ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

FORWARD OSMOSIS MEMBRANE BIOREACTOR PERFORMANCE IN WASTEWATER TREATMENT

M.Sc. THESIS

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Department of Environmental Engineering

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

İLERİ OSMOZ MEMBRAN BİYOREAKTÖRÜN ATIKSU ARITIM PERFORMANSI

YÜKSEK LİSANS TEZİ

Hajar GHASEMZADEH MOMEN
(501121805)

Çevre Mühendisliği Anabilim Dalı

Çevre Biyoteknolojisi Programı

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Hajar GHASEMZADEH MOMEN, a **M.Sc.** student of ITU **Graduate School of Science, Engineering and Technology** student ID **501121805**, successfully defended the **thesis** entitled "**FORWARD OSMOSIS MEMBRANE BIOREACTOR PERFORMANCE IN WASTEWATER TREATMENT**", which she prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

Thesis Advisor:	**Prof. Dr. Suleyman OVEZ** İstanbul Technical University	.....
Jury Members:	**Prof. Dr.İsmail KOYUNCU** İstanbul Technical University	.....
	Dr.Murat EYVAZ Gebze Technical University	.....

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To my lovely father and patient mother

FOREWORD

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JUNE 2015

Hajar GHASEMZADEH MOMEN
(Agriculture Engineer)

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ABBREVIATIONS

CA	: Cellulose Acetate
COD	: Chemical Oxygen Demand
EPS	: Extracellular Polymeric Substances
F/M	: Food To Microorganism
FO	: Forward Osmosis
FTIR	: Fourier Transformation Infrared Spectroscopy
FOMBR	: Forward Osmosis Membrane Bioreactor
HF	: Hollow Fiber
HRT	: Hydraulic Retention Time
IMBR	: Immersed Membrane Bioreactor
MBR	: Membrane Bioreactor
MWCO	: Molecular Weight Cutoff
MF	: Microfiltration
MLSS	: Mixed Liquor Suspended Solids
NF	: Nanofiltration
RO	: Reverse Osmosis
SEM	: Scanning Electron Microscopy
SMP	: Soluble Microbial Products
SRT	: Sludge Retention Time
SS	: Suspended Solids
SVI	: Sludge Volume Index
TFC	: Thin Film Composite
TKN	: Total Kjeldahl Nitrogen
UF	: Ultrafiltration
VSS	: Volatile Suspended Solids

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FORWARD OSMOSIS MEMBRANE BIOREACTOR PERFORMANCE IN WASTEWATER TREATMENT

SUMMARY

Water shortage is a most critical issues in our world. In 21st century, interest of water is getting higher step by step in light of expanding population and constrained supplies. Treatment and reuse of accessible water is a vital issue.

Likewise, treatment and reuse of available water is an important issue for providing water. Subsequently, it is important to recover water from utilized water or wastewater to beat the water lack issue through cutting edge innovations, for example, reverse osmosis process and membrane bioreactor. At present, membrane innovation is favored for wastewater recovery as a result of its high contaminant dismissal and water efficiency. Forward osmosis membrane bioreactor (FO-MBR) is a blend of forward osmosis (FO) and membrane bioreactor (MBR) to treat wastewater. It requires lower energy when compared to the conventional MBR, because of using natural process.

This work concentrated on imaginative utilizations of osmotically-driven membrane process.

The forward osmosis (FO) procedure is a membrane process that uses the natural osmosis process for water transport from a dilute solution to a concentrated solution over an exceptionally high selective membrane. Forward osmosis membrane bioreactor (FO-MBR) which is the blend of FO and MBR procedure is as of late examined and proposed as an option strategy to treat wastewater as a result of its lower energy necessity and lower membrane fouling with the ordinary MBR. While MBR uses suction power to create effluent, FO-MBR uses an osmotic driving power produced by the draw solution, transporting water through the FO membrane. The transportation of water dilutes the draw solution. At the point when the draw solution is adequately diluted, a post-treatment process, in current study reverse osmosis (RO) is using, could be utilized to reconcentrate the draw solution for reuse in the FO process and at the same time deliver high quality water. It has been reported that both reversible and irreversible membrane fouling were not severe in FO process (Cornelissen et al., 2008).

The novel forward osmotic membrane bioreactor (FOMBR) framework for water reuse was displayed. Test results exhibited high reasonable flux and moderately low reverse diffusion of solutes from the draw solution into the activated sludge.

In current frstudy, according to (Achilli et all., 2009) draw solution is selected NaCl with the conductivity of 50 ms/cm, by using the flatsheet polyamide TFC-FO membrane for FO section and using the Filmtec Dow XLE 4440 RO membrane for RO pocess. The lab scale FOMBR has been operated in 13 cycles, each cycle contain 42 hour for FO process, 2 hour for RO process. It is nice to mention that there is not any waste it this system, because of the circulation in RO system, which is cuse to reconcentrate the draw solution for reuse in the FO process and at the same time produce high quality water.

In this study focused on membrane performance in selective condition, analysis the FO effluent water and also investigate the microorganisms and membrane characterization.

Microbial diversity of the FO-MBR system has been very microscopically monitored and defined. It has been found that gram negative and gram positive bacteria were dominant and survived in the system. There were also some eukaryotic cells such as ciliated and attached protozoa, some nematodes and rotifers in the system. They were used to resist 5-7 mS/cm concuctivity values and very effective removing organic carbon, nitrate and phosphate. It can be said that microorganisms in the activated sludge were euryhaline resisting broad range of conductivity (salinity) concentrations. Filamentous microorganisms, *Nostocoida limicola* and some fungi filamentous forms have been observed under the selective conditions. The floc structure of the activated sludge was very compact, dense and settling very well. Because of the high salinity values, we may conclude as flocs have got bigger and made net structures on the membrane as a blanket so they have not caused pore fouling.

As a result the FO-MBR system was found to remove greater than 85% of organic carbon, 96% phosphate removals and 79% nitrate. Also FO membrane characterization analysis did not express significantly fouling during the operation in FO membrane.

İLERİ OSMOZ MEMBRAN BİYOREAKTÖRÜN ATIKSU ARITIM PERFORMANSI

ÖZET

Su sıkıntısı dünyamızın en kritik problemlerinden birisi olarak gün geçtikçe kendisini göstermektedir. Suya olan ihtiyaç gün geçtikçe artan nüfus ve azalan temiz su kaynaklarının azalması ile 21.nci yüzyılın en önemli problem olarak ortaya çıkmaktadır. Kullanılabilir yüzey sularının arıtımı ve tekrar kullanımı önemli hayati önem taşıyan konuların başında gelmektedir.

Bu problem ilave olarak, suyun kontaminasyonu onun amacına uygun olarak kullanımında yeni problemler ve eksiklikler meydana getirmektedir. Bu problemin giderilmesi, suyun arıtılarak yeni teknolojilerin, metotların ve buluşların, örneğin ters osmoz ve membran biyoreaktör prosesleri gibi, su sıkıntısını gidermede kullanılması büyük önem taşımaktadır. Günümüzde membran teknolojilerdeki gelişmeler ile membranların atıksuyun tekrar kullanılmasında istenmeyen kirletici parametrelerin atıksudan uzaklaştırılmasında etkili ve verimli olarak kullanılabileceğini göstermektedir. Bu yeni uygulamalar içerisinde ileri osmoz membran biyoreaktör (Forward Osmosis Membrane Bioreactor, FO-MBR) ileri osmoz (FO) ile membrane biyoreaktör (MBR) proseslerinin bir karışımıdır ve bu tez çalışmasında kullanılmıştır. İleri osmoz doğal bir proses olarak konvansiyonel ters osmoz membran biyoreaktöre göre daha az enerji gereksinimi gösterir.

Bu tez çalışmasında ozmotik basınç ile çalışan membran proses üzerine yoğunlaşılmıştır.

İleri osmoz (FO) bir membran proses olup doğal osmozu kullanarak suyun az yoğun bir ortamdan daha yoğun bir ortama doğru oldukça seçici bir membrandan geçirerek taşınmasıdır. FO ile MBR proseslerinin bir karışımı olan FO-MBR prosesi daha az enerji ihtiyacı ve konvansiyonel ters osmoz membranlarına göre daha az tıkanma göstermesi bakımından bu çalışmada tercih edilmiştir. Ters osmoz suyu emme gücünü kullanarak taşımasına karşılık, FO-MBR suyu ileriye doğru osmozu kullanarak taşır

ve membrandan geçirir. Suyun taşınması draw çözeltisinin (suyun taşındığı çözelti kısmı, çıkış suyu) konsantrasyonunun azalmasına neden olur. Draw çözeltisinin (çıkış suyunun) kalitesi istendiği seyrelme durumuna geldiğinde işlem amacına ulaşmış olmaktadır. Bu çalışmada ters osmoz çıktı çözeltisi FO sisteminde draw çözeltisinin yoğunlaştırılmasında kullanılmak aynı zamanda çıkış suyu çözeltisi içerisinde konsantrasyon azaltılarak istenilen kaliteye ulaşılmaktadır.

Suyun yeniden kullanımında yeni uygulanan ileri ozmotik membrane biyoreaktör (FO-MBR) sisteminin taslak yapısı burada verilmektedir. Deneysel sonuçlar draw çözeltisinden aktif çamura doğru oldukça iyi kalitede çıkış suyuna ulaşılabildiğini ve katıların tersine difüzyonunun normal olarak sağlanmadığını göstermiştir.

Bu çalışmada 50 mS/cm iletkenlik değerine sahip NaCl çözeltisi draw çözeltisi olarak, TFC-FO düz tabaka membranı FO bölümünde ve Filmtec Dow XLE 4440 RO membranı RO bölümünde kullanılmıştır. Laboratuvar ölçekli pilot FO-MBR sistemi 13 devir (cycle) işletilmiş ve her bir devirde 42 saat FO prosesi ve 2 saat RO prosesi olarak işletilmiştir. Burada herhangi bir atık veya süzüntü suyu çıkmaması önemli olup süzüntü suyu (permeate) tekrar RO sistemine döndürülerek sirküle edilmektedir. Burada amaç hem süzüntü suyu çıktısını önlemek hemde DS (draw solution) konsantre edilerek akının kendisine doğru osmoz ile yönlenmesi FO prosesinde tekrar kullanımı sağlanmakta ve böylece daha yüksek kalitede su temin edilmektedir.

FO-MBR sistemi mikrobiyal çeşitliliği mikroskobik olarak izlenmiş ve tanımlanmıştır. Gram negatif ve gram pozitif bakterilerin sistemde baskın olduğu ve tuzlu ortamda hayatiyetlerini sürdürebildiği anlaşılmıştır. Aktif çamurda ayrıca ökaryotik hücrelerinde varlığı tespit edilmiş özellikle silli ve saplı protozoalar, nematodlar ve rotiferlerin sistemde bulunduğu izleme çalışmalarında görülmüşlerdir. Bu mikroorganizmaların 5-7 mS/cm iletkenlik değerlerinde yaşayabildiği, organik karbon, nitrat ve fosfatı yüksek verimlilikle arıtabildiği ölçülerek bulunmuştur. Bu organizmalara farklı ve geniş tuzluluk değerlerine dayanarak yaşayabildiklerinden geniş tuzluluk değerlerinde yaşayabilen mikroorganizmalar (euryhaline) denebilir. Aktif çamur içerisinde filamentli bakteriler ve filamentli mantar hücreleride tespit edilmiş özellikle *Nostocoida limicola* bakterisi ve bazı filamentli mantarlar seçici koşullarda sistemde gözlenmiştir. Aktif çamur floklarının yapısı oldukça düzgün, sıkı ve iyi çöken özelliklere sahip olduğu tespit edilmiştir. Yüksek iletkenlik değerlerinde

flokların daha sıkı ve büyük olması membran porlarının üstüne bu flokların bir ağ gibi kapatması ve porların içinin biyolojik olarak tıkanmaya sebep olmamışlardır.

Bu çalışma ile belirli seçilmiş koşullarda membran performansı FO çıkış suyu kalitesinin ölçülerek izlenmesi ve aynı zamanda biyoreaktördeki aktif çamur mikroorganizmaların yapısı, koşullara uyum ve adaptasyonu ve membranların tıkanmasına neden olan etkilerinin araştırılması hedeflenmiştir.

Bu araştırma sonucunda FO-MBR sisteminin organik karbonun % 85'ini, fosfatın % 96'sını ve nitratın % 79'unu giderdiği bulunmuştur. Ayrıca FO membranının bu işletim koşullarında önemli bir biyolojik tıkanıklılık (biyofouling) belirtisi göstermediği yapılan membran analiz sonuçlarından anlaşılmıştır.

1. INTRODUCTION

1.1 Importance of The Study

Water scarcity is one of the most important problems of the world. In 21st century, as the worldwide population and industry development, freshwater is turning out to be progressively. As of late, and membrane innovation is broadly considered and applied in water and wastewater treatment to produce clean water. In wastewater treatment field, MBR is a favored decision contrasted and customary treatment forms on account of the it's various preferences, e.g., higher effluent quality, little foot shaped impression, less over abundance activated sludge generation, and so on. (Wisniewski, 2006; Matošićet al., 2008; Wen et al., 2010).

The forward osmosis (FO) procedure is a membrane process that uses the natural osmosis process for water transport from a dilute solution to a concentrated solution over an exceptionally high selective membrane. Forward osmosis membrane bioreactor (FO-MBR) which is the blend of FO and MBR procedure is as of late examined and proposed as an option strategy to treat wastewater as a result of its lower energy necessity and lower membrane fouling with the ordinary MBR. While MBR uses suction power to create effluent, FO-MBR uses an osmotic driving power produced by the draw solution, transporting water through the FO membrane. The transportation of water dilutes the draw solution. At the point when the draw solution is adequately diluted, a post-treatment process, for example nanofiltration (NF) or opposite osmosis (RO), could be utilized to reconcentrate the draw solution for reuse in the FO process and at the same time deliver an astounding item or high quality water. It has been reported that both reversible and irreversible membrane fouling were not severe in FO process using activated sludge as a feed (Cornelissen et al., 2008).

1.2 Mission and Scope of the Study

The population of the world continues to increase in the other side the fresh water is in scarsity condition, so wastewater treatment and reuse it is one of the most importent issues for scientists. Membrane filtration advances have different applications, for example, supplement or supplanting with routine procedures for removing of particulate and organic matter. Additionally, they are integrated with bioreactors, which called as membrane bioreactors (MBRs) and applied for wastewaters reuse (Mansouri et. al., 2009).

In current study has been operated Forward Osmosis Membrane Bioreactor (FO-MBR) because of the high effluent quality of water and energy saving.

The main objective of this study is to investigate the feasibility of FOMBR in treating wastewater into high quality water. The study includes FO membrane performance and using membrane characterization and produced water quality evaluation. The main scope of this research is the treatment performance of FOMBR, the contaminant removal efficiency, and produced high water quality.

2. LITERATURE REVIEW

2.1 Membrane Technology

Importance of "membrane" as word is a parcel limit between two stages. In other way, we can describe membrane as a "channel" which can have the ability to breaking point the vehicle of different segments in a particular manner (Wang et. al. 2010). Membranes procedures are not a late development. Membranes were firstly presented as scientific devices at research centers. Than, they formed into mechanical items and routines (Strathmann et. al., 2006). Mechanical utilizations of manufactured membranes were begun in the 1960s. Anyway, the most punctual study about film phenomena was recorded at the center of the eighteenth century (Strathmann et. al., 1985). Recorded improvements of layer innovation are given in Table 2.1.

Table 2.1 : Historical developments of membranes (pre-1980s) (Wang et. al., 2010).

Year	Development
1784	'Osmosis', permeation of water
1833	Diffusion of gases law
1855	Phenomenological diffusion laws
1860-1880s	Osmotic pressure, semi permeable membranes
1907-1920	Microporous membranes
1920s	Reverse osmosis prototype
1930s	Electrodialysis membranes
1950s	Microfiltration, hemodialysis, ion exchange membranes
1963	Reverse osmosis membranes
1968	Spiral-wound reverse osmosis modules
1977	Thin film composite membranes
1970-1980	RO, UF, MF, electrodialysis membranes
1980s	Industrial gas separation membranes processes
1989	Submerged membrane (membrane bioreactor)

It can be see that the membrane science and innovation have been experienced for a long stretch of advancement in research facility studies. Indeed, even with these studies and applications, membrane innovation has still more space for development to fulfill future applications (Wang et. al., 2010).

2.1.1 Membrane separation technology

Membrane serves as a hindrance in the partition handle that hinders the undesirable particles and broke down solutes, and permits the littler particles or just water to pass through, contingent upon the kind of membrane utilized. There are two membrane filtration designs, to be specific dead-end and cross-stream filtrations (Fig. 2.1). In a dead-end filtration prepare, the influent liquid stream sets out oppositely to the membrane surface and the solutes store on the membrane surface. Occasional interference of the procedure is required to clean or change the membrane because of pore blocking and cake development by the solutes. In a cross-stream membrane filtration transform, the influent liquid streams parallel to the membrane surface. The solutes that store on the membrane surface are sheared off by the influent stream, with productivity that depends on the cross-stream speed. The layer fouling of cross-stream filtration is ordinarily less extreme contrasted and the dead-end filtration.

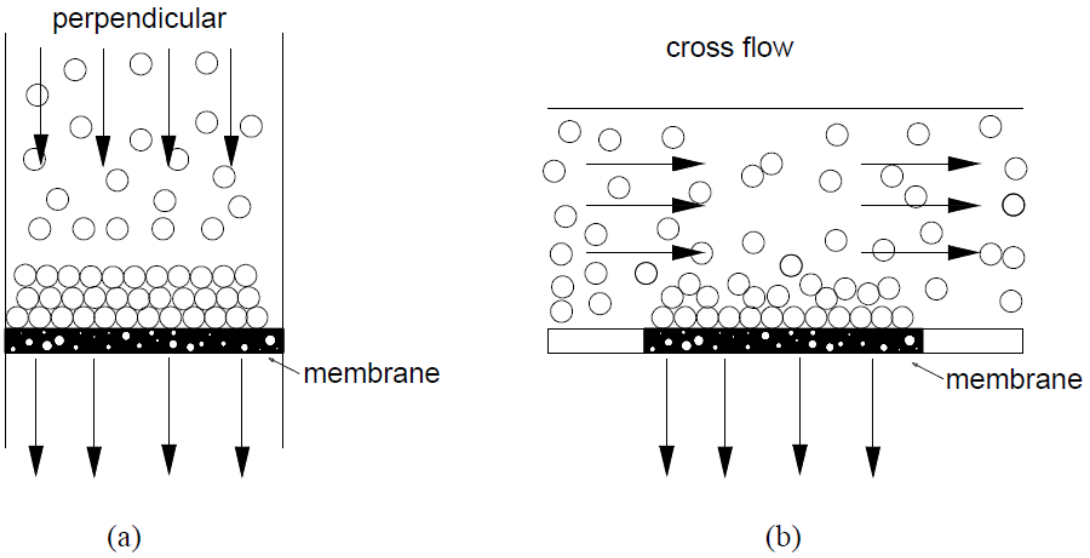


Figure 2.1 : Schematic diagram of two different configurations for membrane filtration: (a) dead-end; and (b) cross-flow. (Zangh, 2011).

2.1.2 Membrane types

A membrane can be characterizedbasicly as a material, which permits components to go through in it. A few components may be more promptly go through from membrane than others as a result of membranes' perm-selectivity. The level of selectivity and penetrability relies on upon the pore attributes (size, appropriation, porosity) of the membrane. Contingent upon the pore structure, weight driven membranes can be characterized by their pore sizes as four key gatherings which are

reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF) layers (Judd, 2011), widely used in different fields. Tables 2.2 and 2.3 show the comparison of membrane structures, materials and characteristics (Cheremisinoff, 2002).

Table 2.2 : Comparison of membrane structures.

Technology	Structure	Driving Force	Mechanism
Microfiltration (0.02-10 um)	Symmetric microporous	Pressure, 1-5 atm	Sieving
Ultrafiltration (1-20 nm)	Asymmetric microporous	Pressure, 2-10 atm	Sieving
Nanofiltration (0.01-5 nm)	Asymmetric microporous	Pressure, 5-50 atm	Sieving
Reverse Osmosis	Asymmetric with homogeneousskin and microporous support	Pressure, 10-100atm	Solution diffusion

Table 2.3 : Membrane materials and characteristics for different type of membrane.

Technology	Materials	Polar Character
Microfiltration	Polypropylene (PP)	
	Polyethylene (PE)	Non polar
	Polycarbonate (PC)	Non polar
	Ceramic (CC)	Non polar
Ultrafiltration	Polysulfone (PSUF)	Non polar
	Dynel	Non polar
	Cellulose acetate (CA)	Non polar
Nanofiltration	Polyvinylidene (PVDF)	Polar
Reverse Osmosis	Cellulose acetate	Polar
	Polayamide	Polar
	Nylon	Polar

- **Microfiltration (MF)**

Ordinary pore size going of MF membranes are somewhere around 0.05 and 10 µm. On account of their pore sizes, MF membranes have high porousness and can be worked in low weight. They can be created from distinctive materials (polymeric, inorganic) furthermore its structure can be symmetric or deviated (Fane et. al., 2011).

- **Ultrafiltration (UF)**

Ordinary pore size running of UF membranes is from 1 to 100 nm. With these sizes, evacuation of bacterias, infections, colloids, and macromolecules from water is

conceivable. Molecular weight cutoff (MWCO) is the mulecular weight of the solute which is commonly in the scope of 1-300 kDa for UF membranes. In the event that MWCO has greater size than this extent, dismissal capacity of layer can be low and pore size can increment. Penetrability has an extent somewhere around 20 and 500 L m^{-2} h^{-1} bar^{-1} and the typical working weight is by and large around 1-5 bar (Fane et. al., 2011).

- **Reverse osmosis (RO)**

Pores of RO membranes are subnanometer and they can uproot little natural particles furthermore broke up particles (counting monovalent particles as Na+ and Cl-). Also, partition properties of RO membrabes are determined as water porousness and sodium chloride dismissal.

RO membranes are partitioned into two fundamental gathering as ocean water RO (SWRO) membranes and saline water RO (BWRO) membranes.

SWRO membranes have high sodium chloride dismissal (>99%) at the same time, low water porousness. Likewise they require high weights. BWRO membranes have low sodium chloride dismissal (>95%) yet higher water porousness and lower working weight than SWROs (Fane et. al., 2011).

- **Nanofiltration (NF)**

NF membranes have similitude with ROs, for example, holding capacity of disintegrated particles and some little natural atoms. NFs have low dismissal rate to monovalent particles or ions, for example, Na (10–90%). Yet, in the event that we contrasted NF and RO, NFs have better water permeabilities at critical low weights. (Fane et. al., 2011).

