.86	170.30	98.01	35	4.4%
0.202	9.44	3.7	17.68	5.86	82.74	153.19	98.51	32	15%
0.174	2.8	7	3.6	4.2	79.91	181.94	97.96	32	9.8%
0.174	2.8	7	16.4	5.8	76.47	182.07	95.29	34	2.5%
0.174	9.2	6.45	16.4	4.6	77.56	158.82	98.43	31	12%
0.174	9.2	6.45	3.6	5.8	82.15	193.56	97.33	31	3.8%
0.777	2.56	4.98	16.55	4.72	78.56	146.78	97.85	36	2.9%
0.898	2.92	3.32	17.45	5.89	76.59	150.18	98.95	39	23%
0.927	9.96	4.43	18	4.41	76.22	137.89	97.60	31	10%
0.960	9.55	5.11	7.140	5.85	82.77	164.64	98.21	35	11%
0.908	2.8	6.45	16.4	4.6	78.97	158.82	98.88	43	20%
0.908	2.8	6.45	3.6	5.8	76.49	193.56	97.79	31	6.1%
0.909	9.60	6.66	6.06	4.97	79.08	182.35	98.90	38	20%
0.908	9.2	7	16.4	5.8	82.13	182.07	97.91	34	12%
0.592	6.27	6.94	11.46	5.30	79.02	183.09	97.79	35	2.2%
1	10	7	18	6	83.73	182.09	97.82	32	17%
0.0833	2	7	2	4	80.96	181.92	97.88	37	14%
0.0833	10	7	2	6	83.73	211.88	96.84	35	5.2%

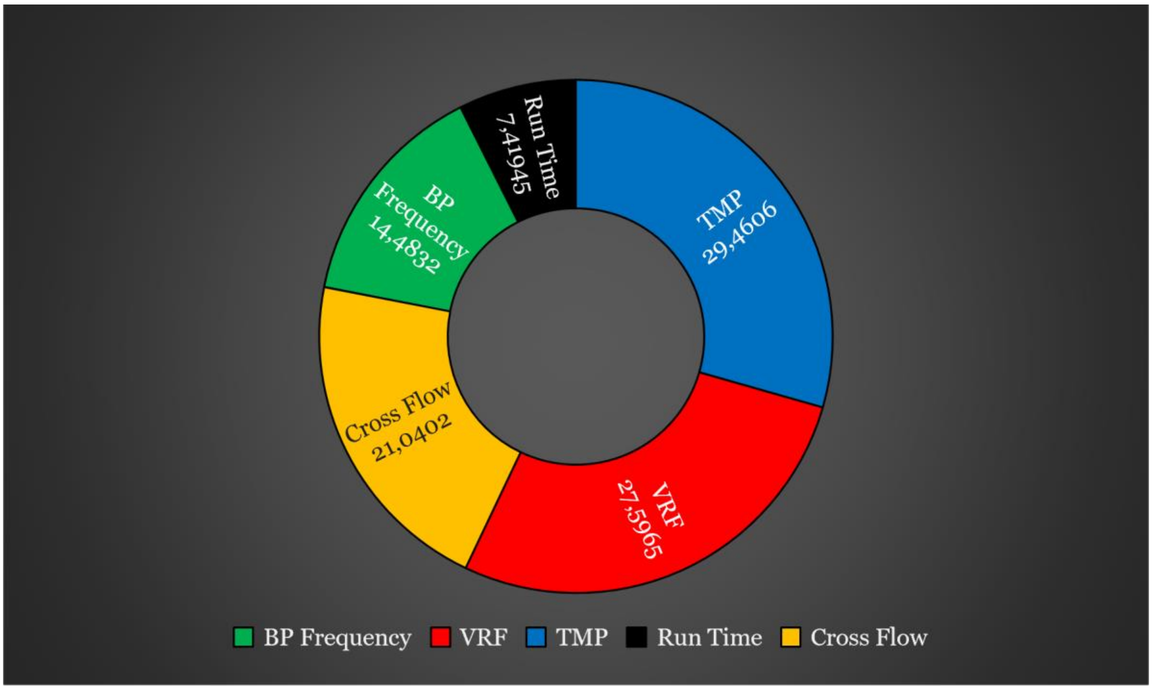

Figure.27) Factors & their Respective influence over experimental worksheet according to maximized flux criterion.

