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(MASTER OF SCIENCE THESIS)

STUDY ON OPERATIONAL CONDITIONS TO MINIMIZE MEMBRANE FOULING IN MBR WASTEWATER TREATMENT PROCESS AT ITOB INDUSTRIAL ZONE

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Yakubu Abdullahi Jarma tarafından Yüksek Lisans tezi olarak sunulan "**Study on Operational Conditions to Minimize Membrane Fouling in MBR Wastewater Treatment Process at ITOB Industrial Zone (ITOB-OSB'de MBR Atıksu Arıtma Prosesine İlişkin Membran Kirliliğini Azaltmak İçin İşletme Koşullarının Optimize Edilmesi)"** başlıklı bu çalışma EÜ Lisansüstü Eğitim ve Öğretim Yönetmeliği ile EÜ Fen Bilimleri Enstitüsü Eğitim ve Öğretim Yönergesi'nin ilgili hükümleri uyarınca tarafımızdan değerlendirilerek savunmaya değer bulunmuş ve **29.07.2016** tarihinde yapılan tez savunma sınavında aday oybirliği/~~oyçokluğu~~ ile başarılı bulunmuştur.

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29 / 07 / 2016

Yakubu Abdullahi Jarma

ÖZET

İTOB-OSB'DE MBR ATIKSU ARITMA PROSESİNE İLİŞKİN MEMBRAN KİRLİLİĞİNİ AZALTMAK İÇİN İŞLETME KOŞULLARININ OPTİMİZE EDİLMESİ

Abdullahi Jarma, Yakubu

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Bu çalışmada, ITOB atıksu arıtma tesisinde arıtılan atıksudaki sertliğin giderilmesi ve MBR membranlarında gözlenen hızlı kirlenmenin minimize edilmesi hedeflenmiştir. Bu amaçla öncelikle ön arıtma yöntemi olarak koagülasyon uygulanmıştır. $Al_2(SO_4)_3$, $FeCl_3$, $Ca(OH)_2$, Na_2CO_3, $NaHCO_3$ ve katyonik polimer koagülant olarak kullanılmıştır. Koagülasyon aşamasında koagülant derişimi, pH, katyonik polimer etkisi detaylı olarak incelenmiştir. Elde edilen sonuçlara göre, atıksudan en iyi sertlik giderimi $Ca(OH)_2$ (A.G.) ile %87 düzeyinde elde edilmiştir.

Tez çalışmasının ikinci aşamasında pilot ölçek MBR sisteminin kurulumu yapılmıştır. Çalışmada, 0.04 mikron gözenek boyutlu, polietilen malzemeden yapılmış Kubota düz plaka MBR membranları kullanılmıştır. Çeşme suyu ve atık su kullanarak gerçekleştirilen testlerde, MBR membranlarındaki kirlenme zamana bağlı akı değişimi izlenerek incelenmiştir. Membranlar temizlenmek için farklı kimyasal yıkama stratejileri uygulanmıştır. Ayrıca arıtma aşamasında antiskalant kullanımının membran kirliliğini önlemede etkin olup olmadığı araştırılmıştır.

Anahtar kelimeler: Membran biyoreaktör (MBR), Membran temizliği, Memran kirliliği, Jar test, Koagülant.

ABSTRACT

STUDY ON OPERATIONAL CONDITIONS TO MINIMIZE MEMBRANE FOULING IN MBR WASTEWATER TREATMENT PROCESS AT ITOB INDUSTRIAL ZONE

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Master ScienceThesis, Department of ChemicalEngineering

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This study aimed to minimize membrane fouling on MBR membranes at ITOB wastewater treatment plant and hardness removal from wastewater. With this aim, first coagulation method was applied as pretreatment. $Al_2(SO_4)_3$, $FeCl_3$, $Ca(OH)_2$, Na_2CO_3, $NaHCO_3$ and cationic polymer as coagulant enhancer are used as coagulants. The effect of coagulant concentration, pH, the use cationic polymer as coagulant enhancer was investigated in details. From the results obtained, it was seen that $Ca(OH)_2$ gave the best rejection with respect to hardness removal (87%) from wastewater.

In the second step of thesis, the construction of a pilot MBR system was established. The membranes used in this study were Kubota flat sheet MBR membranes with a pore size of 0.04 micron made from polyethylene. In the field tests using both tap water and wastewater, the fouling on MBR membrane was investigated by monitoring flux decline vs time. In order to clean membranes, different chemical cleaning strategies were applied. In addition during MBR treatment study, using antiscalant to prevent membrane fouling was investigated in order to understand it is helpful or not

Key words: Membrane Bioreactor (MBR), Wastewater, Membrane cleaning, Membrane fouling, Jar test, Coagulants.

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1. INTRODUCTON

Water plays a complex and multifunctional role in the life of every living on earth, therefore it has functions that cannot be substituted by any other compound. The rapid increase in the population has increased the demand of food, clean air, irrigation, freshwater which is the major cause of the demand of water (UNEP, 2002). For example an adult is expected to drink about 2-4 L water/day while an infant to drink about 0.75 L water/day of water (Micale et al., 2009). Although the water consumption for domestic use varies depending on the countries, culture and belief and region, Coates et al. (2012) reported that 50-500 L water/day is the approximate amount of water that is used for cooking, washing and other domestic use. For agriculture purpose, we need large amount of freshwater especially for irrigation which result to about 90% of the water withdrawn. Water is also needed for farming especially during hot climate.

Energy remains one of the most significant factors for all living. The global energy supply nowadays depend highly on fossil fuel, during the production processes (extraction and refining processes) large amount of water is consumed. As a result of the release of greenhouse gases, there is a need to use renewable energy sources in order to protect the earth for the future generation. (Mielke, Diaz-Anadon and Narayanamurti 2010). Thermal electricity generation (for steam generation and cooling water), hydroelectric power generation (the water that is lost during evaporation) also do consume a lot of water. In the developed nations we do not consume water only for energy purposes but also in the production of paper, sugar, steel, soap, beer, food, plastics, and electronics. Table 1.1 and Figure 1.1 show some statistical values and figures of the water consumed in different sectors and industries.

Table 1.1 Water use per tonne of product produced, selected industries (Coates et al., 2012)

Product	Water use[a]
Paper	80-2,000
Sugar	3-400
Steel	2-350
Petrol	0.1-40
Soap	1-35
Beer	8-25

a. Amount varies with process used.

Source: Margat and Andréassian 2008.

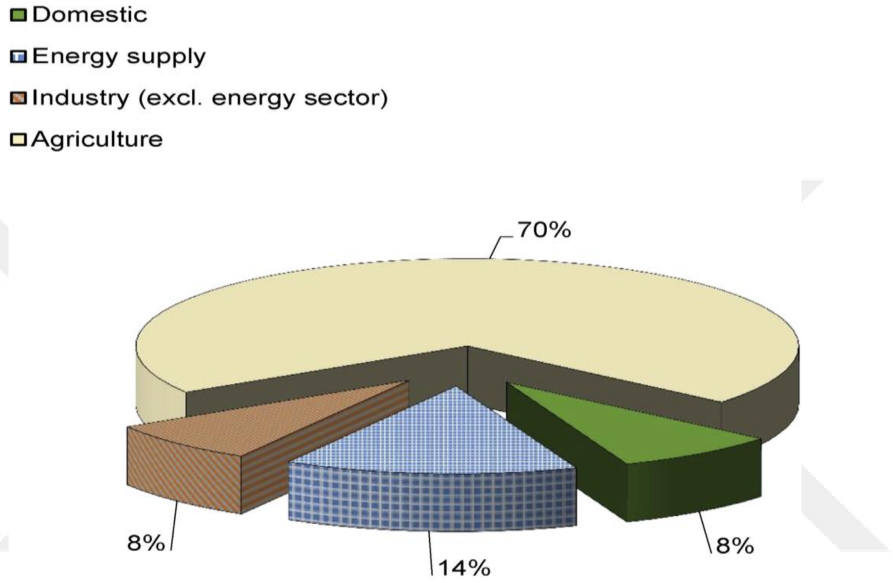

Figure 1.1 Global use of fresh water based on data from UN-Water (Coates et al., 2012).

When we look at the situation in our surrounding, we will find out that the situation is same in any of the developed or developing countries. For example in Izmir, industrial usage of water is one of the parameters that are considered for clean and efficient production (IZKA report, 2012). Below are the statistical data that shows the water consumption from the industrial point of view (Figure 1.2). Metal industries are the highest users of water in their production (72%) followed by food products and beverage industries (7%), chemical industries (6%), textile industries (3%), paper and paper products industries (3%), other non-mineral industries (3%), clothing industries (1%), assembling of imported electric machineries and other apparatus (1%), coal, refined petrol products and nuclear fuel industries (1%) and others (3%) (IZKA report, 2012).

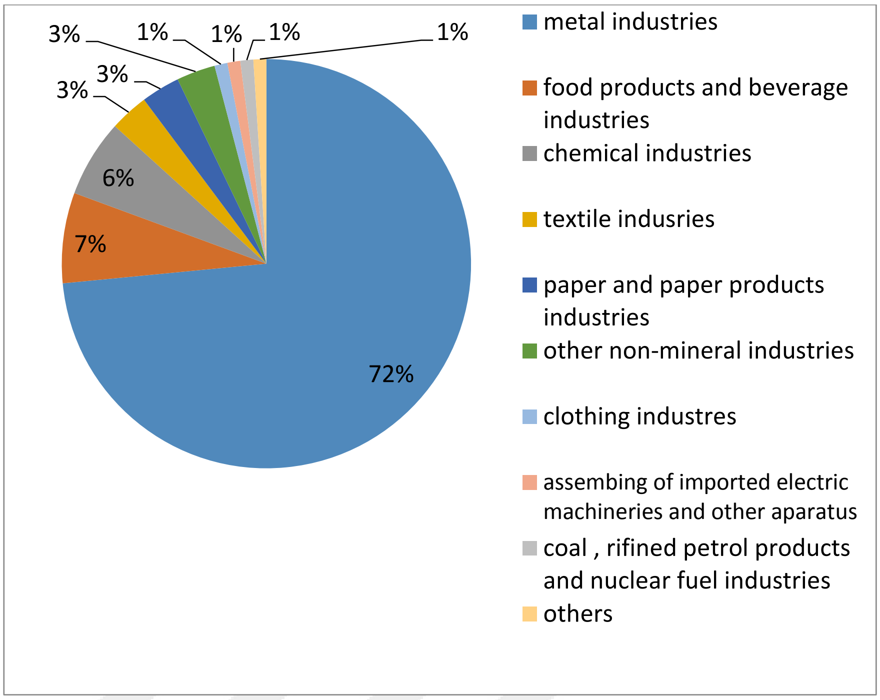

Figure 1.2 Statistical data showing industrial water consumption in Izmir (IZKA report, 2012)

It is clearly seen from the above fact that we use water in every aspect of our life, but the question we should be asking ourselves is, do we have that much freshwater compared to the amount consumed? Is the water in the aquifers enough for the feature generation? Of course, water cannot be destroyed but unfortunately can be polluted. Phosphorus, nitrogen, potassium and magnesium are the major elements used for plants growth, the inefficient use of these elements has led to contamination of the water in aquifers. The release of nitrate into our groundwater has now become a major environmental issue to be dealt with (Xue et al., 2009). High nitrate in groundwater remains a threat to the health because of its responsibility for blue baby syndrome in the new born babies (Fan and Steinberg, 1996 and Pastén-Zapata et al., 2014), and even cancer by the formation of carcinogenic N-nitroso compounds (Weyer et al., 2001). Several studies shows that most of the nitrate contamination in the ground water are coming from different agricultural practices and hydrogeological settings (Güler et al., 2012, Landon et al., 2011 and Stigter et al., 2008). According to United Nations World Water Development Report 3 (UNWWDR 3), the use of fertilizers in agricultural sector grows rapidly worldwide. The excessive release of nitrate and phosphorus to the receiving bodies causes eutrophication and seasonal hypoxia which are part of the threat to the aquatic life. United Nations World Water Development Report3 stated that more than 80% of the sewage discharged in the developing countries has led to pollution in the nearby rivers, lakes and coastal areas. The above mentioned problems are some of the problems only from nitrate release to the environment from agricultural point of view.

As of now we have seen the importance of water to our life and the dangers that will arise if we continue to draw the water from the aquifers without proper management, and also the danger in the contamination of our water sources. One of the topics of discussion today is the proper usage of water so that the next coming generation will be able to benefit from the nature. According to report by IZKA, Izmir is in the border to the countries that are not rich in terms of water (IZKA report, 2008). Therefore, if proper management of water is not considered, it will lead a serious problem. It is reported that the industrial sector consumes about 15.2% of the total water consumed by the city and only 10% of the water discharge is used as recycle (IZKA report, 2008). By increasing the percentage of the recycled water we will definitely decrease the water stress facing the city. Membrane bioreactor (MBR) process is the promising process for municipal/domestic and industrial wastewater reclamation tool.

1.1 Membrane Separations

Before membrane bioreactor (MBR) concept is explained, there is a need to review the term membrane filtration. The membrane filtration is the separation of two or more components from a fluid stream, in other word is the separation of solid or insoluble particles from a liquid stream. Generally membranes are categorized according to: their pore size, their molecular cut-off or the driven force which is generally pressure. Therefore, when the pore size becomes smaller the molecular cut-off decrease and also the driven force to be applied (pressure) increases, hence the energy required is increased that makes the process to become more expensive.

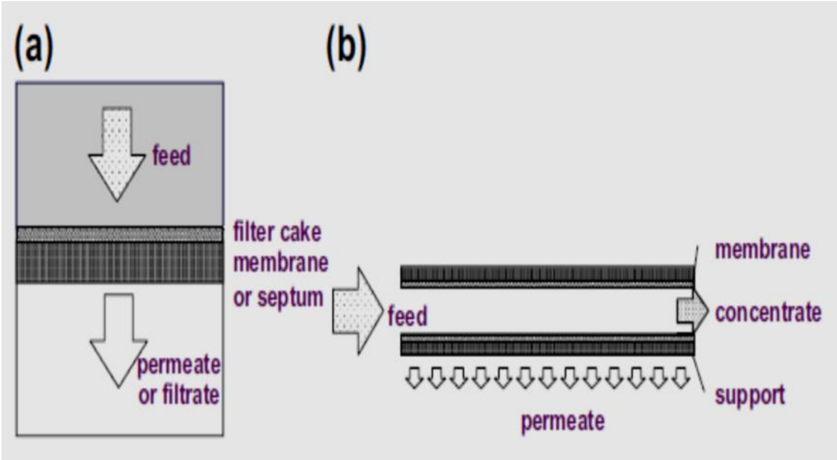

Figure 1.3 (a) dead end and (b) cross flow filtration (Judd, 2011)

During filtration, the wastewater to be separated is passed from the surface of the membrane as shown in Figure 1.3, the product water can be collected vertical to the flow as in the case of cross flow) (Figure 1.3b) or on the same direction with feed as shown in Figure 1.3a (dead end). The product water that passes through the membrane is called the filtrate or permeates and the component rejected by the membrane is called the retentate or concentrate. From Figure 1.3 when we carry mass balance on solute we have

$$Q_f \times C_f = Q_p \times C_p + Q_C \times C_c$$

Where;

Q_f: feed flow rate

C_f: concentration of solute in the feed

Q_p: permeate flow rate

C_p: concentration of solute in the permeate

Q_c: concentrate flow rate

C_c: concentration of solute in the concentrate

In the case of dead end filtration, the concentration term will drop since there is no concentrate collection as in the case of submerged MBR.

The membrane rejection can also be calculated from the solute mass balance as follows:

$$Rej = \frac{C_f - C_p}{C_f}$$

Water recovery of the process can also be calculated as follows;

$$Recovery = \frac{Q_p}{Q_f} \times 100$$

The recovery in the dead end filtration is generally close to 100%, but in the cross flow it varies depending on the design and operational condition.

Flux is the amount of water passing through a unit area of a membrane per time.

$$J = \frac{Q_p}{A}$$

Where Q_p is flow rate of the permeate in L/h, A is the active area of the membrane (m^2) and J is the flux (LMH).

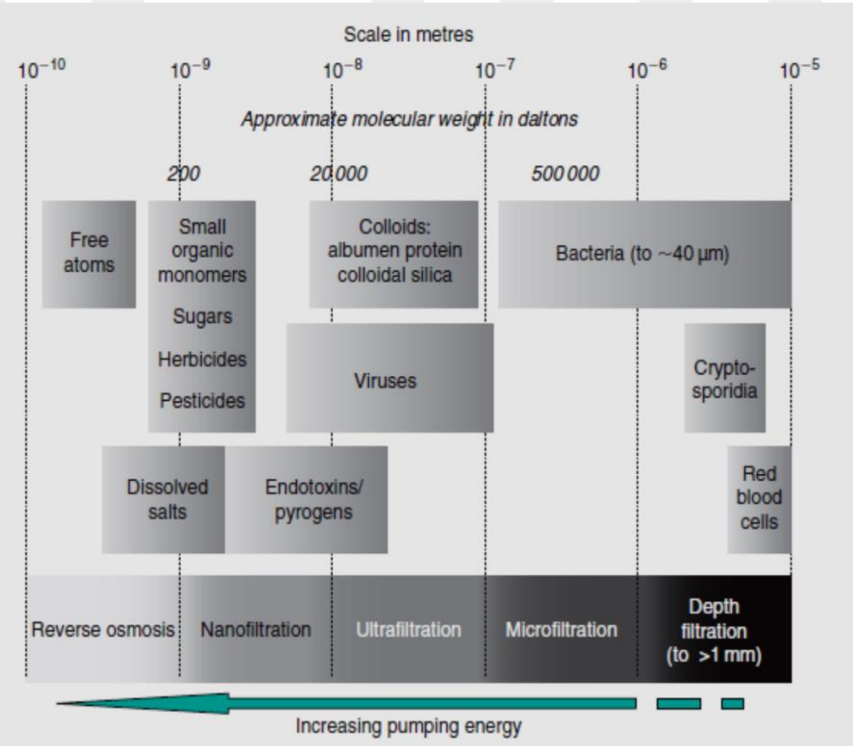

Figure 1.4 Membrane separation processes Overview and ranges of compound rejection (adopted Judd and Jefferson, 2003)

As can be seen from Figure 1.4, basically there are four membrane separation processes:

Reverse Osmosis (RO),

Nanofiltration (NF),

Ultrafiltration (UF),

Microfiltration (MF).

RO membranes are dense and hence RO is a high-pressure membrane process in which a pressure that is greater than the osmotic pressure should be applied to the system before permeate is obtained. Thus, the water flows in the opposite direction to the natural flow across the membrane. The rejected part which is the part of the unconverted raw water is taken away from the system as retentate. RO membranes are the most prepared membranes associated with the rejection of single charged (monovalent) ions, like sodium (Na^+) and chloride (Cl^-). Given that the diameter of these ions is less than 1 nm, in fact the pores of RO membranes are only visible using the most powerful microscopes (Judd, 2006).

NF membranes have a similar principle with the RO processes. The pressure applied in these systems are less compared with the RO systems and also the univalent ions like Na^+ and Cl^- are filtered while the polyvalent ions are retained. In particular, NF membranes are used to remove divalent ions, such as calcium and magnesium that contribute to water hardness, as well as dissolved organic material (Wilf and Klinko, 2001). The rejection of salts by NF membranes is more complicated like in the case of RO membranes, thus the separation mechanism depends on both molecular size and Donnan exclusion effects caused by increase of the acid groups attached to the polymer backbone (Baker, 2004).

UF and MF can separate species by physical or size separation; in short the mechanism can be described as separation that is based on fine screen or sieve filtration. The pore size at the surface of a membrane is highly uniform, with a narrow pore size distribution. The particles with a greater size compared with the porosity of the membranes are rejected, and thus remain on the side of the bulk solution. On the other hand, any particle that has a size smaller than that of porosity of the membranes passes to the filtrate side, which is generally called permeate (Pearce, 2007).

UF can remove the finest particles found in water supply. Depending upon the pore size of the active layer of the membrane; the typical removal capability of UF membranes used for general water treatment is 0.01–0.02 micron. MF membranes exhibit a porosity that is larger than that of the UF membrane which ranges between 0.1–0.2 microns. As can be seen from Figure 6, UF membranes have some advantages over MF membranes because they serve as disinfection barrier, since all bacteria and viruses are rejected by UF membranes while in the case of MF membranes the rejection is only 100% for bacteria but virus can be found in the permeate side.

1.1.1 Membrane materials

According to Judd (2006), the membrane materials are classified into two groups, these are polymeric and ceramic as can be seen in Figure 1.5, although metallic membranes also do exist which are used for very specific applications. Generally membranes are formed from a thin surface layer, which provide the required perm selectivity on top, followed by a thicker porous support for mechanical stability. The membrane material should have a certain qualities like resistance to chemical and thermal attack, pH and oxidants concentrations e t c.

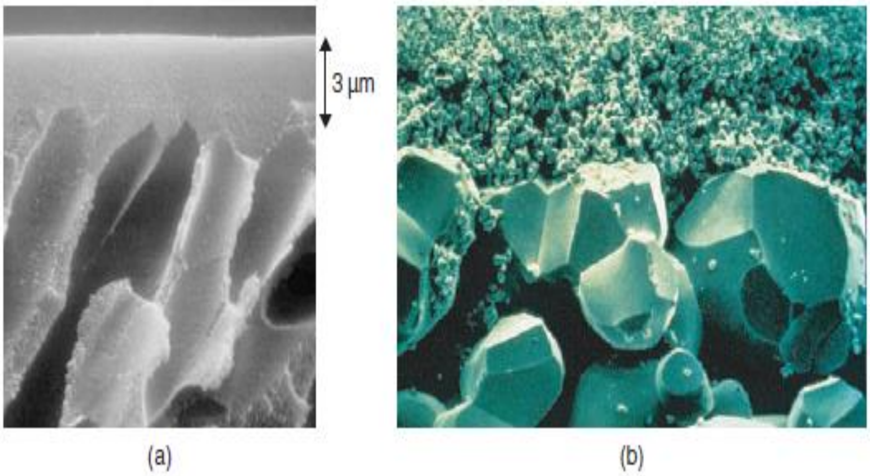

Figure 1.5 Anisotropic UF membranes; (a) polymeric (thickness a "skin" indicated) and (b) ceramic (Judd, 2006)

Although any polymer can be used in forming a membrane, but only a limited number of materials are suitable for the duty of the membrane separation which are:

Polyvinylidenedifluoride (PVDF)

Polyethylsulphone (PES)

Polyethylene (PE)

Polypropylene (PP).