2.2 Forward Osmosis and Reverse Osmosis Process

2.2.1 Forward osmosis process

Osmosis is characterized as the draw in of water through a highly selective membrane by osmotic pressure of the solutions on both sides. Osmosis pressure happens by a distinction in amassing of the arrangements in the both side of the layer. Today utilizations of the osmosis wonder enhance from water treatment and nourishment handling to power era and novel techniques for controlled medication discharge (Cath et al., 2006). A standout amongst the most vital procedures in nature is osmosis process. It is fascinating to acquainted with osmosis process which are happen in nature to point of comprehend the utilizing osmosis prepare in innovation. The osmosis process happens when a low concentration of saline solution will tend to transport to a high concentration of saline solution. There are some simple examples of osmosis processing in nature such as the mechanism of the plant roots when absorb the water from the soil and our kidneys when absorb the water from our blood (Url-2).

For instance, on the off chance that you had a compartment brimming with water with a low salt fixation and another holder loaded with water with a high salt (NaCl) focus and they were isolated by a semi porous membrane then the water with the lower salt (NaCl) focus would start to relocate towards the water holder with the higher salt fixation (Url-2).

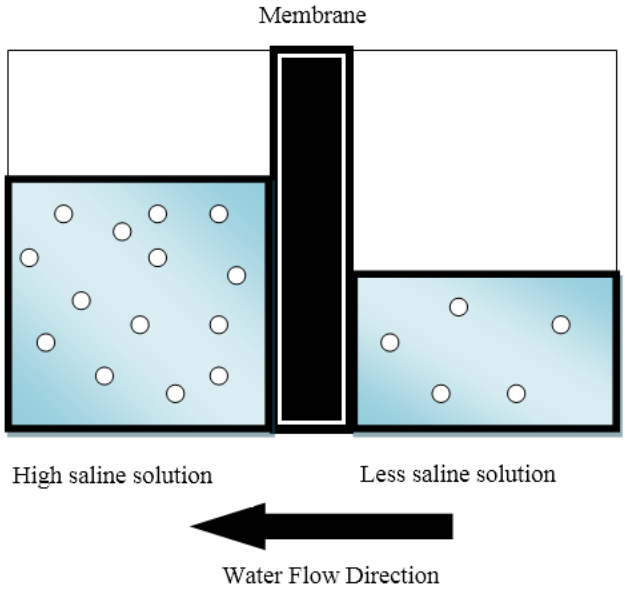


Figure 2.2 : Schematic diagram of forward osmosis.

- **Forward osmosis in membrane technology**

FO membranes role is so important in FO process. The FO membrane is a high selective barrier between two different solutions in concentration of salt, only water molecules allow pass through the membrane. A semi permeable membrane is a membrane that will allow some atoms or molecules to pass but not others. Due to this, after the transportation of the water, the low concentration solution becomes concentrated and the high concentration solution becomes diluted. As a result, the water flux will reduce with time because the effective osmotic pressure across the membrane reduces.

By proceeding in FO process, the difference of the solutions in the concentration becomes decrease. Finally, two solution concentrations reach to about same level in the end of the process (Zangh, 2011).

FO membrane part is so imperative in FO process. The FO membrane is a high specific hindrance between two distinctive solutions in centralization of salt, just water particles permit go through the layer. A semi permeable membrane is a membrane that will permit a few particles or atoms to pass yet not others. Because of this, after the transportation of the water, the low concentration solution gets to be thought and the high concentration solution gets to be weakened or diluted. Therefore, the water flux will diminish with time because the powerful osmotic pressure over the membrane lessens.

By continuing in FO process, the distinction of the solution in the concentration gets to be reduction. At last, the two solution concentrations scope to about same level toward the end of the process (Zangh, 2011).

Recently, in the purpose of wastewater reuse, membrane technologies improve significantly in the field of Forward Osmosis Membrane Technology (Kerusha et. al., 2014).

There are some advantages in wastewater treatment technology with FO process, when compared with conventional membrane processes, such as reverse osmosis (RO), ultra filtration (UF), nano filtration (NF), and micro- filtration (MF), (Huayong et. al., 2014).

FO process in water reuse technology have some advantages such as reductions in operational costs or less energy consumption, reduced lifetime costs and sustainably

in quality of the product water or effluent and reliably produces high quality product water, even in the most challenging conditions. FO has the potential to maintainable treat wastewater sources and produce high quality water (Kerusha et. al., 2014).

- **Osmosis pressure**

As before said, osmosis is as the transport of water through a semi permeable membrane caused by a difference in osmotic pressure of the solution on both sides of the membrane, also the osmosis pressure is a effective element to water flux. Figure 2.3 defined osmosis pressure with increasing in pressure and without adding any pressure.

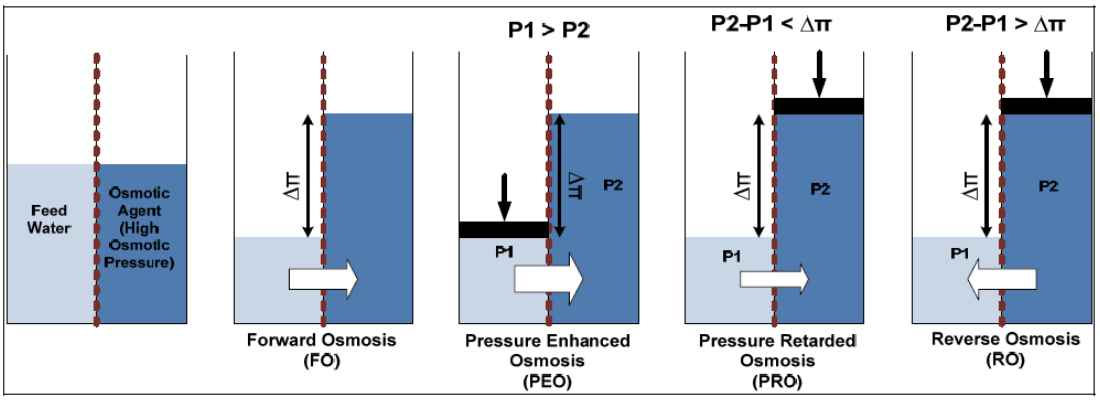


Figure 2.3 : Osmosis pressure (Nicoll et. al. 2013).

In a FO process, draw solution (DS) is used as a high saline solution and feed solution is used as a less saline solution, so water transport from feed solution to DS. Draw solution is one of the key considerations in FO process. As the different chemical compounds such as NaCl, KCl, $MgCl_2$, $CaCl_2$, $MgSO_4$, KN, NH_4HCO_3, with the different concentration generate different osmosis pressure, draw solution selective will be effective in the FO experiment. According to Achilli et. al. study, NaCl solution is recommended for draw solution because of high solubility and relatively easy to reconcentrate to high concentrations and low cast of NaCl (Achilli et. al., 2009).

Figure 2.4 expresses the relationship between DS concentration and osmosis pressure.

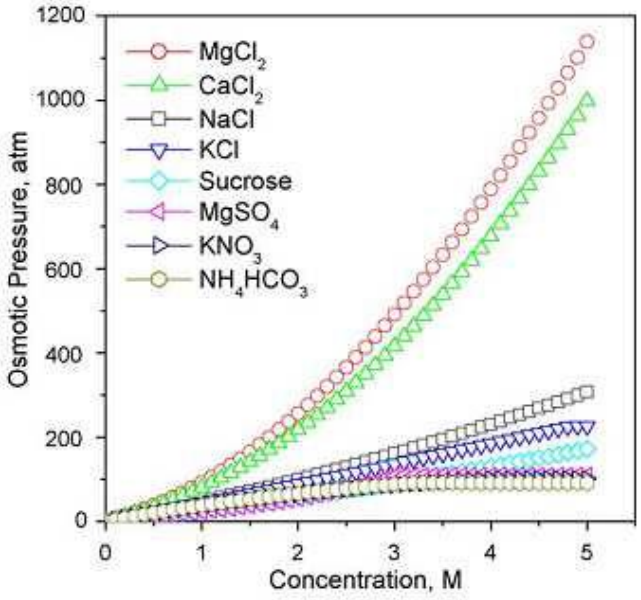


Figure 2.4 : Relationship of osmotic pressures and DS concentration at 25°C (Cath et al., 2006).

A solution osmotic pressure depends on the concentration of dissolved ions in solution and the temperature of solution (Cath et. al., 2009).

TheVan't Hoff equation is using to calculate osmosis pressure (Hoff, 1887) as :

$$\pi = RT\sum iM \qquad \textbf{(2.1)}$$

Where *i* is the dimensionless Van't Hoff factor for the specific ion, *M* is the molarity of the specific ion, *R* is the gas constant (0.08206 $L{\cdot}atm{\cdot}mol^{-1}{\cdot}K\text{-}^{1}$), and *T* is the temperature in Kelvin.

It is worthwhile to mention that, what forward osmosis can do:

It can dilute a solution of higher osmotic pressure with a solution of lower osmotic pressure.

It can concentrate a solution of lower osmotic pressure with a solution of higher osmotic pressure (Nicoll et. al, 2013).

Forward osmosis is used as a "pre-treatment" to reverse osmosis process in wastewater treatment technology with membranes.

2.2.2 Reverse osmosis process

Reverse Osmosis is require to apply vitality or energy, so reverse osmosis are the procedure of osmosis in opposite. As before notice that, osmosis happens in nature without vitality needed, to invert the procedure of osmosis it needs to apply pressure to the more saline solution, in result water transport from less saline answer for more saline . A reverse osmosis membrane is a specific layer that permits to transport of water atoms however not the greater part of broke down salts, organics, microorganisms and pyrogens. It is important to apply energy to reverse osmosis process the utilized energy is more noteworthy than the normally happening osmotic pressure keeping in mind the end goal to desalinate (demineralize or deionize) water simultaneously, permitting pure water through while keeping down a larger part of contaminants.

- **Reverse Osmosis in Membrane Technology**

Membrane technology by using reverse osmosis (RO) is successful to remove a large majority of contaminants from water by pushing the water under pressure through a high selective membrane.

Reverse osmosis (RO) process in membrane technology has been widely expanded for water treatment and reuse. For reverse osmosis process using a high pressure pump to increase the pressure on the salt side of the RO and force the water across the semi-permeable RO membrane, leaving almost all (around 95% to 99%) of dissolved salts behind in the reject stream (Url-2).

Figure 2.5 express the process of Reverse Osmosis. When pressure is applied to the concentrated solution, only water molecules transport through the high selective membrane but the contaminants are not allowed to transport through the membrane.

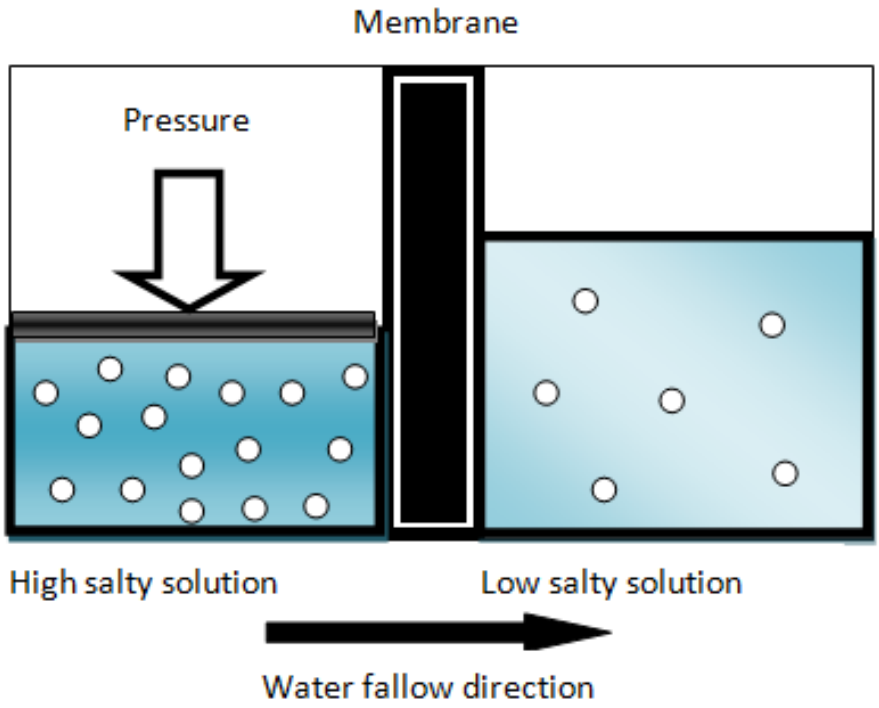


Figure 2.5 : Schematic diagram of reverse osmosis.

Figure 2.6 is a simple diagram that shows how an RO system works im membrane process. As the feed water pumped to RO membrane under enough pressure the water molecules pass through the semi-permeable membrane and the salts and other contaminants are not allowed to pass and goes to drain. The water that makes it through the RO membrane is called permeate or product water and usually has around 95% to 99% of the dissolved salts removed from it (Url-2).

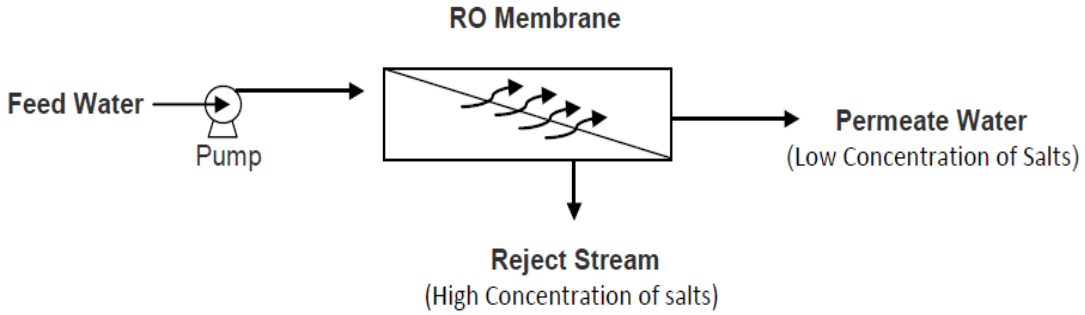


Figure 2.6 : A simple diagram of RO system (Url-2).

2.2.3 FO process versus RO process in membrane technology

There are two main advantages over conventional FO process in versus of RO process lower fouling propensity and lower energy consumption (Nicoll et. al., 2013).

2.2.4 Forward osmosis membranes vs reverses osmosis membrane

Membranes intended for forward osmosis needs to have rather diverse qualities contrasted with customary reverse osmosis films. A slender dismissal layer and a support layer with high porosity and low tortuosity. There are similitudes regarding geometry in that they can be of winding injury, flat sheet, tubular or hollow fiber.

There are preferences and disfavors to each of the distinctive geometries and it is the field of utilization that will direct the most suitable membrane design, despite the absence of by and large financially accessible forward osmosis membranes. In spite of the fact that it is satisfying to note that this circumstance is starting to change as more membrane suppliers enter the business sector, as the potential for forward osmosis based procedures is starting to be figured it out (Nicoll et. al., 2013).

2.3 Membrane Bioreactor

2.3.1 History of MBR

Membrane bioreactor is one of the most popular technologies in wastewater treatment; because of a desirable effluent of MBRs. Membrane bioreactor is the combination of the membrane separation technology and biological treatment process. Bioreactor acts as a biological treatment processor and the membrane is used as a filter in the filtration process, thus the MBRs products high quality of water that is not only low in organic or mineral contaminants, but also free from bacteria, pathogens, and viruses. In MBR technology two configurations of MBRs are using, which are submerged or internal and side-stream or external system. MBR configurations are shown in Figure 2.7.

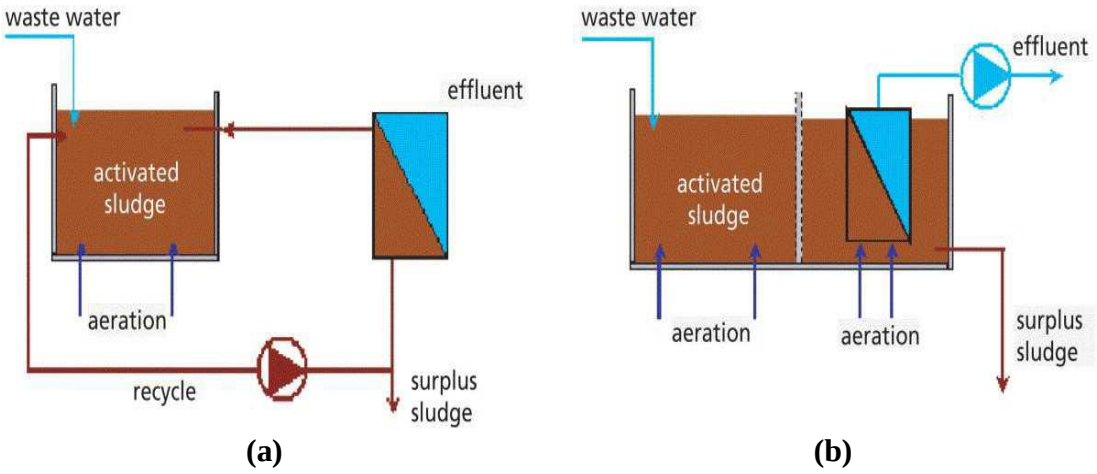


Figure 2.7 : MBR configurations: (a) side-stream; and (b) submerged (Oever, 2005).

Table 2.4 : The largest 20 MBR plant (May 2010) (Judd, 2011).

Project	Technology	Date	PDF ($103 m^3/d$)
Shending River, China	BOW	2010	120
Wenyu River, China	Asahi K/BOW	2007	100
Johns Creek, GA	GE Zenon	2009	94
Beixiaohe, China	Siemens	2008	78
Al Ansab, Muscat, Oman	Kubota	2010	78
Peoria, AZ	GE Zenon	2008	76
Cleveland Bay, Australia	GE Zenon	2007	75
Sabadell, Spain	Kubota	2009	55
San Pedro del Pinatar, Spain	GE Zenon	2007	48
Syndial, Italy	GE Zenon	2005	47
Broad Run WRF, VA	GE Zenon	2008	47
Beijing Miyun, China	MRE	2006	45
NordKanal, Germany	BOW	2004	45
Tempe Kyrene, AZ	Asahi K/BOW	2006	44
Brescia, Italy	GE Zenon	2002	42
Traverse City, MI	Siemens	2004	39
Linwood, GA	Kubota	2007	38
North Kent Sewer Authority, MI	GE Zenon	2008	35
Jinqiao Power, China	GE Zenon	2006	31
Dubai Sports City, UAE	Kubota	2009	30

2.3.2 Advantages and disadvantages of MBR

MBR contain a higher concentration of MLVSS in the mixed liquor than conventional activated sludge (CAS) process, high MIVSS cause to have better performance of biological process in bioreactor, thus organic degradation and removal will be higher in MBRs.

As the membrane technology is replaced of secondary clarification in the CAS, the MBRs need fewer footprints than CAS process, because membrane was used in place of secondary clarification in the CAS process. Therefore, settling problems such as sludge bulking and foaming do not affect treatment.

The excess activated sludge produced by the MBR is fewer than CAS process, which cause to lower cost of treatment and disposal of the excess sludge. In the other word MBRs sludge retention time is high, because of utilizing the membrane separation process to separate the suspended solids from the water.

The effluent quality of MBR is high in terms of turbidity, bacteria, particular and colloidal organic matter.Thus, only water and those particles with smaller size than the membrane pore size can penetrate the membrane and be present in the final effluent (Mutamim et. al., 2013).

Membrane fouling and high cost capital is the key disadvantage of MBRs, which is an obstacle in widespread use of MBRs in wastewater treatment. Membrane fouling affects adversely on membrane technology such as flux reduction. In order to minimize the fouling of the membranes, the additional aeration is used to clean the membrane surfaces in the MBR it is also causing high cost in system.

MBR system needs external suction pump to provide trans membrane pressure (TMP) to produce the effluent. Thus pumping energy is another energy requirement by the MBR system (Choi et. al., 2005).

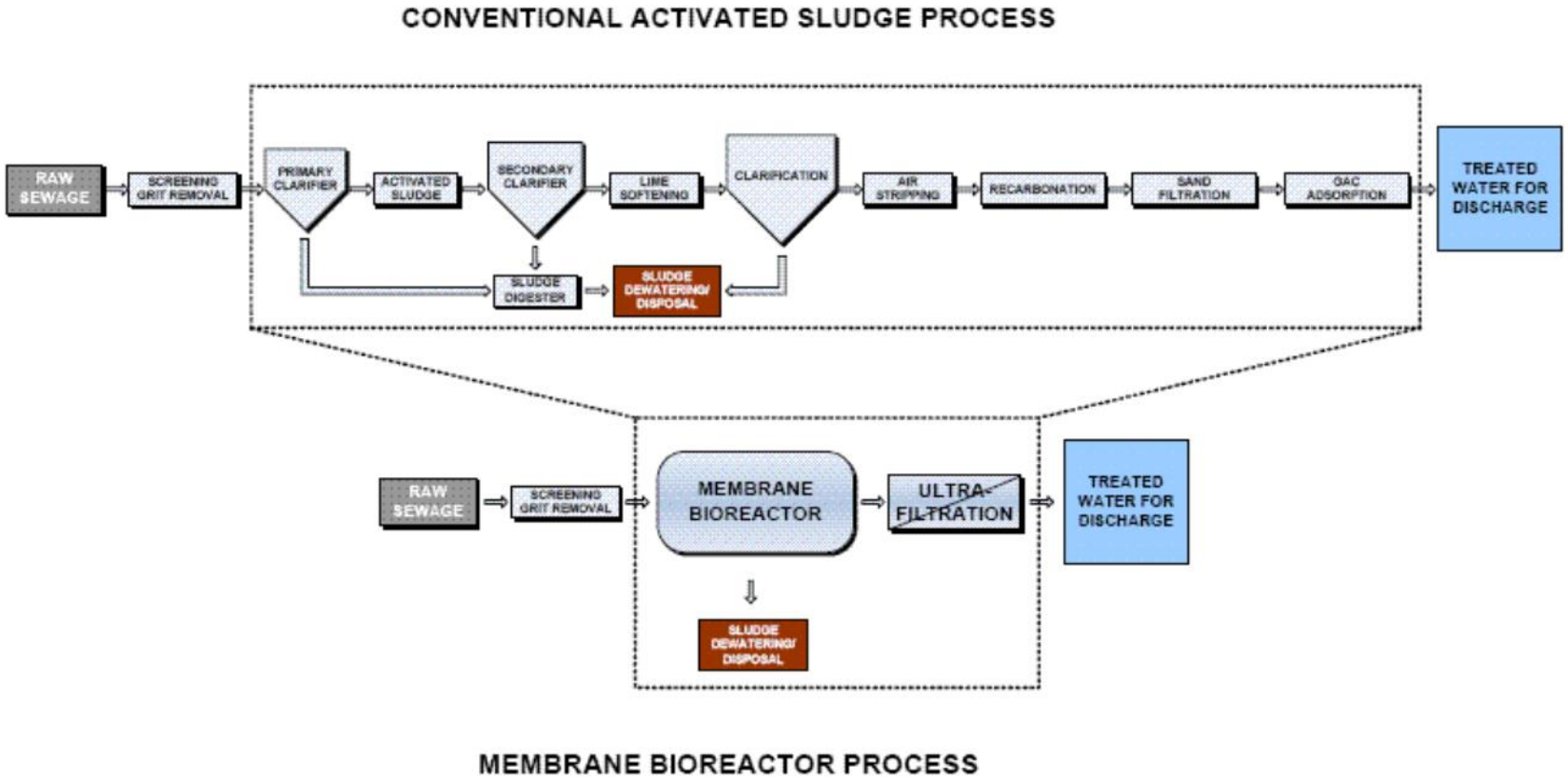


Figure 2.8 : MBR and conventional activated sludge processes (MANN+HUMMEL Group).

