Evaluation of Submerged Anaerobic Fertilizer Driven Osmotic Membrane Bioreactor (AnFDOMBR) for Textile Wastewater Treatment

Submitted by

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A thesis submitted in partial fulfillment of the requirements for the degree of

Master of Science

In

Environmental Engineering

Institute of Environmental Sciences and Engineering (IESE)

School of Civil and Environmental Engineering (SCEE)

National University of Sciences and Technology (NUST)

Islamabad, Pakistan

2019

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"Evaluation of Submerged Anaerobic Fertilizer Driven Osmotic Membrane Bioreactor (AnFDOMBR) for Textile Wastewater Treatment"

submitted by

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Annex A To NUST Letter
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ACKNOWLEDGEMENTS

First and foremost,I would like to thank **ALLAH ALMIGHTY,** the most gracious, the most beneficent, for giving me an opportunity to complete my MS degree and giving me courage and patience throughout the course of my study .This dissertation would not have been possible without the guidance and the help of several individuals who in one way or another contributed and extended their valuable assistance in planning, executing and finally, presentation, of this study.

I would like to express my sincere gratitude to my thesis supervisor **Prof. Dr. Sher Jamal Khan** (IESE-SCEE) for his continuous support throughout my study, for his patience, motivation, enthusiasm, and immense knowledge. His guidance helped me through my research and writing of this thesis. I could not have imagined having a better advisor and mentor for my MS thesis.

Many thanks also go to **Dr. Zeshan Sheikh** and **Dr. Deedar Nabi** (IESE-SCEE) for their guidance and support.

I am grateful to the staff of the Water & Wastewater Lab and Chemistry Lab, for the technical assistance. Last but not least, I would like to express my gratitude to all my family members, MBR group and friends for their support during my research.

Surraya Mehbub Malik

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List of Abbreviations

AMBR	Aerobic Membrane Bioreactor
AnFDOMBR	Anaerobic Fertilizer Driven Osmotic Membrane Bioreactor
AnMBR	Anaerobic Membrane Bioreactor
BOD	Biological Oxygen Demand
BSA	Bovine Serum Albumin
C:N:P	Carbon to Nitrogen to Phosphorus ratio
CER	Cation Exchange Resin
COD	Chemical Oxygen Demand
CST	Cappilary Suction Time
CSTR	Continous Stirred Tank Reactor
DS	Draw Solution
EPS	Extra Cellular Polymeric Substance
FDFOMBR	Fertilizer Driven Forward Osmosis Membrane Bioreactor
FDPAO	Fertilizer Driven Perssure Assisted Osmosis
FO	Forward Osmosis
FS	Feed Side
GDP	Gross Domestic Product
HRT	Hydraulic Rentention Time
IESE	Institute of Environmental Science & Engineering
LMH	Liters per square meter per hour
MAP	Mono Ammonium Phosphate
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids

NF	Nano Filtration
NH4-N	Ammonium Nitrogen
OMBR	Osmotic Membrane Bioreactor
PAI	Population Action International
PN	Protein
PRO	Pressure Retarded Osmosis
PSD	Particle Size Distribution
RO	Reverse Osmosis
SAnMBR	Submerged Anaerobic Membrane Bioreactor
SMP	Soluble Microbial Products
SRT	Solid Retention Time
SSAnMBR	Side Stream Anaerobic Membrane Bioreactor
TP	Total Phosphorus
UF	Ultra Filtartion
WHO	World Health Organization

Abstract

An innovative anaerobic fertilizer driven osmotic membrane bioreactor (AnFDOMBR) was designed to evaluate its feasibility for treating textile wastewater. The results showed that, flux through membrane drops severely with time for mono ammonium phosphate (MAP) under the combined effect of salts accumulation and viscous nature of sludge. Average flux for each cycle was around 1.5 LMH. Salinity in bioreactor was amplified due to accumulation of influent salts and reverse solute flux (RSF) which led to inhibition of microbial activity, more production of soluble microbial products (SMP) and extra cellular polymeric substance (EPS) degradation with time. In FO permeate, COD removal was about 91 ± 4% and dye removal was 91 ± 2% due to high retention of FO membrane. On the contrary, supernatant of anaerobic bioreactor exhibited COD removal was 57 ± 5% and dye removal was 43.7 ± 6% due to deterioration of microbes inside bioreactor. AnFDOMBR has a potential to produce biogas due to biodegradation of organic waste retained by FO membrane.