All the polymers mentioned above can be used to form different type of membranes depending on the method or technique applied. One of the problem with these polymers is they have hydrophobic characteristics, therefore some modifications are done to the active layer using the techniques like chemical oxidation, plasma treatment, organic chemical reaction or grafting to make it hydrophilic (Judd, 2006).

1.1.2 Membrane configurations

This defines the geometry of the membrane, orientation of the permeate or retentate flow and the way it is mounted on the module. It is good to remember that the produced membrane should have some quality as mentioned before like good chemical resistance, low cost, simple maintenance procedure, high surface area, and high fouling resistance and so on. Judd (2006) gave the following configurations below which are based on planer or cylindrical geometry that are used in the MBR technology, also in Figure 1.6 we can see the pictures of these configuration for visual understanding. Considering the way they promote turbulence during filtration and the cleaning protocol that is employed to prevent or to remove fouling from the surface of the membrane:

Plate-and-frame/flat sheet (FS)

Hollow fiber (HF)

(Multi) tubular (MT)

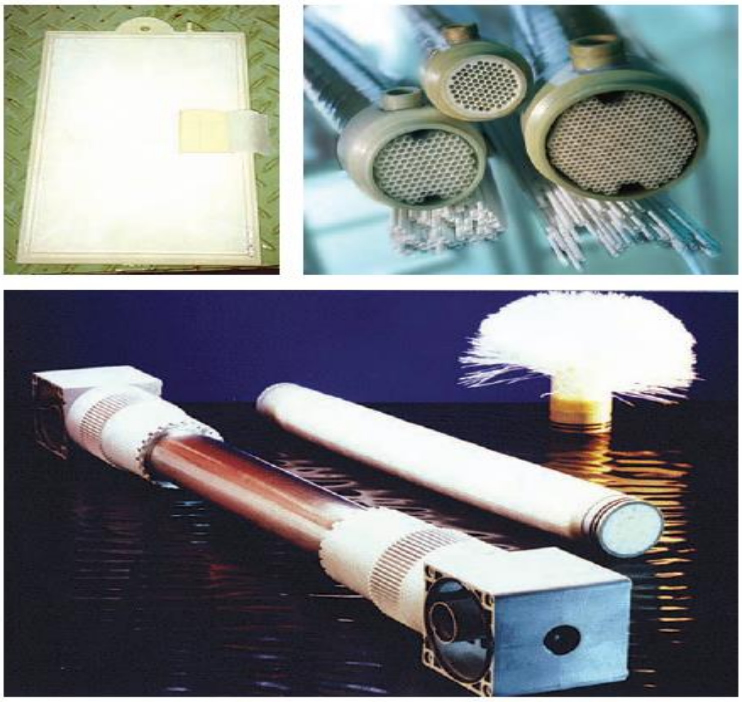

Figure 1.6 From top FS on the left, MT on the right and HF at the bottom (by the permission of Kubota, Wehrle and Memcor).

1.2 MBR processes

MBR is the common method used in the last decade in the treatment of industrial and municipal wastewater as the conventional method .The concept of MBR is similar to that of the conventional method used in wastewater treatment in which the removal dissolved and suspended organic matter are degraded through microorganisms, For the biodegradations of the waste in the wastewater some conditions should prevail in order to keep the microorganisms alive. One of the most important factors is the BOD (biological oxygen demand) or COD (chemical oxygen demand) which are both the ways to measure the amount of oxygen uptake for the oxidation of organic matter. The microorganisms need organic matter (as food) to generate energy for their growth. On the biological process, cellular materials are produced by the microorganisms which are popularly known as soluble microbial product (SMP) and extracellular polymeric substances (EPS), which are subsequently settled by gravity by leaving the clean water. This process can be aerobic which is oxygen dependent or anaerobic (oxygen in depended). The EPS and SMP are one of the factors that lead to the bottleneck of MBR process. The solid-liquid separation in MBR systems is done through a membrane filtration (MF or UF). Thus, MBR is the combination of conventional biological wastewater treatment and the application of membrane filtration instead of clarifiers used in the conventional way.

1.2.1 MBR process in wastewater treatment

The increase in the awareness of the dangers confronting our environment has led to the increase in more and strict policies that are related to environmental protection, for example the pressure that is imposed by the regulatory bodies increases in the limits on pollutants concentration before wastewater discharge. This aspect becomes very important in the activities producing highly saline, recalcitrant, slowly biodegradable and toxic compounds such as fish canning, petrochemical, petroleum, and tannery industries (Munz et al., 2008, De Temmerman et al., 2014, Sharghi et al., 2014, Zhang et al., 2014). For this reason nowadays more attention has been paid to the sector of the MBR technology. According to the previous researches on the MBR, the results shows that excellent efficiency was attained in term of the removal of bacteria and viruses from wastewater (Gunder et al., 1998; Steven et al., 1998; Krauth and Staab, 1993; Lv et al., 2006; Zheng et al., 2005). Due to the presence of membrane filter in the system, we are able to eliminate some suspended substances and big molecules, thereby giving permeate with a turbidity of less than 0.2 NTU (Emmanuel et al., 2004). Although in the recent years the use of membrane in the treatment of wastewater was considered to be unsustainable because of the high operating cost in the sector, the cost caused by low mechanical strength and chemical tolerance of the previous manufactured membranes. However, the increase in the stringent standards and decrease in the costs are making MBR usage more common than the conventional method. In the recent years, MBR application has increased rapidly in the treatment of industrial and municipal wastewaters. Figure 1.7 shows how the use of MBR increased rapidly in United Kingdom and Ireland.

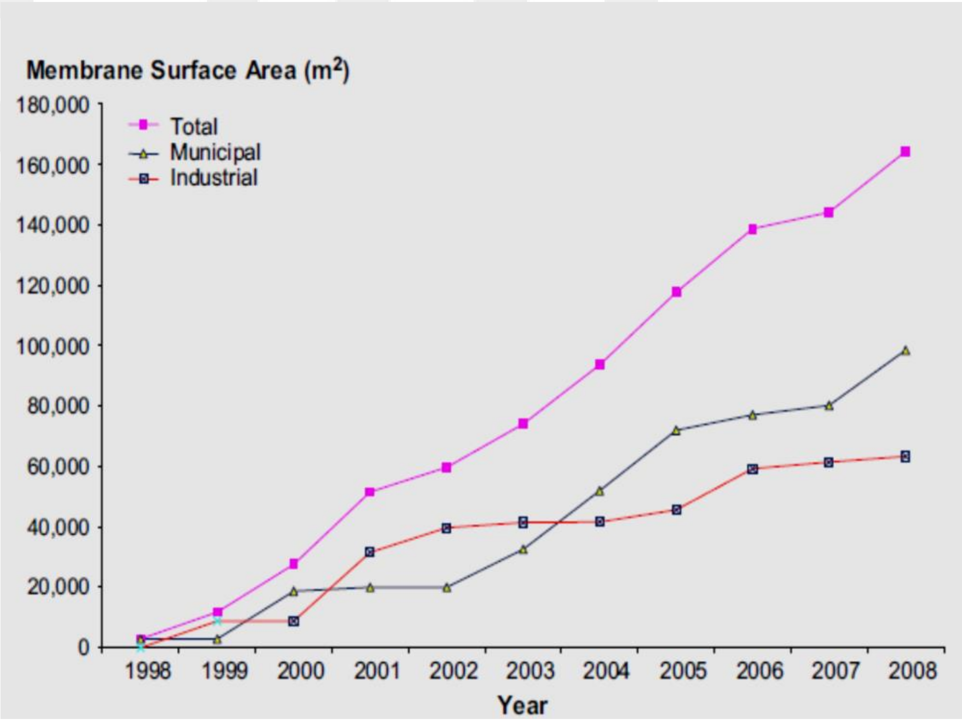

Figure 1.7 Cumulative installed membrane surface area in the United Kingdom and Ireland,Kubota plant (Judd, 2011).

It is clearly seen that MBR have considerable attention especially in the treatment of municipal and industrial wastewater. To date there have been not less than 50 individual MBR membrane suppliers and hundreds of large scale treating more than 100,000 m^3/day worldwide (Wang et al., 2008; Judd, 2011). In addition the sector is expected to increase from in the capacity and the application as well. According to literature, MBR has been used in the treatment of wastewater and water reuse in different industries (Figure 1.8), these include the industries producing wastewater containing heavy metals, and micro-contaminants, pharmaceutical industry, oil industry and refinery, and petrochemical industries (Mousavi et al., 2009, Sadeghi et al., 2010, Qin et al., 2007, Reemtsma et al., 2002, Manafi et al., 2011).

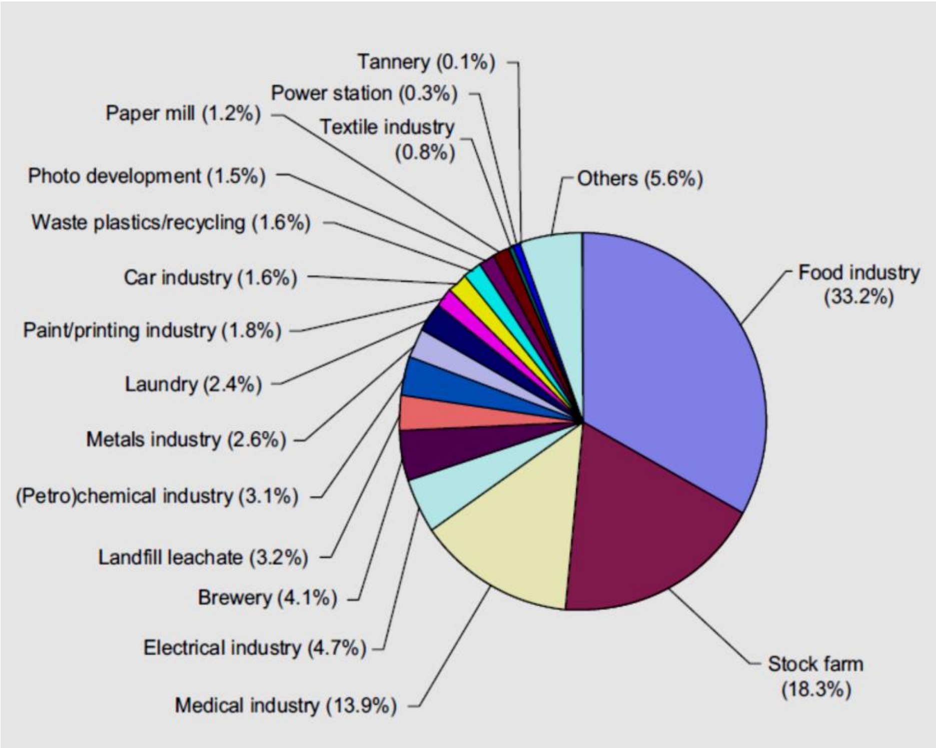

Figure 1.8 Percentage of MBR installation for different sectors in Japan (Judd, 2009)

1.2.2 MBR configurations

Membrane bioreactor configuration used in wastewater treatment can be classified into side-stream and submerged (immersed) configuration. These configurations are based on the way in which the membranes are integrated to the biological treatment. At the present time, the submerged configuration is preferred than the side-stream configuration, because in the side-stream systems, the membranes which are solid-liquid barrier are placed in a separate unit from the biological treatment unit (Figures 1.9 and 1.10), thus the mixed liquor is pumped in to the membrane separation unit. Like in any other biological treatment oxygen is necessary for the biological activities, which is supplied to the reactors in form of air. The main reason of supplying the air is not only for biological activities alone but also to create a shear on the surface of the membranes (to reduce membrane fouling), which make the system to be expensive. According to Judd (2006), the side–stream MBRs required more energy in contrast to the submerged MBRs (Table 1.2). Although both configurations require shear in order to reduce the effect of mixed liquor on membrane fouling, but during pumping in the immersed systems the flocs are broken into smaller size thereby increasing internal clogging (Tardieu et al., 1999; Wisniemick and Grasmick, 1998) which will be discussed in Part 1.4 of this thesis.

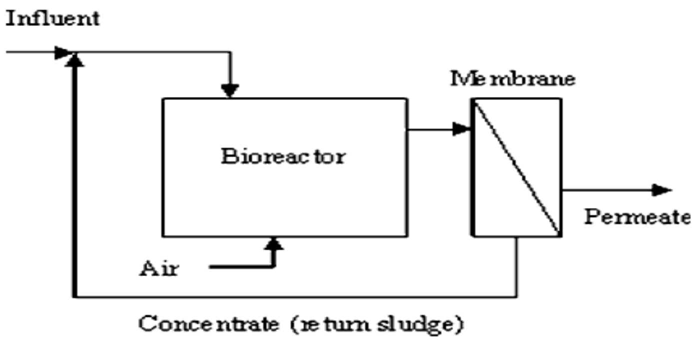

Figure 1.9 Side-stream MBR configuration (Radjenovic et al., 2007)

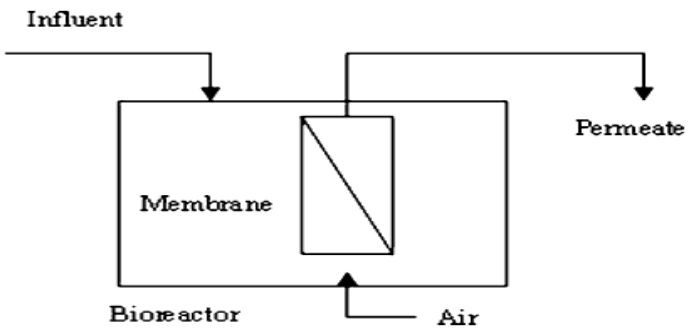

Figure 1.10 Submerged MBR configuration (Radjenovic et al., 2007)

Table 1.2 Comparison of filtration conditions for tubular and immersed MBRs (Radjenovic et al., 2007)

	Side-stream tubular membrane	Submerged membrane
Manufacturer	Zenon	Zenon
Model	Permaflow Z-8	ZeeWeed ZW-500
Surface area [m^2]	2	46
Permeate flux [$L\,m^{-2}\,h^{-1}$]	50–100	20–50
Pressure [bar]	4	0,2–0,5
Air flow rate [$m^3\,h^{-1}$]	–	40
Energy for filtration [$kWh\,m^{-3}$]	4–12	0.3–0.6

1.2.3 Advantages and disadvantages of MBR system

MBR technology creates a good atmosphere in both industrial and domestic wastewater treatment. Some of these advantages are as follows (Judd, 2011):

High sludge age (SRT) applicability,

- Low footprint compare with conventional activated sludge (CAS) systems
- A pathogen barrier, since it gives 100% bacteria and virus free effluent for UF attached MBRs (decrease the cost of disinfection on the process economy)
- Denitrification can be achieved in the system
- Significant removal of some dissolved organic carbon can be achieved
- Further pretreatment is not needed since it can treat a screened sewage
- Much mechanical labor is not needed
- Its ability to produce high quality effluent with low sludge production
- It also gives good resistance to organic loading and hydraulic variation

Disadvantages of MBRs

- Membrane fouling issues resulting to drop on the permeate side.
- It requires high energy input.
- Very complex control system is required.
- No instantaneous start up.
- There is no much experience from large scale system.
- Limited knowledge about membrane fouling.

1.2.4 Comparison of MBR systems to conventional activated sludge (CAS) systems

As mention from the above sections, the principle in the biological treatment in MBR and CAS are same. One of the factors that differentiate MBR from CAS is the presence of membrane for solid-liquid separation, while in CAS, the separation is done in a special device called clarifier in which gravity plays the very important role. Below are some of comparisons of MBR with CAS. In MBR systems, fluctuation in the feed flow rate or concentration has no significant effect on the process, while in CAS systems, variation of these factors can alter the process (Tchobanoglous, 2004).

The operating and capital cost of MBR seems to have exceeds that of CAS systems, but it is clear when high quality water is needed for reuse, the understanding of emerging compounds and their effect the use of MBR can become necessary despite its high cost compared with the CAS (Tchobanoglous, 2004).

Fouling is one of the drawbacks of MBR technology as will be discussed in this thesis, one of the factors causing this, is the presence of EPS since they form a hydrated gel on the surface of the membrane thereby reducing the flow of the permeate. But in CAS this is desirable since it bind the biological flocs thereby making them easy to settle (Judd, 2011).

MBR is able to operate at high solid retention time (SRT) as shown in Table 1.3, this is one of the main advantages of MBR to CAS, this is so because in CAS the settle ability of the sludge is bad at high concentration, thereby making it difficult for the solid-liquid separation at the end of the process (Judd, 2011).

Table 1.3 Typical sludge concentrations (MLSS) as well as solid (SRT) and hydraulic retention times (HRT) of a CAS and MBR process (Kappel, 2014)

	CAS	MBR
MLSS (g/L)	1-3[b]	5-20[b]
HRT (h)	4-8[b]	4-6[b]
SRT (days)	5-15[a]	7-30[a]

a: Judd, 2011, b: Tchobanoglous, 2004

The ability of MBR to operate at high SRT shows the presence of dispersed bacteria which are advantageous in the overall population competition since the ratio of feed to microbial is low and sludge age is high, also the flocs in the MBR are smaller than that of CAS, hence the mass transfer of both oxygen and carbon is high which explain the high quality of the effluent (Radjenovic et al., 2007).

There is an excellence removal of turbidity in MBRs, because all the particulate matter is retained by the membrane unlike the effluent of CAS process. Also the presence of MF/UF membrane helped in removing all non-degradable organic matter and they are discharged with sludge

(Radjenovic et al., 2007). The contribution of membranes in the removal of organic matter is about 30% which is approximately equals to the removal of insoluble fraction by active biomass (Gander, 2000). In another study by Xing (2000), shows that 97% of the total COD was rejected in which about 12% of these were from the membrane separation.

In MBR systems it possible to produce a pathogen free effluent, that make the process advantageous than the CAS system. Radjenovic et al., (2007) reported that 5 log removal of E.coli and enteroccoci can be achieved with MBR. After very long term operation with the membrane especially in the case of submerged MBRs, the biofouling and other fouling means tend to upgrade the membranes to upper class, for example MF is upgraded to UF. Therefore at the start, the removal of phages and spores are inefficient but after some weeks of operation their elimination is possible (Radjenovic et al., 2007; Farahbakhsh and Smith, 2004).

It is known that removal of emerging compounds is one of the topics of discussion today. Their major sources mostly are synthetic chemicals that have being used in cosmetics, industrial and agricultural sectors. Compound like pharmaceutical active compounds (PhVCs), pesticides and industrial chemicals are produced in 100000 t scale worldwide (Radjenovic et al., 2007). After these chemicals are used, large portion of them are discharged to the wastewater treatment plants (WWTPs). The degradation of these compounds is incomplete in CAS, therefore the only answer to this problem is the use of MBR. The figure below (Figure 1.11) gives some rejection of these compounds in CAS (WWTP) and MBR (Radjenovic et al., 2007).

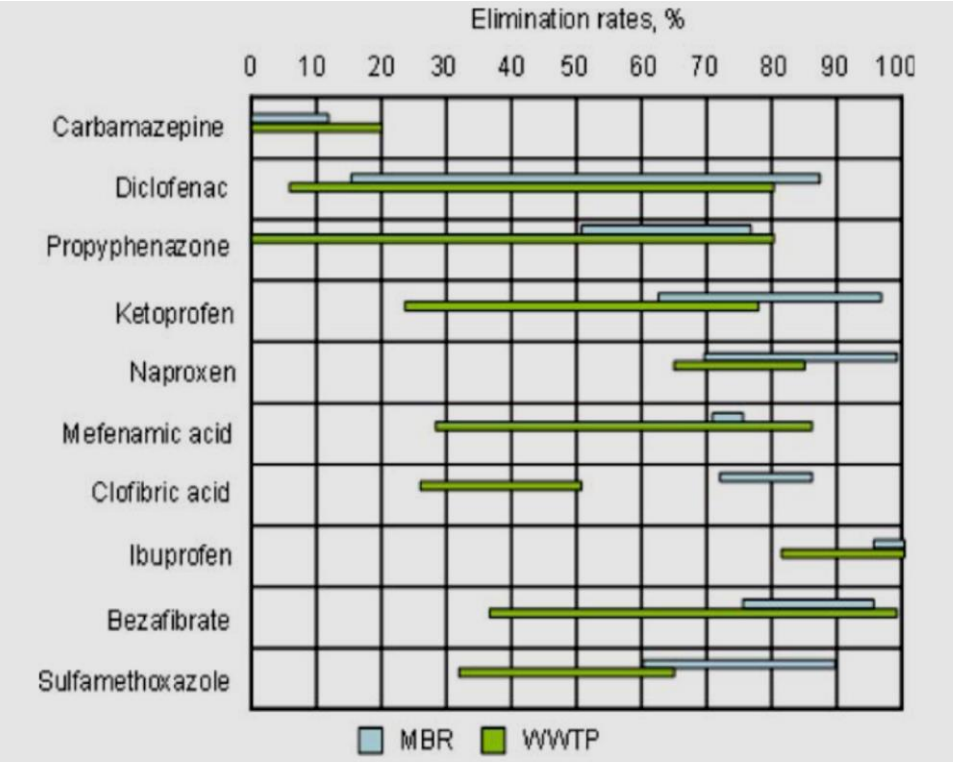

Figure 1.11 Elimination rates of PhACs in MBR and CAS treatment (Radjenovic et al., 2007)

1.3 Fouling problems in MBR

The main disadvantage of MBR technology compared to CAS is the high cost, before the costs include that of the module and the operational cost. Now that there are numerous numbers of researches in the development of membranes with high surface area, moderate temperature resistance, mechanically good, tolerance to corrosion, high pH range and so many qualities compared with old technology. The membrane modules costs have decreased drastically over the last years to about < US$ 50/m^2 (Chaibi, 2000) that makes the capital costs to decrease. Therefore membrane fouling is now the major challenge in this sector. Fouling in MBR is the coverage of surface of the membrane (externally and internally) by accumulated or adsorbed substances during operation, which are attributed to the interaction between the activated sludge components and the membrane. This lead to the loss in the productivity (permeability) due to the loss in the membrane surface area, also increase the production cost since the pressure applied or cross flow velocity will increase.