2.3.3 Fouling in membrane bioreactors

Fouling is a complex mechanism and not entirely understood. All operations and environment conditions are related to eachother and these parameters can change the fouling in many ways. Basicly, it depends on the features of feed solution (nature, concentration and pHect.), properties of membrane (hydrophobicity, charge, roughness, pore size, porosity) and operating conditions (temperature, pressure, cross-flow velocity).

The major problem of membrane processes is fouling is caused by solute adsorbing irreversibly or reversibly on the surface of the membrane or with in the pores of the membrane. It usually causes serious decline in the flux and quality of the permeate ultimately resulting in an increase in the operating pressure with time (Mutamim et. al., 2013; Matin et. al., 2011).

There are various accumulation types on membrane surface such as adsorption, pore blockage, gel/cake layer formation that are cause to fouling. Schematic views of major fouling mechanisms are givenin Figure 2.9.

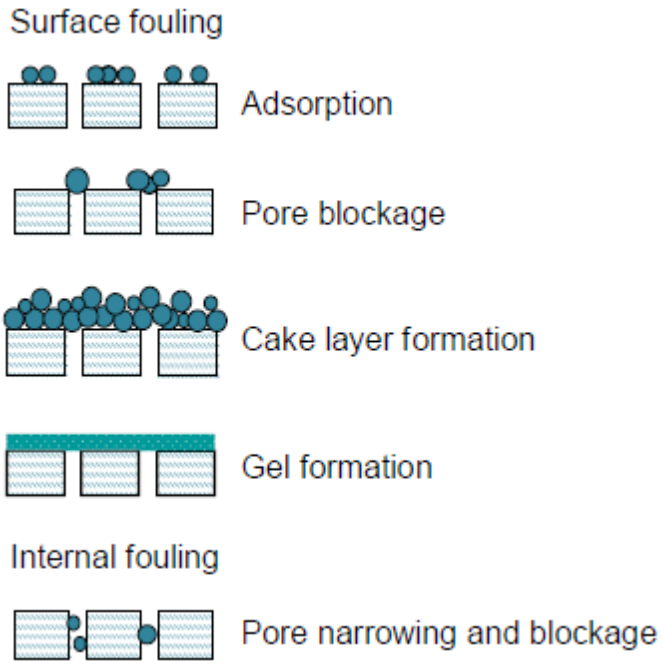


Figure 2.9 : Major fouling types (Yavuz, 2014).

If particules are smaller than the pores, they can enter into the pores. If particle size is about same or bigger than pores, they can be adsorbed to the membrane surface and

may accumulate onto it. Therefore, pores are blocked and obviously, it affects the membrane seperation negatively.

If these particals are cleanable by physical means, it is classified as reversible fouling. If it is not because of the adsorption, it is called as irreversible fouling (Mutamim et. al., 2013).

In other study fouling is classified into two main groups according to fouling material types as scaling (colloidal, organic, inorganic) and biofouling (microbial/biological) (Sillanpää, 2014).

There is a direction relationship between mixed liquor suspended solids (MLSS) and fouling potential. High viscosity decreases membrane permeability. Operating under critical flux can prevent the fouling. Also, there is an inverse relationship between hydraulic retention timeand membrane fouling decreases, when HRT decrease membrane fouling increases. In addition, SMP and EPS are important parameters of fouling mechanism for example, bound EPS influences on specific cake resistance. Especially, SMP is correlated with fouling rate (Menga et. al., 2009; Sillanpää, 2014).

2.3.4 Biofouling

Biofouling can be characterized as an undesired gathering of microorganisms at membrane that may happened bydeposition, development of microorganisms or flocculation. By and large, bacterias are gathered by connection (bioadhesion, bioadsorption) or development. These amassing on layers can be characterized as biofilm membrane. Thus, biofilms are generally made from membrane out of alive and dead microorganisms and their related extracellular items (Guo et. al., 2012).

It can be said that the cell biomass and the extracellular polymeric substances (EPS) reason biofouling. The extracellular polymeric substances (EPS) in microbial organization in corporate charged gatherings (carboxyl, phosphoric, sulfhydryl, phenolic and hydroxyl) and polar gatherings (aromatics, aliphatics in proteins and hydrophobic regions in sugars). Due to the availibility of hydrophilic and hydrophobic gatherings, EPS can be characterized as amphoteric. What's more, this component is critical for microbial totals and their development in

membranebioreactors. The hydrophobic areasin EPS may give the adsorption of natural contaminations (Guo et. al., 2012). EPS are divided into two groups:

- Bound EPS
- Soluble EPS (SMP)

Bound EPS are eliminated by bacterial hydrolysis. Dissolved products of bound EPS are called as soluble EPS in other name soluble microbial products (SMP) that are biodegradable. Each have various organic macromolecules such as polysaccharides, proteins, humic substances, nucleic acids, (phospho) lipids and other polymeric compounds (Guo et. al., 2012).

EPS are directly related with specific cake resistance. If EPS is high, cake resistance will be high, too (Mutamim et. al., 2013). Therefore, it is clear that some microorganisms in the sludge play a significant role in membrane biofouling. Understanding of bioflocculation behaviour and mechanisms of cell attachment in MBRs will be the key component for the biofouling control (Menga et. al., 2009). There are various factors that related to biofouling of MBRs. Table 2.4 describe some important factors and their relationships with biofouling on MBRs.

2.3.5 MBR design parameters

Hydraulic retention time (*HRT*) is calculated as (Url-1). For overall HRT, the reactor volumes are divided by influent flow rate (*Q*):

$$HRT = \frac{\sum_i V_i}{Q_x} \tag{2.2}$$

The solids retention time (*SRT*), which is also called sludge age, is calculated as follow:

$$SRT = \frac{\sum_i X_i V_i}{Q_x X_x} \tag{2.3}$$

whereV_i is individual reactor volume (m^3), X_i is MLSS in each reactor (mg/L), Q_X is excess biosolids removal rate (m^3/d) and X_X is MLSS in the excess biosolids flow (mg/L).

While range SRT is typically 20 days or so, it can go down to 12-15 days.

Organic loading rate is an important design and controlling parameter in biological wastewater treatment process. It is measured by the amount of food provided to a unit amount of biomass (or reactor volume) for a unit period of time.

The F/M is calculated by using the following equation:

$$F/M = \frac{QS_0}{XV} \quad \textbf{(2.4)}$$

Where F/M is food-to-microorganism ratio (g BOD/g MVLSS/day), Q is influent flow rate (m^3/day), S_0 is influent BOD (mg/L), X is MLSS in aeration tank (mg/L) and V is tank volume (m^3).

Table 2.5 : Comparison of preferred ranges of operating parameters in MBR and CAS (Url-1).

Design Parameter	Unit	MBR	CAS
F/M	g BOD/g MLSS/day	0.04-0.12	0.16-0.24
	g COD/g MLSS/day	0.08-0.24	0.32-0.48
	g BOD/g MLVSS/day	0.05-0.15	0.2-0.3
	g COD/g MLVSS/day	0.1-0.30	0.4-0.6
F/V	g BOD/L/day	0.5-1.5	0.6-0.9
	g COD/L/day	1.0-3.0	1.2-1.8
MLSS	g/L	8-12	2-4
MLVSS	g/L	6-10	1.7-3.4
SRT	days	10-30	5-10
SOUR	mg O_2/g MVLSS/hr	2-5	6-12
OUR	mg O_2/L/hr	15-50	20-40
DO	mg/L	1-2	1-2

2.4 Forward Osmosis Membrane Bioreactor (FO-MBR)

Forward osmosis membrane bioreactor (FO-MBR) procedure is a blend of FO procedure and MBR procedure to treat the utilized water into high quality water. A further reconcentration procedure is obliged to reuse the draw solution and produce fresh water.

As indicated in Figure 2.8, the FO membrane module is submerged into the bioreactor and the bioreactor is worked correspondingly to the customary MBR. The contrast between the traditional MBR and FO-MBR is the distinctive membrane modules utilized. The traditional MBR uses a submerged MF or UF membrane

module and outside pressure is utilized to produce the effluent from the mixed liquor. On account of FO process, an attract solution is utilized to course through the FO membrane module in the reactor (Figure 2.10).

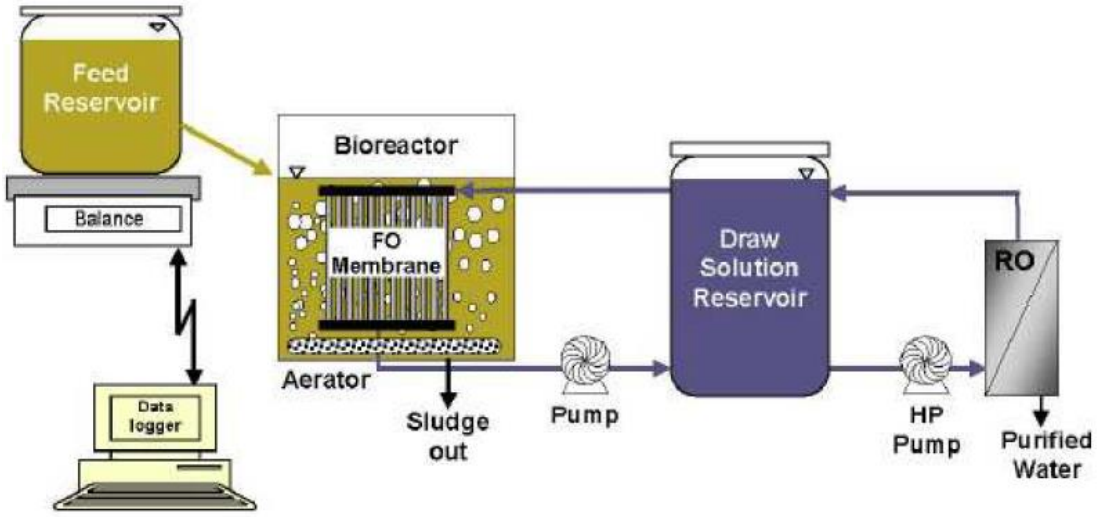


Figure 2.10 : Diagram of FO-MBR process with RO process following (Achilli et. al., 2009).

Through the FO procedure, water particles or molecules are drawn from the mixed liquor into the draw solution and the diluted draw solution will take after back into the D.S tank. After adequate water is drawn through the FO process, the volume of the draw tank increments and the concentration of it reductions. A reconcentration procedure can be utilized to concentrate freshwater from the diluted draw solution (DDS) while permitting the attract solute to be reused in the FO process.

2.4.1 Advantages and challenges

The key preferences of FO-MBR incorporate high contaminant removal proficiency, and low energy prerequisite, low membrane fouling inclination.

A couple of analysts have reported that the fouling of FO membrane is insignificant and reversible (Cornelissen et. al., 2008; Achilli et. al., 2008; Mi and Elimelech, 2009).

The key challenge of FO-MBR includes selection of a suitable draw solution. A suitable draw solution is important for FO process because it is a more effective parameter to water flux.

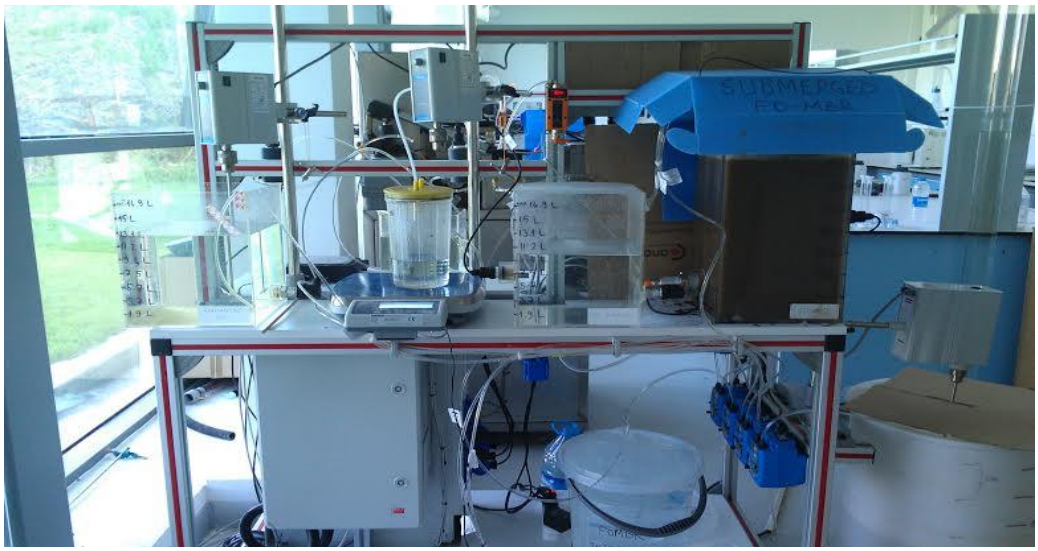
3. METHODS AND MATERIALS

3.1 Introduction

This study was performed in a National Research Center of Membrane Technology which is called MEM_TEK. The MBR system consist of five containers which are called (1) W.W. tank by effective volume of 90 L, a level sensor was applied to maintain the water level constant in the reactor. (2) Bioreactor tank, which is contain the module of TFC_FO membrane, activated sludge, also consisted of a completely mixed aeration. The activated sludge tank was 48 cm, 24 cm, and 24 cm, with an effective volume of 28 L., (3) D.S tank, (4) RO tank, (5) Permeate tank. All the containers are shown in the System diagram in Figures 3.1 and 3.2. The Lab Scale MBR was fed from synthetic domestic W.W. The W.W is made by following compounds: Glucose ($C_6H_{12}O_6$), Urea(CH_4N_2O), (K_2HPO_4), (($NH_4)_2$ SO_4), (Na_2CO_3), As before mention that, the system is equipped to a bioreactor, For launching the MBR, bioreactor filled with activated sludge a wastewater treatment plant (taken from Pasakoy, one of the W.W treatment plants in Istanbul). For reaching the MLSS concentration to 9-12g/L, the activated sludge was feed with nutrient material of synthetic WW. An air diffuser beneath the membrane module was used for aeration and mixing the liquor. The membrane-filtered effluent was continuously removed with a pump to transfer the effluent (DDS) to DS tank for 42 hour, this process call FO process. In the end of the FO process the DS was enough diluted, so employ the RO process to obtain the high effluent which is called ultimate effluent or permeate.

The MBR was operated in 13 cycles, each cycle took 44 hour, 42 hour for FO process +2 hour for RO process. It is interesting to mention that there is not any waste in this lab scale MBR, as there is a circulation in RO system so in the end of the RO process we have some concentrate water. By using concentrate water and permeate can be made the DS solution.

(a)



(b)

Figure 3.1: Lab scale FO MBR (a) RO system (b) FO system.

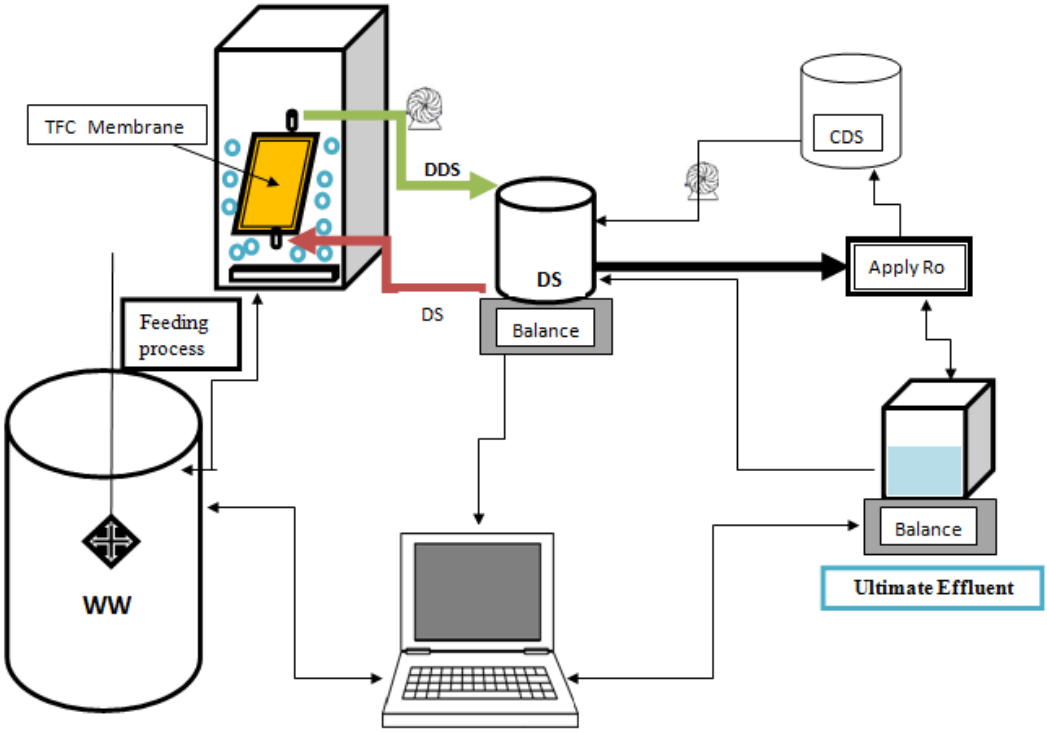


Figure 3.2: Schematic diagram of lab scale FO and RO process

3.2 Materials

3.2.1 Membrane material

The FO membrane used for this research, provided by Hydration Technology Innovations (HTI, Albany, OR, USA). These membranes are unique compared to other semi-permeable membranes (e.g., RO membranes), and have been determined to be the best available membrane for current FO applications (T.Y. Cath, et. al. 2006).TFC membranes with the active layer in the surface, gives the membrane a higher flux.

The RO membrane used is an aromatic polyamide membrane produced for desalination of brackish water by Dow-Filmtec (Midland, MI, USA) under the name BW30. The molecular weight cutoff (MWCO) of the membrane was reported as 98Da, with a sodium chloride rejection of 99.5%, a contact angle of 48° for the virgin membrane, and a zeta potential of -6.1mV at pH 8 (Nghiem and Coleman 2008).

The FO membrane used was a nonporous membrane with excellent solute rejection and was able to retain more than 99% of NaCl solute (Tan and Ng. et. al. 2010). The

porous layer of the membrane faced the draw solution and the selective layer faced the feed solution.

3.2.2 Draw solution

A key consideration in developing an OMBR system is selection of an appropriate DS. The main criterion is that the DS has a higher osmotic pressure than the feed solution. Another important criterion in some FO applications is the availability of a suitable process for reconcentrating the draw solution after it has been diluted in the FO process. Very often, a NaCl solution is selected because it has high solubility and is relatively easy to reconcentrate to high concentrations using a conventional desalination process (e.g., RO or distillation) without risk of scaling (Jellinek and Masuda, 1981).

The FO membrane of the module operated in FO mode, in which the active layer of the FO membrane faced the feed water while its support layer faced the draw solution.

The FO membrane of the module worked in FO mode, in which the dynamic layer of the FO membrane confronted the food water while its support layer faced the draw solution. The module made from a support layer and tow spacer and some other instruments. The using module is express in the following figure, which is contain tow hollow in both top and bottom side of the module to inter the DS and exite the DDS.

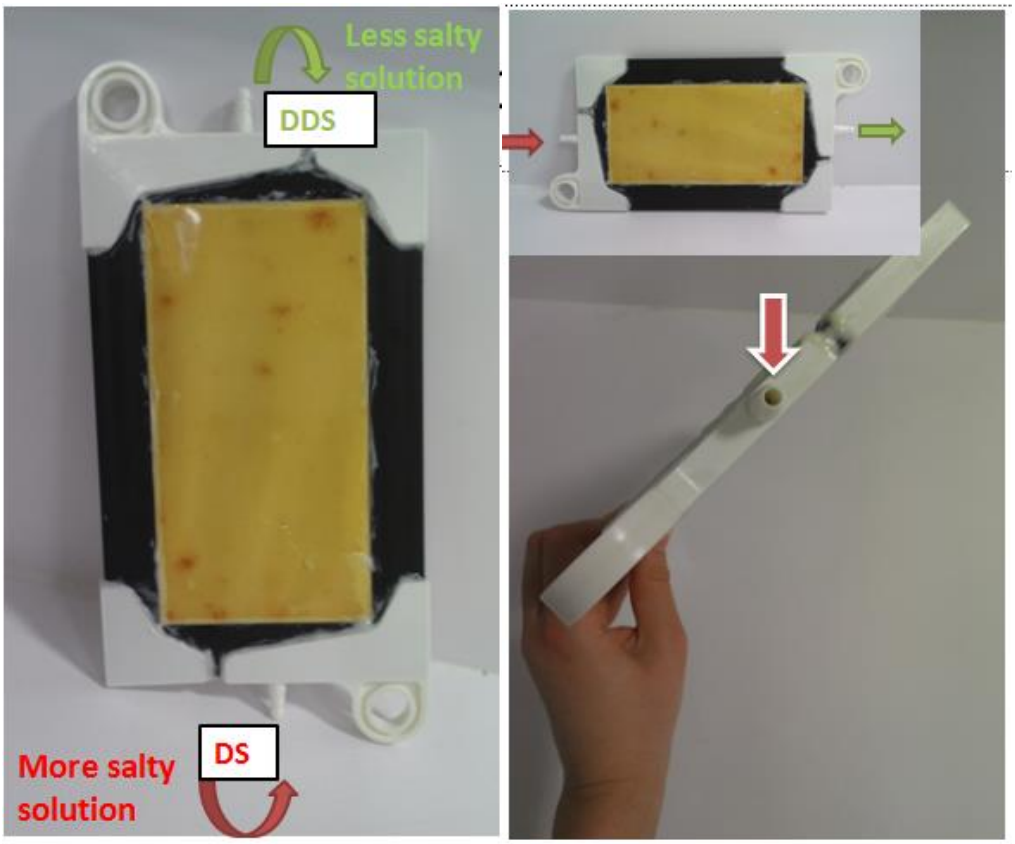


Figure 3.3: Handmade module of membrane.