Keywords

Textile wastewater; Fertilizer driven forward osmosis; Anaerobic bioreactor; Salinity buildup; Color removal

Chapter 1

Introduction

1.1.Background

Scarcity of potable water is going to be a matter of existence and loss of life in the coming era if the issue is not addressed in a proper time frame. According to statistics, about 71 % of earth surface is covered with water out of which only 4 % is fresh water and only 0.5 % of this water is safe for humans. Safe drinkable water is an essential resource for human beings which is being frequently devalued, mishandled and mismanaged, leaving behind a lot of people suffering from water shortage. Currently, safe drinking water is not available to 2.1 billion people worldwide and globally 40 % individuals do not have access to fundamental sanitation (WHO, 2017). Demand for water continues to increases due to hasty urbanization, improved industrial development and better quality living standards. World's water demand estimated to increase by 55 % by 2050, largely due to increasing demand from industrial sector , hydal electricity production and household use as shown in **Figure** 1 (Marchal et al., 2011).

At the global level, textile industries within the industrial sector hold the key reputation in building the world economy and satisfying the individual demands. These textile industries are water demanding with an immense variety of Processes that involve use of considerable use of water.

Average sized Textile industry which have production of 8000 kg of textile every day, consumes 1600 cubic meter of water every day. Dyeing and Printing units consumed 16 % and 8% of this water,respectively (Kant, 2012).

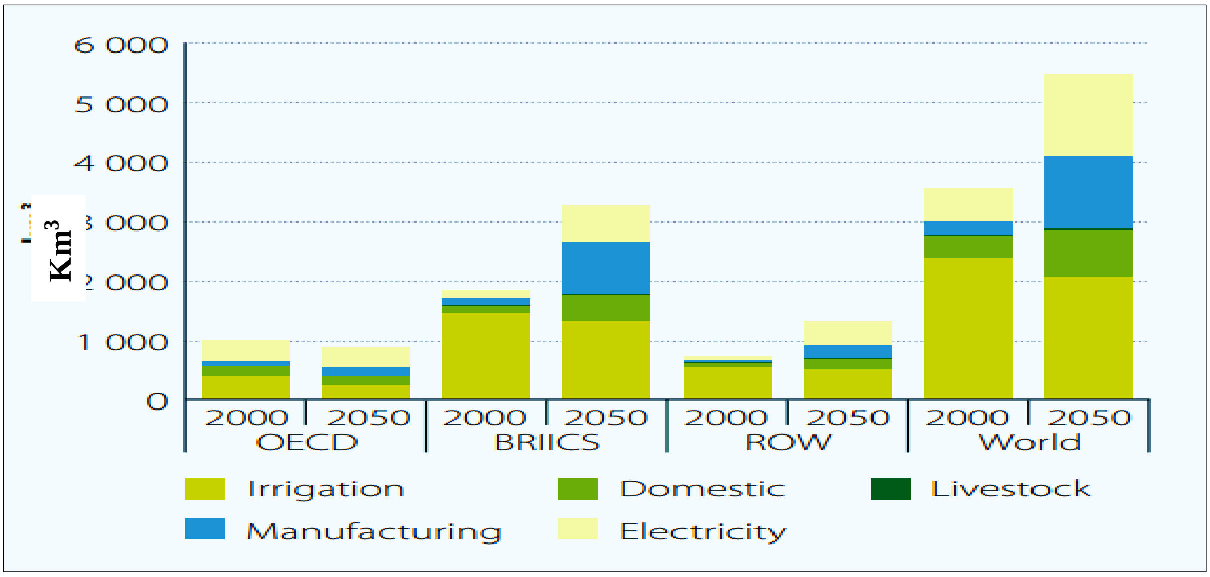

Figure 1 Increase in worldwide water demand by 2050 [1]

Dyeing and finishing units in textile industries produce about 17 to 20 % of industrial waste water, as stated by the World Bank assessments (Holkar et al., 2016). Among the 51% of sewage wastewater generated, textile industries play a major part in releasing contaminated water. According to an estimated calculation textile industries discharge 70 billion tons/year of waste water from dyeing sector only (Siddique et al., 2017).