Non continuous in the productivity due to the back flushing in the case of hollow fiber modules, relaxation in the flat sheet modules which are applied 15-60s every 3-12 min of operation are the operational precautions against fouling (Judd, 2006, Kraume and Bracklow, 2003, Lyko et al., 2008). Frequent maintenance cleaning which is done after 2-7 days of operation and recovery cleaning (once or twice a year) also reduce the productivity due to the time lost during the cleaning (Judd, 2006, Kraume and Bracklow, 2003) it also lead the environmental hazard since the chemicals used during the cleaning may produce some by-products like absorbable organic halogens, THM etc.

Damaging, inefficient or late chemical cleaning might reduce the lifespan of the modules thereby increasing the cost due to the higher replacement costs. Higher energy requirement for aeration which is the major factor attributing to the expensiveness of this process (about 70% of the total energy costs required) (Judd, 2006; Verrecht et al., 2008 Kraume and Bracklow, 2003).

Fouling is categorized based on the attachment strength of the foulant to the membrane or the way that is followed in recovering the permeability of the membrane, these includes;

Reversible fouling

Irreversible fouling

Residual and

Irrecoverable.

Reversible fouling: in other terms removable or temporary fouling (Enegess et al., 2003, Le-Clech et al., 2003), are fouling caused by the loose attachment of the fouling material to the membrane surface and can be removed by physical cleaning method, e.g., back flush or strong shear force, relaxation, or with a soft brush.

Irreversible fouling: The internal clogging or pore blockage of the membrane due to the adsorption colloids is called irreversible fouling or physically irreversible fouling. Membrane relaxation or backwashing as it is done in hollow fiber modules has no significant impact on the flux decline with operation time, because of the continuous accumulation of irreversible fouling on the surface of the membranes. It is also good to remember that this type of fouling cannot be removed by physical cleaning like relaxation but only with the help of chemical cleaning (chemically enhance backwash on daily basis or maintenance cleaning). The formation of strong matrix of fouling layer with solutes during a continuous filtration will result in reversible fouling and later transformed to irreversible fouling layer e.g., formation of gel layer under long term sub-critical flux operation (Wang et al., 2008).

Residual fouling: This type of fouling cannot be removed by air sparging or other chemical methods, this concept was proposed by Kraume et al., (2009) and Judd (2011). They defined this fouling as the fouling that cannot be removed by chemically enhance backwash or maintenance cleaning but only by recovery cleaning.

Irrecoverable fouling: This type of fouling cannot be removed even by chemicals, because after a long operation with a membrane the membrane is fouled by the above mentioned reasons hence the original (unused) membranes permeability is never recovered. There is always a remaining resistance which is defined as irrecoverable fouling (Judd, 2011; Resosudarmo et al., 2013). Sometimes it is defined as permanent or long term irreversible fouling, the unrecovered fouling will continue to accumulate over a number of year, therefore this defines the life of the membrane (Ayala et al., 2011). Table 1.4 shows the types of fouling and the size of foulant that can cause membrane fouling with time.

Table 1.4 Typical ranges of the different fouling rates occurring at full scale (Drews, 2010)

Category	Fouling rate in mbar/min	Time frame
Reversible fouling (cake filtration)	0.1–1	10 min
Residual fouling	0.01–0.1	1–2 weeks
Irreversible fouling	0.001–0.01	6–12 months
Irrecoverable fouling	0.0001–0.001	Years

1.3.1 Foulants

Foulants as the name implies are the compounds that are responsible for fouling in membranes. According to (Meng et al., 2009, Spettmann et al., 2010) fulants can be classified into biofoulants, organic foulants, and inorganic foulants. The nature of these foulants gives us a hint which our MBR plants should be operated, the nature of the chemicals to be selected for the cleaning of the fouled membranes.

1.3.1.1 Biofoulants

Sometimes referred as bio-solids, are formed due to the growth of microorganisms and deposition on the surface of the membranes. According to some literatures bio-fouling is not only about the attachment of microbial cells on the surface of the membrane but also the adsorption of the organic matters produced by microorganisms during biodegradation (Malaeb et al., 2013). Bio-foulants can be classified into organic foulants which are soluble microbial products (SMP) and extracellular polymeric substances (EPS). In biofouling, the dominant foulants are biosolids but also part of organic matters can be found.

1.3.1.2 Organic foulants

Organic foulants are proteins, polysaccharides, monosaccharide, humic acids and other organic substances (either soluble or colloidal) which are found from feed water or released from the biological activities during biodegradation. Although SMP and EPS are described as the major membrane foulants in MBR, but metal cations also interact with some of the functional groups of the biopolymers to form chelating polymers (organic-inorganic complexes) (Wang et al., 2008). For example in the presence of alginate, a severe membrane fouling can be observed when the feed water contained calcium (Myat et al., 2014).

1.3.1.3 Inorganic foulants

The presence of metal ions like Ca^{2+}, Mg^{2+}, $Fe^{3+}$and Al^{3+}, and the anions like CO_3^{2-}, SO_4^{2-} and OH^- can react cause chemical precipitation. Inorganic fouling occurs due to the accumulation of inorganic precipitates like metal hydroxides on the surface of the membranes, or blocks the pores of the membranes, the precipitations are formed when the concentration of the chemical species exceeds their saturation concentrations (Zhang et al., 2012), also biological precipitation of inorganic-organic complexes can be observed as mentioned earlier.

1.3.2 Pre-treatment methods to prevent fouling

Pre-treatment of wastewater is done for several purposes, for example pre-treatment is necessary for the safety of the equipment in the plant (membrane, pumps, diffusers etc.). It is also of significant importance in order to remove biologically non-degradable materials. Membrane fouling is the bottleneck of MBR technology as mentioned in the above section; its prevention is one of the research topics nowadays. In MBR coagulants has been used for agglomeration of colloids to form larger particles, because the tendency of colloids to be absorbed by the membrane is high, also fine particle clog the pores of the membrane thereby causing irreversible fouling. Adsorbent has also been used in order to reduce the organic load on the microorganisms inside the reactor. The use of supplementary dose of PAC, coagulant, adsorbent has been reported by (Park, Choo and Lee, 1996).

Ferric chloride, aluminum sulphate (alum) and powdered activated carbon (PAC) are the coagulants widely used for coagulation in MBR. These coagulants can be use simultaneously with the biological treatment and the membrane separation at the same time or can be used in the pretreatment step before the MBR unit. For example, Holbrook et al. (2004) added alum directly into the MBR and reported that the concentration of carbohydrate soluble microbial products (SMPc) lower than that of the MBR in which alum was not added. In another study by Lee et al. (2001), it was reported that the size of particles in the reactor has been reduced from 0.1 to 2 μm by the addition of alum to the reactor. Ferric chloride is the key element which enhances the production of iron-oxidizing bacteria that is responsible for the degradation of H_2S (Park et al., 2005). One of the disadvantages of using these coagulants in the MBR unit, is the production of excess sludge in the unit, also there is a possibility of the ferric precipitate to foul the membrane. It was mention in a study by Adham et al. (2004) that ferric phosphate and K-jarosite ($K\text{-}Fe_3(SO_4)_2(OH)_6$) has some fouling effect on the membrane, therefore it is of vital important to pre-treat the feed before the MBR unit.

1.3.3 Operational precautions against fouling

A lot of struggles have been made for the mitigation of membrane fouling by the membrane surface modification, use of coagulants and adsorbent, the use of some inert nanoparticles, changing the operational conditions. Some of the operational conditions to be considered in alleviation of membrane fouling are as follows:

1.3.3.1 Aeration and crossflow velocity

Aeration is done in MBR for the purpose of providing the microorganisms with oxygen, to maintain the homogeneity of the activated sludge in the reactor and to ameliorate fouling (Dufresne et al., 1997). The type of flow in the MBR happens to be solid-liquid-gas non-Newtonian flow, since it comprises solute, colloids and particulates. Therefore, the theoretical analysis to describe the event taking place become complicated, but the general antifouling effect of the air scouring on the membrane can be explained (Le-Clech et al., 2006). The bubbles that flow on the vicinity of the membrane induces a tangential shear on the membrane, hence increases back transport on the particles that tend to foul the membrane. Air scouring does not have significant effect on small particles of less than 0.1μm (foulants that cause irreversible fouling), since the dominant transport phenomenon in such case is transport by diffusion, the particles greater than 1 μm are prevented from the surface of the membrane. As mention earlier aeration is one of the factors that make MBR technology to be costly, but is very important in controlling fouling which is major drawback of this technology. Aeration has effect on the flux but there exist a point in which it has no effect on the flux, therefore an optimum aeration has to be determined. Also further increase in the aeration rate can lead fouling, since the flocs will break to smaller pieces thereby resembling side stream MBR (in the case of submerged MBR). According to Le-Clech et al. (2006) it was observed that a 3 m/s crossflow velocity (CFV) is sufficient in MF (0.3 μm) and 2 m/s for UF (kDa). Based on the crossflow velocity (CFV) in tap water in the riser section of an internal loop-airlift MBR system, Liu et al. (2003) set up a modified model to determine the CFV in the case of activated sludge as shown in the following equation:

$$CFV_{as} = 1.406 \times CFV_{tp}^{1.226} \times \mu^{-0.147}$$

Where μ is the viscosity of the MLSS, CFV_{tp} is the crossflow velocity in tap water can be estimated using the procedure given by Liu et al. (2003).

1.3.3.2 Solid retention time (SRT)

SRT and consequently the ratio of feed to microorganism (F/M) is one of the operational parameter that determines the performance of MBR. Because when MBR is operated at higher SRT, there will be increase in the MLSS in the reactor, although it is mentioned that will not increase the fouling rate (Zhang et al., 2006). At low SRT, the production of EPS is high. In a study conducted by Ng et al. (2005), it was stated that when the SRT was lowered from 10 to 2 days (F/M ratio from 0.5 to 2.4 g COD/MLVSS and MLSS of 7.8 to 6.9 g/L) the fouling rate increased about 10 times of the 2 days SRT. They suggested that the F/M ratio should be maintain below 0.5. It is good to keep in mind that higher SRT does not always define the good performance of the MBR due to the following reasons

i) The bioflocs formed may be broken to smaller pieces and hence fouling rate will increase,

ii) It can lead to the accumulation of biologically non-degradable substance like hair which were not removed from the pretreatment unit and at the end such substances can clog the membranes,

iii) It leads to the accumulation of toxic substances thereby killing the living bacteria responsible for the biotreatment.

1.3.3.3 Relaxation

Relaxation plays a vital role in the back transport of particles away from the membrane. During the operation there is a flow only on the direction of the permeate flow and with time bioflocs tend to block pores of the membranes. For that purpose, there is a need for the back transport of the accumulated particles. Therefore during relaxation no permeate withdrawal from the system occurs. The period of relaxation is important in the operation of MBR because when the interval of the application is high, the fouling layer will be compact thereby making it difficult for its removal. In the case of hollow fiber (HF) systems it can also be applied Judd, (2011). It is recommended that relaxation should be applied for 1-2 min in every 8-15 min of operation, in the case of hollow fiber HF backflushing is usually employed at 1-3 times the operational flux.

1.3.3.4 Flux conditioning

While defining the operational flux it is important to keep in mind that there always exists a balance between the foulants from the membrane and to the membrane. The balance gives a hint about the architecture and the extent of the fouling. In MBR, there always a flux in which further increase in the driving for does not has an impact on the flow which is known as critical flux. Therefore it is important to determine this value at the beginning of each experiment. Fouling does not occur rapidly at low flux, but that is not the solution since the lower the productivity the higher the cost of the process. Also when a system is run at a low and constant flux, it is obvious that fouling will not show up immediately but after some time the bottom of the layer formed on the membrane cannot be removed easily.

1.3.3.5 Unsteady state operation

The variation on the feed flow and the shift in the oxygen supply in MBR causes fouling propensity. The sudden stress on the cell in MBR can cause by high MLSS concentration resulting low dissolved oxygen, therefore it is important to check the COD of the wastewater before it can be fed to the reactors. Based on the experiments carried out on biological suspension at different impulses in a batch test, it was observed that exogenous phases were followed by starvation period, these are characterized by the ratio of the feed substrate concentration to the mixed liquor volatile suspended solid (MLVSS) concentration (So/Xo), the equation below can be used to calculate the ratio (Lobos et al., 2005).

$$\frac{S_o}{X_o} = HRT \times \left(\frac{F}{M}\right)$$

Where S_o is thefeed substrate concentration and X_o the mixed liquor volatile suspended solid (MLVSS) concentration.

The main idea behind using low F/M in the MBRs is that, at low F/M ratio it is considered to be starvation period. In this period, the production of protein soluble microbial product (SMP_p) is low, hence less severe fouling tendency. According to Ried et al. (2006) the sudden change in the salinity of the feed will result into membrane fouling, for example when a shock sodium chloride was injected to the MBR system, the production of protein extra-cellulose substances (EPS_p) and carbohydrate soluble microbial product (SMP_c) increased.

Cleaning of MBR membranes

Proper cleaning in MBRs is another way mitigate fouling, the major cleaning protocols include;

Physical cleaning

Chemical cleaning

Physical cleaning: using this technique we are able to remove only the reversible fouling as can be seen in Figure 1.12. Generally this method is applied during operation and before chemical cleaning. Air sparging, regular backwashing for the back washable membranes and relaxation are the methods used during physical cleaning. But the conditions like time for the sparging, the interval and the flow rate of the air should be optimized. Same with the backwash and relaxation, the frequency and the time should be determined (Judd, 2011).

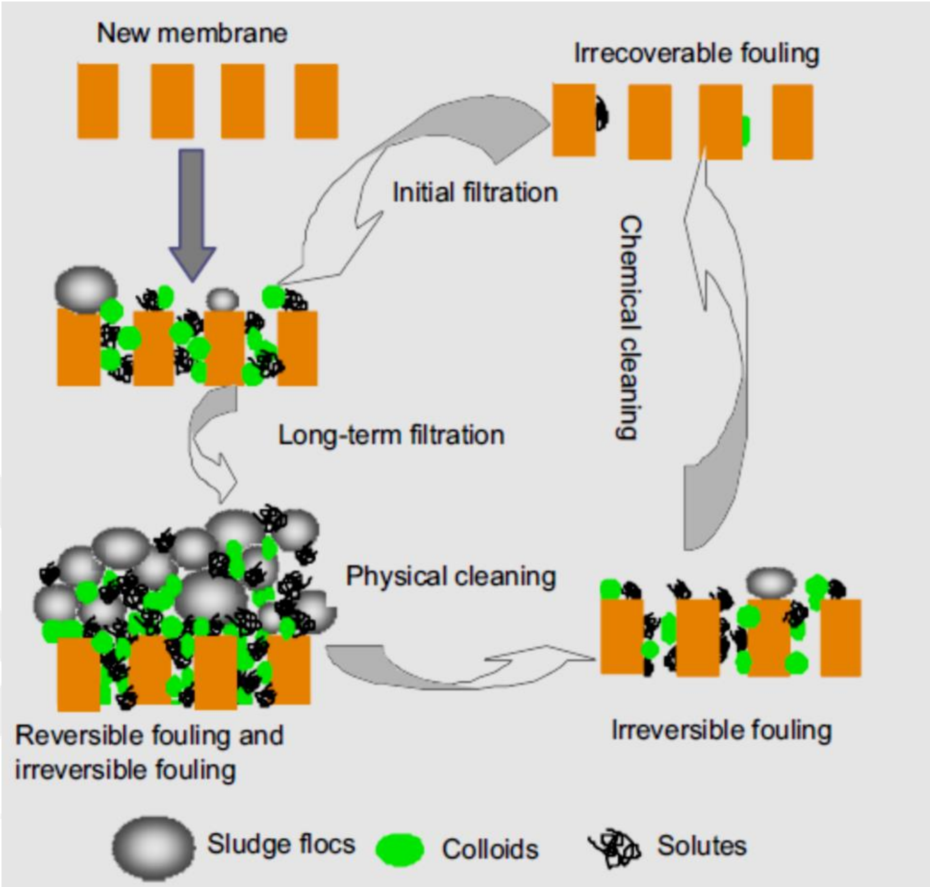

Figure 1.12 Fouling and cleaning effect of membranes in MBR (Meng et al., 2009)

Chemical cleaning: in the physical cleaning, the backwashing, relaxation and air scouring can only remove the reversible fouling, that means with time there will be accumulation of the irreversible fouling on the membrane. Therefore, physical cleaning alone cannot solve the problem of fouling, but with the use of proper chemical and proper protocol we can alleviate membrane fouling. From Figure 1.12 we can see that even after chemical cleaning there exist some fouling on the membrane (irrecoverable), therefore proper protocol is very important in order to prevent the accumulation of irrecoverable fouling. Below are some protocols recommended by Judd (2011):

Chemically enhance backwash (CEB) on daily basis, Maintenance cleaning with higher concentration than that of the CEB generally on weekly basis, and Recovery/intensive chemical cleaning (once or twice a year).

Generally CEB cleaning is done to prolong the cleaning frequency of intensive and recovery cleaning. It is obvious that whenever we use higher concentration on the membranes we cause some changes in the active layer of the membrane. For this reason, the membrane suppliers usually provide some protocols so as not course catastrophic deterioration of the membrane active layer. The maintenance cleaning as recommend by Judd (2006) should take up to 30 min with a moderate concentration (0.01 wt.%) of NaOCl. The intensive cleaning protocol for different membrane suppliersar summarized in Table 1.5, but these protocols can vary according to treatment plant.

Table 1.5 Intensive chemical cleaning protocols for four MBR suppliers (Judd, 2006)

	Type	Chemicals	Concentration (%)	Protocols
Mitsubishi	CIL	NaOCl	0.3	Backflow through membrane (2h) + soaking (2h)
		Citric acid	0.2	
Zenon	CIP	NaOCl	0.2	Backpulse and recirculate
		Citric acid	0.2-0.3	
Memcor	CIP	NaOCl	0.01	Recirculate through lumens, Mixed liquors and in tank air manfolds
		Citric acid	0.2	
Kubota	CIL	NaOCl	0.5	Backflow and soaking (2h)
		Oxalic acid	1	

CIL: cleaning in line where chemical solutions are generally backflow (under gravity) inside the membrane. CIP: cleaning in place where membrane tank is isolated and drained; the module is rinse before being soaked in the solution and rinsed to remove excess of chlorine

1.4 ITOB Organized Industrial Wastewater Treatment Plant

ITOB OSB is an organized industrial zone at Menderes, Izmir, Turkey. The Zone is located 32 km distance from the city centre, 12 km to the Menderes District and 18 km to the Izmir Adnan Menderes Airport. Cumaovası - Aliağa Railway line passes along the region. Therefore, transportation to the organized zone is possible from all part of the city. Figures 1.13 and 1.14 shows the entrance and top view of ITOB Organized Industrial zone.

There are 360 unit parcels ranging from 1.000 m^2 to 30.000 m^2 based on 250 hectares of land in the region.

Figure 1.13 A view of ITOB OSB entrance

Figure 1.14 Top view of ITOB Organized Industrial Zone

The companies in the Organized Zone mainly operate in food, iron and steel industry, chemical, plastics, machinery and electrical electronics. When the Zone is fully operated, it is expected that the employment will reach 15,000 people compared with the current numbers of employees which is approximately

4500. It is good to mention that all the management and technical (like power, water and waste management) are being provided without any external assistance.

1.4.1 ITOB OSB Wastewater Treatment Plant

Among the industrial zones in Turkey, ITOB industrial wastewater treatment plant is the first wastewater plant that started using MBR technology in treating the wastewater coming from the industrial zone. ITOB wastewater treatment plant was established in 2008 with the aim of treating the wastewater coming from the Organized Industrial Zone with a design considering that the zone operates in full capacity. The design capacity of the wastewater treatment plant is 8,000 m^3/day. Since 2008, the wastewater treatment plant operates with a 2,000m^3 /day. By time, the number of factories is expected to increase in the region, and then the capacity of the plant will also be increased to its maximum capacity.

The process consists of physical and biological separations of wastewater. The MBR membranes used are Kubota EK400 membranes made from polyethylene. The porosity of the membranes is 0.4 μm, but the quality of the treated water is in the range of ultrafiltration treated wastewater, because during the process there is a formation of bio-film on the surface of the membranes.

The treated water quality is very good in terms of color, COD content, phosphorus content as well as nitrogen with the help of MBR process and obeys the permissible levels provided by Water Pollution Control Standards of Ministry of Environment and Urbanization, Republic of Turkey for the discharge of industrial wastewater, water pollution control standards for the discharge of industrial waste water. Furthermore, because T.R. State Hydraulic Works' drying channel is used as receiving environment. ITOB OSB management has put a lot effort in collaboration with Ege University to see that the treated water is further treated so that it can be used in the industries as either process water or as irrigation water, by doing that they will reduce the amount of the withdrawn water from the underground source.

1.4.1.1 Coarse Grid and Screening Unit

This part is the first physical separation, Flow rate and pH of input wastewater which comes from various industries are measured first. Wastewater passes through coarse grids screen the large particles which may be dangerous to the equipments. Pore diameters of coarse grids are 15 mm, therefore all particles larger that the pore size of the coarse grid are eliminated in this unit. In these sections COD and BOD values are reduced to about 40%. Figure 1.15 shows a closer look at the coarse grid unit

Figure 1.15 Coarse grid

1.4.1.2 Elevation Center

After the separation of the large particles, the pump units in elevation center are used to send wastewater from grid unit to aerated sand and grease trap unit, and to compensate for the loss of load, as a result of the energy loss of the wastewater between the process units.