3.2.3 Activated sludge preparation and characterization

As the FO tank (activated sludge tank) volume is about 28 L, filled the tank with A.S (given from Pasakoy, one of the waste water facilities in the Istanbul) around 25 L. Aeration and feeding process are implied to A.S, in order to support the microorganism's activities. The microbiological samples have been taken by weekly. The samples were observed under microscope with a visualisation attachment unit, using wet-mount technique, Gram and Neisser staining methods.

3.2.4 Suspended solids and volatile suspended solids (SS and VSS)

To purpose of this experiment is to determine the SS concentration gravimetrically in a wastewater sample. First, weight a glass fiber filter to obtain the initial weight, filtering the certain amount of sample by apply vacuum. Rinse the sample container with small volume of DIW to ensure that all SS collected on the filter. Remove the filter and put it in the oven, then dry it at least one hour at 105 ^{o}C, the wait to cool in the desiccators and weight it. (In this step can be measured the SS), for VSS again

put the filter in oven at least 30 min at 550 °C, cool in the desiccator and again weight it.

$$SS = ((A - B) \times 1000) / \text{volume of the sample ml} \quad \textbf{(3.1)}$$

Where A is the weight of filter and residual (mg) and B is the weight of filter (mg).

SS and VSS experiments were measured three times in a week.

3.2.5 Chemical oxygen demand (COD)

To determine the COD of a sample-closed reflux method used. COD is a collective environmental parameter, which is quantifying the organic matters including a sample by Standard potassium dichromate digestion at low PH and sulfuric acid reagents, in the presence of catalyst silver. The COD test run at 150°cc for 2 hours, during the test organic nitrogen is oxidizing to ammonia nitrogen.

Residual dichromate was back titrated with ferrous ammonium sulfate (FAS) with a certain normality to determine the amount of dichromate was consumed to oxidize organic matters of the sample. The end point of titration is detected by ferroin indicator solution.

1. Filtration the sample by micro filters (0.45 μm)

2. Diluted the sample with pure water x2 or x5 times. The diluted sample must be 250 ml

3. Add 1.5 ml standard potassium dichromate digestion

4. Add 3.5 ml sulfuric acid reagents

5. Heat the sample at 150°cc for 2 hours

6. Waiting to cool the sample

7. Titration the sample by adding 3 drop of ferroin as an indicator, until color turn from blue to red

8. A sample blank was treated in the same manner to eliminate the possible interferences

The results are reported in terms of mg/l COD

$$\text{Mg/l COD} = \left((\text{A} - \text{B}) \times \text{Normality of FAS} \times (\text{diluted times of sample}) \times 8000\right) / \text{sample volume mL} \quad \textbf{(3.2)}$$

Where A is volume of sample blank and B is the volume of sample in mL.

COD experiment was measured three times in a week.

3.2.6 Sludge volume index

SVI is one of the important parameters used for the determination of the settling properties of the activated sludge systems. To measure the SVI pure the sample, which is given from A.S in a 1000 mL graduated cylinder, then wait for 30 min to settle the sludge, recode the settling level of the sludge.

The results are reported in terms of (mL/g SS).

$$\text{SVI} = (\text{volume of settled sludge } (\text{ml/l}) \times 1000) / \text{MLSS mg/}L \quad \textbf{(3.3)}$$

3.2.7 Total kjeldahl nitrogen (TKN)

Steps of digestion procedure: Excess sample expelled, leaving concentrated sulfuric acid to attack the organic matter. Copious white fumes form in the flask at the time sulfuric acid reaches its boiling point-digestion is just beginning at this stage. Mixture turns black, owing to the dehydrating action of the sulfuric acid on the organic matter. Oxidation of carbon occurs. Boiling during this period is characterized by extremely small bubble formation due to the release of carbon dioxide and sulfur dioxide. Complete destruction of organic matter indicated by a clearing of the sample to a "water-clear" solution. Digestion should be continued for at least 20 min after the samples appear clear to ensure complete destruction of all organic matter. TKN in this study is calculated by following equation:

$$\text{TKN} = (\text{amount of titration} \times 0.02 \times 14 \times 1000) / \text{sample volume} \quad \textbf{(3.4)}$$

3.2.8 Viscosity

Viscosity of mixed liquire of A.S was measured by AND vibro viscosimeter SV-10 (UK) (Figure 3.4). After the calibration with distilled water at 25ºC, viscosity value of mixed liquire of A.S was used to determine at room temperature. The A.S viscosity measured one time in a week during the operation period.

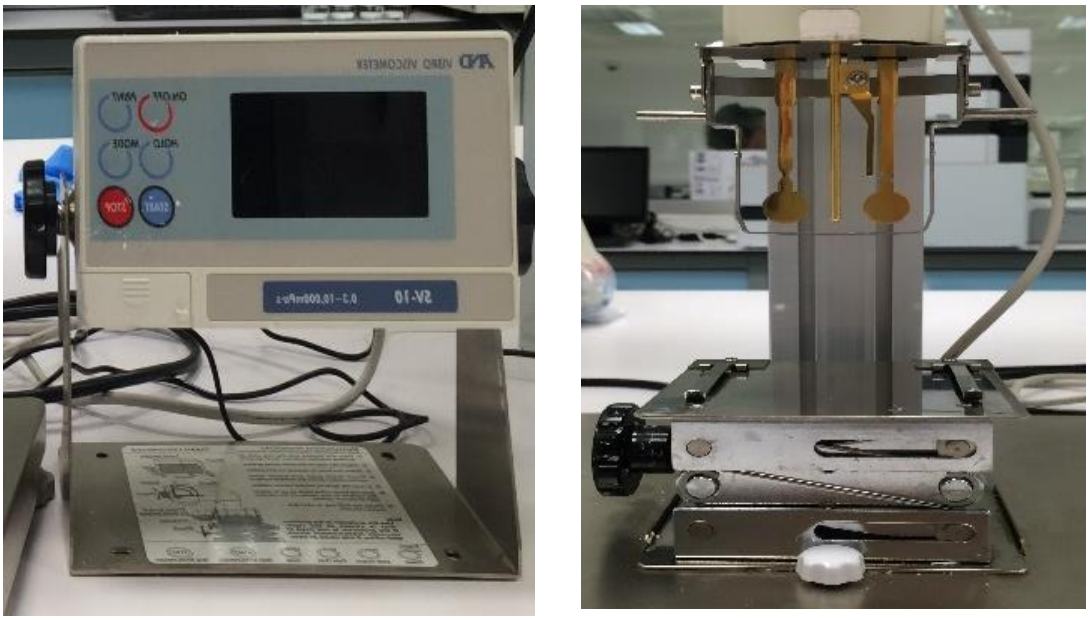


Figure 3.4: Viscosity measurement setup.

3.2.9 Temperature

Ambient temperature measured 3 times in a day by a simple thermometer, to determine relationship of the A.S microorganisms by temperature variation during operation period.

3.2.10 Microscopic examination

- **Microscopic examination (wet mounting):**

In this method, the A.S live sample is investigated under the microscopic with different objectives. The microscopic study was down one time in a week, and taken some micropictures.

- **Microscopic examination by sample staining:**

The wet mounting slides using to staining after draying according standard methods, Sampleswere examined under Olympus BX50 model microscope and microphotos taken from original samples by Cameram model attachment. The slideswere dried out and than stained by using Gram Staining and Neisser Staining methods. Stained slides were also examined microscopically and microphotos of the biological diversity and floc structures were taken and tried to be identified. Different objectives (4X, 10X, 20X, 40X, 100X) and magnifications (40X, 100X, 200X, 400X and 1000X) were used to observe microorganisms and floc structures of the activated sludge.

3.2.11 SMP and EPS

SMP and EPS analysis were made with samples taken from the mixed liquor from the bioreactor. The important point in EPS and SMP analysis is to separate these components without damaging the cells. In order to achieve this physical-chemical (sodium hydroxide-formaldehyde) extraction method was used. Formaldehyde prevents the cell rupture while it reacts with the nucleic acid and amino, hydroxyl, carboxyl and sulfide groups of proteins present in the cell wall. Hence, cell forms are preserved. NaOH increases the pH, which increases the solubility of EPS in water and increases the amount of EPS to be extracted from the cells).

At the first stage of the analysis, sludge sample is centrifuged at a lower speed allowing bacteria to be removed from the medium. The supernatant is centrifuged at high speed and the SMP released from the cell to the medium is physically separated from the water phase. Separation of EPS, which is attached to the bacterial cells, is possible by adding the above-mentioned chemicals during the procedure. After separation of SMP and EPS, samples are analyzed for protein and carbohydrate contents.

Samples are taken from the bioreactor for EPS and SMP analysis, while permeate sample was analyzed only for SMP. Samples of 5 ml volumes are taken and placed in Eppendorf tubes. These samples are centrifuged for 10 minutes at 4℃ at 4000 ×g in order to remove the suspended solids. The supernatant obtained is transferred to a sterile tube and re-centrifuged for 20 minutes at 4℃ at 13200 ×g. The supernatant obtained from this physical separation is analyzed for soluble protein and carbohydrate. The sum of soluble protein and carbohydrate corresponds to the SMP

(free EPS) present in the medium. In order to determine the bound EPS, the residue retained after the first centrifuge is completed to 5 mL volume by adding distilled water. 6 µL formaldehyde (%37) is added and the solution is kept at 4°C for 1 hour. Following this, 500 µLNaOH (1N) is added and the solution is kept at 4°C for another 3 hours. The suspension is centrifuged at 13200 ×g and 4°C for 20 minutes. The supernatant obtained from the chemical extraction is analyzed for carbohydrates and proteins, the sum of which will give the bound and/or extracted EPS. Lowry method is used for the analysis of proteins and phenol-sulfuric acid method is used for the analysis of carbohydrates.

According to the Lowry method (Lowry et. al., 1951), three main solutions were prepared to be used for the analysis (Solutions A, B and C). For preparation of Solution A, 2.86 g of NaOH and 14.31 g of Na_2CO_3 are dissolved in distilled water and completed to a final volume of 500 mL. For preparation of Solution B, 1.42 g of $CuSO_4.5H_2O$ is dissolved in 100 mL distilled water. For the preparation of Solution C, 2.85 g of Na_2tartarate.$2H_2O$ is dissolved in 100 mL distilled water. Lowry solution is prepared on the analysis day by mixing of Solutions A, B and C with the ratio of 100:1:1 (A:B:C). 0.7 ml of the Lowry solution is added to 0.5 ml of sample, the mixture is mixed rigorously and let still for 20 minutes at room temperature at dark. Folin solution is prepared in parallel by adding 5 ml of 2N folin to 6 ml of distilled water. 0.1 ml of folin solution is added to each 0.5 ml sample after which the samples are mixed rigorously and let still for 30 minutes at room temperature at dark. At the end of this period the samples are colored from light to dark blue according to their protein contents. The colorimetric analyses were made by using Spectro Pharmacia LKB Nova Spec II UV spectrophotometer at 660 nm against a blank sample. In order to assure reproducibility two analysis samples were prepared from the original sample. The calibration curve is given in Figure 3.5.

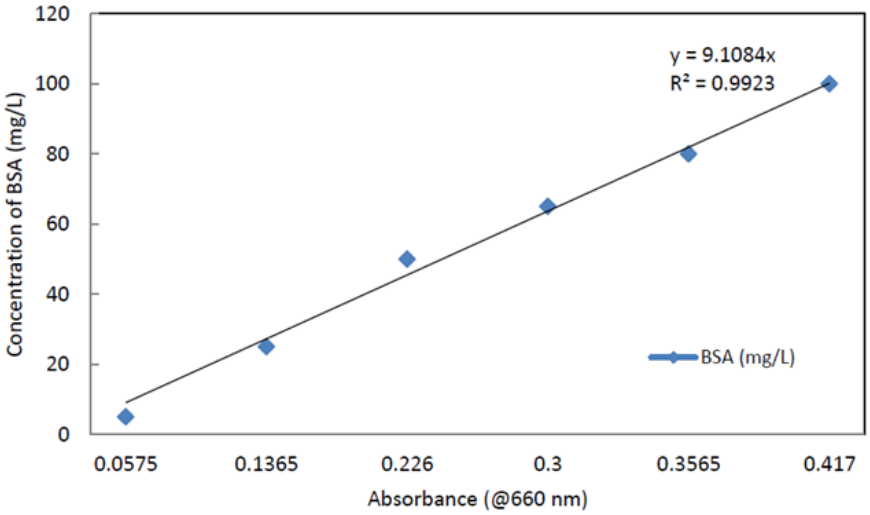


Figure 3.5: Protein Calibration Curve.

3.2.12 Carbohydrate analysis method

Modified "phenol-sulphiric acid method" (Dubois et. al., 1956) was used for the analysis of carbohydrates. 25µl of 80% phenol solution and concentrated H_2SO_4 (95-97 %) were used of phenol solution (80%) and 2.5 ml H_2SO_4 (95-97%) were added to 1 mL of sample and waited in a water bath for 15 min. Colors of the samples were varied from light yellow to dark yellow according to their carbohydrate concentrations. Measurements were done at 490 nm wave length by UV spectrophotometer.

Glucose was used as the standard carbohydrate solution for the calibration.The absorption-concentration graph was drawn with the obtained values. Calibration graph is given at Figure 3.6.

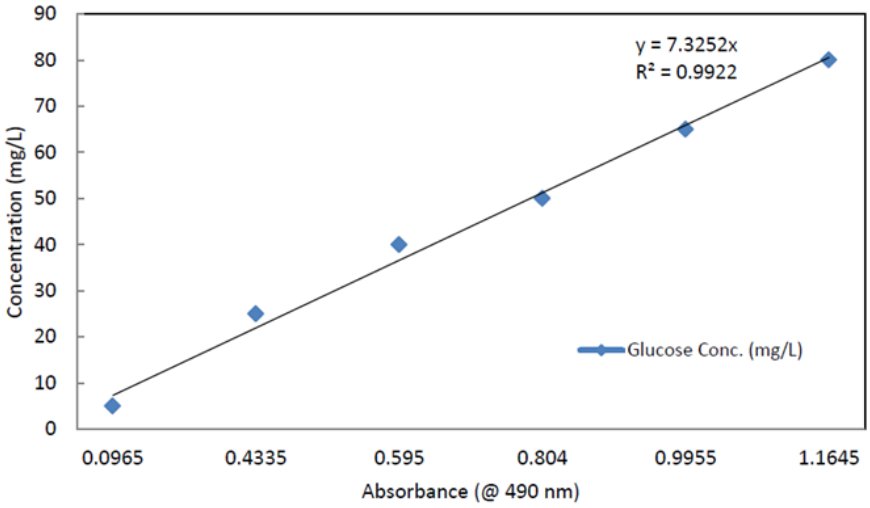


Figure 3.6: Carbohydrate Calibration Curve.

3.2.13 Water flux

The water flux figuring was in view of the progressions of the draw tank weight. The expanded water volume was acquired by isolating the expanded draw tank weight with the water density. Equation (3.5) showed the water flux calculation.

$$F_w = \frac{V_i}{A_m t} \tag{3.5}$$

Where F_w is water flux (Lm-2h-1), V_i *is* increased water volume (L), A_m is effective membrane area (m^2) and t is operation time (h).

3.2.14 Conductivity

The conductivities of the samples were recorded otomaticaly during laboratory-scale FO-MBR experiments.

3.2.15 Reverse salt flux

Reverse salt flux is a pointer for the FO film capacity in holding the solutes. FO membrane shows distinctive capacities in dismissing diverse solutes. Subsequently the reverse salt fluxes were measured during FO process. A low reverse salt flux demonstrates a high FO membrane rejection of the tried draw solute while a high reverse salt flux mirrors a low rejection performance.

In this study to see the reverse salt flux, measured the conductivity in the start point of each cycle and measured in the end of cycle.

3.3 Membrane Characterization

3.3.1 Contact angle

To have the capacity to discuss how hydrophobic or hydrophilic a membrane is contact edge estimations were directed utilizing Attension T200 Theta (Figure 3.7). Somewhere around 2µl and 5µl refined water was dropped onto dry layer surface in air. Information's were gathered from 3 distinct focuses. Contact edge was utilized to gauge the contact point of the FO membrane. The film test was appended to the surface with twofold-sided tape to avoid ingestion of the water drop into the material, to take an unfaltering picture of the drop and compute with exactness the contact angle.

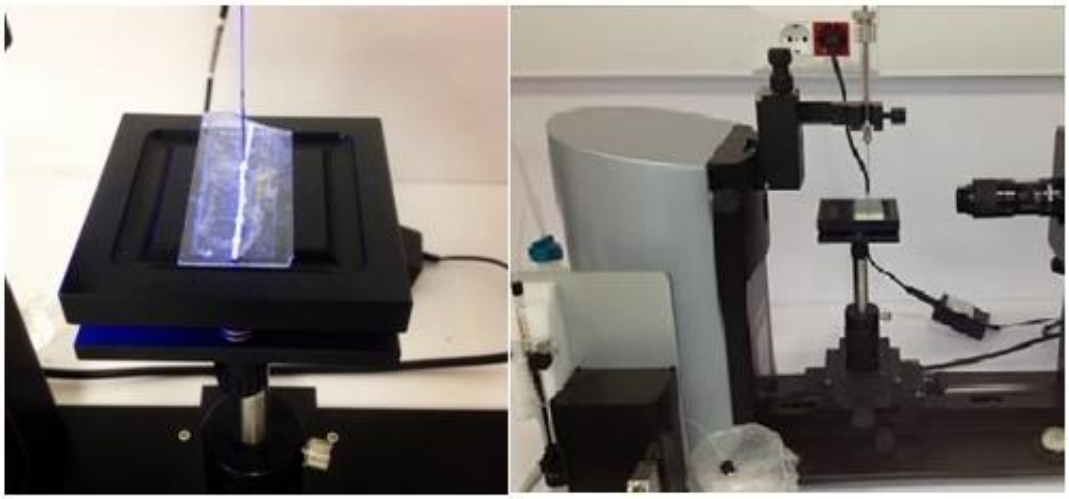


Figure 3.7: Contact angle measurement setup.

3.3.2 Scanning electron microscopy (SEM)

Morphology of hollow fiber membranes was characterized by FEI Quanta FEG 200. Membranes were prepared by inserting them into liquid nitrogen and cut. Then it was coated about 3-4nm with Palladium and Gold (Pd-Au) by using Quorum SC7620 ion sputtering equipment.

Morphology of membranes was portrayed by FEI Quanta FEG 200 SEM Films were arranged by embeddings them into fluid nitrogen and cut. At that point it was covered around 3-4nm with Palladium and Gold (Pd-Au) by utilizing Quorum SC7620 particle sputtering hardware.

SEM device where in MEM-TEK is shown in Figure 3.8.

Figure 3.8: SEM set up (FEI Quanta FEG 200).

3.3.3 Zeta potential of membranes

Streaming potential measurements were made using the Anton PAAR SurPASS Electro-kinetic Analyzer (Figure 3.9) (Şengür, 2013).

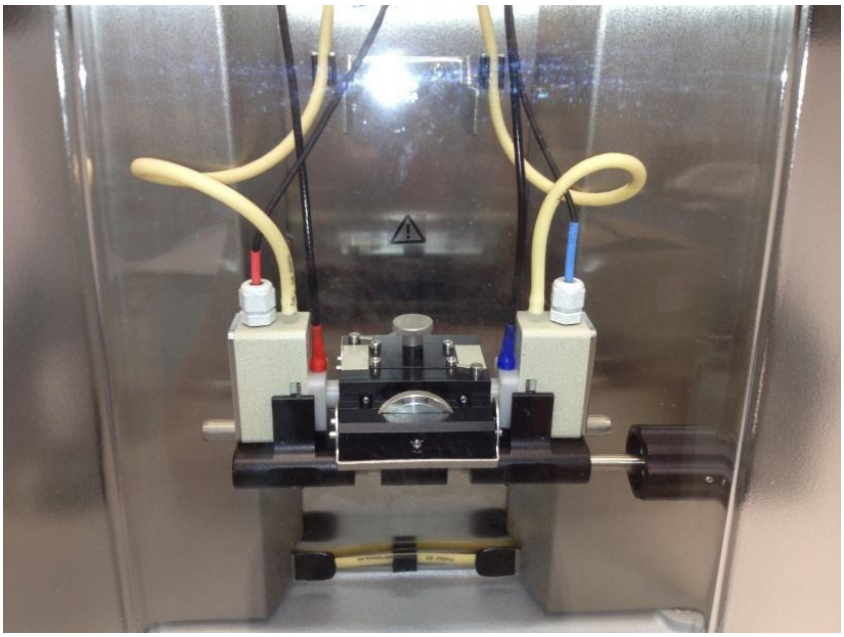


Figure 3.9: Electrokinetic analyzer cell.

3.3.4 Fourier transformation infrared spectroscopy (FTIR)

Surface functionalization of membranes was characterized by using Perkin Elmer Spectrum 100 FTIR Spectrophotometer (Figure 3.10). Before measuring hollow fiber membranes, a background spectrum was conducted to decrease instrumental and atmosphoric contributions to a minimum level (Şengür, 2013).

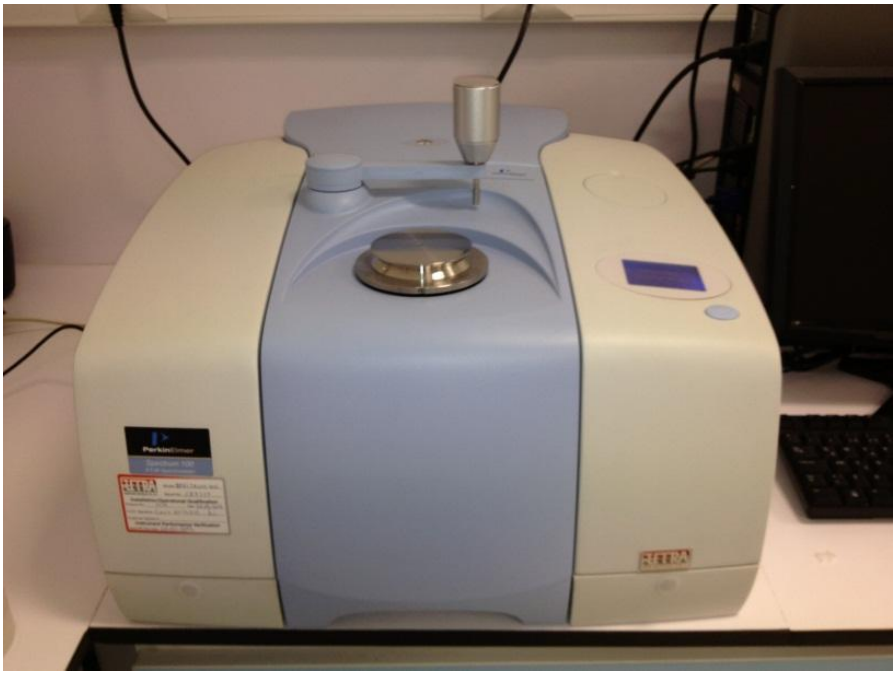


Figure 3.10: FTIR spectrophotometer.