Detergents, caustic, dyes, fixing agents, sizing agents, oils, latex and glues and many other in-organics are being used in textile industry for different purposes as shown in **Figure** 2 . Effluents coming from textile industries contain residual of all these chemicals and for that reason are not effectively handled in traditional wastewater treatment plants (Yukseler et al., 2017).

Global water consumption by each sector is shown in **Figure** 3; agriculture sector utilizes 70% of the available fresh water out of which 15 to 35 % of water is not used sustainably.

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[1] Note: Blue water demand was only taken into account in this graph and it does not reflect rain supported agriculture. OECD (Organization for Economic Co-operation and Development). BRIICS (Brazil, Russia, India, Indonesia, China, South Africa). ROW (rest of the world). Source: OECD Environmental Outlook to 2050

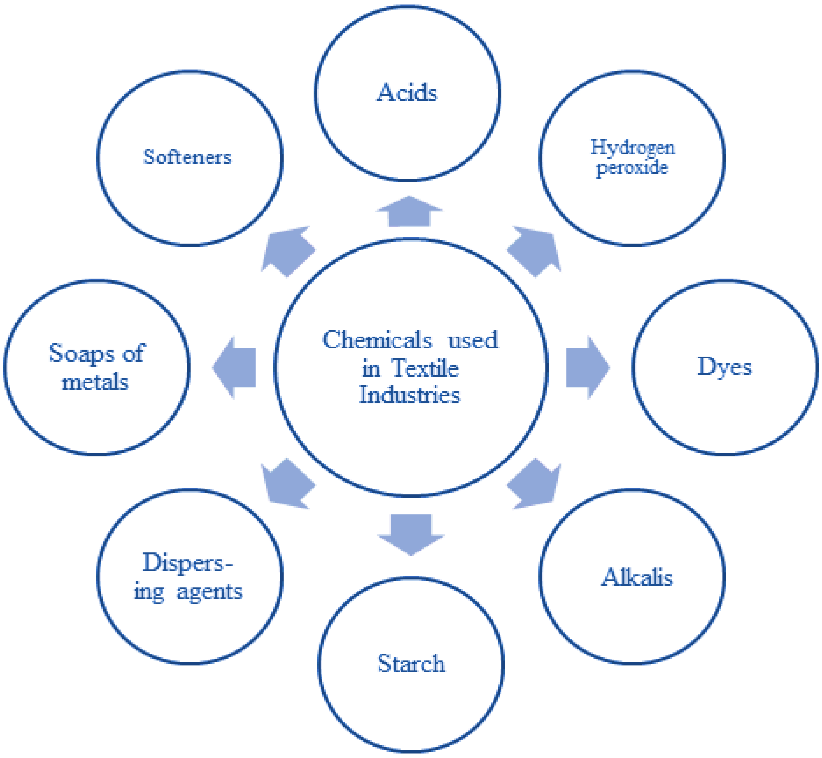

Figure 2 Hazardous Chemicals Used in Textile Industry (Katheresan et al., 2018)