1.4.1.3 Aerated Sand and Grease Trap Unit

In next step, water comes to sand and grease trap unit, the separation of sand and grease is necessary since we know that sand cannot be degraded via the microorganisms and for the grease it will take very long time for the biodegradation which is not feasible in the wastewater treatment plant. The separation in this unit is done by gravity that is less dense material (which is grease) is gathered from the top by stripping. On the other hand, denser materials (sand particles) are settled down and gathered.

1.4.1.4 Equalization Basin

In equalization basin (Figure 1.16), water is mixed by an aerator, for homogenization of water. The main reason of the aerator is *not* aeration, but mixing since the wastewater is coming from different industries and about 40% of this water is domestic wastewater. Therefore it is important to make the wastewater equal so that we can decide which nutrient is missing for the survival of the microorganisms and for the pH adjustment.

Figure 1.16 Equalization basin

1.4.1.5 Chemical Additive Units

After these physical separation steps, some chemicals are added to the process adjust the wastewater to a desired condition, the chemicals are prepared in a separate unit as shown in Figure 1.17 before dosing to the wastewater. These chemicals are;

- $Al_2(SO_4)_3$ is used to reduce phosporus and pH lowering.

- NaOH is used to increase pH value.

- Polyelectrolyte is used to form a chain between suspended materials and cations. Therefore bigger particules are precipitated.

- Urea (CH_4N_2O) is a source of N_2.

- NaClO membrane cleaning.

Figure 1.17 Chemical preparation unit

1.4.1.6 DeNO$_x$ Unit

After chemical adjustment, wastewater passes through DeNO$_x$ unit. In this unit, NO_x molecules are converted to N_2 gas by de-nitrification bacteria and this gas is released to the atmosphere.

1.4.1.7 Aeration Basin

Aeration increases oxygen content in the water. Oxygen is needed by bacteria to break down dissolved or suspended organic materials. In water treatment, aeration helps to oxidize certain compounds into a form that settle in the water which can then be filtered out as shown in Figure 1.18. Aeration can help in the removal of gases in water.

Figure 1.18 Aeration basin

1.4.1.8 MBR Unit

This unit is the heart of the wastewater treatment process because the biological activity and the solid-liquid separation occur at this unit (Figure 1.19). Pre-treatment steps for wastewater are applied before sending it to this unit in order to protect the MBR membranes. This includes the rejection of non-degradable and oily substances and also large particles are rejected in order not to damage the membranes. Wastewater is then sent to the MBR tanks. These tanks contain membranes which are submerged into the sludge and these membranes are Kubota EK 400 type MBR membranes and each MBR tank contains 2000 of them, pore sizes of membranes are 0.4 μm.

Figure 1.19 MBR tank

Sludge Recycle Tank and Decanter

Sludge solution in the system (dead bacteria etc.) which comes from MBR tanks and aeration basin are stored in sludge recycle tank. This sludge is sent to decanter. In decanter, sludge and water are separated by centrifugal. While organic solid waste sludge is taken, water from sludge solution is sent to the coarse grid, again.

1.4.1.9 Treated Water Storage Tank

Finally, after MBR process, treated water from MBR tanks is collected in storage tank. After that, this water is discharged as 2nd quality water to the environment. Finally Figure 1.20 shows the overall Flow diagram of ITOB wastewater treatment plant.

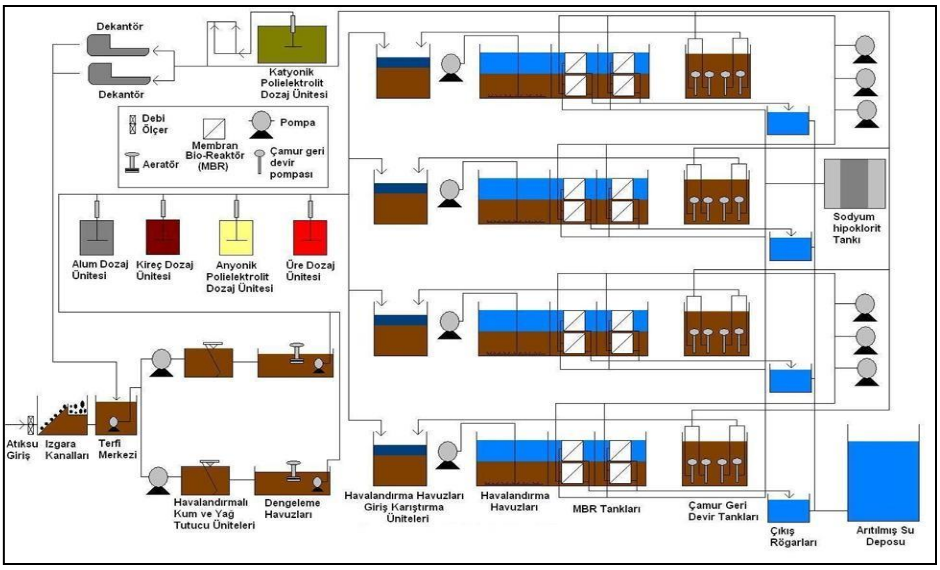

Figure 1.20 Flow diagram of wastewater treatment plant at ITOB Organized Industry Zone

1.4.2 Problems in ITOB Wastewater treatment plant

The main problem that ITOB wastewater treatment plant is facing is fouling and scaling on the surface of the membranes which causes flux decline with time. They find it difficult to identify the root of these problems because optimization of operational conditions in trying to solve the problems in full scale system is not possible. As stated from the manager of the plant, inorganic scaling is dominant in reducing the flux in the MBR treatment plant.

1.5 Aim of this study

This study aimed to explore the problems of ITOB wastewater treatment plant and to find feasible solutions to these problems. In the first step of this research, coagulation prior to MBR unit is investigated in order to minimize scaling problems on the surface of the membranes due to the hardness in wastewater. The use of antiscalant and the effect of antiscalant on minimizing membrane fouling during MBR operation is also investigated in the second step of this research.

2.0 MATERIALS AND METHODS

2.1 Jar Test

Velp scientifica flocculator FCS was used for jar tests as shown in Figure 2.1, it is equipped with 6 beakers (1 L capacity each).

Figure 2.1 Velp scientific flocculator FCS

During the experiment, pH and temperature were measured by the means of Hach Lange potable digital pH meter, conductivity, salinity and TDS were measure by Hach Lange conductivity meter as shown in Figure 2.2. Total hardness measurement was done by Hach Lange Kit 327, TSS and color measurements were done by colorimetric method.

Figure 2.2 Digital pH and conductivity meter

2.1.1 Experimental

Before starting the experiment, stock solutions were prepared (i.e 2000 mg/L of $FeCl_3$, $Al_2(SO_4)_3$ and $Ca(OH)_2$). The samples used in these experiments were taken from the equalization unit of ITOB Organized Industrial Zone wastewater treatment plant. The wastewater was well mixed before the samples were taken. $Al_2(SO_4)_3$ was used from the first set with the concentrations of 125, 250, 500, 700 and 800 mg/L. The pH, conductivity, temperature and salinity of the sample wastewater were recorded. Then the above mention concentration was put into the available beaker which has a total volume of 1000 mL. The rapid mixing was done at 150 rpm for 5 min. followed by slow mixing at 30 rpm for 15 min then 30 min. for settling. In the following sets the coagulant used were $Ca(OH)_2$, $FeCl_3$, $Al_2(SO_4)_3$ and $Ca(OH)_2$ (analytical grade) with the concentrations of 125, 250, 500, 750, 1000 mg/L using same procedure as mention above. Then the pH was adjusted with HCl and $Ca(OH)_2$, one set was done with the concentration of 500 mg/L each then 1000 mg/L ($Al_2(SO_4)_3$, $FeCl_3$ and $Ca(OH)_2$). pH was then adjusted with NaOH and HCl.

Same procedure was followed as in the case of wastewater (concentration effect) using the three coagulants, in the case of tap water we have used two additional coagulants ($NaHCO_3$ and Na_2CO_3) which have not been used in wastewater have been used.

2.2 MBR pilot system

Optimization of operational parameters in the MBR system is one of the factors that will help in the prevention of membrane fouling. For this purpose, MBR pilot system was designed to optimize the operational parameters in ITOB wastewater treatment plant.

2.2.1 The reactor

The reactor is the heart of the process because the biological degradation of the waste as well as the solid liquid separation took place at this unit. The reactor is made up of stainless steel with a capacity of 50 L volume (Figure 2.3).

Figure 2.3 Side view of the reactor

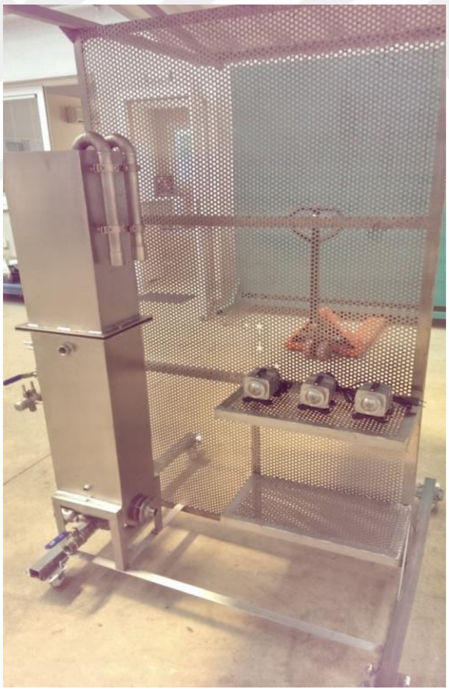

Figure 2.4 Another side view showing the reactor inlet

From the outside view (Figure 2.4), we can clearly see the reactor consists of two inlets to the reactor, one is coming from the big MBR which provides fresh feed and the other one is coming from the recycle stream. The ratio of the recycle to the reactor is one of the parameters that could be investigated as an operational parameter. On the side of the reactor there are also probes which will measure the conductivity and the pH of the wastewater inside the reactor. We also have four streams from the side connected to a single stream for the collection of permeate from the reactor then to the storage tank as shown in Figure 2.5. There is also a tap for taking a sample from the permeate stream. Figure 2.6 shows the position of aerator (placed at the bottom of the reactor) to ensure uniform distribution of air in the reactor.

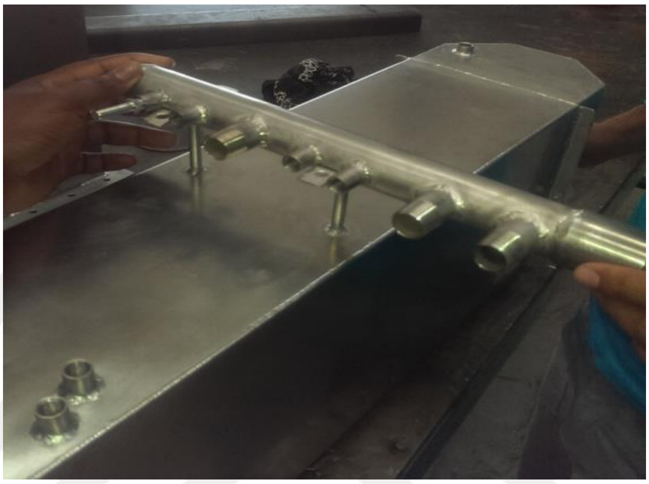

Figure 2.5 Permeate collector

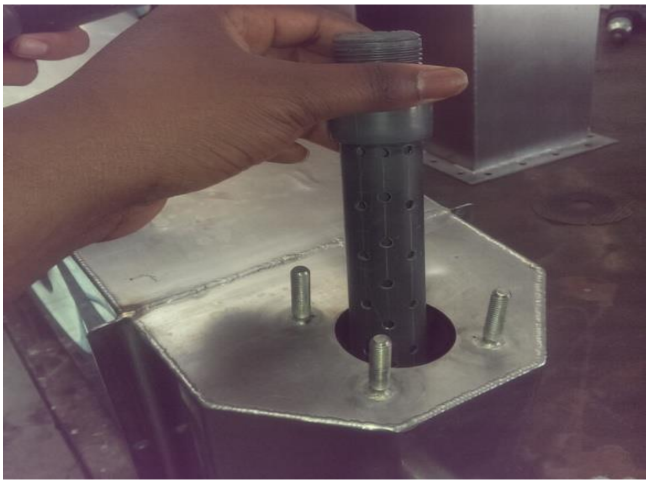

Figure 2.6 Position of aeration tube

At the bottom of the reactor we have three streams which will be used for

- Recycling and excess sludge removal
- Aeration

- Emptying the reactor in case of any failure in the pumps or after chemical cleaning of the membranes.

The aeration is another operational parameter that will be investigated in our experiments. There are two pipes with an opening of 5 mm (Figure 2.8) and 2 mm diameter (Figure 2.7) in order to control the size of the bubbles. Also, the amount of the oxygen supply to the reactor is another parameter for our study.

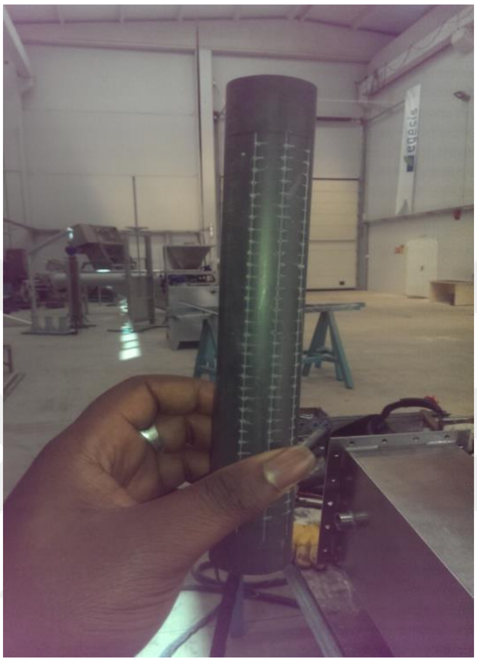

Figure 2.7 Aeration tube with 2 mm diameter

Figure 2.8 Aeration tube with 5 mm diameter

From inside, the membranes are placed at the center of the reactor. The membranes are also placed in a way that they are supported to make them unshakeable when the air is blown into the reactor (Figures 2.9 and 2.10).

Figure 2.9 Support for the membrane

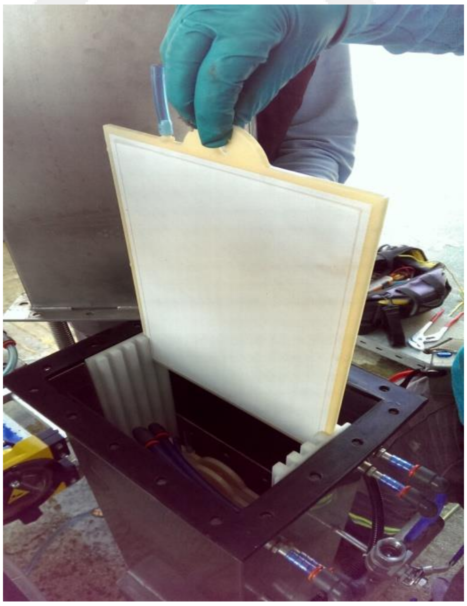

Figure 2.10 Position of the membranes inside the reactor

Figure 2.11 Feed pump

Figure 2.12 Recycle pump

The above pumps were used for taking the fresh feed from the ITOB MBR which is about 4.5 m height into our pilot system (Figure 2.11) and also for taking some portion of the sludge into the main reactor while some part of will be taken back to the system as recycle stream (Figure 2.12). Furthermore, the ratio of the recycle to the reactor can be optimized by the help of this pump.

Figure 2.13 Blowers

There are three Resun ACO-001 electromagnetic air pumps used as blowers (Figure 2.13), for aeration to the reactor. Their function is to supply air for metabolic activities of the micro-organisms and also to prevent gel formation on the surface of the membranes. Excess air supply into the reactor leads to power consumption which makes the system to be expensive, therefore the aeration intensity is another important. For that reason two blowers were used while the third one was kept as reserve in case of failure from any of the two. There is a flow meter as shown in Figure 2.14 to control the air flow with a by-pass to return the excess air.

Figure 2.14 Air flow meter

Figure 2.15 Permeate storage tank

At the back side of the system there is a 200 L storage tank as shown in Figure 2.15 for the collection of permeate before discharging to the canalization. Therefore, there are two streams one the inlet to the storage tank and the other one the exit to the canalization with a level controller.

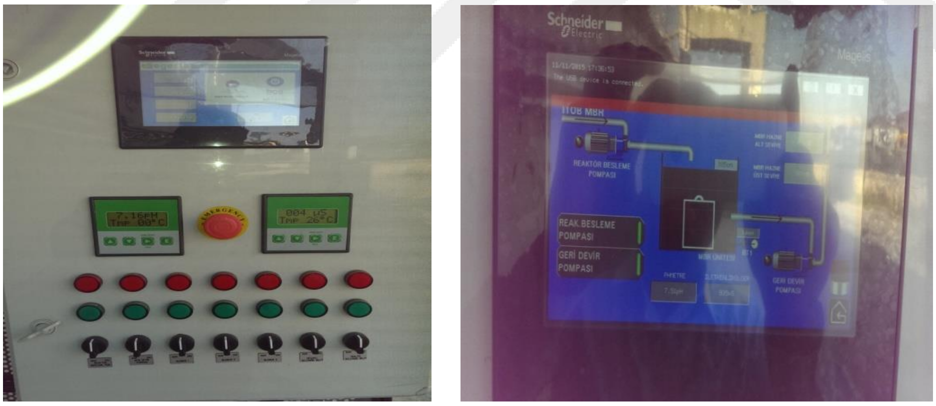

Figure 2.16 Control panel

Figure 2.17 pH and the conductivity display

The control panel in Figure 2.16 shows us the view of what is happening in the system. From there, we are able to learn the conductivity, temperature and the pH of our system (Figure 2.17), these values are also recorded into a flash drive. It enables us to enter the level at which we want to study, the driving force for separation in this system is the hydrodynamic pressure of water inside the reactor. From the screen, we are also able to learn the trans-membrane pressure (TMP) of the system. TMP is the measure of the pressure needed to press water through a membrane, therefore is a parameter to us that shows whether our membrane is fouled or not, when it increase rapidly that means our membrane is fouled. We can also set the TMP at which when exceeded an alarm will appear on the screen. We can on/off the pumps, blowers, valves and even the whole system from the screen.

Figure 2.18 Front view of the MBR pilot system

Figure 2.18 shows our constructed pilot MBR in its final shape. Figure 2.19 shows the first trial of our membranes after soaking in water for 24 h. From Figure 2.20, we can see how permeate is collected in the permeate tank before discharge.

Figure 2.19 First permeate from the MBR pilot system using tap water

Figure 2.20 Back view of the MBR pilot system

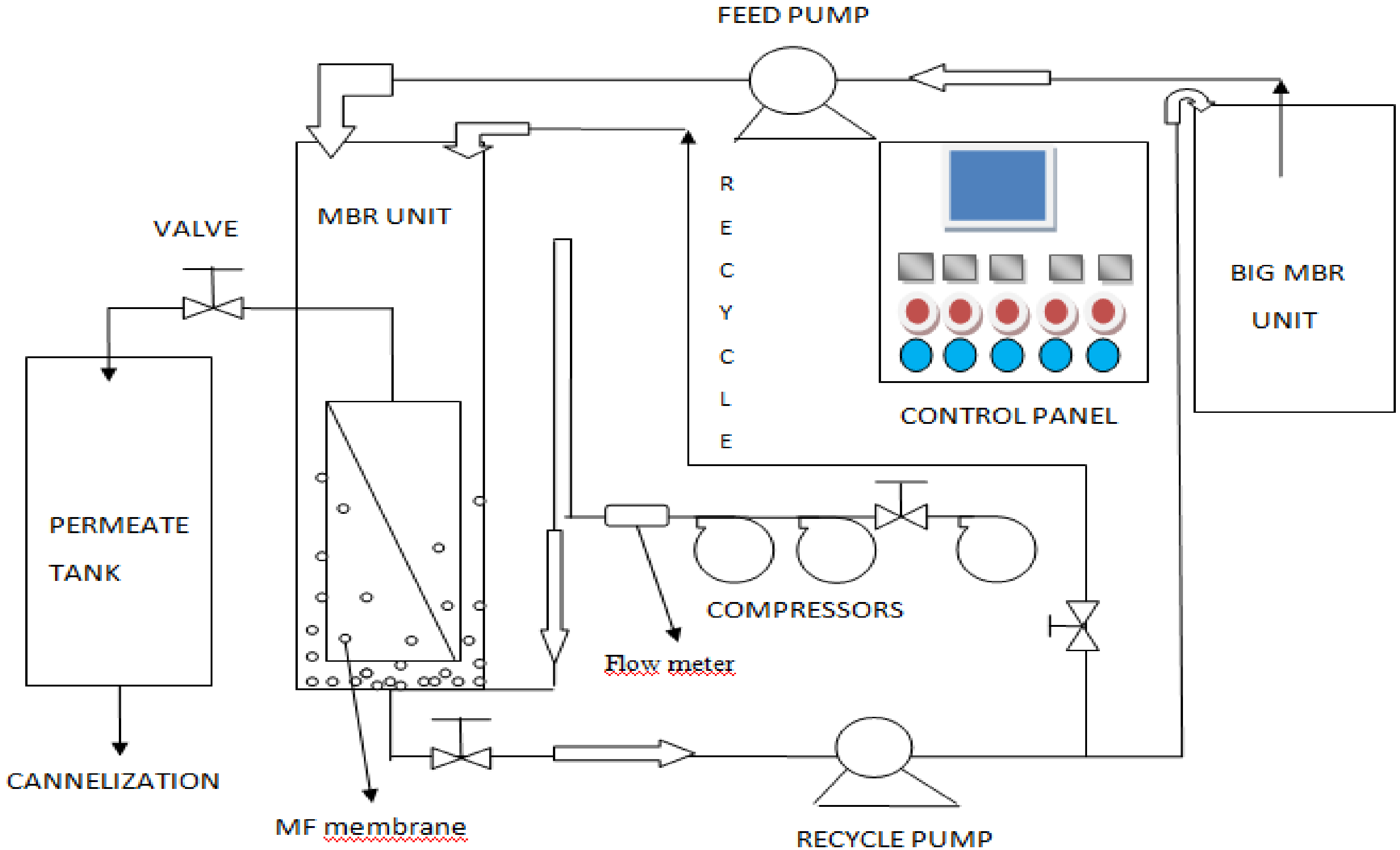

Figure 2.21 Flow diagram of the MBR pilot system

The ready-prepared chemicals used in our analyses are essentially for COD, PO_4-P, NO_3-N, NH_4-N and NO_2-N. The chemicals used were listed below:

- COD Test kits (LCK Hach Lange)
- Phosphate Test kits (LCK Hach Lange)
- Nitrate Test kits (LCK Hach Lange)
- Nitrite Test kits (LCK Hach Lange)
- Ammonium Test kits (LCK Hach Lange)

The measuring ranges of the kits used are summarized in Table 2.1.