4. RESULTS AND DISCUSSION

The FO-MBR study focused on the system performances in terms of contaminant removal efficiencies, water fluxes, and FO membrane performance in lab scale FOMBR. As before mention that, before carrying out the experiment, an ideal draw solution and a proper FO membrane module design had to be selected. A suitable module design would enhance the water flux produced by the draw solution when it went in the module. A perfect draw solution assumed a noteworthy part in the execution of the FO-MBR as it could create a high water flux and produce a last water of brilliant utilizing a suitable reconcentration procedure, bringing about a low general expense.

4.1 MBR Application Results

4.1.1 COD results

COD is one of the most important parameters in wastewater treatment. In Sutton et. al., study the biomass concentration was kept at around 3900- 4600 mg/L VSS and the COD removal rate during this period was an average of 96.7% 97%. Obviously, this COD removal rate was much higher than the conventional MBR (Sutton et al., 2004), which might be resulted from the differences in the membrane configuration: microfiltration versus FO membrane (non-porous). This result was consistent with (Achilli et. al., 2009) that the semi-permeable FO membrane exhibited 98% rejection of TOC due to its non-porous composition compared with microporous membranes with typically 28%, 87% retention ability. FO process demonstrated better water quality because of a double barrier, which exhibited remarkable removal efficiency for salts (above 95%), ammonia (74%), nitrate (78%), sulfamethoxazole (90%), carbamazepine (83%), trace organics (w80%) and so on (Alturki et al., 2012; Cath et al., 2009; Heo et al., 2013; Jin et al., 2012). 95.0% removal efficiencies of organic matter were achieved in (Zhimin et. al., 2008) study, also in current study biomass concentration was kept at around 9000- 16000 mg/L SS and COD removal efficiency

is calculated 87%. In current study, COD results during the FO-MBR operation is shown in Table 4.1.

Table 4.1 : COD results during the FO-MBR operation.

Day of FO-MBR operation	COD in(w.w) mg/L	COD (A.S) mg/L	COD (FO effluent) mg/L	COD (RO effluent) mg/L
1	580	350	127	<4
4	520	334	110	<4
7	550	243	182	<4
10	--	--	--	<4
13	515	228	60	<4
16	520	232	57	<4
19	520	240	89	<4
22	523	237	92	<4
25	531	243	112	<4
28	530	248	105	<4
31	525	233	87	<4
34	525	238	90	<4
37	523	260	78	<4

From each cycle, COD removal percentages were given at table 4.2. Average removal values for FO 86%, A.S 52% were calculated percent of COD removal.

Table 4.2 : COD removal efficiency.

Day of FO-MBR operation	A.S COD% removal	FO COD %removal	RO COD %removal
1	40	85	>98
4	36	86	>98
7	56	84	>98
10	--	--	>98
13	56	90	>98
16	55	90	>98
19	54	88	>98
22	55	88	>98
25	54	85	>98
28	53	87	>98
31	56	88	>98
34	55	87	>98
37	50	90	>98

4.1.2 COD versus temperature

As it is clear from Figure 4.1, when the ambient temperature was high, the COD removal efficiency results would be better. It can be concluded that in higher temperatures, COD removal would be better generally, may be because microorganisms have increased activity and better performance in high temperature.

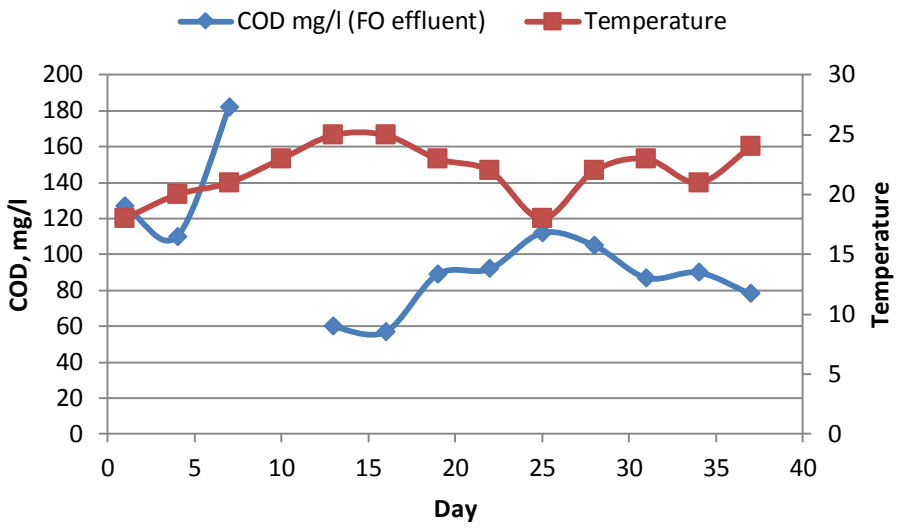


Figure 4.1: COD variation versus temperature.

4.1.3 SS and VSS

The aeration tank equipped with submerged membrane was operated with the concentration of biomass at MLSS 10,000 mg/L in the beginning and then the biomass concentration increased gradually to 16,000 mg /L at a month of continuous operation as shown in figure 4.2. The aeration tank in higher than one of the influent. This indicates that organic solids in the wastewater was converted into soluble organic. So it is abviouse from Figure 4.2 SS and VSS accumulation was occure during the study. These results are similar to (Zhao, et. al., 2009; Jeong, et. al., 2005) studies.

Average amount of suspended solids and volatile suspended solids of A.S were 11570 and 81250 mg/l in order of term. Daily amounts of SS and VSS were presented in the Figure 4.2.

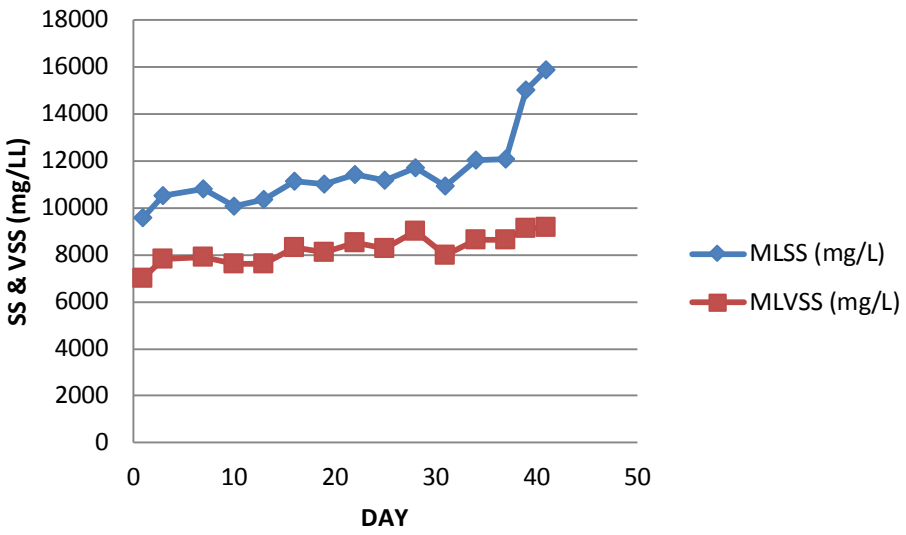


Figure 4.2: SS and VSS values of A.S.

4.1.4 Viscosity

The viscosity variation of activated sluge was increased with low slope during the exprimentation. Salinity increases could lead to an increase in mixed liquor viscosity and a reduction in oxygen solubility (Lay, et al., 2010).

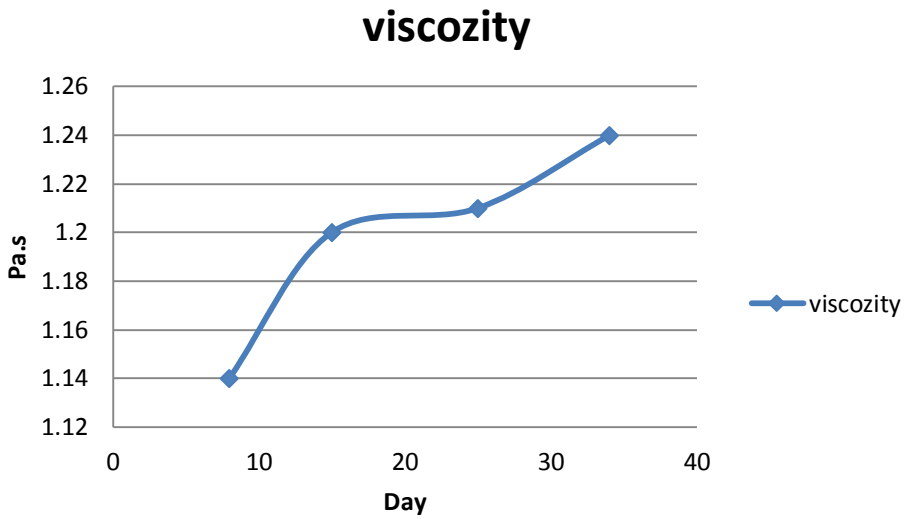


Figure 4.3: Viscosity variation in operation period.

4.1.5 Turbidity

In a result, the turbidity removal in FO effluent was about 99% during the FOMBR operation. The results in detail are shown in the below table (Table 4.3).

Table 4.3 : Turbidity results in FOMBR

Day of FO-MBR operation	Diluted A.S turbidity (NTU)	Average	X Dilution	A.S turbidity (NTU)	Diluted draw solution turbidity (NTU)	Average
6	278	288	25	7200	4.1	4.6
	292				6.5	
	294				3.1	
16	161	158.5	25	3962	3.0	3.3
	165				3.5	
	155					
	153					
26	170	163	25	4010	3.1	3.3
	167				3.6	
	152					
34	150	142	25	3761	3.1	3.1
	148				3.2	
	130					

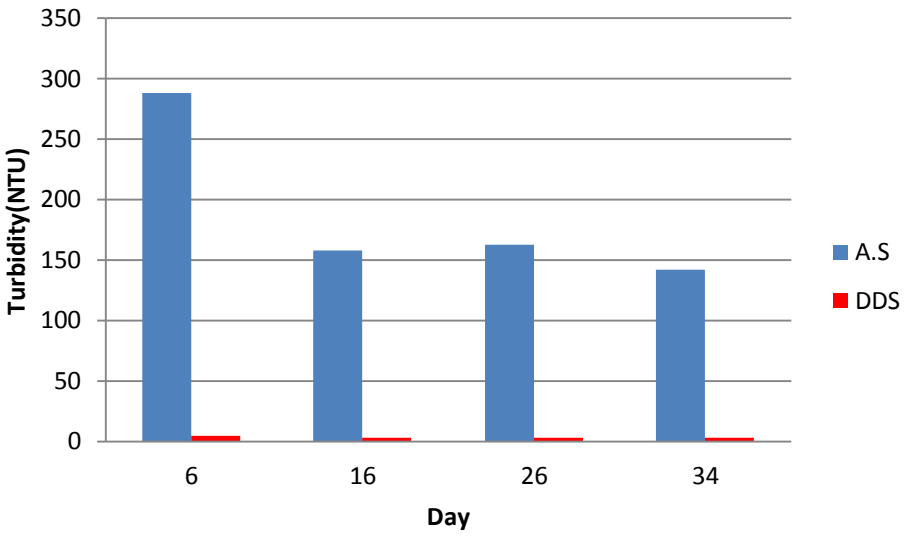


Figure 4.4: Turbidity removal (efficiency).

Non-consumable reuse applications require consistent water quality, suspended solids and turbidity, to diminish the probability of bacterial contamination and to protect potential clients and receiving environments. Membrane bioreactors (MBRs) worked with low-pressure microfiltration (MF) or ultrafiltration (UF) membranes are

perfect advancements for most non-consumable reuse applications on account of the predominant and reliable gushing quality that they deliver compared to traditional clarification processes. For the same reason, UF membranes give a phenomenal pretreatment to reverse osmosis (RO) membranes by lessening colloidal, organic, and biological fouling on RO membranes. Yet, the dismissal of low molecular weight constituents, including trace organic compounds, ions, and viruses by UF membranes is limited. This deficiency might restrict indirect or direct potable reuse applications that require a multiple barrier treatment approach to ensure that public health is not compromised. In this study the turbidity removal in FO effluent was about 99% during the FOMBR operation it is shown in Figure 4.4.

4.1.6 Total kjeldahl nitrogen (TKN)

The Total Kjeldahl Nitrogen if A.S was not change significantly in this period. The results are shown in Table 4.4.

Table 4.4 : TKN results in A.S tank.

Day of FO-MBR operation	TKN (mg/L)
1	513.2
9	494.2
21	483.4
29	480.6

4.1.7 Nitrate and phosphate analysis

Whereas a shift in the population of non-saline resistant species of *Nitrosomonas europaea-lineage* and *Nitrosomonas eutropha* to saline resistant species (e.g. *Nitrosococcus mobilis-lineage*) was also observed (Chen et al., 2003). In fact, in the treatment of saline wastewater, different levels of tolerance by nitrifiers were observed. Nitrifiers can survive in a highly saline system through acclimation to the salt environment at the concentrations of 0–40 g/l NaCl. For example, the adaptation of microorganisms was determined to be the key factor towards high-efficiency nitrification at elevated salt concentrations (Bassin et al., 2011). In fact, adapted biomass was found to be less sensitive to highly saline environments (Campos et al., 2002). However, severe or complete inhibition would occur when the salt level increased to 40 g/L and beyond (Rothschild and Mancinelli, 2002; Moussa et al.,

2006). In general, efficient nitrification at elevated salt concentrations of less than 40 g/L should be achieved through microbial adaptation.

According to Table 4.5 the average amount of nitrate of A.S, FO, RO were 55.4, 12.1 and 5.87 mg/L in order of terms. In addition, it is clear from Figure 4.5 the removal efficiency is 79% and 90%, in FO and RO effluent respectively.

Table 4.5 : Nitrate results in A.S., FO and RO effluent.

Week	A.S Nitrate mg/L	FO Nitrate mg/L	RO Nitrate mg/L
3	42.8	16.2	6.1
12	43.8	11.8	6.8
22	68	10.1	5.3
33	67	10.6	5.3

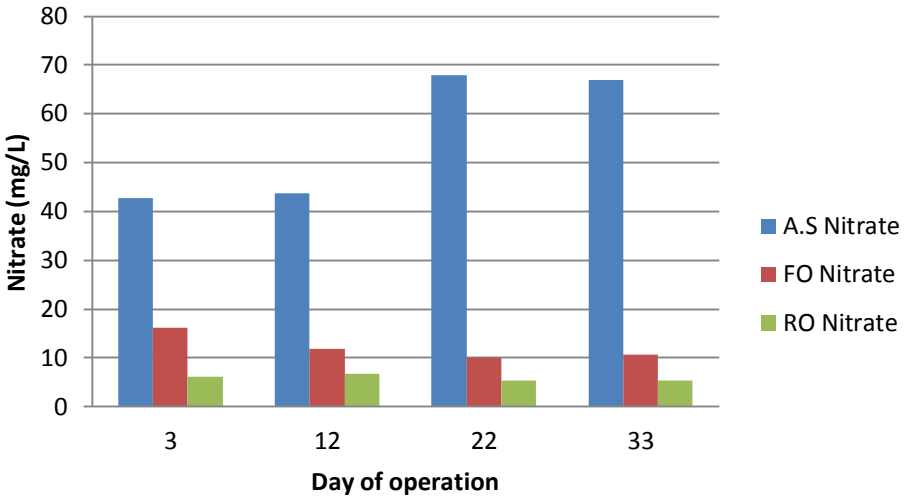


Figure 4.5 : Nitrate removal (efficiency)

According to Table 4.6 the average amount of phosphate of A.S., FO, RO were 32.47, 1.39 and 0.03 mg/L in order of terms. Also the removal efficiencies were calculated 96% and 99%, for FO and RO effluent respectively, it is clear from Figure 4.6.

Table 4.6 : Phosphate results in A.S. FO and RO effluent.

Week	A.S. Phosphate mg/L	FO Phosphate mg/L	RO Phosphate mg/L
3	41.2	1	0.006
12	29.8	1.3	0.01
22	28.6	1.8	0.009
33	30.3	1.5	0.008

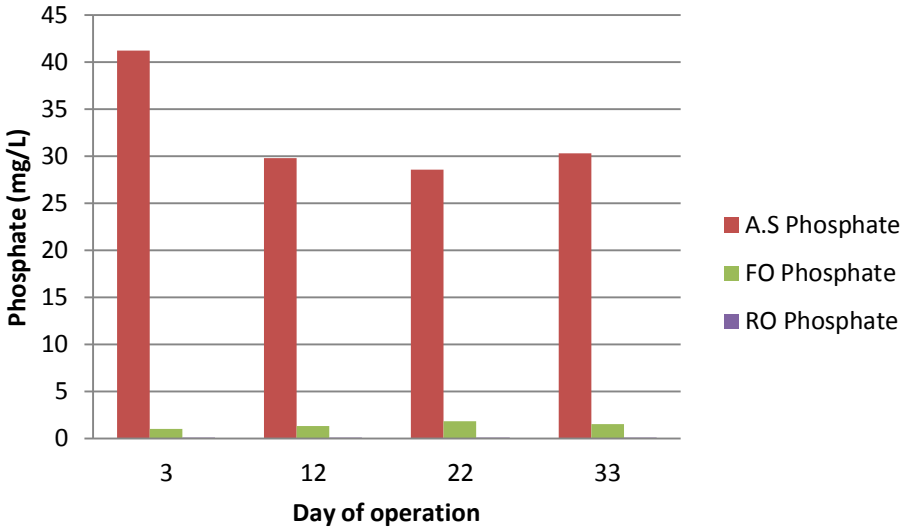


Figure 4.6: Phosphate removal (efficiency).

Phosphorus is a key nutrient that stimulates the growth of planktonic cyanobacteria and algae, and must be removed from wastewater to avoid eutrophication in natural waters. Various techniques have been developed for phosphorus removal, among which the most widely adapted ones are chemical flocculation and enhanced biological phosphorus removal (EBPR) (Shu et al., 2006; Bowden et al., 2009).. Recovering phosphorus from wastewaters is a desirable alternative to provide sustainable phosphorus supplies. It has been estimated that 15–20% of world's phosphorus demand could be satisfied by its recovery from municipal wastewater (Yuan et al., 2012. Currently, technologies for direct recovery of phosphorus form municipal wastewater are still not available. Recently, there has been increasing interest in a novel integration of forward osmosis (FO) and biological process for wastewater treatment, known as the osmotic membrane bioreactor (FOMBR) (Wang et al., 2014; Kim, 2014). In this process, FO membranes are used instead of microporous membranes. Water is transported from the mixed liquor into the draw solution (DS) under a driving force of natural osmotic pressure (Achilli et al., 2009;

Chung et al., 2012). The usage of FO membrane in OMBR brings along some unprecedented advantages such as higher theoretical water flux and low fouling potential (Achilli et al., 2009; Yap et al., 2012; Qiu and Ting, 2014). More importantly, the FO membrane allows high rejection of various containments and mineral salts, and hence results in very high quality produce water. This high rejection feature of the FO membrane may be exploited to facilitate the removal and recovery of phosphorus from municipal wastewater. The rejection of PO_4-P in the feed wastewater by the FO membrane could result in several fold concentration of PO_4-P with in the bioreactor. To date, the potential of using F-OMBR for direct recovery of phosphorus from municipal wastewater has not been explored.

4.1.8 EPS and SMP

EPS, consisting of a variety of organic substances such as polysaccharide, protein, lipids and nucleic acids, had significant impacts on the sludge properties and membrane fouling (Wang et al., 2009)., while the SMP is in sludge supernatant.

Before the stable state of the FOMBR, the salinity and sludge properties having a strong impact on the variations of EPS would change all the time. There is strongly negative correlation between membrane permeability and SMP content. (Reid et al., 2006; Wang et al., 2009; Hai et al., 2011).

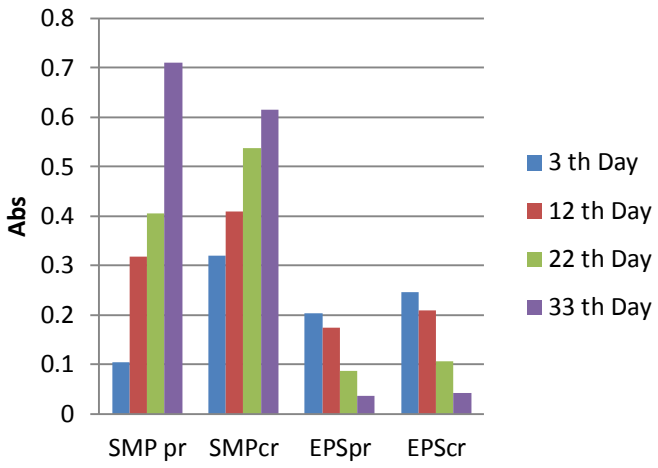


Figure 4.7: SMP and EPS variations.

Notable increases in the content of SMP and EPS with increasing salinity loading were observed in the saline-MBR, (Laspidou and Rittmann, 2002; Yogalakshmi and

Joseph, 2010). It is noteworthy that the EPS content of the saline-MBR gradually decreased to that of the control system by the end of the experimental period. The decreased EPS content was probably because of the increased solubility of EPS fractions (e.g., protein and carbo- hydrate) at high salinity (Zhang et al., 2014). Thus, a more dramatic increase in the SMP content was observed when the NaCl loading was stabilized at 16.5 g/L.The SOUR of the biomass in the control-MBR was stable at approximately 2.8 mg O_2/g MLVSS h during the experiment. However, a lower SOUR value (2–2.5 mg O_2/g MLVSS h) was observed in the saline-MBR with increasing the NaCl concentration.The EPS and SMP variations in current study are similar to (Zhang et al., 2014) the results variations are shown in Figure 4.7.

4.1.9 FO flux

The results for the flux through the forward osmosis membrane are not a direct measurement. Nevertheless, the change in weight through each cycle is the most straightforward way to determine flow, with the effective area of the membrane (190m^2) will give an accurate estimation of the flux. The usage of FO membrane in FOMBR brings along some unprecedented advantages such as higher theoretical water flux and low fouling potential (Achilli et al., 2009; Yap et al., 2012; Qiu and Ting, 2014).For the FO process, the fluxes varied from 6.2 to 2.6 L/m^2-h, with an average of 3.75 L/m^2-h (Valladares, 2011). In this study the average flux of cycle 8 (21th, 22nd, day of FO-MBR operation) was calculated as 3.3L/m^2-h. Unfortunately we could not record the FO flux precisely due to the malfunction of the automation system of the setup.