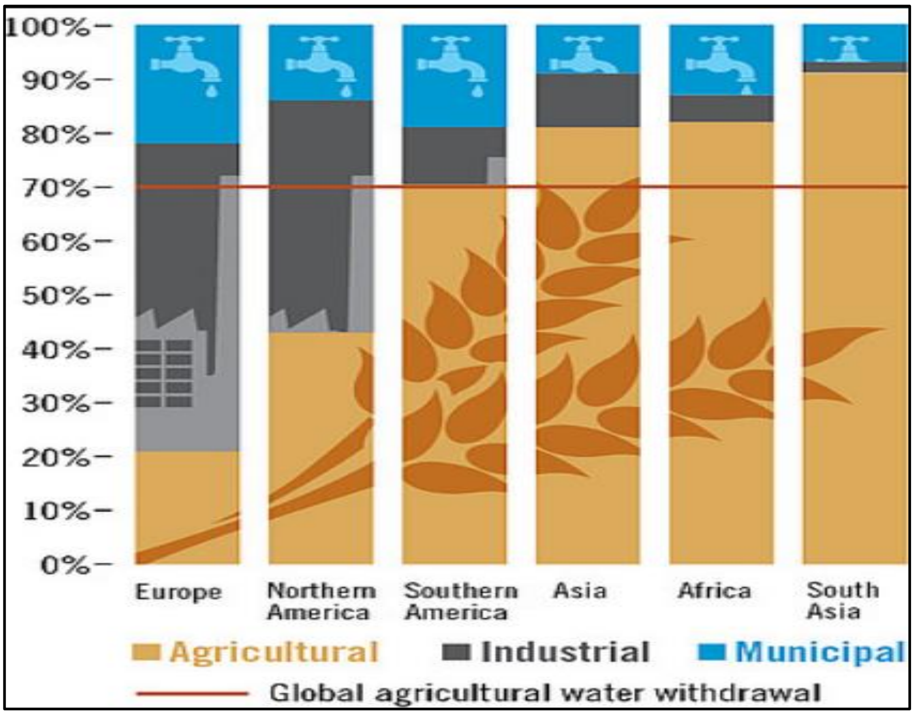

Figure 3 Global Water Footprints for Different Sectors (Source: Aquastat,2014)

Pakistan has turn into a water deficient country due to withdrawing ground and surface water reserves, natural famines and overuse of fresh water for domestic as well as industrial purposes instead of using it for agriculture purposes (Ensink et al., 2004).

Currently, annual water accessibility in Pakistan is about 935 m^3 per person it will further reduce upto 860 m^3 per person (EXPRESS, 2019) . **Table**-1 gives the examination of per person water-accessibility up to the year 2025 in some chosen nations of the World, including Pakistan (Kahlown and Majeed, 2003). In 1996-97 per capita water availability was 1.299×10^3 cubic meter (m^3) which decreased up to 1.1×10^3 m^3 per capita in 2006. It is predictable that water accessibility will be less than 700 m^3 per capita by 2025 in contrast to the international standard of 1000 m^3 per capita (Martin et al., 2006).

Table 1 Water Accessibility for Per Capita in Particular Countries (m^3) (Kahlown and Majeed, 2003)

Country	1995	1990	2025
China	4597	2427	1818
Mexico	11396	4226	2597
Philippines	13507	5173	3072
Iraq	18441	6029	2356
USA	14934	9913	7695
Pakistan	2490	1672	837

Current situation of Pakistan shows that the country is approaching conditions of persistent water scarcity. For now, the difference between requirement and availability of water has intensified to the point where it initiating restlessness between the federal unions. During recent years, prolonged drought in the country declined fresh water resources and it highlighted the significance of building of new water sources and water conservation strategies to use limited resources of water carefully (Kahlown and Majeed, 2003).

1.1.1. The textile industry in Pakistan

Textile industrial sector is classified among the oldest sector in Pakistan and even with its in-built strengths; it is ruining its competitiveness to other countries, especially in Southeast

Asia countries. The industry is in serious require of economic and hi-tech investments. However, in line with latest authorized facts, the Pakistan textile industry contributes over and above 60 percent to the country's overall trades that sums to round 5.2 billion US dollars. The textile industry provides round 46 percent of the overall yield generated in the country. In Asia, Pakistan is the eighth largest trader of fabric goods. Overall significant addition of textile industry in Gross Domestic Product is 8.5 % (GoP, 2008). Inside country it covers service to 38 % of the labor and that amounts to about 15 millions (Iqbal et al., 2010).

The accessibility of essential crude material for textile industry, cotton, has assumed a main job in the development of the industry. In production and consumption of cotton, Pakistan comes at fourth number in the world. In context of this Pakistan's textile industry is very dependent on agriculture sector of Pakistan. Both these sectors are major contributors to GDP of the country but both these sectors are major consumer of water resources as shown in **Figure** 4.

It is estimated that around 670 industries are working under textile sector. From these 670 industries, 300 industries are established in Karachi only, and the other 370 industries are working in various areas of Punjab (Aslam et al., 2004).