Table 2.1 Measuring range of the kits

Type of kit	Measuring Range
COD Test	100-200 mg/L
	15-150 mg/L
	5-60 mg/L
Phosphate Test (as PO_4-P)	2-20 mg/L
	0.05-1.5 mg/L
Nitrite Test (as NO_2-N)	0.015-0.6 mg/L
	0.0015-0.03 mg/L
Nitrate Test (as NO_3-N)	0.23-13.5 mg/L
Total Nitrogen (TN)	20-100 mg/L
	1-16 mg/L
Ammonium Test (as NH_4-N)	2-47 mg/L
	0.015-2 mg/L

Before analyzing COD, PO_4-P and total nitrogen, a Hach Lange LT200 thermo-reactor was used for standard and special digestion as shown in Figure 2.22, while Hach Lange DR3900 Bench top VIS spectrophotometer was used to take the final reading (Figure 2.23).

Figure 2.22 Hach Lange LT200 digester

Figure 2.23 Hach Lange DR3900 spectrophotometer

Table 2.2 Properties of the pilot MBR system

Reactor capacity (L)	50
Membrane active area (m^2)	0.11
Nominal pore size of membrane (micron)	0.4
Membrane type	Plate and frame
Membrane material	Kubota-PE
Diffuser diameter (mm)	5
MLSS (g/L)	12-19
HRT (h)	3
SRT (Day)	37

Table 2.3 Properties of influent wastewater

Parameter	**Influent**
Conductivity (mS/cm)	s
pH	7.05-7.69
Salinity (%)	1.52-2.05
TSS (mg/L)	580-1341
NH_4-N	19.70-44.30
PO_4-P (mg/L)	2.12-18.20
Total Nitrogen	35.8-75.30
NO_3-N (mg/L)	0.514-10.956
NO_2-N (mg/L)	0.112-0.921
COD (mg/L)	262-1006
Color (Pt-Co)	1697-2304

Table 2.2 shows the properties of the pilot MBR system and the conditions during the study. Table 2.3 shows the characteristics of the influent wastewater.

3.0 RESULTS AND DISCUSSION

3.1 Jar tests

3.1.1 Hardness rejection (wastewater tests)

Table 3.1 shows the effect of concentration on hardness rejection. As the concentration of the coagulant increases, the rejection also increases. The highest rejection was obtained with 1000 mg/L (47.90%), the remaining rejections were 38.81, 23.78, 15.38, 6.99 for 750, 500, 250 and 100 mg/L of $Al_2(SO_4)_3$ respectively.

Table 3.1 Hardness rejection obtained with $Al_2(SO_4)_3$

Coagulant concentration (mg/L)	Final Hardness (mg/L)	Final pH	Rejection (%)
Blank	500.50	7.55	-
100	465.50	7.29	6.99
250	423.50	6.93	15.38
500	381.50	6.53	23.78
750	306.25	6.06	38.81
1000	260.75	5.05	47.90

Table 3.2 shows the effect of concentration on hardness rejection by $FeCl_3$ and the results show a similar trend with that of $Al_2(SO_4)_3$. As the concentration of the coagulant ($FeCl_3$) increases, the rejection also increases. The highest rejection was obtained with 1000 mg/L (43.87%), the remaining rejections were 34.07, 17.88, 8.83, 6.99% for 750, 500, 250 and 100 mg/L of $FeCl_3$, respectively.

Table 3.2 Hardness rejections results obtained with $FeCl_3$

Coagulant concentration (mg/L)	Final Hardness (mg/L)	Final pH	Rejection (%)
Blank	509.08	7.55	-
100	473.48	7.35	6.99
250	430.76	6.98	8.83
500	388.04	6.50	17.88
750	311.5	6.01	34.07
1000	265.22	5.11	43.87

Table 3.3 shows the effect of coagulant ($Ca(OH)_2$) concentration on hardness rejection, The highest rejection was obtained with 1000 mg/L (77.42%), the remaining rejections are 62.09, 44.12, 16.99, -12.42 % for 750, 500, 250 and 100 mg/L, respectively. At the pH of 8.09, the results showed a negative rejection which means there was an increase in the hardness when $Ca(OH)_2$ was used at lower pH. This result shows that in the case of $Ca(OH)_2$, pH also played a vital role in hardness removal. As the pH increases, the rejection of hardness also increases.

Table 3.3 Hardness rejections results obtained with $Ca(OH)_2$

Coagulant concentration (mg/L)	Final Hardness (mg/L)	Final pH	Rejection (%)
Blank	544.68	7.72	-
100	612.32	8.09	-12.42
250	452.12	8.29	16.99
500	304.38	8.60	44.12
750	206.48	9.06	62.09
1000	123.00	9.73	77.42

Table 3.4 shows hardness rejections with respect to the three coagulants ($Ca(OH)_2$, $Al_2(SO_4)_3$ and $FeCl_3$) Jar test analysis was repeated in case of each coagulant to be sure of the results obtained. The highest rejection recorded was obtained with $Ca(OH)_2$ (Analytical grade) which is 87.44% using 1000 mg/L coagulant concentration without adjusting pH followed by 86.85% of rejection by $Al_2(SO_4)_3$ at 500 mg/L of concentration. But in next step, the pH was adjusted with 1M NaOH (Table 3.4). The least rejection was obtained by $FeCl_3$ (Table 3.4) 41.24% at 1000 mg/L, but when the pH was increase with 1M NaOH up to 9.70, the rejection increased to 81.48% as can be seen from Table 3.6, even though the concentration of $FeCl_3$ was 500 mg/L.

Table 3.4 Hardness rejections by various coagulants from wastewater

Coagulant	Final pH	Concentrations of coagulant (mg/L)	Hardness rej.(%)
$Al_2(SO_4)_3$	5.05	1000	49.60
	5.16	1000 (repeated)	45.62
$FeCl_3$	5.11	1000	43.87
	5.14	1000 (repeated)	41.24
$Ca(OH)_2$	9.73	1000	83.17
	9.68	1000 (repeated)	87.44

In Table 3.5, pH of all the samples was adjusted with $Ca(OH)_2$ (Analytical grade) with the exception of the blank solution and that of $Ca(OH)_2$ itself. The rejection values were 62.66, 71.73 and 72.57% for $Al_2(SO_4)_3$, $FeCl_3$ and $Ca(OH)_2$ respectively as shown in Table 3.5.

Table 3.5 Hardness rejection at high pH

Coagulant	Coagulant concentration (mg/L)	Adjusted pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	7.43	476.00	-
$Al_2(SO_4)_3$	500	9.00	177.00	62.66
$FeCl_3$	500	9.00	134.00	71.73
$Ca(OH)_2$	375	9.00	130.00	72.57

Table 3.6 shows the effect of pH on hardness rejection The pH of the solutions was adjusted with NaOH this time not with $Ca(OH)_2$. pH was adjusted by taking pH of $Ca(OH)_2$ as basis because highest hardness rejection was obtained with this coagulant as shown in Table 3.2. As shown in Table 3.6 $Al_2(SO_4)_3$ gave the highest rejection (86.85 %), followed by $Ca(OH)_2$ (84.24 %) and $FeCl_3$ (81.48 %).

Table 3.6 Hardness rejections from wastewater by adjusting the pH

Coagulant	Coagulant concentration (mg/L)	Final Hardness (mg/L)	Adjusted pH	Final pH	Rejection (%)
Blank	-	540.00	8.07	8.07	-
$Al_2(SO_4)_3$	1000	71.00	9.50	9.37	86.85
$FeCl_3$	1000	100.00	9.50	9.41	81.48
$Ca(OH)_2$	1000	85.10	9.50	9.45	84.24

It is known from the literature that $Al_2(SO_4)_3$ and $FeCl_3$ are effective at pH range of 6-7 as coagulant. But when we looked at the results given in Tables 3.1, 3.2 and 3.6, we can see that the result contradict the theory. For this purpose, the role of coagulants at elevated pH was investigated, initially all the pH of the solutions were adjusted to 9.70 with NaOH including the wastewater. Table 3.7 shows hardness rejection from wastewater. The highest rejection was found with $Al_2(SO_4)_3$ as 87.73%, followed by $FeCl_3$ (81.65%) then without coagulant (70.48%). Without coagulant addition, the rejection was quite good. However, with the help of coagulant we have made a remarkable hardness rejection.

Table 3.7 Effect of type of coagualants at elevated pH on hardness removal

Coagulant	Coagulant Concentration (mg/L)	Adjusted pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	-	454	-
$Al_2(SO_4)_3$	1000	9.70	55.7	87.73
$FeCl_3$	1000	9.70	83.3	81.65
Without Caogulant	-	9.70	134.0	70.48

After coagulation, the coagulated particles tend to settle down. To increase the settleabiliy of the sludge, a cationic polymer was used as shown in Figure 3.1, Table 3.8 shows the effect of cationic polymer addition on hardness rejection. The highest rejection was obtained with $FeCl_3$ together with 30 mg/L of cationic polymer (52.89 %), $FeCl_3$ together with 60 mg/L of cationic polymer (52.69 %), followed by $Al_2(SO_4)_3$,with 60 mg/L cationic polymer (51.86 %) and $Al_2(SO_4)_3$,with 30 mg/L cationic polymer (49.79%). The cationic polymer alone gave 38.02 % of hardness rejection. As can be seen, the optimum amount of cationic polymer that can be used with $Al_2(SO_4)_3$ and $FeCl_3$, when 60mg/L is.

Table 3.8 Effect of cationic polymer on hardness removal from wastewater

Coagulant	Concentration of coagulant (mg/L)	Cationic Polymer (mg/L)	Final pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	-	7.93	484	-
$Al_2(SO_4)_3$	1000	60	5.77	233	51.86
$FeCl_3$	1000	60	5.35	229	52.69
Cationic polymer	1000	-	5.95	300	38.02
$Al_2(SO_4)_3$	1000	30	5.84	243	49.79
$FeCl_3$	1000	30	5.56	228	52.89

Figure 3.1 Coagulation after cationic polymer addition

According to the results in Table 3.9, the highest hardness rejection was obtained at elevated pH For this purpose, the effect of pH on hardness rejection along with cationic polymer addition was investigated and the results obtained are shown in Table 3.9. Initially pH of all solutions was adjusted to 9.5. As can be seen, the highest rejection was obtained with $Al_2(SO_4)_3$ with 30 and 60 mg/L of cationic polymer as 81.72 and 84.65%, respectively. In case of $FeCl_3$ with 30 and 60 mg/L cationic polymer the rejections were 75.99 and 78.48%, respectively. For $Ca(OH)_2$ with 60 mg/L of cationic polymer the rejection was 24.45%. These results showed that cationic polymer does not affect hardness rejection when pH was adjusted to 9.5, but it only enhances the settleability of the sludge as shown in Figure 3.2.

Table 3.9 Effect of pH on hardness rejection along with cationic polymer addition

Coagulant	Coagulant concentration (mg/L)	Cationic polymer (mg/L)	pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	-	8.27	454	-
$Al_2(SO_4)_3$	1000	60	9.60	69.7	84.65
$FeCl_3$	1000	60	9.41	97.7	78.48
$Ca(OH)_2$	1000	60	9.43	343.0	24.45
$Al_2(SO_4)_3$	1000	30	9.65	83.0	81.72
$FeCl_3$	1000	30	9.58	109.0	75.99

Figure 3.2 Effect of pH adjustment on sludge sedimentation along with cationic polymer addition (during sedimentation)

3.1.2 TSS rejection from wastewater

Table 3.10 shows TSS rejection values obtained with $Al_2(SO_4)_3$ jar test, TSS rejection increased as the concentration of $Al_2(SO_4)_3$ increased. The highest rejection was observed with 1000 mg/L as 99.05%

Table 3.10 TSS rejection with $Al_2(SO_4)_3$

Coagulant concentration (mg/L)	Final TSS (mg/L)	Final pH	Rejection (%)
Blank	224	7.55	-
100	125	7.29	44.20
250	92	6.93	58.93
500	44	6.53	80.36
75	11	6.06	95.09
1000	3	5.05	99.00

Table 3.11 shows TSS the rejections with $FeCl_3$ jar test. TSS rejection increased as the concentration of $FeCl_3$ increased. The highest rejection was observed at 1000 mg/L as 99.11%.

Table 3.11 TSS rejection with $FeCl_3$

Coagulant concentration (mg/L)	Final TSS(mg/L)	Final pH	Rejection (%)
Blank	224	7.55	-
100	125	7.35	44.20
250	90	6.98	59.12
500	41	6.50	83.36
750	9	6.01	97.19
1000	2	5.11	99.11

In terms of TSS rejection, $Ca(OH)_2$ gave the worst rejection as can be seen in Table 3.12. The highest rejection was observed with 1000 mg/L as 91.67%

Table 3.12 TSS rejection with $Ca(OH)_2$ from wastewater

Coagulant concentration (mg/L)	Final TSS (mg/L)	Final pH	Rejection (%)
Blank	324	7.72	-
100	308	8.09	4.94
250	283	8.29	12.65
500	314	8.60	3.09
750	308	9.06	4.94
1000	27	9.73	91.67

Table 3.13 shows TSS rejections with respect to the three coagulants ($Ca(OH)_2$, $Al_2(SO_4)_3$ and $FeCl_3$). Jar test analysis was repeated twice. The highest rejections were obtained with $FeCl_3$ and $Al_2(SO_4)_3$.

Table 3.13 TSS rejections by three coagulants

Coagulant	Coagulant concentration (mg/L)	TSS rejection (%)
$Al_2(SO_4)_3$	1000	99.00
	1000 (repeated)	96.26
$FeCl_3$	1000	99.11
	1000 (repeated)	98.93
$Ca(OH)_2$	1000	91.67
	1000 (repeated)	81.82

Table 3.14 shows the effect of cationic polymer on TSS rejection. Two different concentrations of cationic polymer (30 and 60 mg/L) were added into $Al_2(SO_4)_3$ and $FeCl_3$. Two coagulants showed a similar rejection with respect to TSS removal in the presence of cationic polymer. Cationic polymer alone gave 41.63% of TSS rejection.

Table 3.14 Effect of cationic polymer on TSS rejection from wastewater

Coagulant	Coagulant concentration (mg/L)	Cationic polymer concentration (mg/L)	Final pH	Final TSS (mg/L)	Rejection (%)
Blank	-	-	7.93	245	-
$Al_2(SO_4)_3$	1000	60	5.77	8	96.73
$FeCl_3$	1000	60	5.35	3	98.78
Cationic polymer	1000	-	5.95	143	41.63
$Al_2(SO_4)_3$	1000	30	5.84	3	98.78
$FeCl_3$	1000	30	5.56	3	98.78

TSS was highly rejected with $Al_2(SO_4)_3$ in the presence of 60 mg/L cationic polymer and with $Al_2(SO_4)_3$ in the presence of 30 mg/L cationic polymer. $FeCl_3$ gave also high TSS rejection irrespective of the cationic polymer concentration. The least rejection was found in $Ca(OH)_2$ in the presence of 60 mg/L cationic polymer as depicted in Table 3.15.

Table 3.15 Effect of pH on cationic polymer addition for TSS rejection

Coagulant	Coagulant concentration (mg/L)	Cationic polymer (mg/L)	Final pH	Final TSS (mg/L)	Rejection (%)
Blank	-	-	8.27	199	-
$Al_2(SO_4)_3$	1000	60	9.60	1	99.50
$FeCl_3$	1000	60	9.41	10	94.97
$Ca(OH)_2$	1000	60	9.43	28	85.93
$Al_2(SO_4)_3$	1000	30	9.65	3	98.49
$FeCl_3$	1000	30	9.58	10	94.97

3.1.3 Color rejection from wastewater

The highest rejection in term of color was observed with $Al_2(SO_4)_3$ (98.96%) as shown in Table 3.16.

Table 3.16 Color rejection by using using $Al_2(SO_4)_3$ as coagulant

Coagulant concentration (mg/L)	Final Color (Pt-Co)	Final pH	Rejection (%)
Blank	1340	7.55	-
100	785	7.29	41.42
250	544	6.93	59.40
500	294	6.53	78.06
750	110	6.06	91.79
1000	14	5.05	98.96

The highest rejection in term of color was observed with $FeCl_3$ 1000 mg/L of (99.40%) as shown in Table 3.17.

Table 3.17 Color rejection by using $FeCl_3$

Coagulant concentration (mg/L)	Final Color (Pt-Co)	Final pH	Rejection (%)
Blank	500	7.55	-
100	320	7.19	45.00
250	219	6.87	62.20
500	105	6.46	83.00
750	51	6.00	94.80
1000	38	5.09	99.40

The highest rejection in term of color was observed with 1000 mg/L of $Ca(OH)_2$ (89.54%) as shown in Table 3.18.

Table 3.18 Color rejection using $Ca(OH)_2$

Coagulant concentration (mg/L)	Final Color (Pt-Co)	Final pH	Rejection (%)
Blank	1730.00	7.72	-
100	1610.00	8.09	6.94
250	1480.00	8.29	14.45
500	1380.00	8.60	20.23
750	1085.00	9.06	37.28
1000	181.00	9.73	89.54

Table 3.19 shows color rejection with respect to the three coagulants ($Ca(OH)_2$, $Al_2(SO_4)_3$ and $FeCl_3$). Jar test analysis was repeated twice. Both $FeCl_3$ and $Al_2(SO_4)_3$ gave a high color rejection in the presence of cationic polymer.

Table 3.19 Color rejection using three different coagulants

Coagulant	Coagulant concentration (mg/L)	Color rejection (%)
$Al_2(SO_4)_3$	1000	98.86
	1000 (repeated)	97.58
$FeCl_3$	1000	99.40
	1000 (Repeated)	97.76
$Ca(OH)_2$	1000	89.54
	1000 (Repeated)	85.05

Table 3.20 shows the effect of cationic polymer on color rejection, two concentrations of cationic polymer were added into wastewater containing coagulants. Cationic polymer alone showed a poor performance with respect to color rejection (45.45%). An optimum of 30 mg/L of cationic polymer was found for two coagulants ($Al_2(SO_4)_3$ and $FeCl_3$).

Table 3.20 Effect of cationic polymer on color rejection

Coagulant	Coagulant concentration (mg/L)	Cationic polymer concentration (mg/L)	Final pH	Final Color (Pt-Co)	Rejection (%)
Blank	-	-	7.93	1320	-
$Al_2(SO_4)_3$	1000	60	5.77	87	93.41
$FeCl_3$	1000	60	5.35	94	92.88
Cationic Polymer	1000	-	5.95	720	45.45
$Al_2(SO_4)_3$	1000	30	5.84	46	96.52
$FeCl_3$	1000	30	5.56	63	95.23

Color was highly rejected with $Al_2(SO_4)_3$ in the presence of 60 mg/L cationic polymer and with $Al_2(SO_4)_3$ in the presence of 30 mg/L cationic polymer (97.60 and 96.58% respectively), $FeCl_3$ gave similar rejection irrespective of the cationic polymer concentration. $Ca(OH)_2$ in the presence of 60 mg/L cationic polymer gave a color rejection of 92.81% as depicted in Table 3.21.

Table 3.21 Effect of pH on cationic polymer addition on color rejection by various coagulants

Coagulant	Coagulant concentration (mg/L)	Cationic polymer concentration (mg/L)	pH	Color (Pt-Co)	Rejection (%)
Blank	-	-	8.27	1168	-
$Al_2(SO_4)_3$	1000	60	9.60	28	97.60
$FeCl_3$	1000	60	9.41	117	89.98
$Ca(OH)_2$	1000	60	9.43	84	92.81
$Al_2(SO_4)_3$	1000	30	9.65	40	96.58
$FeCl_3$	1000	30	9.58	108	90.75

3.1.4 Hardness rejection (Tap water tests)

Coagulation prior to MBR unit is one of the methods used to remove inorganic foulants in wastewater. However, excessive sludge production remains the main drawback to this process. In the case of ITOB organized industrial zone the hardness comes from raw water (well water) fed to the industries, if this water is soften before giving to the industries, this will drastically reduce the inorganic fouling problem in the MBR unit. For this purpose, jar tests were carried out with tap water. Since tap water does not contain suspended solid, it will not produce large amount of sludge.

Table 3.22 shows the results of hardness rejection obtained with $Ca(OH)_2$ in tap water. Unfortunately we were able to observed rejection only at lower concentration (125 mg/L) with 17.57% of hardness rejection. The negative results may are attributed to the addition of excess Ca^{2+} into the tap water. The reason why the pH was kept at 7.5 is that, when this method is considered there will be no need of pH adjustment while sending the tap water to the industries.