5) Conclusion & Recommendations:

5.1) Conclusion:

The Literature Study revealed the various configurations of the Membrane Filtration process highlighting the parameters influence and modifications needed to obtain good effluent quality results and optimize the cleaning performance of membranes employed in Wastewater Treatment.

The Wastewater Factorial Tests illustrated the highest obtainable flux of 274 LMH stable over longer durations of Runtime with a mean value of 140 LMH, COD reduction results were unstable with a huge variance which were found out to be resulting of high variance of Feed flow quality from Sedimentation Tank. The Turbidity Reductions were averaging about 98% reductions with the lowest value being 90.46%.

The model was fitted in MODDE according to PLS which showed less than significant fit for COD reductions, therefore, compromising the Prediction and optimization of COD Reductions for further simulations. However, it fits best for Membrane flux response and significantly good for Turbidity reduction response with Q2 values for Membrane Flux and Turbidity reductions being 0.64 and 0.23 thus having a significantly better predictive quality as to COD reduction Response.

With the help of Prediction plots and Contour plots zones of maximum performance were found out, w.r.t each corresponding factor.

Generally, the backpulsing was found out to be an insignificant factor over the experimental run time but there have been some experiments with lower run time where backpulsing was found to be having a significant effect, and flux increase was observable.

Finally, Optimization simulations were done for a case scenario where the Membrane flux response was maximized to a high value of 300 LMH and the highest obtainable value was 211.885 LMH, it also displayed the Factor influence with TMP, VRF, Cross Flow, BP Frequency and Runtime being the most influential factors for that particular optimization in respectively.

5.2) Recommendations

As a direct follow up of this thesis project, the best results and optimization configurations must be performed again with further cross-checks against predicted values and error reporting.

To exercise better control over Reduction Responses, Feed & Effluent sampling can be increased and if allowed to happen both after the start of the experiment and before the test end.

Inclusion of other influential factors investigated in the literature review, essential for the membrane cleaning process would result in a better understanding of Membrane Performance.

It would be interesting to verify the results by testing a similar factorial design with a similar membrane of different manufacturers, employing a different and stronger Cleaning agent.

A series of SDI tests should be performed on Optimized Factorial experiments generated by MODDE to follow up with earlier unfinished SDI tests and obtain working configuration.

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Appendixes

Appendix 1.) Cross Flow Calculations

	Velocity in the channel (m/s)										
	4,0	4,2	4,4	4,6	4,8	5,0	5,2	5,4	5,6	5,8	6,0
membrane design	**Volume flow [m³/h] per membrane element**										
10/6 single- channel (Ø 6 mm)	0,40	0,43	0,45	0,46	0,49	0,51	0,53	0,55	0,57	0,59	0,61
1/16 single- channel (Ø 16 mm)	2,9	3,1	3,2	3,3	3,5	3,6	3,8	3,9	4,1	4,2	4,3
14/8 single- channel (Ø 8 mm)	0,7	0,75	0,79	0,83	0,87	0,9	0,94	0,98	1	1,05	1,09
7/6 *multi- channel* (Ø 6 mm)	2,9	3,0	3,1	3,3	3,4	3,6	3,7	3,8	4,0	4,1	4,3
37/2 *multi- channel* (Ø 2 mm)	1,7	1,8	1,8	1,9	2,0	2,1	2,2	2,3	2,3	2,4	2,5
19/3.3 *multi- channel* (Ø 3,3 mm)	2,3	2,5	2,6	2,7	2,8	2,9	3,0	3,2	3,3	3,4	3,5
19/4*multi- channel* (Ø 4 mm)	3,4	3,6	3,8	3,9	4,1	4,3	4,4	4,6	4,8	5,0	5,15
19/6 *multi- channel* (Ø 6 mm)	7,7	8,1	8,5	8,8	9,3	9,7	10,0	10,4	10,8	11,2	11,6
37/3.8 *multi- channel* (Ø 3,8 mm)	6,0	6,34	6,6	6,9	7,3	7,6	7,8	8,2	8,4	8,8	9,1
61/2.5 *multi- channel* (Ø 2,5 mm)	4,3	4,5	4,7	4,9	5,2	5,4	5,6	5,8	6,0	6,2	6,5
85/3.3 multi- channel (Ø 3.3 mm)	10,5	11,0	11,5	12,0	12,6	13,1	13,6	14,1	14,6	15,1	15,7
19/8 *multi- channel* (Ø 8 mm)	13,7	14,4	15,1	15,8	16,5	17,2	17,9	18,6	19,2	19,9	20,6