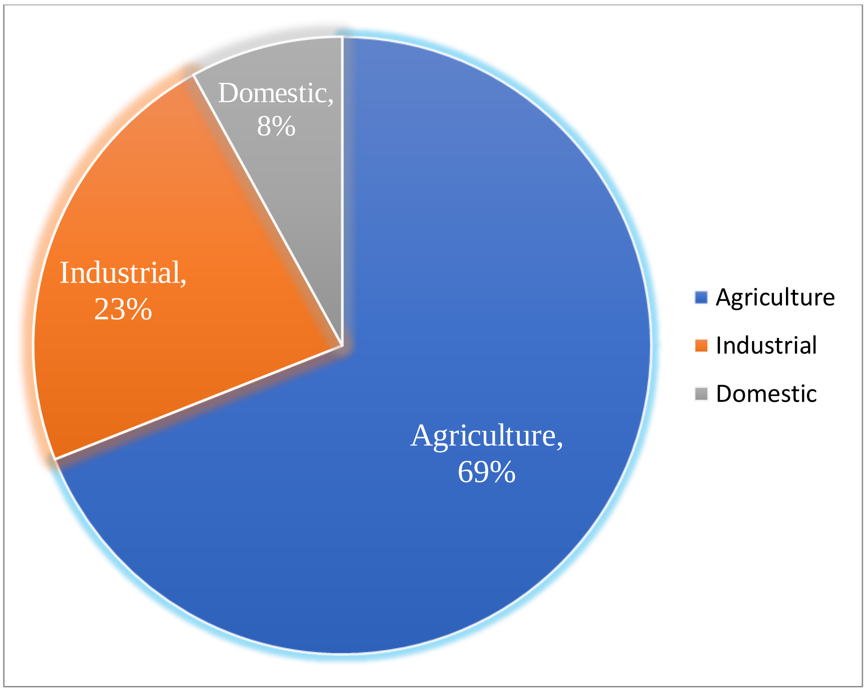

Figure 4 Sectorial Water Demand of Pakistan (Khoso et al., 2015)

Mostly, wastewater from these industries is released into surface water bodies either in sewerage system or into the surface water reservoirs. If substantial treatment of this wastewater is done and it meets the permitted quality standards then it is accessible for further consumers downstream as it can be used for ground water recharge or used for agricultural activities.

In agricultural country like Pakistan, the reuse of water is a consistent interest in light of the fact that plenty of water is needed for agricultural purposes. Thus, management of water resources is a difficult task for us as freshwater reservoirs are depleting quickly and water is becoming contaminated with industrial effluents (Siddique et al., 2017). The countries surroundings the Mediterranean sea, that are stressed out by water scarcity, have taken into account wastewater reuse as a feasible substitute for agriculture sector. Currently, researchers focused on sustainable wastewater treatment technologies are aiming to minimize stress on available water resources (Wang et al., 2017) .

Before reusing industrial wastewater for irrigation, its sufficient treatment is needed not merely to care for the humans and plants but also improve the worth of the yields cultivated through wastewater reuse. Variety of treatment options for feasibility of wastewater reuse for agricultural purposes were studied by several researchers (Alderson et al., 2015; Ferro et al., 2015).

For reclamation of wastewater, the use of membrane bioreactors has been increased globally due to stringent environmental regulations for effluent quality, reduced footprints , and high removal efficiencies by Membrane bioreactors. (Zhang et al., 2014) .Membrane bioreactor (MBR) is a hybrid technology that combines membrane filtration and conventional activated sludge process. For retention of suspended solids in bioreactor, microfiltration and ultrafiltration membranes are used in a conventional membrane bioreactor (Gu et al., 2015) . Advance treatment methods (for example reverse osmosis (RO), nano-filtration (NF) or advanced oxidation), needed are as a tertiary treatment for wastewater reuse, and because wastewater might contain contaminants such as dyes ,pharmaceutics, heavy metals, and trace organic contaminants that are not treated properly by conventional techniques (Kim et al., 2016).

For high strength wastewater especially of industrial wastewater, researchers have used anaerobic membrane bioreactor. In Anaerobic Membrane bioreactor (AnMBR), biological anaerobic treatment combines with membrane separation. Advantages of Anaerobic Membrane bioreactor involve less sludge production and less energy requirement, as there is no aeration required like aerobic membrane bioreactors and generating methane in process byproducts. Although membranes has high treatment efficiencies but some of the pollutants will remain in permeate certainly which limit the use of membranes (Gu et al., 2015).