4.1.10 Conductivity

The FO membrane allows high rejection of various containments and mineral salts, and hence results in very high quality produce water. Figure 4.8 is express the conductivity of end point of each cycle, as it is clear from the figure The salinity removal efficiency would be better by operation time. The draw solution was diluted from 50 ms/cm to 15 ms/cm in average.

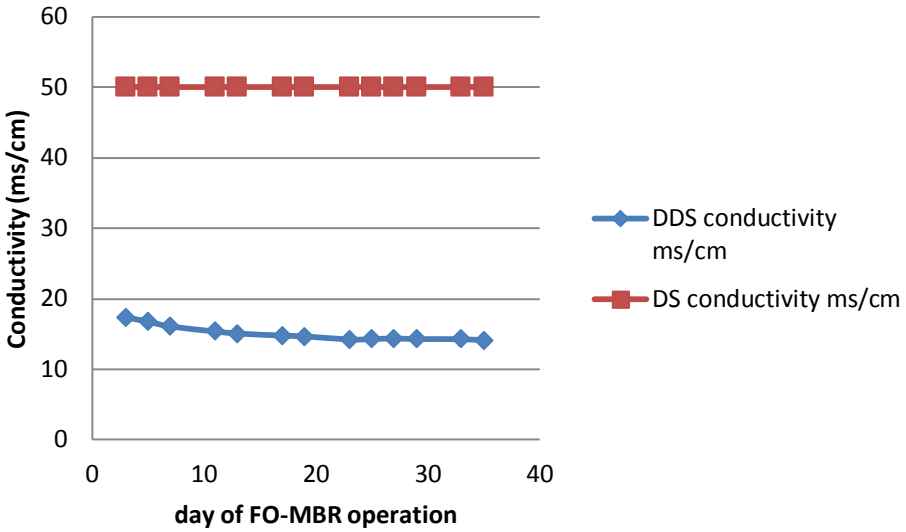


Figure 4.8: Salinity removal (efficiency).

4.1.11 Reverse salt diffusion

As it is clear from Figure 4.9 the reverse salt diffusion was not a significant changing during the FO-MBR operation, so there is not important reverse salt diffusion. There is a peak after cycle 3 it is related to diluted of the AS tank before cycle 4 because of high salinity. As a result, there is a little salt accumulation in the A.S tank, and it can affect the other parameters such as EPS and SMP, microorganism activities, COD removal and nutrient removals.

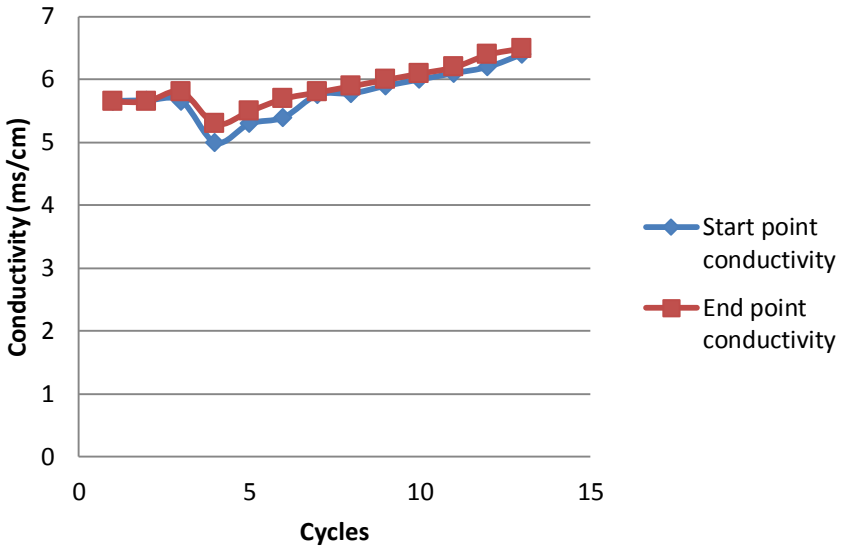
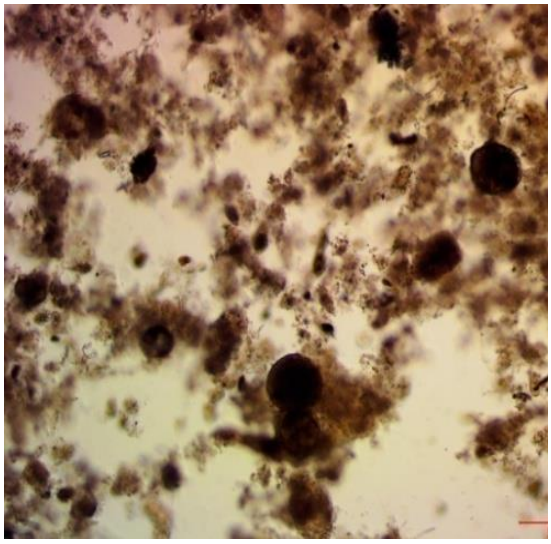


Figure 4.9: Reverse salt diffusion in each cycle.

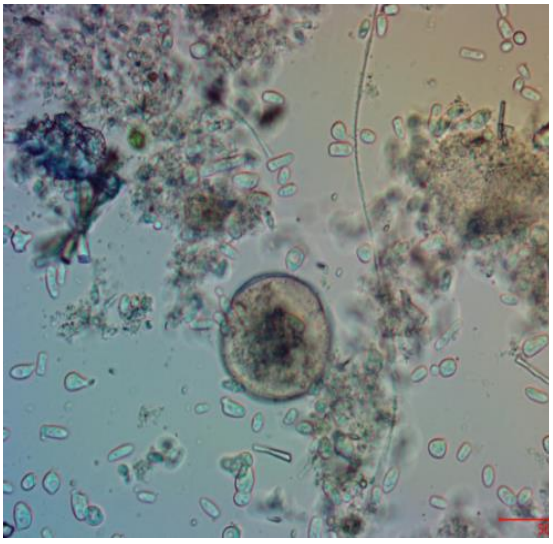
4.1.12 Microscopic study

Activated sludge samples have been observed under microscope and monitored throughout the study. Sample microphotos of microorganisms and activated sludge structure were given below Microphotos 1-16 with parallel to the operation time.

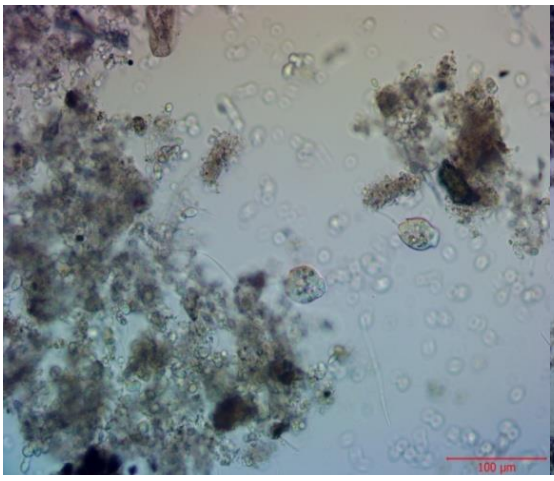
4.1.12.1 Wet mounting



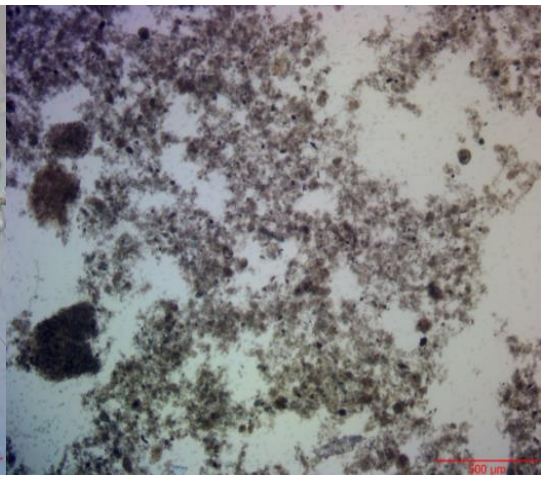
Microphoto 1: General view of activated sludge, flocs and other organic materials, original sample (first week sample), bright field microscopy, 200X.



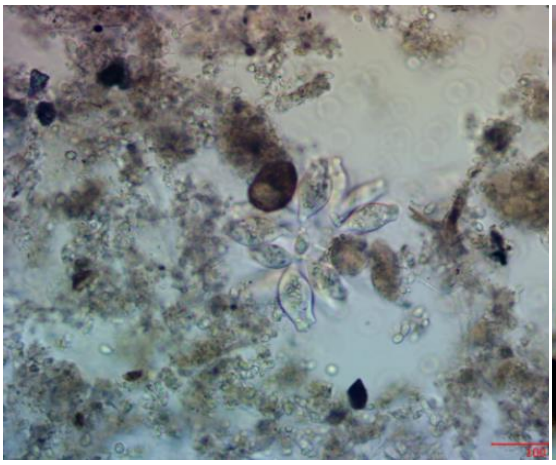
Microphoto 2: General view of activated sludge, flocs, free bacteria, eukaryotic cells, filamentous microorganisms, original sample (first week), phase contrast microscopy, 400X.



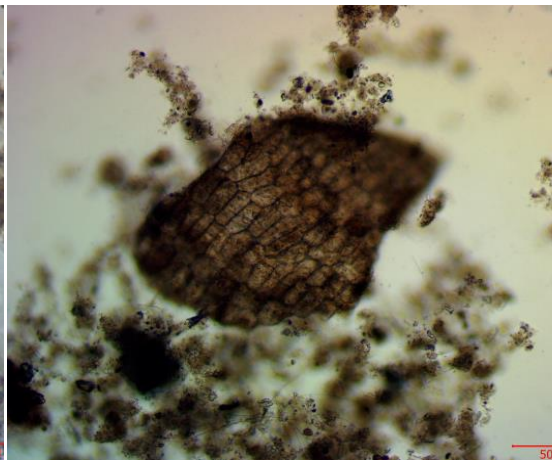
Microphoto 3: General view of activated sludge, flocs and attached protozoa, *Vorticella* spp., original wet mount sample (first week), bright field microscopy, 400X.



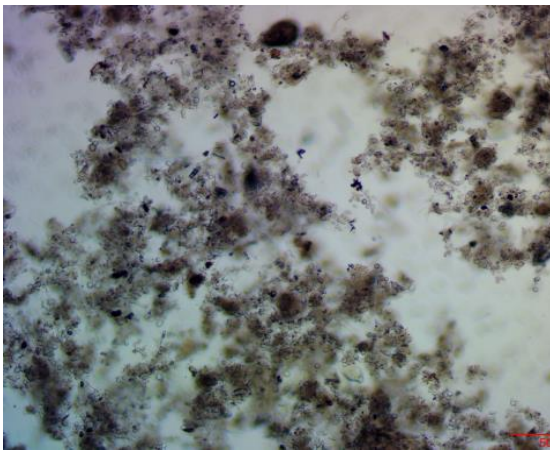
Microphoto 4: General view of activated sludge, flocs that are very loose and scattered, as it is seen flocs have some holes, original sample (first week), bright field microscopy, 200X.



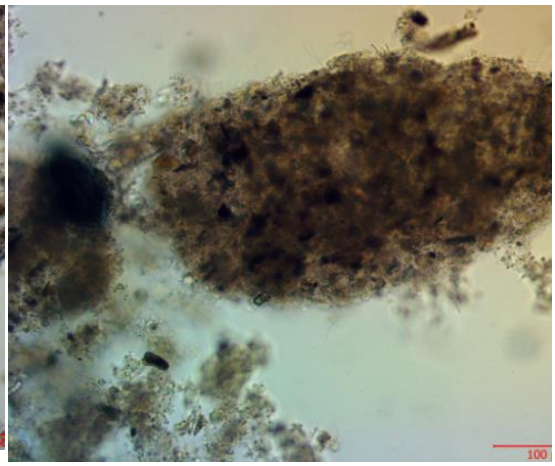
Microphoto 5: General view of activated sludge, flocs and attached protozoa, *Opercularia* spp. colony, original wet mount sample (second week), bright field microscopy, 400X.



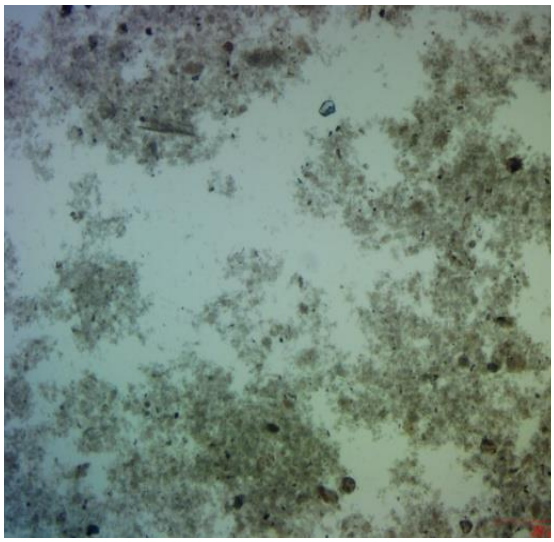
Microphoto 6: General view of activated sludge, flocs and some organic particules, skin cells, original wet mount sample (second week), bright field microscopy, 200X.



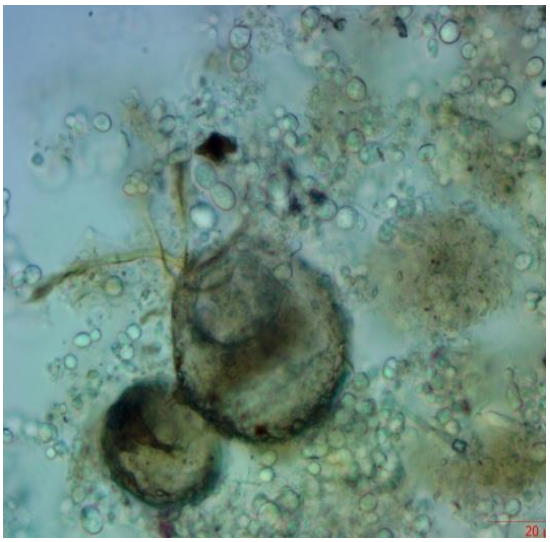
Microphoto 7: General view of activated sludge, loose flocs original wet mount sample (second week), bright field microscopy, 200X.



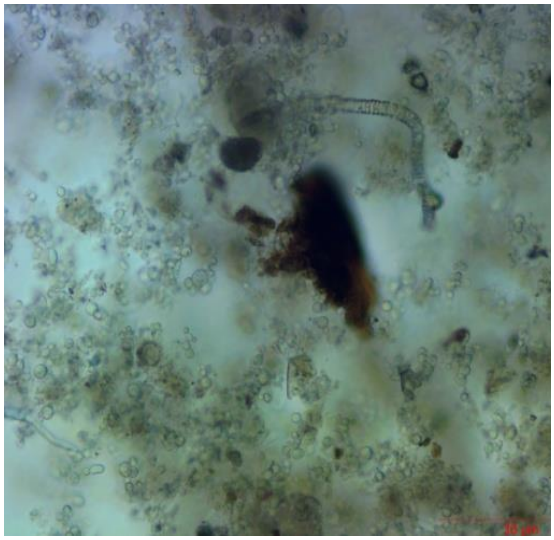
Microphoto 8: General view of activated sludge, compact floc, original wet mount sample (second week), bright field microscopy, 200X.



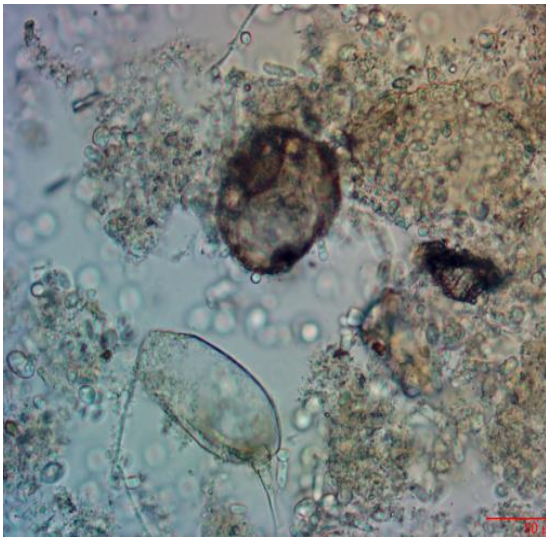
Microphoto 9: General view of activated sludge, loos flocs, original wet mount sample (third week), bright field microscopy, 200X.



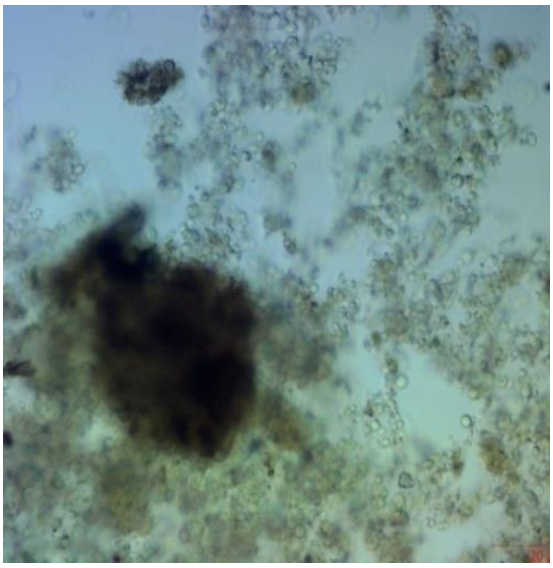
Microphoto 10: General view of activated sludge, flocs, eukaryotic cells, original wet mount sample (third week), bright field microscopy, 400X.



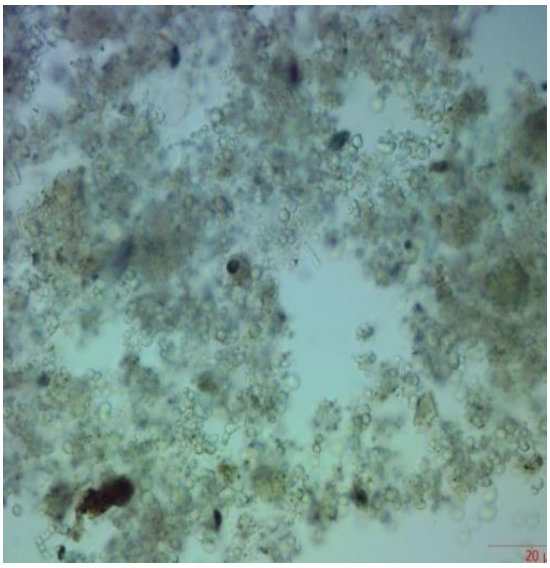
Microphoto 11: General view of activated sludge, good flocs, *Aeolosoma worm spp*., original wet mount sample (third week), bright field microscopy, 200X.



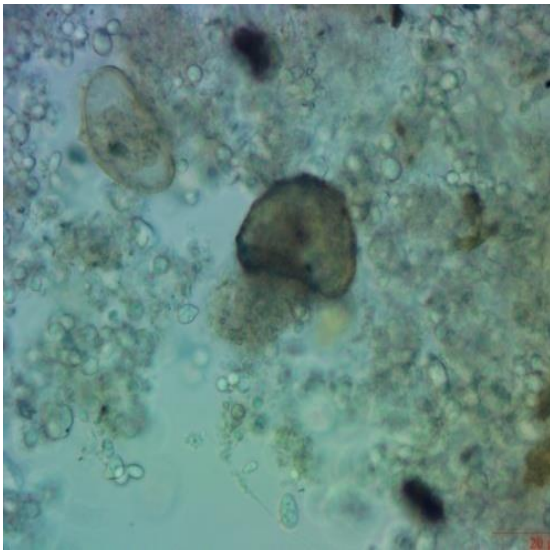
Microphoto 12: General view of activated sludge, flocs, rotifer (*Philodina* spp.) original wet mount sample (third week), bright field microscopy, 400X.



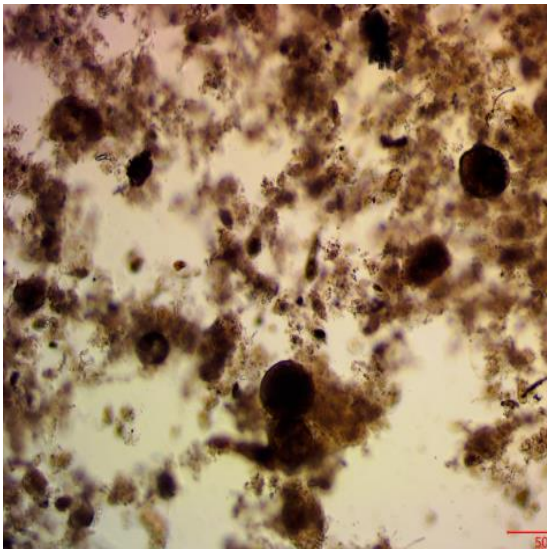
Microphoto 13: General view of activated sludge, good flocs, original wet mount sample (fourth week), bright field microscopy, 100X



Microphoto 14: General view of activated sludge, loose flocs, original wet mount sample (fourth week), bright field microscopy, 100X



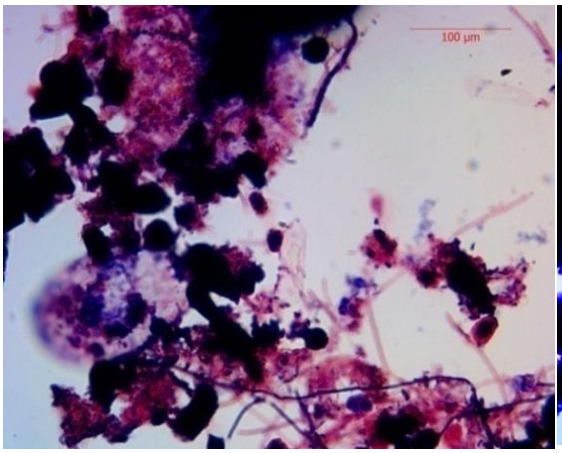
Microphoto 15: General view of activated sludge, loose flocs, eukaryotic cells, original wet mount sample (fourth week), bright field microscopy, 400X



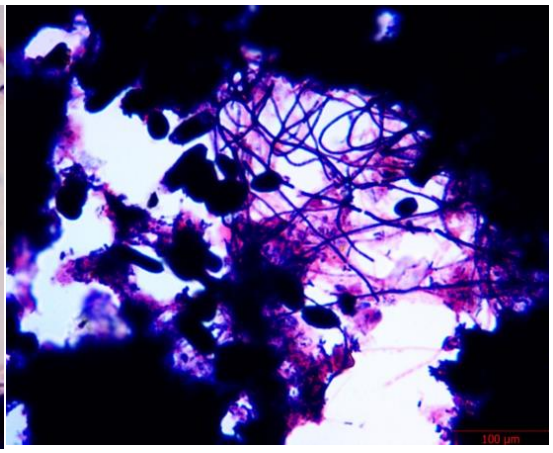
Microphoto 16: General view of activated sludge, loose flocs, original wet mount sample (fourth week), bright field microscopy, 200X

Figure 4.10: Wet mounting results during the study.