Table.20) Membrane Cross flow Spreadsheet w.r.t allowable Volume Flow

For Calculating the amount of flow passing through the membrane with the help of regulating the flow velocity in the channel the above Table.20) is used, here type 7/6 Multi-Channel membrane channel is selected to undergo the pilot experiments and ranges for the Velocity across the channel as chosen during the factorial design would be 4-6 meters per second. However, the amount of Volume flow for the same range of Velocity is varying and in the pilot setup which is already outputting 1m^3/h of flow through the retentate pump the overall Value obtained from this table is deducted and the remaining Volume flow is converted into l/ min (units)of the cross flow pump on the Pilot plant.

Henceforth, adjusting the values of allowable cross flow by regulating the valve beside the pump ensures in maintaining that value throughout the entirety of the experimental duration.

Appendix 2.) CIP Ultrasil 25

ULTRASIL™ 25

PROPERTIES

Form	liquid	Alkalinity:	
Color	light yellow	Active as Na_2O	13.2%
Foam	none	Total as Na_2O	15.0%
Odor	chlorine	pH 1% solution	12.5
Specific gravity @ 68°F	1.250	Pounds per gallon	10.4
Available chlorine	2.6%	Kilograms per liter	1.2

Formula contains not more than 0.5% phosphorus, 0.18 gms. per gal. average recommended use concentration.

TO USE

For normal CIP cleaning of RO/UF membrane systems, prepare a 0.5–1.0% solution or 7–13 ounces ULTRASIL 25 for each 10 gallons (55–102 mls/10 l) of water. Circulate throughout the system for 20 minutes at a temperature of 120°–140°F. (49°–60°C). Note: observe the recommendations of the manufacturer of the membrane material for maximum temperature.

Consult your Klenzade representative for specific use instructions and recommended dispensing equipment.

For cautionary and first aid information, consult the Material Safety Data Sheet (MSDS) or product label.

Figure.28) Ultrasil 25 Chemical Properties from (Swat.net.au, 1992)

Ultrasil 25 is a liquid, chlorinated detergent developed by ECOLAB specifically for cleaning chlorine resistant reverse osmosis and Ultrafiltration Membranes.

Its main purpose is to remove fouling agents from chlorine tolerant RO & UF membranes while ensuring a uniform membrane flux thus enhancing membrane life which ensures quick penetration of soil protein layer and resulting in the removal of protein build-up (Swat.net.au, 1992).

Secondly, since it’s in a liquid-form it allows easier access to the cleaning process reducing the time allowed to dissolve the cleaning agent providing a one-step circulation procedure making the whole process easier and faster (Swat.net.au, 1992).

Thirdly it doesn’t need an additional solvent such as water, therefore, it can be ideally used by its own however considering economical and just usage of cleaning agent it is typically diluted to a certain limit before usage (Swat.net.au, 1992).