Now, researchers are taking interest in use of high retention membranes in bioreactor to overcome this limitation. These membranes can also retain hydrolyzed organic contaminants, which are generally smaller. It increases their retention time in bioreactors, which ultimately enhanced their biodegradability and permeate quality.

A technology that draws attention to among the high retention MBRs is osmosis membrane bioreactor (OMBR) integrating Membrane Bioreactor (MBR) with natural forward osmosis (FO) process. Forward osmosis is a membrane filtration process where process water permeates from dilute solution to a concentrated draw solution by using natural osmotic process through a semi-permeable membrane (Cath et al., 2006).

The previous studies on osmotic membrane bioreactors (OMBR), main focus have been on aerobic wastewater treatment (Yap et al., 2012) , and few researchers have been worked on osmotic membrane bioreactor (OMBRs) under anaerobic condition for treating wastewater (Chen et al., 2014; Li et al., 2018; Tang and Ng, 2014; Wang et al., 2017).

The concept of hybrid Forward Osmosis and AnMBR was initially proposed by (Achilli et al., 2009; Chekli et al., 2016; Wang et al., 2016) in their relevant studies. Osmotic Membrane Bioreactor attributed to effective removal of contaminants, low fouling tendency and irreversible fouling but also has demerits such as reverse solute flux inhibits the biological treatment processes (Achilli et al., 2009).

Due to expensive recovery process of diluted draw solution, recently fertilizer-driven forward osmosis membrane bioreactors (FDFOMBR) have gained attention since diluted draw can be used for irrigation directly (Phuntsho et al., 2011; Phuntsho et al., 2012). Instead of different organic and inorganic draw solutes, FDFO process utilized fertilizers as an osmotic agent and it gets diluted during operation. Phuntsho et al initially research on single fertilizer, which doesn’t meet nutrients criteria, after that they introduced new concept of blended fertilizers as draw solution to meet crops nutrients criteria (Phuntsho et al., 2012). The extensive dilution of diluted draw solution still needed to meet nutrients ranges for fertigation. To deal with this issue Nano-filtration is integrated with this process as tertiary treatment to meet nutrients criteria in view of the fact it has 80-90% rejection rates these rates are lower than Reverse Osmosis rejection rates (Phuntsho et al., 2013). But Nano filtration is energy extensive treatment and must oppress the osmotic gradient of diluted draw solution. For improving final dilution of fertilizer draw solution, Fertilizer-drawn Pressure Assisted Osmosis (FDPAO) was recently developed. (Kim et al., 2016). In this study, we proposed novel Anaerobic Forward Osmosis Membrane Bioreactor (AnFDOMBR) for textile wastewater treatment.

1.2.Problem Statement

Textile wastewater reuse is only possible after proper treatment due to its severe contamination. Reuse criteria and concentration of pollutants defined the type of treatment needed. Conventional treatment processes (e.g. adsorption, photo-catalytic oxidation, advance oxidation (electro-chemical oxidation), and microbiological or enzymatic breakdown) are not practically successful in treating textile wastewater. Different combinations of these processes used to meet stringent effluents standards and textile sector standards. To focus on this thought-provoking problem, Forward Osmotic Membrane Bioreactor technology is rising as a favorable and efficient approach. This study based on the design and construction of Anaerobic Forward Osmosis Membrane Bioreactor (AnFDOMBR) and evaluation of different parameters to check its feasibility for treatment of textile wastewater.

1.3.Objectives of Study

- Establishment of Anaerobic Fertilizer Driven Osmotic Membrane Bioreactor (AnFDOMBR).
- Evaluation of AnFDOMBR in term of treatment performance and membrane fouling.
- Influence of salinity buildup on sludge characteristics in AnFDOMBR.

1.4.Scope of Study

Operation of FD-OMBR was coupled with Anaerobic process to treat the synthetic textile wastewater and to check the stability of:

- Water flux
- Conductivity of Mix Liquor

Optimized operation with molar concentration of 1M for draw solution Mono ammonium Phosphate (MAP).The effects of salt accumulation on:

- Membrane biofouling in term of SMPs and EPSs
- SludgeCharacteristics in term of PSD and cappillary suction time.

The contaminant removal efficiency, product water quality and water productivity of the whole system with the AnFDOMBR operating with MAP DS will be studied.