Table 3.22 Effect of coagulant concentration on hardness removal from tap water

Coagulant concentration ($Ca(OH)_2$)	Adjusted pH	Final hardness (mg/L)	Rejection (%)
Blank	-	424	-
1000 mg/L	7.50	1430	-237.26
750 mg/L	7.50	1210	-185.38
500 mg/L	7.50	850	-100.47
250 mg/L	7.50	483.5	-14.03
125 mg/L	7.50	349.5	17.57

The pH was kept at 7.5 with the same reason as in $Ca(OH)_2$ also. It has been mentioned in the literature that the optimum pH for $FeCl_3$ is around 6-7 for coagulation. The hardness rejection showed a similar trend with wastewater test. The rejection increase as the $FeCl_3$ concentration increased. The highest hardness rejection was observed with 1000 mg/L concentration as 45.52% (Table 3.23).

Table 3.23 Effect of coagulant ($FeCl_3$) concentration on hardness removal

Coagulant concentration ($FeCl_3$)	Adjusted pH	Final pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	7.46	424.0	-
1000mg/L	7.50	7.65	231.0	45.52
750mg/L	7.50	7.69	258.5	39.03
500mg/L	7.50	7.57	316.5	25.35
250mg/L	7.50	7.53	360	15.09
125mg/L	7.50	7.5	381.5	10.02

Table 3.24 shows hardness rejection from tap water with $Al_2(SO_4)_3$. The rejection values showed a similar trend with that of wastewater test. Rejection increased with coagulant concentration. Hardness rejection was highest with 1000 mg/L of coagulant as 54.40%.

Table 3.24 Effect of coagulant concentration ($Al_2(SO_4)_3$) concentration on hardness removal

Coagulant concentration ($Al_2(SO_4)_3$)	Adjusted pH	Final pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	7.30	569.06	-
1000 mg/L	7.50	7.53	259.50	54.40
750 mg/L	7.50	7.47	304.50	46.49
500 mg/L	7.50	7.39	333.00	41.48
250 mg/L	7.50	7.32	385.50	32.26
125 mg/L	7.50	7.32	413.50	27.34

Hardness rejection values obtained with Na_2CO_3 are given in Table 3.25. The rejections showed a similar trend with other coagulants, as the concentration increased, the hardness rejection also increased with the exception of 750 and 1000 mg/L of coagulant. Highest hardness rejection was observed with 750 mg/L as 76.71%. In this case, pH was not kept at 7.5 as in the case of alum and other coagulants because it was mentioned in the literature that the optimum pH of this coagulant is around 9.

Table 3.25 Effect of Na_2CO_3 concentration on hardness removal from tap water

Coagulation concentration (Na_2CO_3)	Adjusted pH	Final pH	Final Hardness (mg/L)	Rejection (%)
Blank	-	7.54	395.00	-
1000 mg/L	8.50	8.44	96.00	75.70
750 mg/L	8.50	8.46	92.00	76.71
500 mg/L	8.50	8.22	130.00	67.09
250 mg/L	8.50	8.23	140.00	64.56
125 mg/L	8.50	8.18	234.00	40.76

Table 3.26 shows hardness rejection values with $NaHCO_3$. As the concentration increased, hardness rejection also increased. The highest rejection

was observed with 1000 mg/L of $NaHCO_3$ 62.28%. The rejections with 1000 mg/L and 750 mg/L of coagulant are found to be very close. Therefore, while selecting the coagulant concentration, 750 mg/L of coagulant can be selected as optimal dosage. The reason of using initial pH 8.5 is same as given in the case of Na_2CO_3.

Table 3.26 Effect of $NaHCO_3$ concentration on hardness removal from tap water

Coagulant concentration ($NaHCO_3$)	Adjusted pH	Final pH	Hardness (mg/L)	Rejection (%)
Blank	-	7.54	395.00	-
1000 mg/L	8.50	8.58	149.00	62.28
750 mg/L	8.50	8.27	157.00	60.25
500 mg/L	8.50	8.24	193.00	51.14
250 mg/L	8.50	7.47	259.00	34.43
125 mg/L	8.50	7.43	295.00	25.32

3.2 Pilot MBR tests

Before tap water test, the membranes were soaked into tap water for 24 hours for conditioning and the measured flux was 190 LMH as shown in Figure 3.3 Tap water test was considered first to understand the main source of scaling on the surface of the membranes. Therefore, preliminary tap waster test was considered first which lasted for 2 months, the flux profile of this test is depicted in Table 3.27.

Table 3.27 Flux profile of preliminary tap water MBR pilot test

Days	**Flux (LMH)**	**Days**	**Flux (LMH)**	**Days**	**Flux (LMH)**	**Days**	**Flux (LMH)**
1	190.00	17	12.37	39	4.22	56	2.78
2	180.00	27	12.00	40	4.18	57	2.63
3	109.09	28	10.21	41	4.09	58	2.54
4	98.18	29	8.41	42	4.27	59	2.26
5	40.91	30	6.94	43	4.12	60	2.12
6	38.18	31	5.86	44	3.59	63	1.92
10	36.36	32	4.98	46	3.72	56	2.78
13	17.04	33	4.33	52	3.68	57	2.63
14	15.68	34	4.29	53	3.15	58	2.54
15	13.64	35	4.27	54	3.12	59	2.26
16	12.95	38	4.23	55	3.03	60	1.92

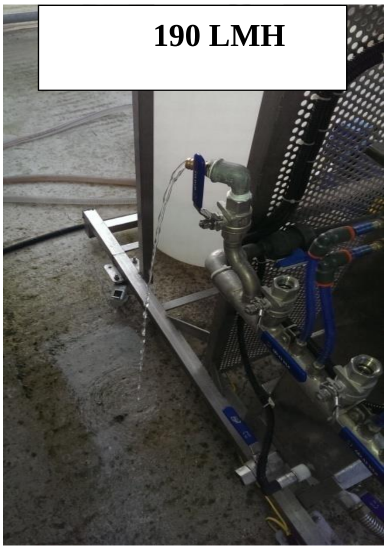

Figure 3.3 Permeate flow of virgin

3.2.1 Preliminary study

Figures 3.4 show the fouled and new membrane after 2 months of operation with tap water. Figure 3.5 shows the flow from the fouled membrane before chemical cleaning (almost no flow). The fouled membrane has a chalky color indicating a fouling from inorganic (mainly $CaCO_3$ deposition).

Figure 3.4 Photo of new and fouled membrane

Figure 3.5 Permeate flow from fouled membrane after 60 days of operation

Chemical cleaning after preliminary test

Since our membranes were not contacted with wastewater, therefore we can say that only inorganic fouling exists on our membranes and this shows that only acid cleaning can remove the foulants. Generally, the membrane manufacturers do give a recommended cleaning procedure, however, this may vary according to wastewater treatment plant (since the characteristic of wastewater cannot be same everywhere). However, one should be careful with the recommended pH and temperature because these parameters affect the chemical structure of the membranes thereby decreasing the performance of the membrane. To find the optimum condition of membrane cleaning, a trial and error method is generally adopted (Kappel C., 2014). For this purpose our membranes were cleaned as follows:

The membranes were cleaned ex-situ with 3000 mg/L citric ($C_6H_8O_7.H_2O$) at the 2.5 pH, temperature was 7^oC and the operation time was 2.5 hours. After cleaning with citric acid, rinsing with tap water was carried out and the membranes were inserted back to the reactor. The membrane recovery was calculated with the equation given below

Membrane recovery = J/J_o *100

Where J and J_o are the flux of cleaned and virgin membrane, respectively.

The flux after citric acid cleaning was 22.5 LMH from the initial flux which was 1.92 LMH. That means we recovered about 11.84% of the virgin membrane. This shows that the concentration of the citric acid used was not enough to remove the scaling from membranes. Table 3.28 gives the summary of how chemical cleaning was carried out.

When the membranes were cleaned, the temperature was around 2°C therefore, we need to recalculate the flux values since it changes with temperature as recommended by Yigit (2007). The equation below was used to calculate the normalized flux.

$$J(20℃) = \frac{Q_p \times Exp^{-0.0239\times(T-20)}}{A}$$

Where J is the normalized flux at 20°C, Q_p (L/h) is the flow rate of the permeate, T(°C) is temperature and A is the membrane area (m^2).

Table 3.28 Summary of citric acid cleaning

Date	Citric acid concentration (mg/L)	Cleaning procedure	Contact time (h)	Normalized flux (LMH)	Membrane recovery (%)
27/01/2016	3000	The membranes were cleaned ex situ with citric by adjusting the pH to 2.5 (temperature was 7^{o}C). After cleaning with the acid, followed by rinsing with tap water, membranes were inserted back to the reactor.	2.5	22.5	11.84
28/01/2016	4000	The membranes were cleaned ex situ with citric by adjusting the pH to 2.5, the temperature was 7^{o}C.	3	22.5	11.84
	7000	The concentration was increased, but the pH was same with the previous cleaning, also the temperature was increase to about 20^{o}C.	0.5	24	12.63
29/01/2016	8000	Cleaning was conducted by drawing the chemical solutions as permeate by maintaining the pH at 2.5 and the temperature at 20^{o}C, followed by cleaning with a tap water, then the membranes were inserted back to the reactor.	2.5	26	13.68
01/02/2016	10000	COP cleaning was done by keeping the pH at 2.5 and the temperature at 25^{o}C, we stopped the chemical cleaning because we observed a membrane ageing.	1.3	47.78	26.25

Figures 3.6 and 3.7 show a clear view of fouled and cleaned membranes. In the first citric acid cleaning trial, we can say that the membranes are physically cleaned but when we looked at the flux which was 22.5 LMH (Figure 3.8), it shows a lot has to be done in order to recover the membranes from the initial flux which was 190 LMH**.**

Figure 3.6 Membrane during 3000 mg/L citric acid cleaning

Figure 3.7 Chemically cleaned membranes after 3000 mg/L citric acid cleaning

When 4000 mg/L of citric acid solution was used for cleaning, there was no improvement in the flux and the measured flux was 22.5 LMH. The concentration of citric acid was increased to 7000 mg/L but the pH was same with the previous cleaning. Temperature has a significant role in membrane cleaning as it increases the rate of the reaction between the chemical reagent and the foulants. For this purpose, the temperature was increased to about 20^{o}C in order to increase the speed of the reaction. The membranes remained in the solution for 30 min after that the flux was measured and was found to be 24 LMH which is close to the previous findings. This shows that the concentration of the citric acid used was not enough for the foulants. Figure 3.9 shows the permeate flow after the chemical cleaning in this step. The contact time between the citric acid and the membranes was shortened from 3 h to 0.5 h because, we were afraid to cause some changes in chemical structure of the membranes. Even after using 7000 mg/L of citric acid the changes in the flux was very small, this shows that the membranes were severely fouled.

Figure 3.8 Permeate flow after the first cleaning after 3000 mg/L citric acid cleaning

Figure 3.9 Permeate flow after the second cleaning.

We decided to do the cleaning by drawing the chemical solutions as permeate for 2 hours from the membranes. During cleaning, the pH and temperature was kept at 2.5 and 20^{o}C, respectively. Also the concentration was increased to 8000 mg/L and cleaning period was only 1 h. The flux was measured as 26 LMH which is still low compared with the flux of the virgin membrane which is 190 LMH. Figure 3.10 shows the flow from the membranes after cleaning with 8000 mg/L citric acid. This cleaning also was not enough. The increment of the citric acid concentration was adopted from Wang et al. (2014) in which higher concentration of citric acid was used (up to 1.5 %) without causing any damage to the membranes.

Figure 3.10 Permeate (citric acid solution) flow during chemical cleaning

For the above mentioned reason, a 10000 mg/L solution of citric acid was prepared for COP cleaning by keeping the pH at 2.5 and the temperature at 25^{o}C for 1 hour 20 min. The flux measured was 47.78 LMH (Figure 3.11) then chemical cleaning was stopped because we started to observe a membrane ageing. After 1 hour flux decreased to 42.27 LMH after 2.5 hours. Flux recovery was about 30% and remained constant for that day. Figure 3.12 show a close view of the membranes just after 2 days of operation after 8000 mg/L citric acid cleaning.

Figure 3.11 Flow from cleaned

Figure 3.12 Photos of membranes before last cleaning (after 2 days of operation)

3.2.2 MBR pilot study with tap water without antiscalant addition

Figure 3.13 shows the normalized flux of our study with a tap water as our feed. Immediately after the chemical cleaning, we observed that the measured flux decreased rapidly, and then almost constant flux then it decreased again. After the chemical cleaning, the membranes were clean but when they are inserted into water, accumulation of flocs on the pores of the membrane occurred. That was the reason of the sharp drop in the flux immediately after chemical cleaning (Meng et al., 2009). After rapid build-up of the flocs, the system reached steady state in 14th day and after that the flux was almost constant until 21th day. After 21th day the flux began to decrease that means, during the steady state of operation there was an accumulation of foulants on the membrane. From 21th day it began to have some negative effects on the permeability, then we can say that there is a membrane fouling. Figure 3.13 and Table 6.1 (in Appendix) show the normalized flux values of our experiment vs time the calculated normalized flux values with their corresponding temperature.

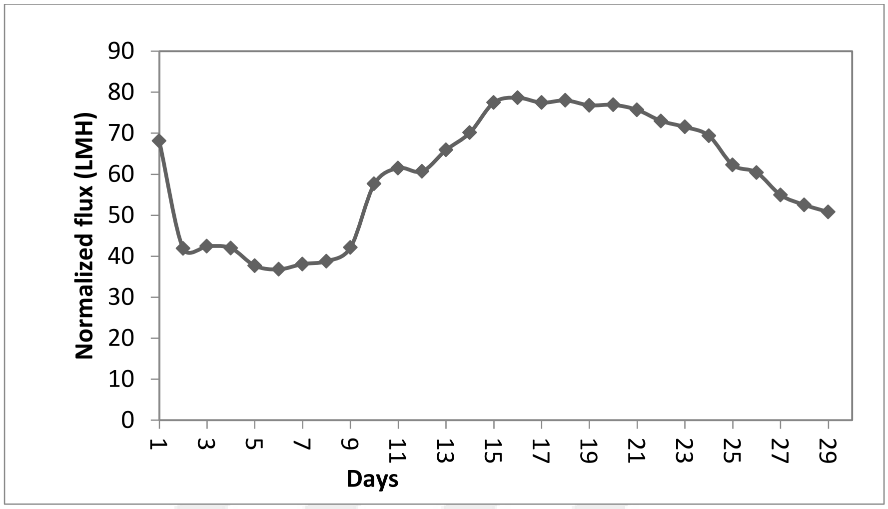

Figure 3.13 Normalized flux values of tap water study

Chemical cleaning after a test without antiscalant addition on tap water

Before chemical cleaning, the measured flux was 43 LMH. The permeate flow from the membranes after 29 days of operation can be seen in Figure 3.14.

Figure 3.14 Flow of the permeate before cleaning after 29 days of operation in tap water

Figure 3.15 shows the photo of our fouled membranes after 29 days of operation.

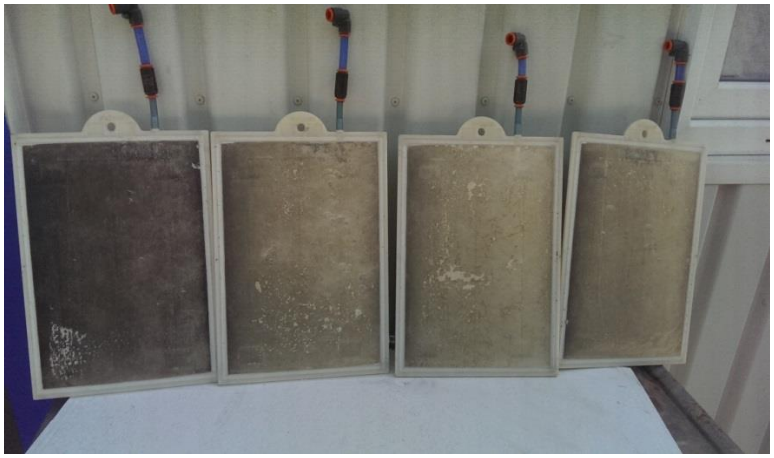

Figure 3.15 Fouled membranes after 29 days of operation in tap water

We wonder about the color of the fouling on the surface of the membrane which seems not to be only $CaCO_3$. For this purpose, the foulant extracted during chemical cleaning was analyzed by EDX (Energy Dispersive X-ray spectroscopy) system. Thus, the main component of the precipitate is determined as $CaCO_3$ (21.5 wt%) and the results are shown in Table 3.29

Table 3.29 Analysis of foulant on membrane surface

Element	Content rate (wt %)
Ca	8.614
Si	0.168
K	0.062
Zn	0.059
Fe	0.052
Cu	0.021
Br	0.017
S	0.011
Sr	0.05
Ti	0.003
C	91
Total	100.057

In the preliminary study section, it was mentioned that some deterioration on some part of the membrane surface was observed. This might likely due to the high concentration of citric acid used during the cleaning (10000 mg/L). Figure 3.16 shows a close look at the damaged part on the membrane surface.

Figure 3.16 Deformation on membrane surface by high concentration of citric acid

The membranes were cleaned ex-situ as shown in Figure 3.17 with 4000 mg/L citric acid and the pH was adjusted to 2.5 during cleaning. The membranes were soaked into 4000 mg/L citric acid solution for 3 hours, during cleaning. The foulants were easily removed from the membranes surface as can be seen in Figure 3.18, this shows that the fouling was not severe also the ambient temperature ranges from -5 to 13^{o}C. Therefore, we can say that precipitation was not high as in the preliminary section since the solubility of $CaCO_3$ decreases by increasing the temperature. The membranes were cleaned with tap water and then inserted back to the reactor for another study.

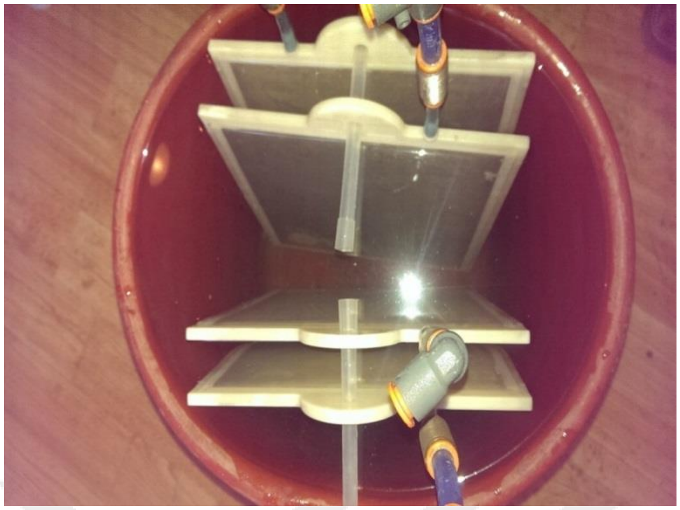

Figure 3.17 Membranes inside a citric acid solution during chemical cleaning

Figure 3.18 shows a chemically cleaned and fouled part a membrane. While in Figure 3.19 we can see chemically cleaned membranes. The obtained normalized flux was 140 LMH. When we looked at the flow after cleaning we can see that there is an improvement in the flux (Figure 3.20). As we cleaned the membranes frequently, the remaining foulants from the previous study were possibly removed also. While cleaning the membranes frequently, one should be very careful, because excessive cleaning can lead to the damage on the active layer of the membrane.

Figure 3.18 Fouled and chemically cleaned part of a membrane after tap water test in the absence of antiscalant

Figure 3.19 Cleaned membranes after tap water test in the absence of antiscalant

Figure 3.20 Flow of the permeate after cleaning after tap water test in the absence of antiscalant

3.2.3 MBR pilot study with tap water in the presence of antiscalant

Table 6.2 (in Appendix) and Figure 3.21 shows the flux values and normalized flux values of tap water study by, respectively using antiscalant. The flux values shows a similar trend with the previous study (without antiscalant) in the sense that, the immediate flux after chemical cleaning was high then shows a flactuation for two days after that the flux values were almost constant (that means that the system has reach a steady state).

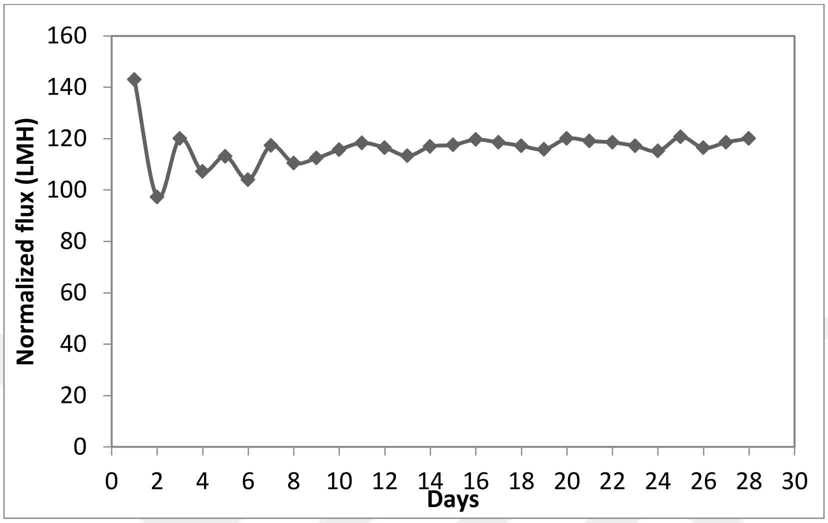

Figure 3.21 Normalized flux values vs. time for the study with tap water in the presence of antiscalant

3.2.4 MBR pilot test with wastewater in the presence of antiscalant

Figure 3.22 shows the flux of the study in the presence of antiscalant. At the beginning of the experiment the calculated flux was 84 LMH. A day after start up of the system, a sharp drop in the flux was observed due to the accumulation of colloids and biomass on the surface of the membranes, followed by gradual decrease in the flux until 21st day of the experiment in which a second sharp drop began to appear (8 LMH) indicating that the membranes are fouled. In the next day, a chemical cleaning was applied exsitu as follows:

A physical cleaning was employed to remove the foulants. This was done by hand without applying much pressure on the membranes in order not to cause damage on the membranes. The measured flux was 72 LMH with tap water.

The membranes were soaked into a 3000 mg/L hypochlorite solution for 45 min followed by 15 min backwash (by gravity), the normalized flux with tap water (after rinsing with tap water) was 147 LMH.