Appendix 3.) PLS Design Data

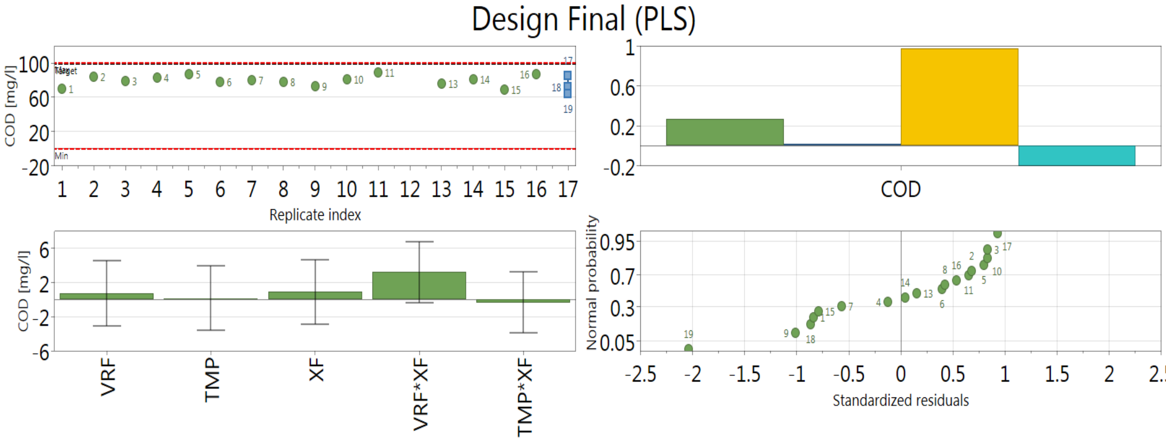

Figure.29) Designed Model elements COD

Going by the Designed model elements for COD model fit shown above through Figure.29) displays the replicate index where highest possible reductions were observed for the Experiment No. 2,5,11 and 16 respectively and while standardized residuals display the ratio of a difference between observed vs the expected count to the expected count of the standard deviation of the expected count, which can be troublesome to understand however the Positive Standardized residuals offer better-understanding insights into the experiments hence in the case of COD model the experiments no. 14,13,6,8,16,2,5,3,10 offered better COD results that were similar to what was expected by the outcome of the experiment however the perfect results were obtained in case of Experiment No.17). While the Experiments with the negative standardized residuals such as no.4,7,15,1,18,9 offered worse results respectively with the worst result being of experiment no.19. Also, for Coefficient plot the VRF* XF was the only coefficient that seemed to have a signature model term.

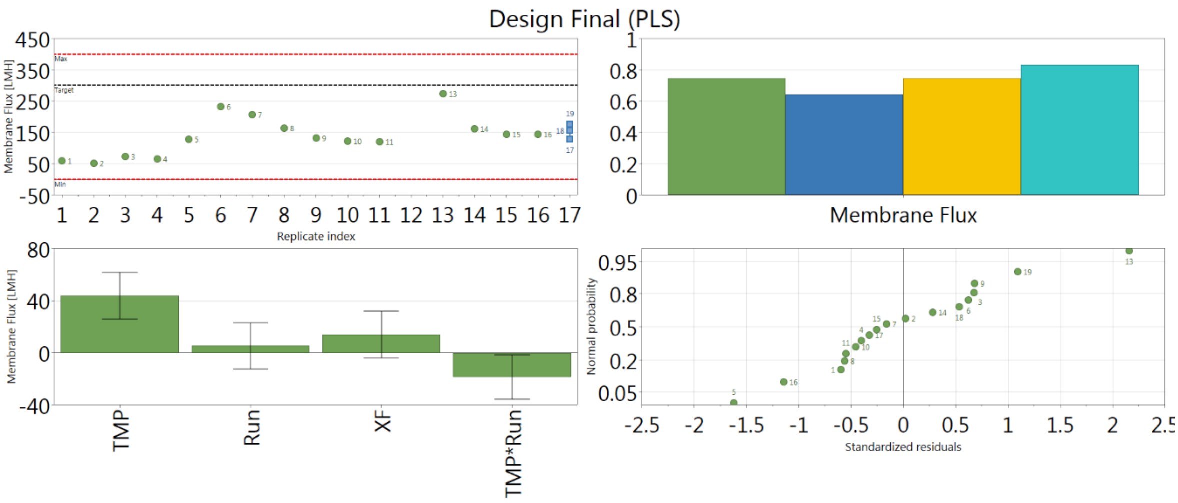

Figure.30) Designed Model elements Membrane Flux

The model fit for Membrane flux was the best observable fit across all the model response fits for all the responses with highest observable values for experiment no.6,7,13 and 19 respectively as all these values were more than 150 LMH. Analyzing Standard Residuals is similarly as with COD the best-case observable was for experiment no.13 with Flux rating of 274 LMH while the lowest was experiment no.5 with a meager 50 LMH Flux. For Coefficient analysis both the TMP & TMP*Run were found out to be significant model terms. Model Produced overall a good fit and all the values of R2 & Q2 were >0.6 which is explainable based on the Standardized Residual lying mostly between -0.5 to 0.75.