Chapter 2

Literature Review

2.1. Process involved in Textile Industry

Several processes are involved in textile industries to transform raw fibers into final product. Some of the processes involved in wet textile industries are covered up in **Figure** 5 (Vigo, 2013).

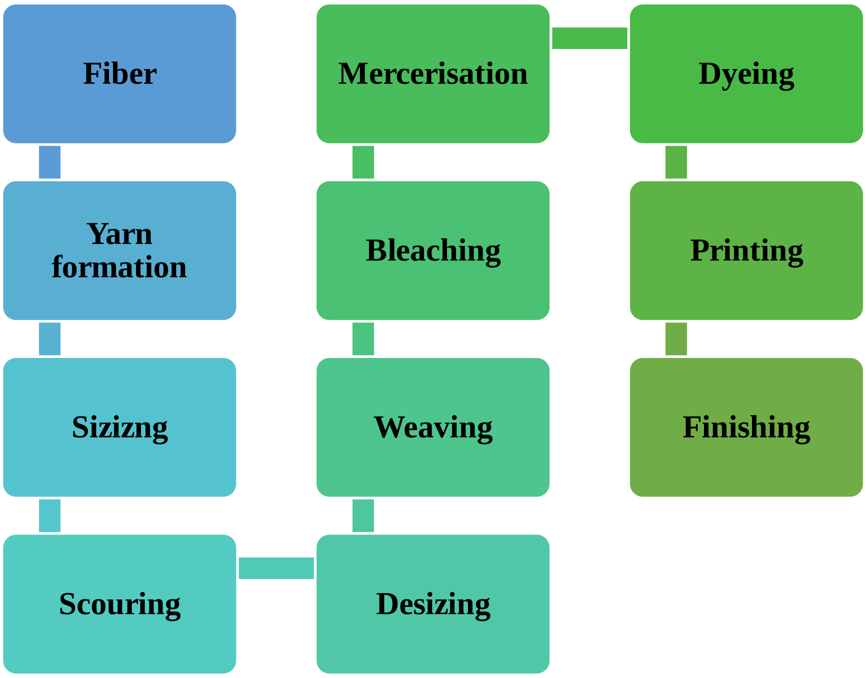

Figure 5 Flow diagram of Wet Processing of fiber Adapted from (Vigo, 2013)

2.2. Treatment processes for textile wastewater

Wastewater from textile industriescontain dyes, metals, detergents caustic soda etc. Usually, these effluents are treated biologically in activated sludge plant to meet discharge standards but not in order to reuse it.Wastewater coming out from textile industries has a high amount of color ,high chemical oxygen demand (COD) and total dissolved solids (TDS) concentration. Biological processes do not easily treat reactive dyes coming from cotton dyeing industries. These color compounds are toxic for aquatic species leading to environmental imbalance. Rivers severe as a drinking water source, so before releasing these effluents into rivers, various treatment processes (**Figure** 6) like physical, chemical, biological and hybrid treatment process are used to treat it effectively and economically.(Praveen Kumar and Bhat Sumangala, 2012) These technologies are discussed one by one in following section.