After soaking in hypochlorite the membranes were soaked into 2000 mg/L of citric acid solution for 45 min then 15 min backwash (by gravity) followed by rinsing with tap water, the measured flux with tap water was 188 LMH which is very close to the measured flux with the virgin membrane. The test was continued with the wastewater in the presence of antiscalant, the flux measured with the wastewater was 92 LMH which is greater than the flux at the beginning of the experiment. This happened due to the decrease in the MLSS in the reactor. A day after the cleaning, we experienced a sharp drop in the flux which may be due to presence of fine particles in the biomass. The flux and dates of the study in the presence of antiscalant are shown in Table 6.3, in the presence of antiscalant, the membranes seems to be less biofouled as can be seen in Figure 3.23

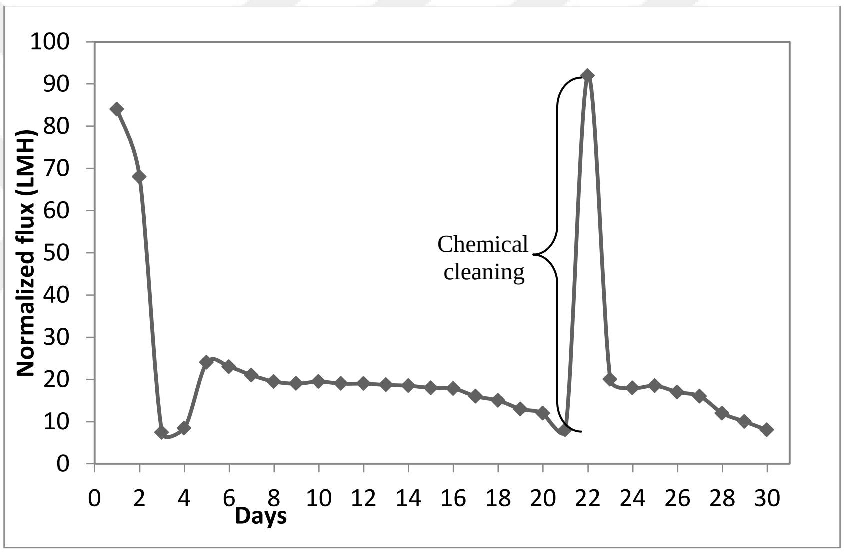

Figure 3.4 Normalized flux Vs time plot for pilot MBR study with wastewater in the presence of antiscalant

Figure 3.23 Membranes at the end of the study in the presence of antiscalant

3.2.4.1 Quality analysis of MBR permeate

Table 3.30 shows quality analysis results of MBR permeate such as conductivity, salinity, dissolved oxygen (DO), pH and MLSS from the MBR pilot tests performed in the presence of antiscalant.

Table 3.30 Some quality analysis values taken everyday

Days	MLSS (g/L)	DO (mg/L)	Cond (ms/cm)	pH	Sal (%).
1	17.50	7.25	2.63	7.53	1.47
2	18.10	5.68	2.91	7.47	1.37
3	43.24	4.93	2.65	7.38	1.46
4	27.65	5.83	2.87	7.45	1.45
5	19.30	6.74	3.11	7.53	1.49
6	19.75	6.24	3.45	7.5	1.58
7	20.50	5.86	3.38	7.53	1.76
8	19.50	5.48	3.41	7.62	1.77
9	17.60	4.23	3.27	7.57	1.79
10	19.65	7.98	3.58	7.58	1.76
11	17.95	6.50	3.47	7.43	1.84
12	17.95	6.72	3.51	7.48	1.80
13	18.50	6.61	3.35	7.41	1.87
14	19.35	6.95	3.42	7.56	1.91
15	20.65	7.17	3.54	7.48	1.85
16	19.85	7.25	2.87	7.55	1.90
17	20.15	6.95	3.05	7.63	1.89
18	19.75	6.78	2.25	7.48	1.95
19	9.18	7.18	2.75	7.68	1.98
20	18.75	7.10	2.89	7.56	1.85
21	18.91	6.95	3.25	7.67	1.95
22	19.45	6.65	3.51	7.45	1.88
23	18.90	5.98	3.45	7.23	1.92
24	19.75	6.25	3.15	7.52	1.83

Figure 3.24 shows color rejections from the study in the presence of antisclant. As can be seen, almost all the rejections were above 98% indicating well function of the membranes. The minimum color in the permeate was 19 Pt-Co while the maximum color was 35 Pt-Co. One of the reason to obtain a clear MBR permeate is due to the formation of biofilm on the surface of the membrane which serve as a prefilter before the membranes. Figure 3.25 shows TSS rejection in the MBR pilot study with antiscalant. The results showed a similar trend with color rejection in which all the influent TSS were rejected by MBR (not less than 99%, even 100% in some cases). The rejection increased when the membranes were fouled. Of course this is good to some extent since almost all the TSS is rejected with the help of the biolayer formed on the membrane during the process. However if membranes are not clean this will lead to a severe fouling which may be remove even by chemical cleaning as shown in the preliminary section of this thesis.

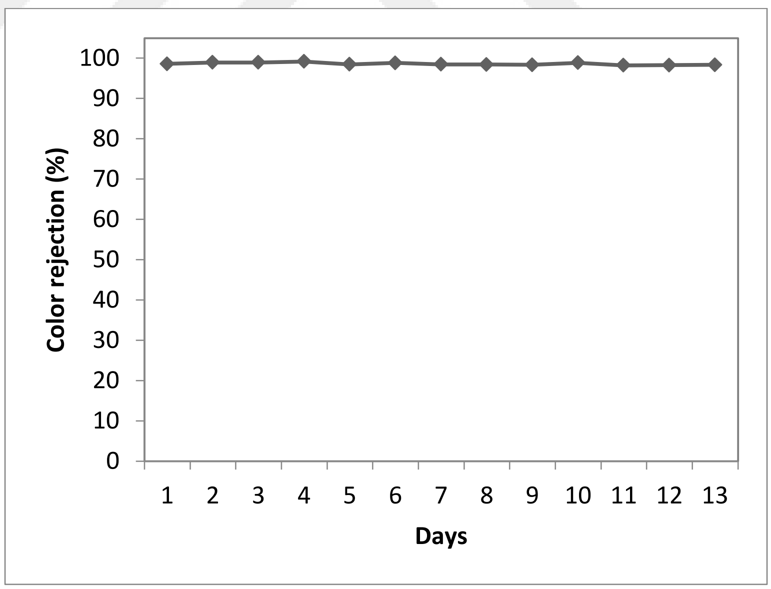

Figure 3.24 Color rejection in the presence of antiscalant

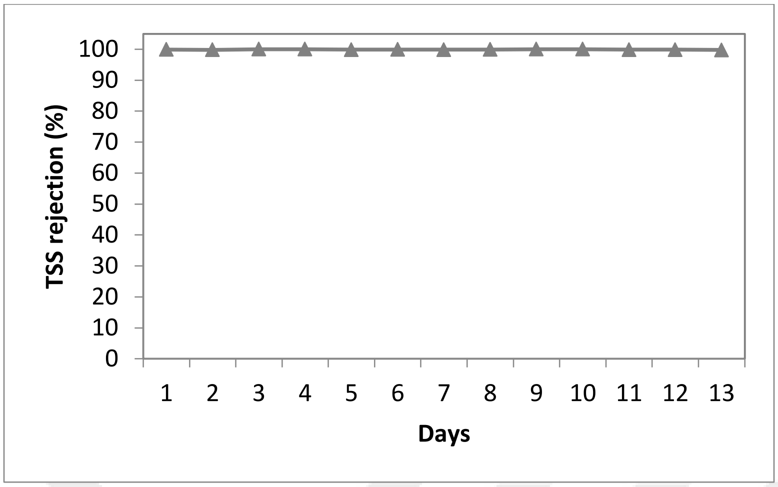

Figure 3.25 TSS rejection by MBR pilot system in the presence of antiscalant

Figure 3.26 shows rejection values of NH_4-N, throughout the study NH_4-N were rejected more than 98%. This is possible because in MBR NH_4-N is usually rejected independent of STR (Huang et al., 2001). In Table 3.31 shows the values of NO_3-N in the influent and the effluent. As depicted, nitrate in the effluent was greater than that in the influent. The reason behind the increase in the NO_3-N instead of decrease was due to ineffient denitrification in the MBR tank, this can be enhanced when aeration is done at intermediate rates so that some part in the tank will operate as anoxic chamber (Radjenovic et al., 2007). NO_2-N was highly rejected as shown in Figure 3.27. This is possible because NO_2-N is removed by converting NH_4-N into NO_2-N in an oxic process, in the MBR system, since air is continuously supplied into the MBR chamber. In wastewater treatment, nitrogen is usually removed by the conversion of NO_3-N into N_2 gas and this requires an anoxic process which is lacks in the MBR chamber. That is why we could not achieve a high total nitrogen removal in our system as depicted in Figure 3.28. An average rejection of 33% was obtained. Phosphorus in wastewater is usually found in phosphate form and it is usually removed by adsorption on the biomass or by precipitation (Radjenovic et al., 2007). Both precipitation and adsorption require a pH adjustment or the presence of calcium ion. In our wastewater, the calcium concentration was above 140 mg/L. That is the reason of high PO_4-P rejection in our system as depicted in Figure 3.29.

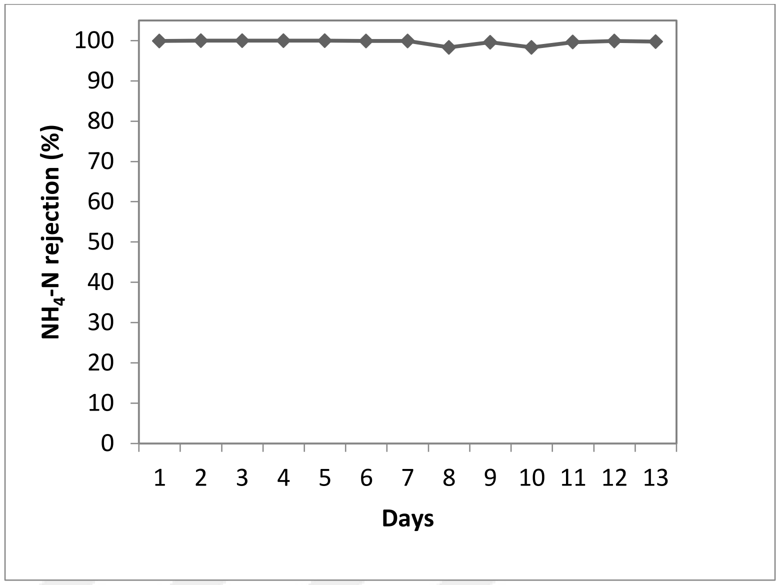

Figure 3.26 NH_4-N rejection in the presence of antiscalant

Table 3.31 Inlet and exit concentration in the study with antiscalant

Date	Inlet NO_3-N (mg/L)	Permeate NO_3-N (mg/L)
1	0.714	6.57
2	0.956	7.51
3	0.871	6.53
4	0.652	5.78
5	0.794	5.89
6	0.941	7.15
7	0.892	6.11
8	0.921	7.54
9	0.827	6.81
10	0.853	6.42
11	0.918	6.58
12	0.869	6.73
13	0.768	7.21

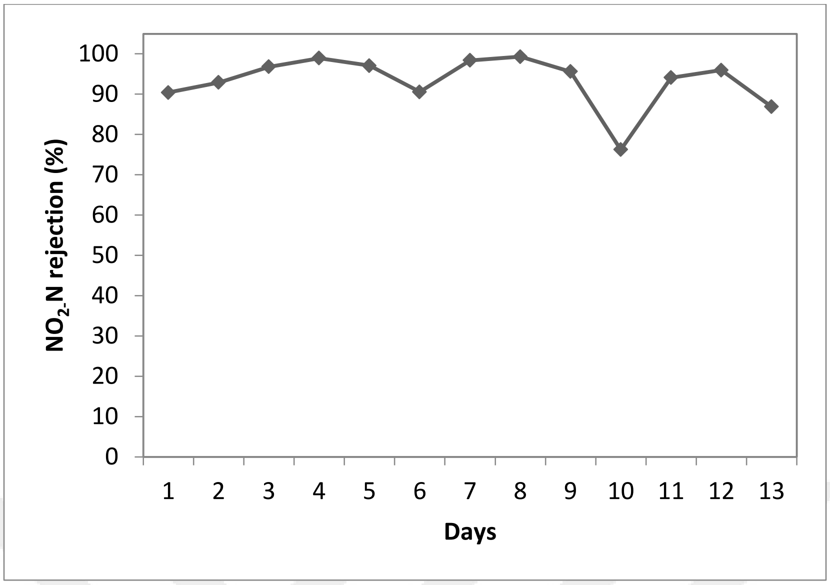

Figure 3.27 NO_2-N rejection in the study with antiscalant

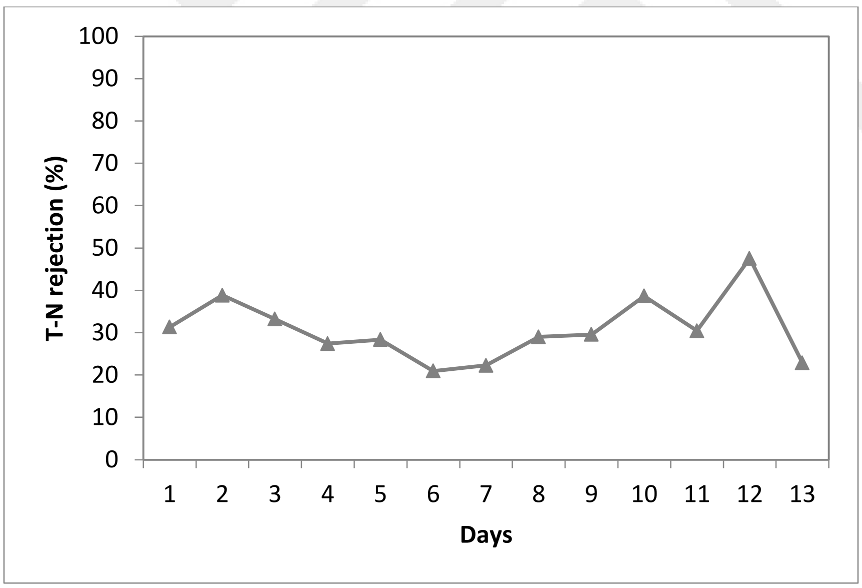

Figure 3.28 T-N rejection in the study with antiscalant

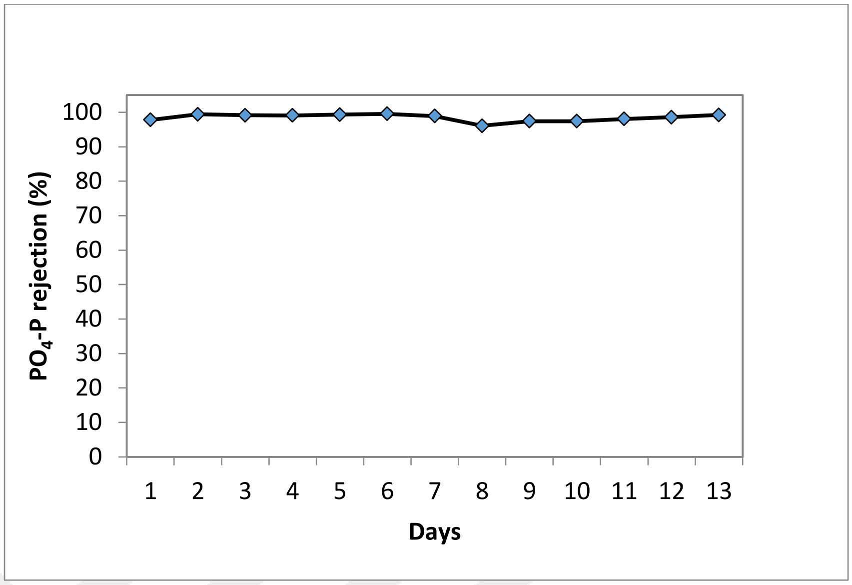

Figure 3.29 PO_4-P rejection in the presence of antiscalant

COD is a measure of organic matter in the wastewater. For this purpose, its removal will improve the quality of the effluent water in wastewater treatment processes. In our study, an average COD removal of 94% was achieved with respect to COD rejection as shown in Figure 3.30. Although the mechanism of COD removal in wastewater is scarce, but generally the rejection is greater than 90% (Radjenovic et al., 2007).

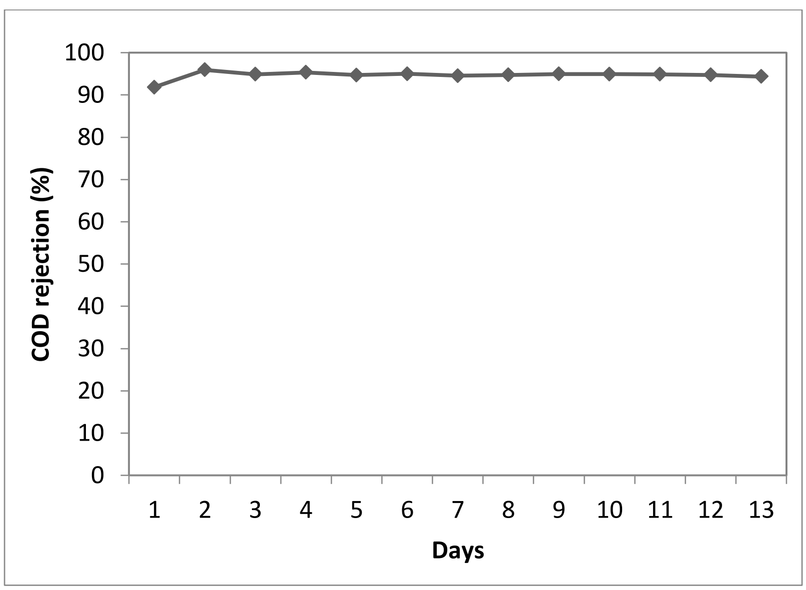

Figure 3.30 COD rejection by MBR pilot system in the presence of antisclant

3.2.5 MBR pilot study with wastewater in the absence of antiscalant

In Figure 3.31, the MBR pilot flux profile of the study carried out with wastewater without antiscalant was shown. As can be seen, the flux drop in the first 8 days of the experiment (from 84 to 8 LMH) unlike in case of the study with antiscalant. The reason of the flux drop may be due to the presence of the fine particles or inorganic foulants, for that reason we consider to clean the membranes in situ with 300 mg/L of hypochlorite for (chemically enhance backwash) 30 min followed by washing with 300 mg/L of citric acid for 30 min. The flux after the cleaning was 46 LMH.

The initial flux after chemical cleaning and the initial flux the beginning of the experiment were different. The reason might be due to incomplete removal of the foulants. The system continues to work without antiscalant until 21st day in which the flux drops from 46 to 8.18 LMH (which means cleaning is required). The membranes were cleaned insitu (chemically enhanced backwash) with 500 mg/L of hypochlorite for 30 min followed by 500 mg/L of citric acid for 30 min. The concentrations of hypochlorite and citric acid increased from 300 to 500 mg/L because, more fouling on the membranes was expected compared to the previous fouling.

After the second cleaning, the experiment continued until 29th day (the end of the experiment), the flux dropped from 27.27 to 9 LMH, the values of the flux and their corresponding dates are given in Table 6.4. The membranes were cleaned ex situ with the same procedure as cleaned before starting the experiment. The membranes seems to be more biofouled as can be seen in Figure 3.32 unlike in the presence of antiscalant as shown in Figure 3.22.

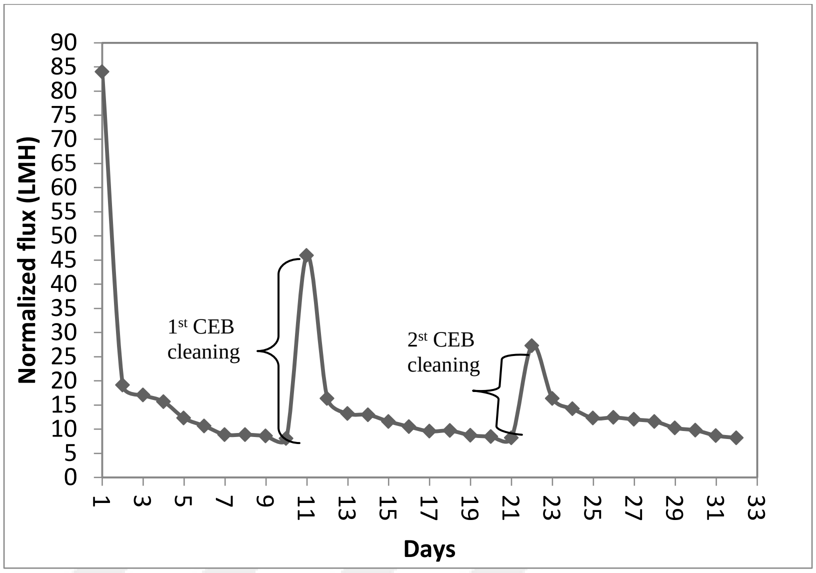

Figure 3.31 Pilot MBR without antiscalant

Figure 3.32 Membranes at the end of the MBR test in the absence of antiscalant

3.2.5.1 Quality analysis of the MBR permeate in the absence of antiscalant

Table 3.32 shows some quality analysis results such conductivity, salinity, dissolved oxygen (DO), pH and MLSS from the MBR pilot tests performed in the absence of antiscalant.