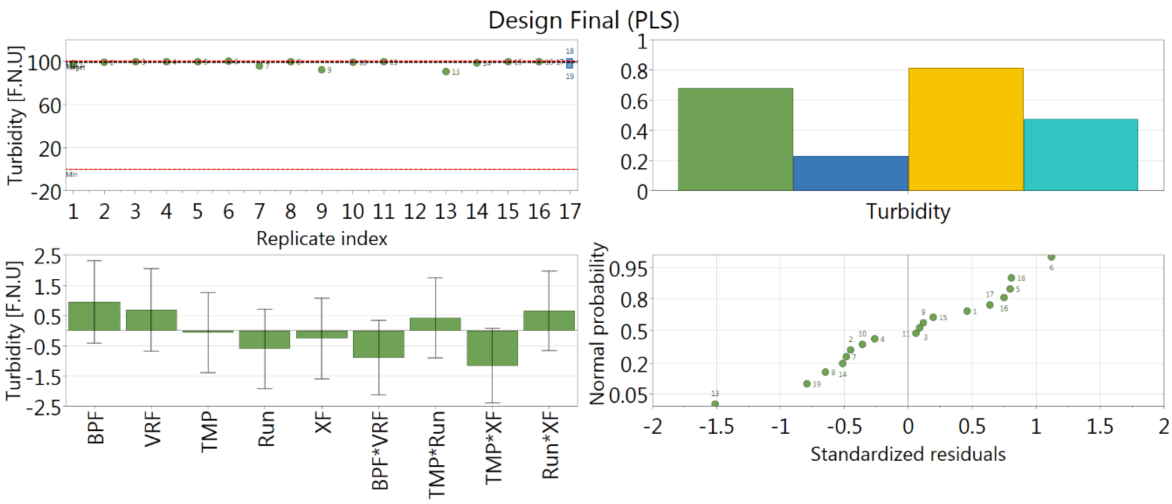

Figure.31) Designed Model elements Turbidity

Turbidity Reduction model offers us an in detail that most of the reductions are lying close to the maximum set limit reductions with permeate turbidity reduced up to 1 FNU. Which marks and qualifies the permeate free of suspended solids, most microorganisms, and colloidal solutions that we come across in typical municipal wastewater feed. One thing additionally could be taken form the turbidity model is that despite varying feed of input wastewater the results were expected uniform reductions barring some exceptions mainly across experiments no.9 & 13. Briefly glancing over the standardized residuals gives the information of typical distribution with lying in the range of -0.5 & 0.75. While the single most significant term amongst the coefficient chart is TMP*XF.

Table.21) Additional Model Statistics for Membrane Flux

Membrane Flux	*DF*	*SS*	*MS (variance)*	*F*	*p*	*SD*
Total	18	415323	23073.5			
Constant	1	356062	356062			
Total corrected	17	59261.2	3485.95			59.0419
Regression	4	44264.5	11066.1	9.59274	**0.001**	105.196
Residual	13	14996.7	1153.59			33.9646
Lack of Fit	11	13816.2	1256.02	2.12793	**0.363**	35.4403
(Model error)						
Pure error	2	1180.51	590.254			24.2951
(Replicate error)						
	N = 18	Q2 =	0.642	Cond. no. =	1.231	
	DF = 13	R2 =	0.747	RSD =	33.96	
	Comp. = 2	R2 adj. =	0.669			