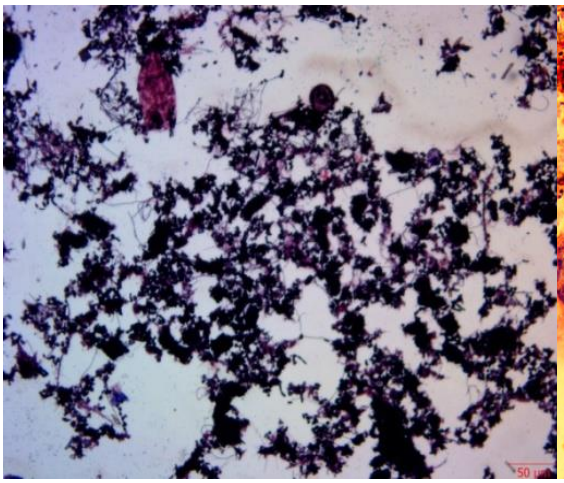
4.1.12.2 Gram and Neisser staining results



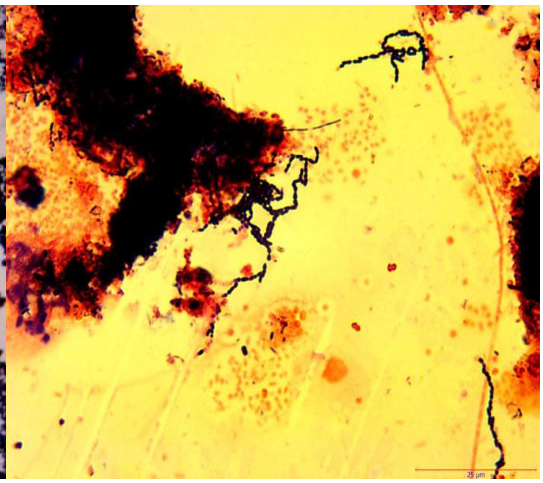
Microphoto 17: View of activated sludge, Gram staining sample (first week), Gram negative and Gram positive filamentous microorganisms, bright field microscopy, 1000X



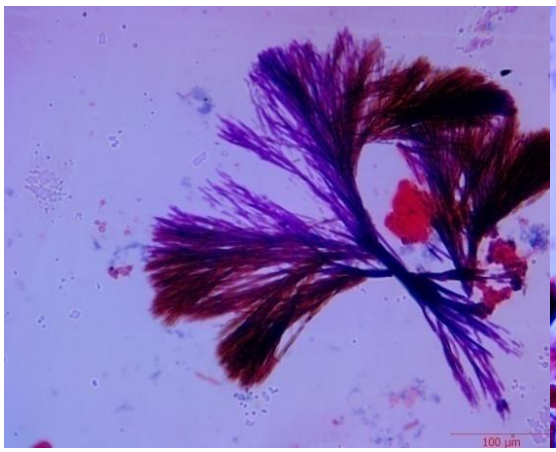
Microphoto 18: View of activated sludge Gram staining sample (first week), flocs, Gram (+) filamentous bacteria and eukaryotic cells, bright field microscopy 1000X.



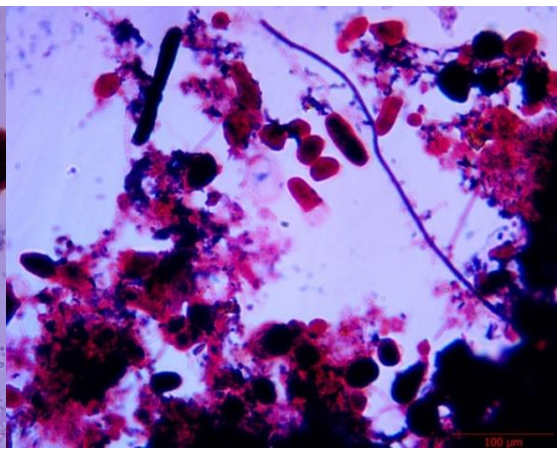
Microphoto 19: View of activated sludge Gram staining sample (first week), loose flocs, filamentous microorganisms connecting flocs together, bright field microscopy, 200X.



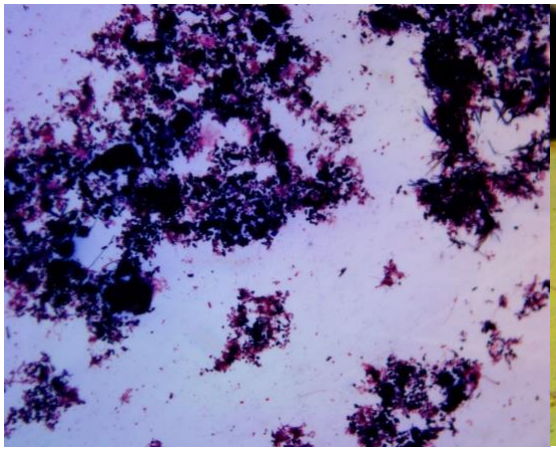
Microphoto 20: View of activated sludge Neisser staining sample (first week), flocs, *Bacillus spp.* filamentous chain bacteria (dark color, blue), bright field microscopy, 1000X.



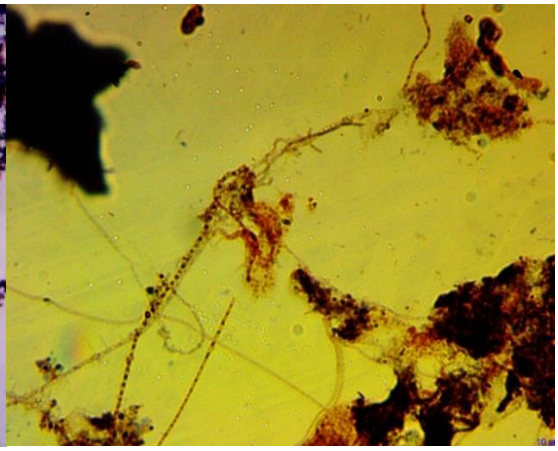
Microphoto 21: View of activated sludge Gram staining sample (second week), filamentous, soil bacteria *Actinomycetes* spp., bright field microscopy, 1000X.



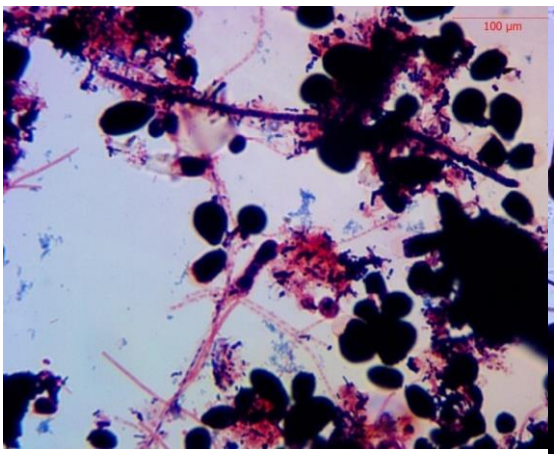
Microphoto 22: View of activated sludge Gram staining sample (second week), flocs, Gram (+), and Gram (-) filamentous bacteria, protozoa, eukaryotic cells, (fungi cells and spores), bright field microscopy, 1000X.



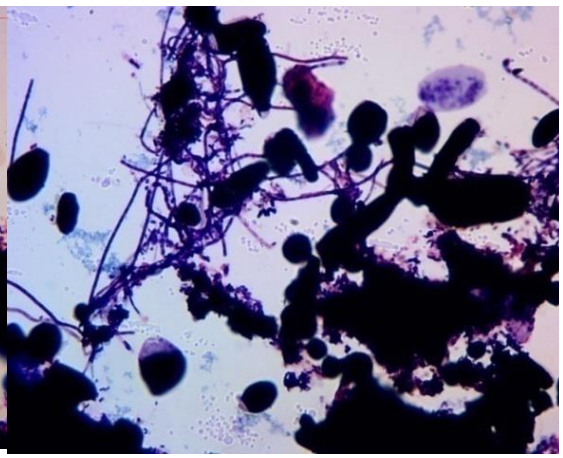
Microphoto 23: View of activated sludge Gram staining sample (second week), flocs, bright field microscopy, 100X.



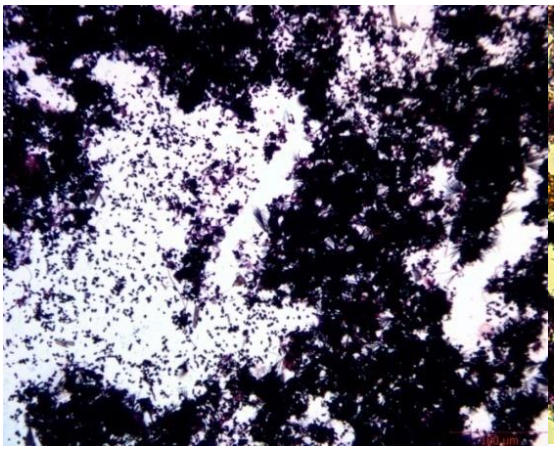
Microphoto 24: View of activated sludge Neisser staining sample (second week), eukaryotic cells -*Microthrix parvicella* filaments incluiding granules in cell filaments, bright field microscopy, 1000X.



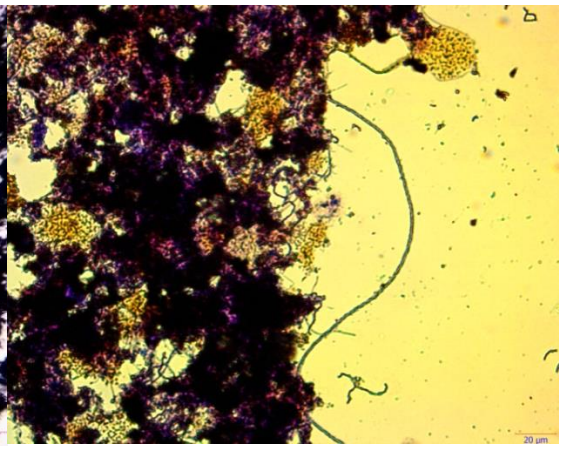
Microphoto 25: View of activated sludge Gram staining sample (third week), flocs, Gram positive and negative filamentous bacteria and eukaryotic cells (fungi cells and spores), bright field microscopy, 1000X.



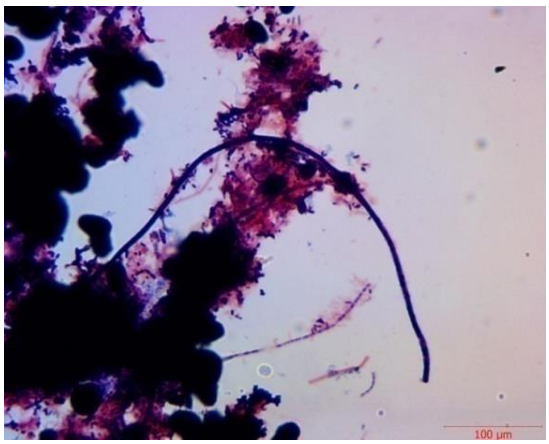
Microphoto 26: View of activated sludge Gram staining sample (third week), flocs, bundle of Gram positive filamentous bacteria, and eukaryotic cells, bright field microscopy, 1000X.



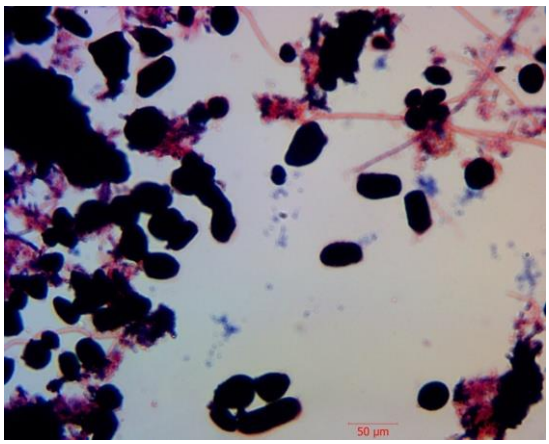
Microphoto 27: View of activated sludge Gram staining sample (third week), flocs, Gram (-) and Gram (+) filamentous microorganisms, bright field microscopy, 40X.



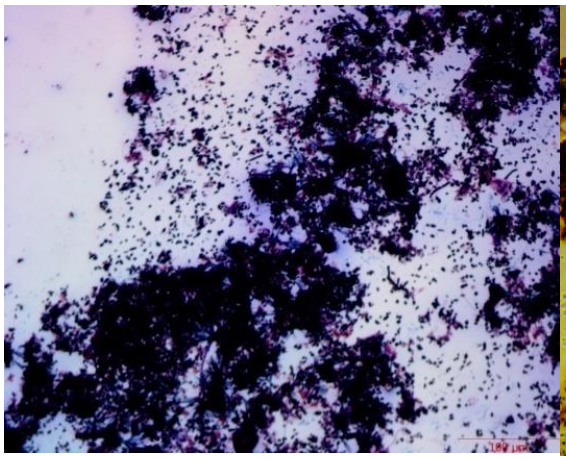
Microphoto 28: View of activated sludge Neisser staining sample (third week), flocs, filamentous bacteria and eukaryotic cells, bright field microscopy, 1000X.



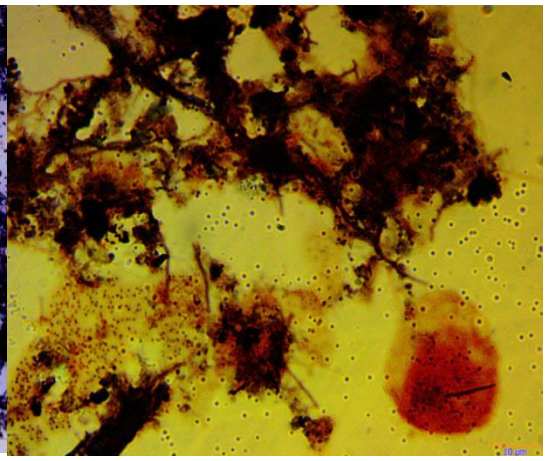
Microphoto 29: View of activated sludge Gram staining sample (fourth week), flocs, Gram (-) and Gram (+) filamentous bacteria, bright field microscopy, 1000X.



Microphoto 30: View of activated sludge Gram staining sample (fourth week), *Noistocida limicola* I, flocs, Gram (-) and Gram (+) filamentous microorgansisms, eukaryotic cells, bright field microscopy, 1000X.



Microphoto 31: View of activated sludge Gram staining sample (fourth week), loose flocs, Gram (-) and (+) filamentous microorganisms, bright field microscopy, 40X.



Microphoto 32: View of activated sludge Gram staining sample (fourth week), flocs, Gram (-), and Gram (+) filamentous microorganisms, bright field microscopy, 1000X.

Figure 4.11: Gram and Neisser staining results during study.

- **Microbiological Examination of the FO-MBR System**

5 L of activated sludge was provided from Pasakoy Wastewater Treatment Plant (Pasakoy WWTP) which has been treating domestic wastewater of Istanbul. The activated sludge has been monitored during the operation of the FO-MBR system for 13 weeks. Activated sludge is a mixture of microorganisms including bacteria,

protozoa, fungi, viruses, filamentous microorganisms, nematodes, rotifers and etc. Bacteria species are very activated and dominated in the system. Almost 90-95% of biomass in the system is constituted by bacteria and 2-3% is formed by protozoa species. Other groups of microorganisms are found almost 1-2% depending upon wastewater characteristics and system configurations. Pasakoy WWTP activated sludge was used as seeding for this system and it microorganisms were very diverse and their performances were very good its microbiological characterization was very similar to conventional activated sludge diversity.

Activated sludge bacteria in the FO-MBR system was determined by using microscopic examination and visualization system. Microbiological samples taken from the aeration unit of the system by weekly. Sample were taken from aeration unit of the system and examined under microscope using 4X, 10X, 20X, 40X and 100X objectives. Samples were first examined on the original sample preparing wet-mouth slides (alive samples) and taken some micro-photos of microorganisms and floc structures. Also prepared slides were dried out and then Gram and Neisser staining methods were applied. Stained slides were also examined under bright field microscope and micro-photos were taken and used for characterization of the activated sludge. The samples were examined for biological diversity and floc structure characterization of the system.

Examination of original activated sludge samples under phase contrast and bright field microscopy on wet-mouth slides was performed to determine especially floc structure, floc size, eukaryotic cells and prokaryotic cells specifically filamentous microorganisms that can cause sludge bulking and foaming and adversely affect settling properties of activated sludge causing solids separation problems.

Floc structure of the activated sludge was classified as strong and greyish-white color, little bit different than normal domestic wastewater activated sludge color which is usually pale brownish as it can be seen in the wet mouth slides of Microphotos 1-16. Flocs had a few of filamentous microorganisms and floc dimensions was usually about 0,1-1 µm diameter. They were not so compact structure but they were little bit loose just like as sponge appearance which had some pores and holes. Flocs were made up by *Zoogloea ramigera* bacteria species having fill of other bacteria species in a zooglea gelatinous net structure, just like similar to fingers of glove.

Bacterial diversity was very rich in the FO-MBR system activated sludge including Gram negative and Gram positive bacteria species having different morphological structures such as, coccus, bacillus, vibrio, cocco-basil, spiral and helical shapes, found in different sizes, lengths and dimensions. Cocci shaped bacteria were found morphologically as diplococci, tetracocci, streptococci, staphylococci and sarcina types (Eikelboom, 2000). There have also been found some soil originated bacteria species like *Actinomycetes* spp., which is seen on the Microphotos 21.

Filamentous microorganisms have been observed and defined in all samples especially on gram stained slides. *Microthrix parvicella* Gram positive in blue-purple color and *Nostocoida limicola* I Gram negative in pink color have been detected in flocs and bulk solution of the activated sludge. *Microthrix parvicella* was seen as primary dominant filamentous bacteria species having 5-10 filaments per floc and *Nostocoida limicola* I was secondary dominant filamentous species in the system. These filamentous bacteria species have been found in the low Food/Mass ratio systems. They cause bulking and foaming problems in the activated sludge aeration units increasing the surface area of the flocs and decreasing the density of the flocs. During the operation of the system, these filamentous species were always seen and their subjective amounts were defined as "common" (subjective score 3) filaments according to method developedby Jenkins et al. 1993, which is subjective scoring classification of the filaments in the activated sludge. *Microthrix parvicella* is given in Microphoto 24 and *Nostocoida limicola* I is given in Microphoto 30.

Fungus species *Aspergillus* spp. have been found in the system as filaments and spore forms. Fungi species normally can be found in the activated sludge systems. They can prefer little bit acidic pH conditions, usually 5,5-6,5 range. In this study because of high salinity concentrations, we can say that the draw solution has provided suitable conditions for fungi species. They could able to resist salinity conditions in the systems and survived under FO-MBR operational conditions. Fungi species and their spores were observed throughout the operation of FO-MBR system. Fungi filaments and cells are given in Microphotos 22 and 25.

Protozoa species are eukaryotic cells and were observed especially in original samples before drying out and staining of slides at the starting of the FO-MBR system. They were gradually disappeared in 2 weeks after started up and later during the operation time. Ciliated protozoa such as *Paramecium* spp., *Chilodonella* spp., *Colpidium* spp.

and *Lionotus* spp. have been frequently observed in the first days of the FO-MBR system operation. These ciliated free swimming protozoa have fed on bacteria cells especially consuming free bacterial cells but not much of attached ones. Their dimensions were in the range of 20-200 µm width and length depending on species and numbers of them have gradually decreased during the operation. The numbers of ciliated protozoa were very few at the last days of the operation, only *Colpidium* spp. and *Lionotus* spp. have resisted to higher salinity concentrations.

Some attached protozoa species have also been detected in the system as a small colony groups. The most observed attached species of protozoa were *Vorticella* spp. and *Opercularia* spp. They had been observed as 3-5 cell colonies attaching on the flocs. Their number were increased in colonies (10-20 cells in a colony) with increasing salinity concentrations thru the operation (Microphotos 3, 5, 9). Increasing cell number in a colony can be explained resisting to environmental stress conditions here that it was increasing salinity (conductivity) concentration.

Other type of protozoa species for example crawling ciliated protozoa *Aspidisca* spp. and *Euplotes* spp. have been observed in the system in a few numbers per ml of activated sludge. They have been survived in the system during the operation time for 13 weeks.

Flagellated protozoa group has been represented by only *Bodo* spp. which is a small approximately 10-20 µm length and very active form in/on activated sludge flocs.

Metazoa group of eukaryotic cells has been also represented by rotifers such as *Euchlanis* spp. and *Philodina* spp. (Microphoto 12).

Nematodes are multicellular free living roundworms feeding on bacteria and present in the fresh domestic wastewater. These cells have been found in a few numbers early stages of the activated sludge system and then disappeared later stages of the FO-MBR system.

The activated sludge of the FO-MBR system was changed to more stable biological forms with increasing salinity (conductivity) concentration. The biological diversity has been decreased throughout the first 6-8 weeks and then biological diversity has been conserved and survived without decreasing in the biomass amount. The resistant species have been survived and activated in the FO-MBR system that those species can be classified as euryhaline (able to tolerate a wide range of salinities)

microorganisms. The disappeared species of microorganisms at the started up period of FO-MBR can be classified as stenohaline microorganisms which cannot tolerate a wide fluctuation in the salinity.

Osmosis, as it is known that it is a way of transportation of solids and solvent molecules through a semi-permeable membrane into a higher solute concentrationregion from a lesser solute concentration region. In this research draw water conductivity had have high salinity concentration, microorganisms were tended to lose their content towards to the draw solution or higher concentration medium. Microorganisms who were resisted to high conductivity values they were only able to live and survive in this condition and these microorganisms are called as euryhaline or salt lovers. Some prokaryote species especially archaic bacteria are able to live in high salinity medium such as in 2-10% salinity concentrations. Many other prokaryote bacteria and eukaryote species cannot survive in that much salinity concentrations.

The stable biological structure at the last part of the activated sludge have helped to membrane quality being protected and preventing biofouling of the membrane. Because of preventing biofouling for a longer time in the activated sludge, the life spun of the membrane was increased for providing better effluent quality and on flux and performance was continued for a longer time operation.

Extracellular polymeric substances (EPS) and Soluble Microbial Products (SMP) are produced by great percentage of microorganisms especially by bacteria species. These substances usually cause biofilms and biofouling on the surfaces and pores of membranes. Because these substances decrease life spun of membranes, EPS and SMP should be prevented to increase membrane performance and efficiency. In the FO-MBR system operation, EPS and SMP amount were decreased and levels of them were under normal activated sludge operation during the test. So this result has reflected in the system increased and longer life span, not closed pores and prevented biofouling of the membrane structure.