Table 3.32 Quality of MBR permeate obtained in the absence of antiscalant

Date	MLSS (g/L)	DO (mg/L)	Cond (ms/cm)	pH	Salinity (%).
1	8.00	4.23	3.68	7.48	1.97
2	8.05	7.98	3.79	7.54	200
3	8.15	6.50	3.58	7.50	1.88
4	8.23	6.72	3.41	7.51	1.85
5	8.25	6.61	3.23	7.28	1.75
6	8.41	6.95	3.15	7.39	1.65
7	8.65	7.10	2.95	7.35	1.58
8	8.68	6.95	3.01	7.23	1.55
9	8.73	6.65	2.92	7.05	1.52
10	8.89	5.98	3.14	7.15	1.61
11	8.92	6.25	3.43	7.38	1.68
12	8.96	6.95	3.35	7.45	1.82
13	9.12	7.17	2.88	7.58	1.97
14	9.48	7.25	2.65	7.43	2.05
15	9.72	7.25	3.14	7.48	1.78
16	9.68	5.68	3.61	7.53	1.92
17	9.88	4.93	3.26	7.47	1.96
18	9.97	5.83	3.18	7.24	1.79
19	10.15	5.98	3.32	7.18	1.81
20	10.35	5.63	3.12	7.53	1.93
21	10.45	6.50	2.97	7.34	1.8
22	10.86	6.72	2.87	7.56	1.77
23	11.5	6.61	2.68	7.62	1.74
24	11.41	6.95	3.08	7.38	1.81
25	11.56	6.47	2.75	7.51	1.83
26	11.3	5.93	3.17	7.69	1.89
27	11.15	5.87	3.21	7.46	1.98
28	11.25	5.61	2.94	7.58	1.88
29	11.5	4.98	3.28	7.67	1.72
30	11.75	5.43	3.18	7.67	1.84
31	12.24	5.36	3.06	7.62	1.59

Figure 3.33 shows color rejections from the study in the absence of antiscalant. As can be seen, almost all the rejection was above 98% indicating well function of the membranes. The minimum color in the permeate was 21 Pt-Co while

the maximum was 61 Pt-Co. The reason why there was poor performance in this study compared with the previous study (MBR in the presence of antiscalant) was due to the decrease in the MLSS which make it difficult for the membranes to reject small particles. Figure 3.34 shows TSS rejection in the study without antiscalant. The results showed a similar trend with color rejection in which all the influent TSS were rejected by MBR (not less than 99%, even 100% in some cases). The TSS rejection increased when the membranes are fouled, of course this is good to some extent since almost all the TSS are rejected with the help of the biolayer formed on the membranes as explained in the previous study (MBR in the presence of antiscalant).

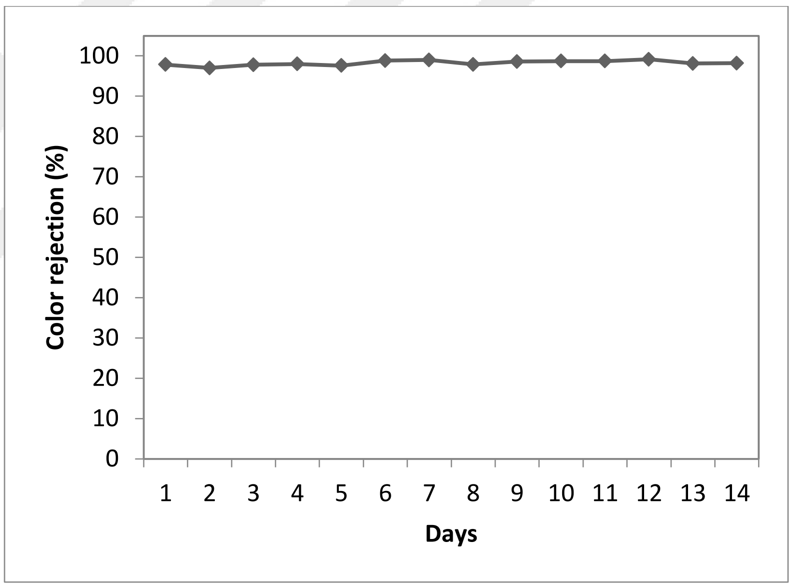

Figure 3.33 Color rejection by MBR pilot system in the absence of antiscalant

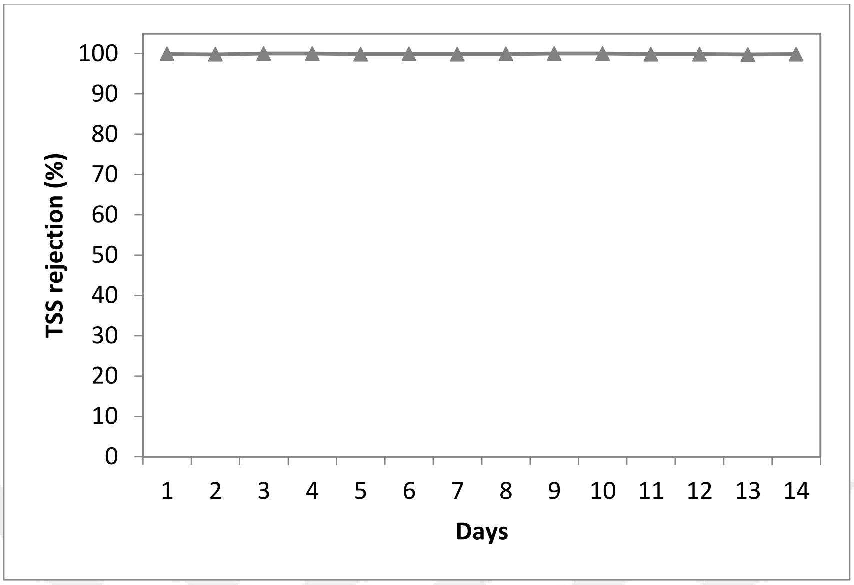

Figure 3.34 TSS rejection by MBR pilot system in the absence of antiscalant

Figure 3.35 shows rejection values of NH_4-N. Throughout the study NH_4-N was rejected more than 99% like in the previous study. This is possible because in MBR system, NH_4-N is usually rejected independent of STR as explained in the study with antiscalant. Table 3.33 shows the values of nitrate nitrogen in the influent and the effluent, as depicted nitrate in the effluent was greater than that in the influent. The reason behind the increase in the nitrate instead of decrease was due to ineffient denitrification in the MBR tank. NO_2-N was highly rejected as shown in Figure 3.36, this is possible because NO_2-N was removed by converting ammonia nitrogen into nitrite in an oxic process. In wastewater treatment nitrogen is usually removed by the conversion of nitrate into N_2 gas and this requires an anoxic process which is lacked in the MBR chamber. That is why we could not achieve high total nitrogen removal in our system as depicted in Figure 3.37 (an average rejection of 33% was observed like in the previous study). Phosphorus in wastewater is usually found as phosphate and is usually removed by adsorption into the biomass or by precipitation (Radjenovic et al., 2007). Both precipitation and adsorption requires a pH adjustment or the presence of calcium ion. In our wastewater, the calcium concentration was above 140 mg/L. That is the reason of high phosphate rejection in our system as depicted in Figure 3.38.

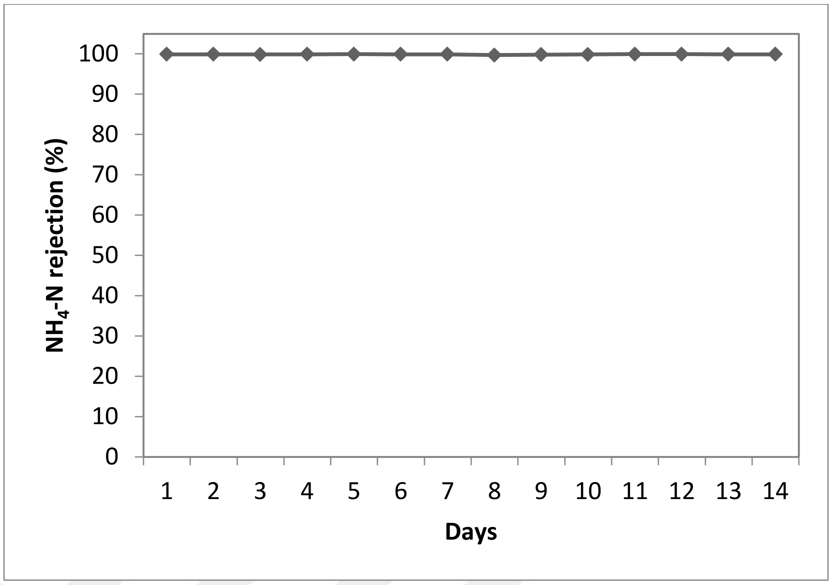

Figure 3.35 NH_4-N rejection in the absence of antisclant

Table 3.33 Inlet and exit concentration in the study without antiscalant

Date	Inlet NO_3-N (mg/L)	Permeate NO_3-N (mg/L)
1	0.750	29.88
2	0.5140	20.84
3	0.5170	24.60
4	0.510	28.56
5	0.625	31.88
6	0.535	28.88
7	0.556	30.88
8	0.664	30.00
9	0.547	29.88
10	0.521	38.32
11	0.534	29.88
12	0.601	40.40
13	0.566	40.00
14	0.518	29.88

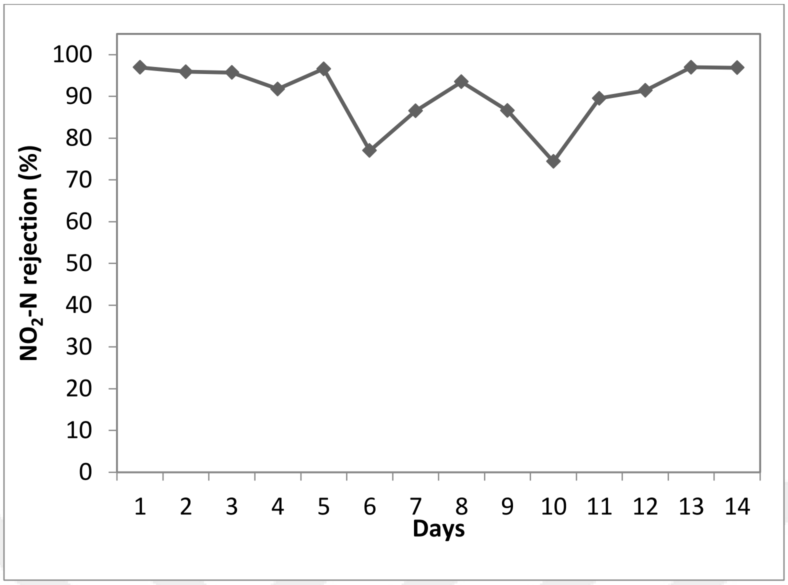

Figure 3.36 NO_2-N rejection in the absence of antisclant

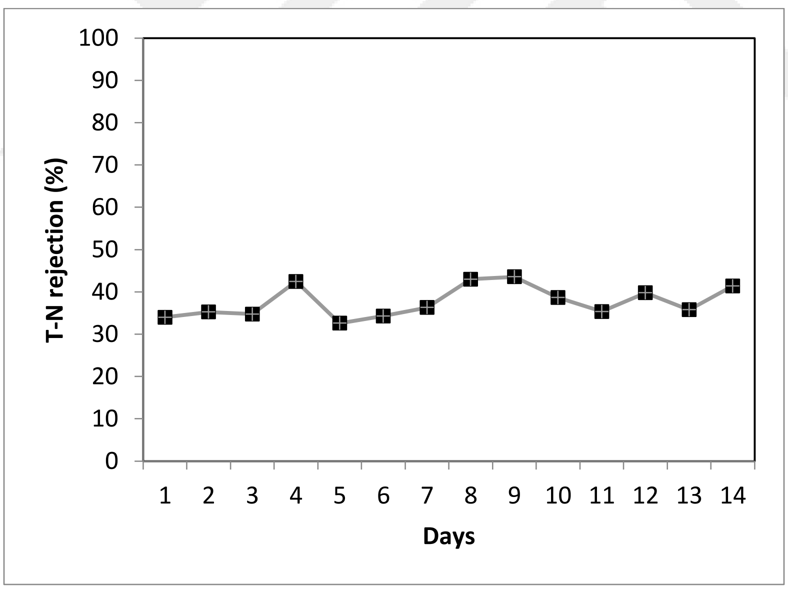

Figure 3.37 T-N rejection in the absence of antisclant

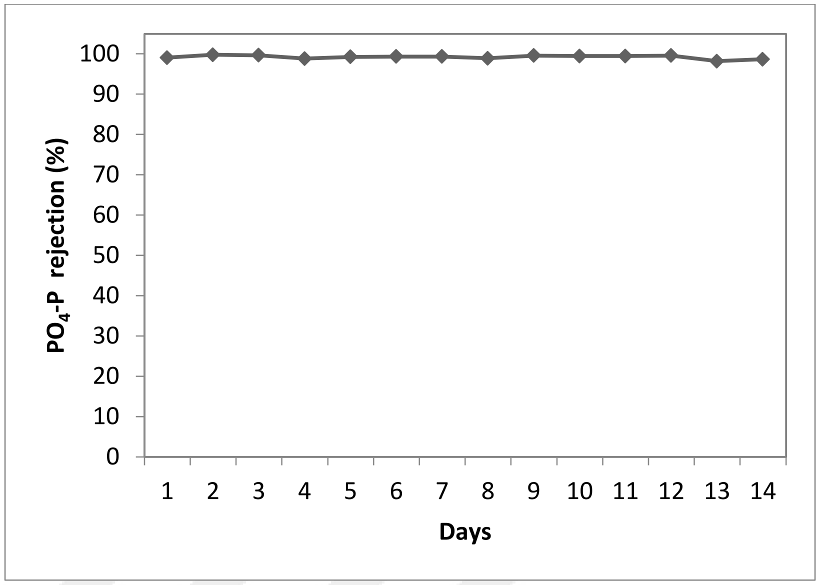

Figure 3.38 PO_4-P rejection in the absence of antiscalant

COD is the measure of organic matter in the wastewater, for this purpose its removal will improve the quality of the effluent water in wastewater treatment processes. In our study, an average COD removal of 94% was achieved as shown in Figure 3.39.

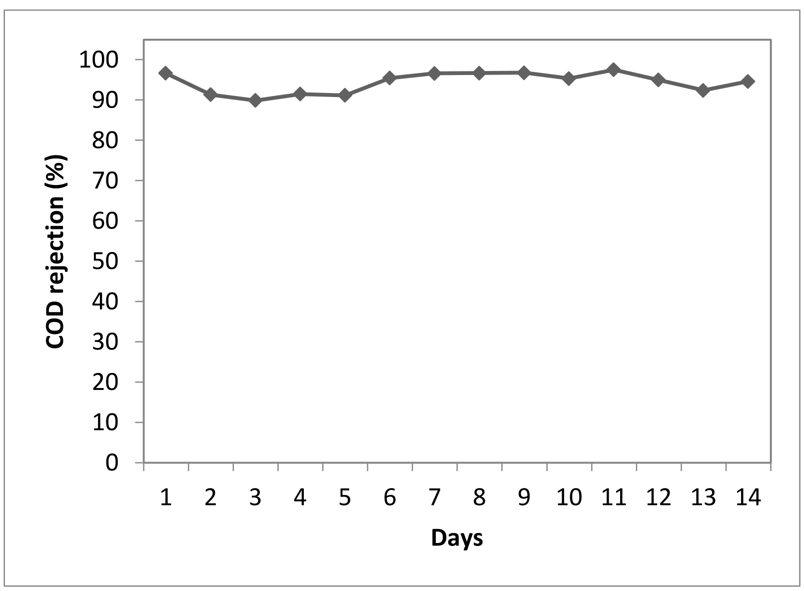

Figure 3.39 COD rejection by MBR pilot system in the absence of antiscalant

4.0 CONCLUSIONS

This study consist of two parts, optimizing the conditions for hardness removal in wastewater prior to MBR process and optimization of operational conditions to minimize membrane fouling on MBR membranes at ITOB Organized industrial wastewater treatment plant.

For coagulation tests, five coagulants and one coagulant enhancer were used ($Al_2(SO_4)_3$, $FeCl_3$, $Ca(OH)_2$, Na_2CO_3, $NaHCO_3$ and cationic polymer as coagulant enhancer) to remove hardness from wastewater prior to MBR unit. The tests were conducted first with wastewater with $Al_2(SO_4)_3$, $FeCl_3$, $Ca(OH)_2$ and cationic polymer to find the optimum concentration. It was concluded that the higher the coagulant concentration, the higher the hardness removal from the wastewater. The highest hardness rejection was found with 1000 mg/L of $Ca(OH)_2$ with an average hardness rejection of 78%. However, as the coagulant concentration decreased 100 mg/L, the hardness rejection was found to be negative. $Al_2(SO_4)_3$ and $FeCl_3$ were not good coagulants for the removal of hardness at pH below 7 even when coagulant enhancer was used. But they were very good coagulants in the rejection of TSS and Color especially in the presence of cationic polymer.

When the pH was adjusted to 9.0, the best coagulant was found to be $Ca(OH)_2$ again with a hardness rejection of 73% at 375 mg/L of coagulant. However, there was increase in hardness rejection with $Al_2(SO_4)_3$ and $FeCl_3$ from 47.90% and 43.87% to 62.66% and 71.73% as the pH increased to 9.50, similar rejections were found with all the coagulants (82-86%). It was concluded that $Al_2(SO_4)_3$ and $FeCl_3$ helps in hardness removal at pH above 7.0.

Jar test analyses were conducted with tap water also considering that it will produce less sludge. In this part $Al_2(SO_4)_3$, $FeCl_3$, $Ca(OH)_2$, Na_2CO_3 and $NaHCO_3$ were used. Hardness rejections were similar with the wastewater rejections (that is 46% for $FeCl_3$ 55% for $Al_2(SO_4)_3$). It was concluded that the optimum coagulant concentration with respect to Na_2CO_3 was 750 mg/L with a rejection of 76% and Na_2CO_3 was found to be better coagulant than $NaHCO_3$.

In the second part of this research study, the MBR pilot tests were conducted with tap water first to understand the source of scaling on MBR membranes. The pilot MBR, tests were carried out in the presence/absence of antiscalant

In the presence of antiscalant, the MBR system worked longer with less decrease in flux. It was concluded that the use of antiscalant in MBR system does not affect the bio-oxidation of the organic matter in the reactor.

If proper cleaning (either physical or chemical) is not applied, it will lead to the deterioration of the active layer on the other hand. When we used 1% of citric, acid we observed some membrane ageing. Therefore, less of acid concentration should be used while cleaning the membranes thereby inceasing the contact time.

According to the quality analyses results for MBR effluent, a 95% of COD rejection, 98% of total P-phosphate rejection, 40% total N-rejection, 99% of total suspended solid rejection and 98% of color rejection in the presence or absence of antiscalant were obtained.

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APPENDIX

Appendix1. Normalized flux valus in the absence of antiscalant tap water test (Figure 3.12).

Appendix 2. Normalized flux values with tap water in the presence of antiscalant (Figure 3.20).

Appendix 3. Normalized flux values with wastewater in the presence of antiscalant (Figure 3.21).

Appendix 2. Normalized flux values with wastewater in the absence of antiscalant (Figure 3.30).

Table 6.1 Normalized flux valus in the absence of antiscalant tap water test (Figure 3.12)

Days	Temperature (^oC)	Normalized Flux (LMH)	Flux (LMH)
1	2	68.14	47.73
2	1	41.87	26.59
3	2	42.46	27.61
4	3	41.97	27.95
5	8	37.69	28.30
6	10	36.80	28.98
7	10	38.10	30.00
8	12	38.80	32.05
9	12	42.10	34.77
10	15	57.63	51.14
11	18	61.51	58.64
12	20	60.68	60.68
13	21	65.91	67.50
14	23	70.13	75.34
15	24	77.46	85.23
16	25	78.65	88.64
17	24	77.46	85.23
18	22	78.00	81.82
19	19	76.81	75.00
20	17	76.91	71.59
21	16	75.70	68.80
22	15	72.99	64.77
23	18	71.52	68.18
24	18	69.37	66.14
25	15	62.26	55.91
26	14	59.02	51.14
27	15	54.94	48.75
28	14.5	52.52	47.73
29	13	50.78	42.95

Table 6.2 Normalized flux values with tap water in the presence of antiscalant (Figure 3.20)

Days	Temperature (^oC)	Flux (LMH)	Normalized flux (LMH)
1	21	146.46	143.00
2	18	92.73	97.27
3	16	109.09	120.03
4	21	109.77	107.18
5	19	110.46	113.13
6	22	109.09	104.00
7	18.5	113.18	117.31
8	20	110.45	110.45
9	19	109.77	112.43
10	17	107.72	115.73
11	15	105.00	118.33
12	14	100.91	116.47
13	16	102.95	113.28
14	15.5	105.00	116.92
15	15	104.32	117.56
16	14	103.64	119.62
17	16	107.73	118.53
18	17	109.09	117.20
19	18	110.45	115.86
20	16	109.09	120.03
21	15	105.68	119.10
22	16	107.73	118.53
23	17	109.09	117.20
24	14	109.77	115.15
25	16	108.41	120.72
26	15	107.05	116.38
27	15	109.09	118.61
28	14	110.45	120.09
29	13	109.09	119.00

Table 6.3 Normalized flux values with wastewater in the presence of antiscalant (Figure 3.21)

Days	Normalized flux (LMH)
1	84.00
2	68.00
3	7.43
4	8.45
5	24.00
6	23.00
7	21.00
8	19.50
9	19.00
10	19.50
11	19.00
12	19.00
13	18.70
14	18.50
15	18.00
16	17.85
17	16.00
18	15.00
19	13.00
20	12.00
21	8.00
22	92.00
23	20.00
24	18.00
25	18.50
26	17.00
27	16.00
28	12.00
29	10.00
30	8.00

Table 6.4 Normalized flux values with wastewater in the absence of antiscalant (Figure 3.30)

Days	Normalized flux (LMH)
1	84.00
2	19.09
3	17.05
4	15.68
5	12.27
6	10.64
7	8.86
8	8.86
9	8.59
10	8.05
11	45.95
12	14.32
13	13.23
14	12.95
15	11.59
16	10.50
17	9.55
18	9.68
19	8.73
20	8.45
21	8.18
22	27.27
23	16.36
24	14.18
25	12.27
26	12.41
27	12.00
28	11.05
29	10.24
30	9.75
31	8.65
32	8.18

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