Annexure 4.) Plots

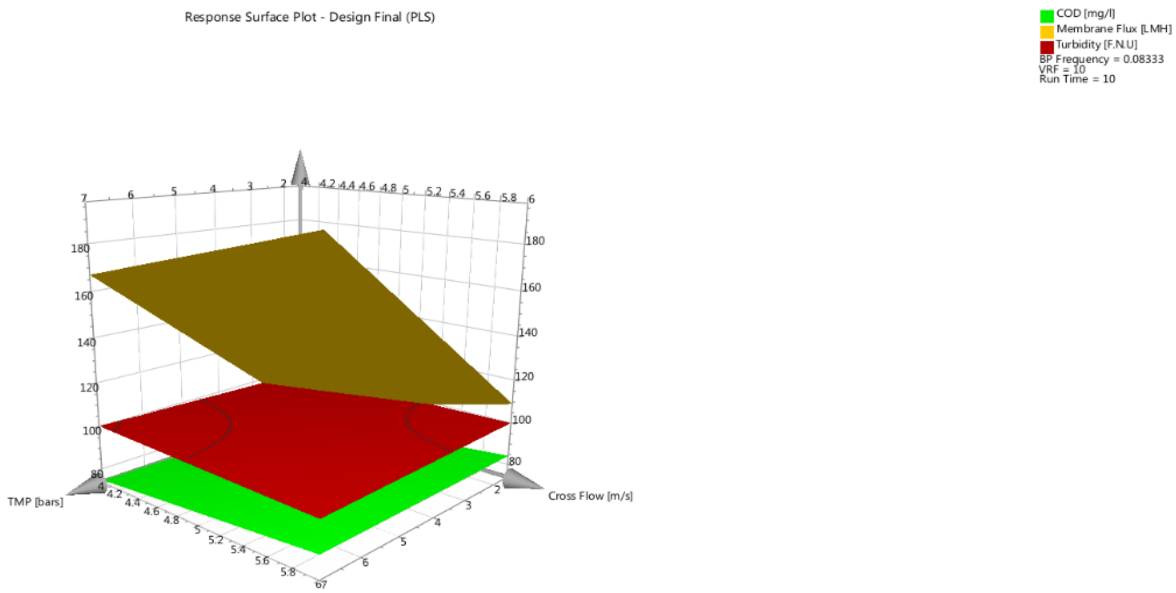

Figure.32) Response Surface Plot (PLS) of COD, Membrane Flux & turbidity vs TMP, and Cross flow on three respective axes.

The above-mentioned Figure.32) displays the 3D surface graph which plots COD, Membrane flux & Turbidity responses in colors green, brownish-yellow and red respectively at Back pulsing Frequency of 5 minutes, VRF 10 and Runtime of 10 hours. It develops from the results of the factorial experiments discussed in Section 4.X) earlier. High pressure & low XF corresponding towards better COD & Turbidity reductions but suboptimal Membrane Flux response. While maximum Membrane Flux could be obtained with a combination of high TMP along with high XF. The essential thing to be taken from this plot is that it’s not possible to maintain a record high reductions and better flux values all in one possible configuration.

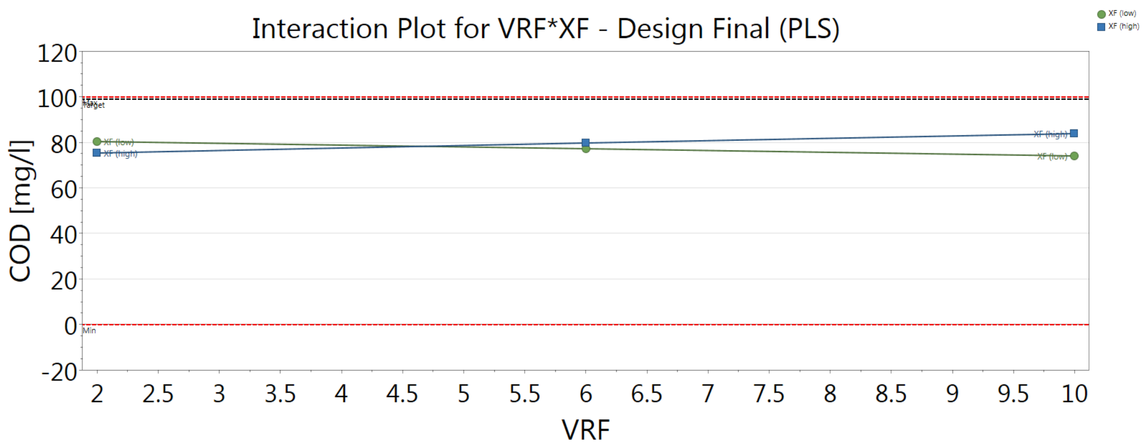

Figure.33) Interaction Plot COD vs VRF*XF

Shown above through Figure.33) an interaction plot that shows the low and high cross values varied plotted across varying VRF values. Both high and low configurations offer a difference of about 5 % reductions in COD at lower VRF and about 10% at higher VRF values respectively. The common point is achieved at a VRF 4.75. Although the higher XF is preferred across all other configurations for sake of uniformity and maximizing response results across all responses higher XF & VRF values are preferable with an estimated COD reductions of about 82%.