4.2 FO Membrane Characterization

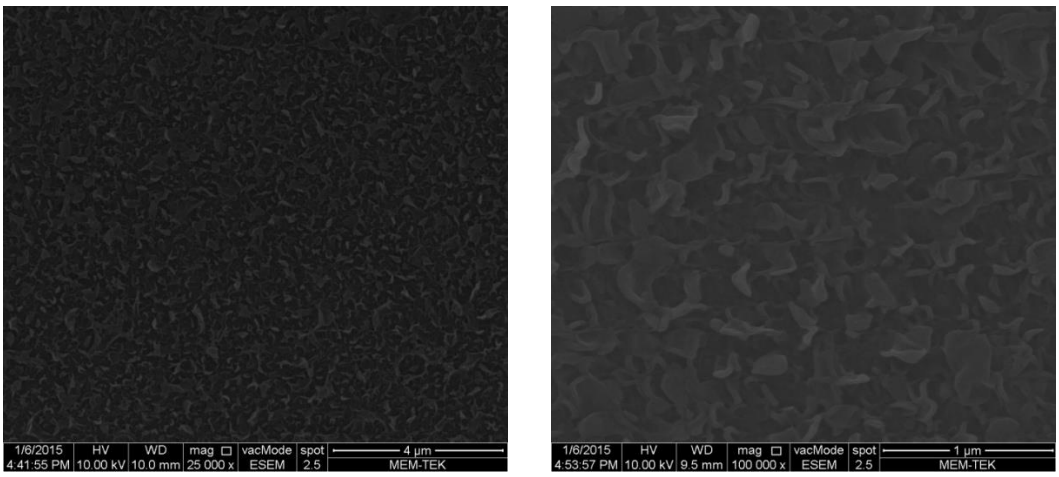
The selective FO membrane with the selected spinning condition was tested for assessing the characterization of their structures and anti biofouling features. Compared with pressure driven processes, FO was a relatively low fouling treatment

option for the absence of hydraulic pressures, and the foulant compaction might be milder due to the utilization of osmotic pressure to extract water (Achilli et. al., 2009). Results were given at the following sections.

4.2.1 Scanning electron microscopy (SEM)

Images of membrane surfaces (Figure 4.12) were taken by 25000X, 100000X, magnification to obtain general morphology of the membranes whether they had defects on them or not. The membrane was placed in pure water for 30 min, then dry it compleatly and used in SEM setup, also the membrane after operation is placed in pure water more than 24 hour, dry it well then can be observed surface morphology of the membrane. As before mention that, there is no significantly fouling on FO membrane when compare the images of both initial and used membrane.

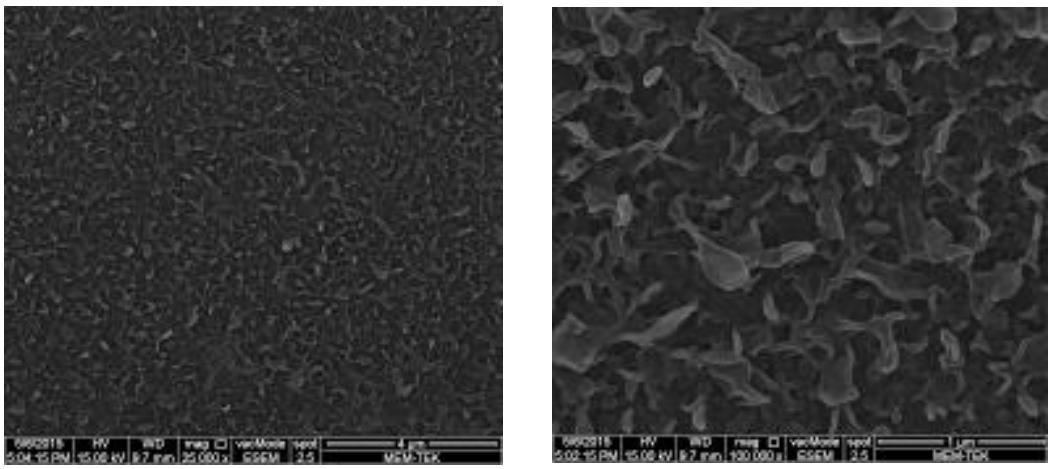
Initial new FO membrane SAM photograph is shown in Figure 4.12. and FO membrane photograph after operation is given in Figure 4.13.



(a) (b)

25000X 100000X

Figure 4.12: FO membrane before operation.



(a) (b)

25000X 10000X

Figure 4.13: FO membrane after operation

4.2.2 FTIR

Initial TFC (polyamide) FO membrane FTIR is shown in Figure 4.14 and the used membrane FTIR is shown in Figure 4.15. Both spectra are shown in Figure 4. 16.In the spectra, all used samples showed high absorbance at 1660 and 1580 cm^{-1}, which may be assigned to amides I and II in protein (Kimura et al., 2005; Qiu and Ting, 2014; Ramesh et al., 2006). Fig. 4.16 indicates that the peaks present at 1100 and 1013 cm^{-1}were due to symmetric and asymmetric C–O stretching in polysaccharides (Kimura et al., 2005; Ramesh et al., 2006). N–H stretching was observed at the peak at the wavelength of 3305 cm−1 (Zularisam et al., 2006). Through the FTIR spectra, it was evident that biological macromolecules including both proteins and polysaccharides were present in foulants.

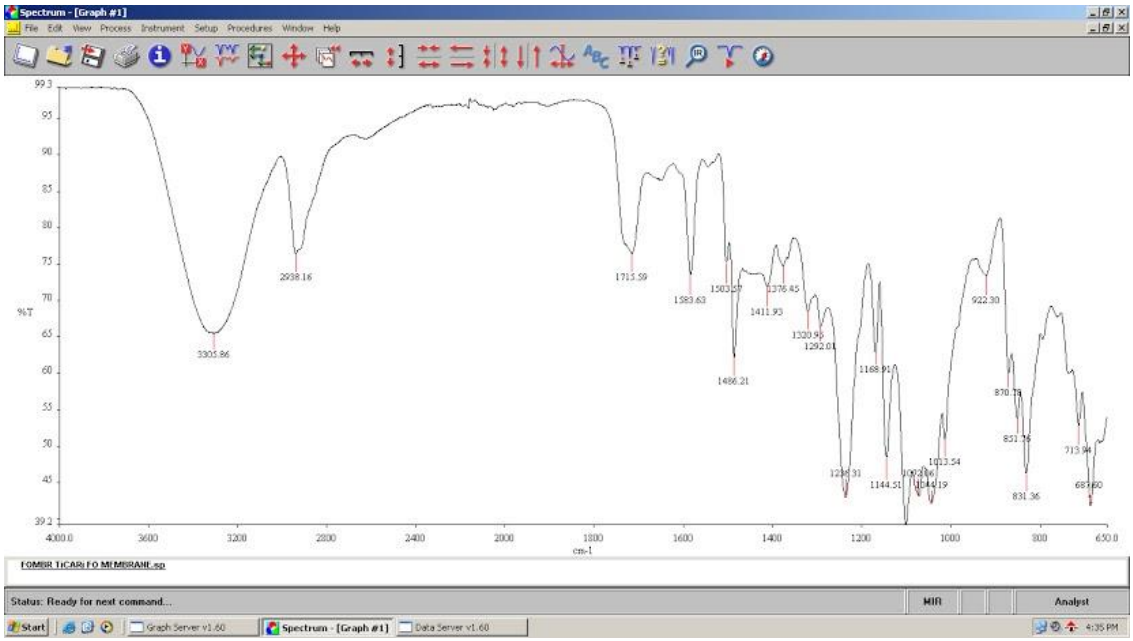


Figure 4.14: FO membrane FTIR before operation.

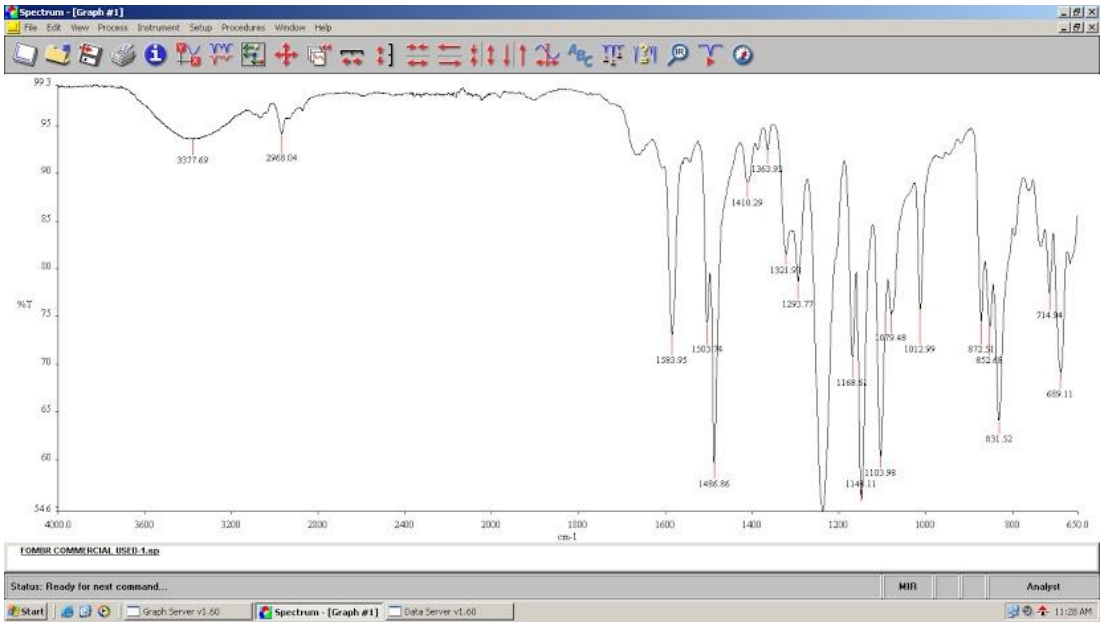


Figure 4.15: FO membrane FTIR after operation.

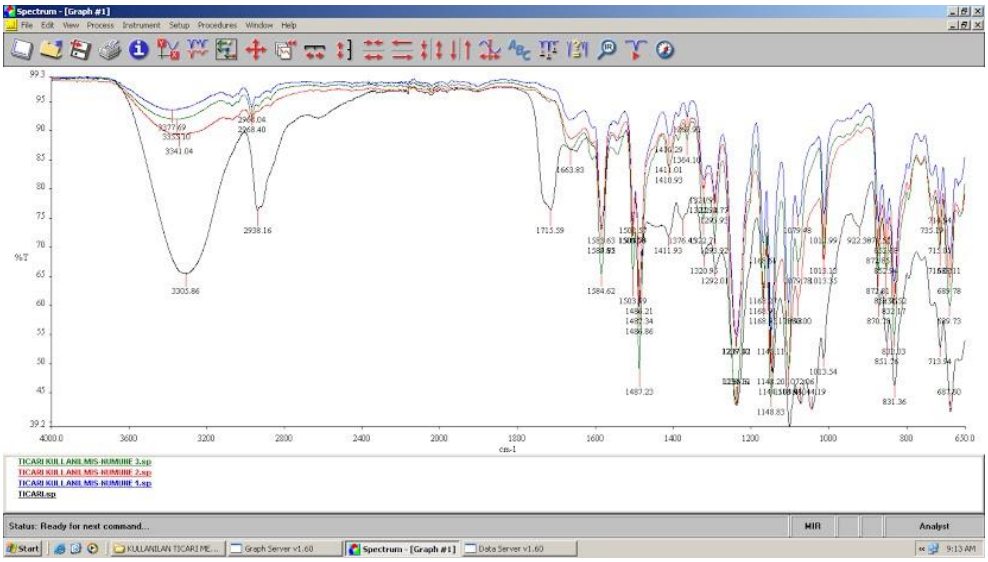


Figure 4.16: FO membrane FTIR before and after operation.

4.2.3 Contact angle

Contact angles of membranes were measured for investigating their hydrophobicity characteristics. In this study it is obvious from contact angles result which are shown in Table 4.7 the hydrophobicity characteristics was not significantly change. This indicates that the organic foulants affected the surface hydrophobicity similarly despite likely variations in the local DS concentration and flow distribution.

Table 4.7 : Contact angle results from FO membrane.

before using		after using	
contact angle	st. Deviation	contact angle	st. Deviation
41.61	2.30	25.15	4.43
27.39	2.19	23.31	4.90
25.45	2.84	24.74	4.59
33.94	3.37	22.16	3.42
27.16	3.13	23.54	2.00
		30.01	4.36
		29.37	1.58
		30.80	2.65
		26.92	3.85
		21.66	3.36
		21.72	7.33
		16.50	4.57
		16.12	5.87
Average of results:			
32.53	2.75	24.83	3.82

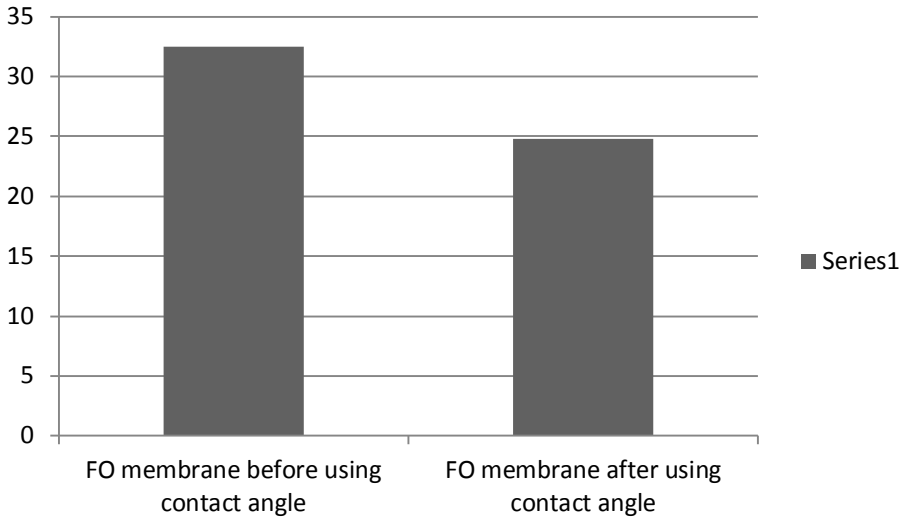


Figure 4.17: FO membrane variation in contact angle during the operation.

4.2.4 Surface charge of membranes

Zeta potential measurements were conducted to quantify the surface charge of the membranes and investigate the result of fouling on membrane surface charge (Figures 4.18 and 4.19). FO membranes exposed to domestic wastewater effluent became more negativelycharged than the virgin FO membrane. These results corroborate findings from contact angle measurements of the FO membrane.Recent research on the effect of membrane surface charge and solute permeation behavior indicates that the increasing negative charge could affect the type of coupled solute diffusion observed (Coday et. al., 2013). Namely, increasing negative surface charge appears to favor sodium ion permeation and may partially account for relatively high reverse salt flux observed during operation.

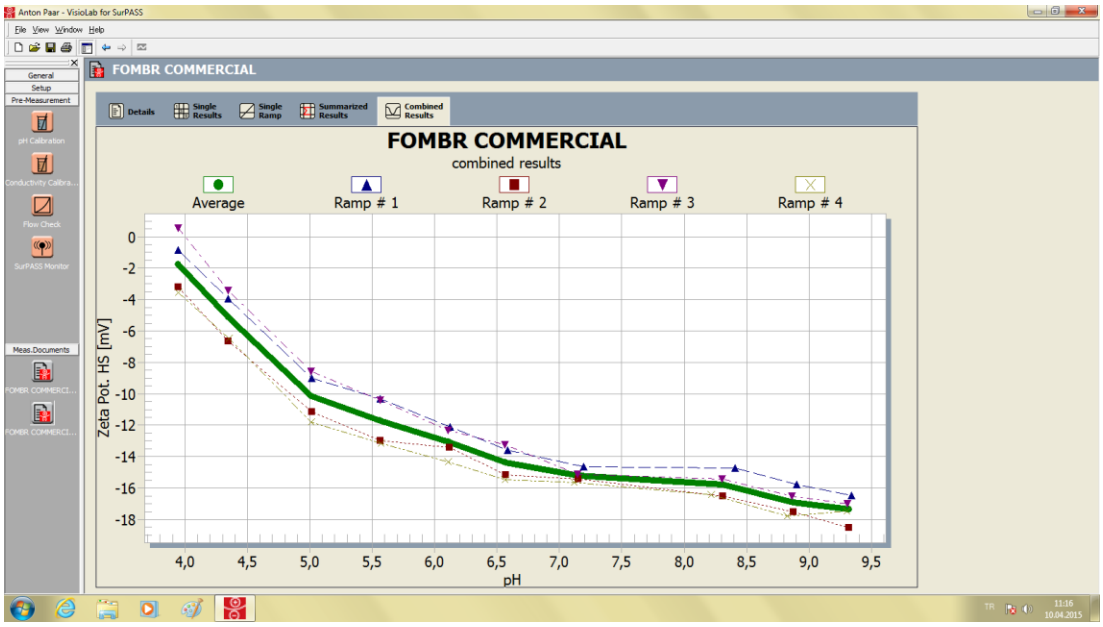


Figure 4.18: FO membrane Zeta potential before operation.

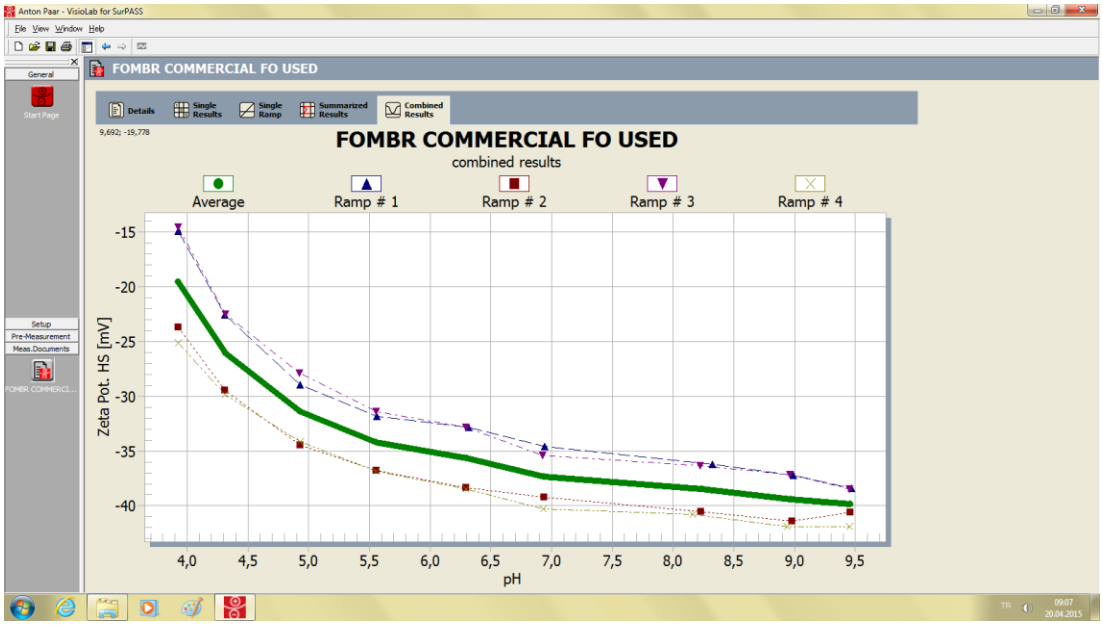


Figure 4.19: FO membrane zeta potential before operation.

5. CONCLUSION

FO-MBR is proposed as an option for wastewater treatment in light of the fact that less energy is obliged when contrasted with the traditional MBR. This study researched the execution of FO-MBR on treating municipal wastewater and in addition the FO membrane fouling affinity. It demonstrated the considerable capability of FO-MBR in municipal wastewater treatment. The experiments of FOMBR were conducted, with optimal FO module configuration and a suitable draw solution for the FO process according to (Achilli, et. al., 2009 and Zhang, 2011) studies.

In this study, during the FO-MBR operation, when the ambient temperature was high the COD removal efficiency results would be better. It be concluded that in higher temperatures, COD removal would be better generally, may be because of microorganisms have a better performance in high temperature. SS and VSS occumulation was occurred. The viscosity variation of activated sluge was increased with low slope during the experimentation. The decreased EPS content was probably because of the increased solubility of EPS fractions (e.g., protein and carbo- hydrate) at high salinity (Zhang et al., 2014). Thus, increase in the SMP content was observed when the NaCl loading was stabilized at 15 g/L. In this study the average flux of cycle 8 (21th, 22nd, day of FO-MBR operation) was calculated as 3.3L/m²-h. The salinity removal efficiency would be better by operation time. The draw solution was diluted from 50 ms/cm to 15 ms/cm in average, there is a little salt accumulation in the A.S tank.

FO-MBR system microbiological structure had very diverse at the start up period of the system and then microbiological diversity has decreased but population of the dominated species has increased. These microorganisms, conductivity resisting microorganisms including Gram negative and Gram positive bacteria, some eukaryotic cells, attached protozoa, fungi, worms and rotifers have survived in the FO-MBR system. Euryhaline species were resisted 5-7 mS/cm conductivity values and they were also very effective removing organic carbon, nitrate and phosphate.

Filamentous microorganisms, *Nostocoida limicola* and some fungi filamentous forms have been dominated the system throughout the operation. The floc structure of the activated sludge has been increased the quality and got compacted, densed and settled very well. It may be concluded that flocs have got bigger and made net structures on the membrane as a blanket so they have not caused pore biofouling increasing the life span of the membrane. The further study can concentrate on the specific euryhaline species in the FO-MBR and resistance limits for conductivity parameter.

The FO membranes were able to reject most of the micropollutants, 87% COD removal, and more than 99% turbidity removal, also the removal efficiency were 79% for nitrate, removal efficiency were 96% for phosphate.

The flux for the FO process using as feed and seawater as an average of 3.3 L/m^2-h. The draw solutionwas diluted from 50 ms/cm to 15 ms/cm, creating a feed for the RO process that can be desalinated at low pressure (15-17 bars), producing apermeate with conductivity lower than 5µs/cm. Backwash schemes for the FO-MBR were sufficient for the FO membrane cleaning, with 5 days interval.

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CURRICULUM VITAE

Name Surname: Hajar Ghasemzadeh Momen

Place and Date of Birth: Oroumieh - Iran, 12/09/1986

E-Mail: hajar.ghasemzadeh@gmail.com

B.Sc.: Agriculture Engineering, Urmia University

M.Sc.: Environmental Biotechnology, Istanbul Technical University

Professional Experience and Rewards:

MEM-TEK National Research Center of Membrane Technology of Istanbul Technical University (ITU), 2014-2015.

(January 2014 – May 2014) TUBITAK 1001 - COST Action Project (European Cooperation in Science and Tech.) Scholarship (High Performance Forward Osmosis Membrane Manufacturing and Determination of Their Wastewater Treatment Performance).