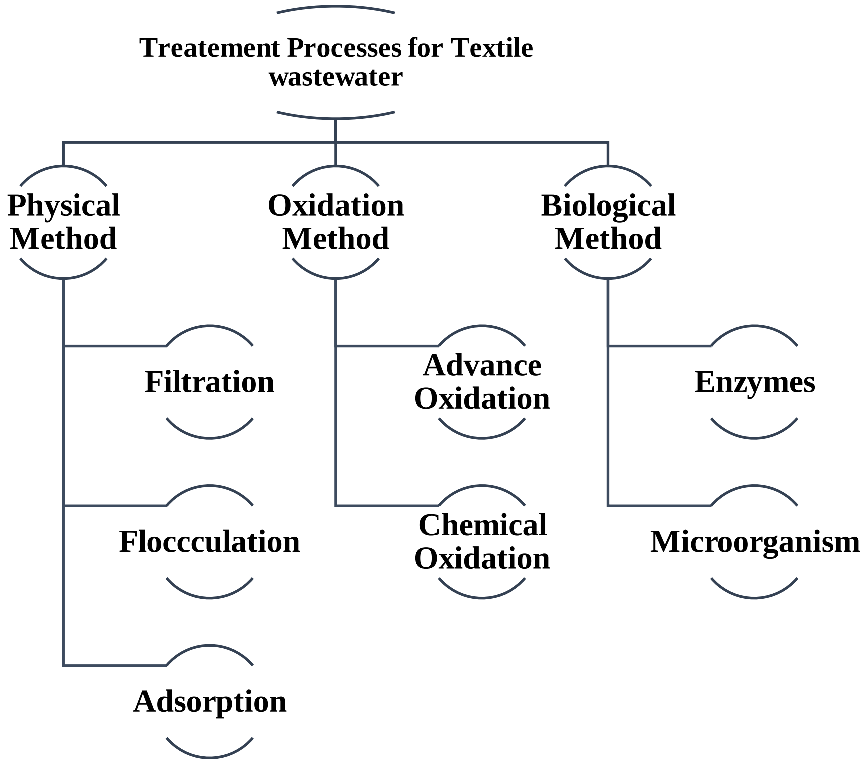

Figure 6 Different options for textile wastewater treatment Adapted from (Holkar et al., 2016)

2.2.1. Biological Processes

The biological process treats only organic material in textile wastewater. Efficiency of biological treatment depends on organic loading rate, microbial population, temperature and oxygen concentration of system. Biological processes, classified into three categories based on oxygen requirement, which are as follow: aerobic, anaerobic, and facultative. The hybrid system of anaerobic and aerobic treatment is generally applied in which utilizes anaerobic treatment to reduce chemical oxygen demand of textile wastewater, subsequently the aerobic treatment is used to polish effluent coming from anaerobic treatment (Wang et al., 2011).

The biological processes for the entire degradation of textile effluent have advantages for example: (i) cost effective, (ii) environmentally friendly, (iii) minimum sludge generation, (iv) minimum requirement of water contrasted to physical or oxidation processes (Hayat et al., 2015).

2.2.2. Oxidation Methods

2.2.2.1. Advanced oxidation processes

In this process, sufficient amount of •OH (hydroxyl radicals) are generated. These radicals are used as strong oxidant. This oxidant shows rapid oxidation reactions as compared to conventional oxidizing agents such as H_2O_2 and $KMnO_4$. When •OH radical reacts with dyes it has high reaction rate constant (Asghar et al., 2015). Complex organic and in organic compounds present in textile effluent are oxidized by hydroxyl radicals. Processes as photo-catalytic oxidation and reaction between Ferric ions with hydrogen peroxide (Fenton chemistry) are also include in AOP processes. Combined flocculation of dyes and reagent molecules results in generation of iron sludge which is the basic negative aspect of Fenton process (Holkar et al., 2016).

2.2.2.2. Chemical Oxidation

This method use oxidants like Ozone and Hydrogen peroxide.O_3 and H_2O_2 forms strong •OH radical at basic pH. Due to strong oxidizing power of these radicals, they can efficiently destroy the conjugated bonds of dye radicals among other functional groups. Ozone is used in its gaseous state in ozonation process therefore this process does not increase volume of wastewater and does not result into production of sludge. However, main demerit of ozonation process is production of toxic byproducts and cost of process (Miralles-Cuevas et al., 2017).

2.2.3. Physical treatment

2.2.3.1. Coagulation–flocculation

Disperse dyes coming out from textile industry can be removed by coagulation-flocculation processes. These processes have minimal discoloring efficiency for effluents, which contains reactive and vat dyes. Minimal discoloring efficiency and excessive sludge production are demerits of these processes which limit their application (Liang et al., 2014)

2.2.3.2. Adsorption

Adsorption techniques have attained considerable interest due to remarkable color removal competence for wastewater containing different types of dyes. During an adsorbent selection for dye removal, the basic properties which must be taken into account are high affinity, regeneration ability of compound and adsorbent (Jadhav and Srivastava, 2013). Activated carbon adsorbs variety of dyes. Nevertheless, some factors that limit its use are its high cost and regeneration complexity. Recently for cost effective practical use of adsorption method, number of researchers used a cheap price adsorbent material such as peat, bentonite clay, fly ash, and polymeric resins. A few researches also tried numerous organic resources like wheat residue, treated ginger waste, groundnut shell, charcoal, date stones, and potato plant waste for the color removal of textile wastewater. These adsorbents have several challenges such as adsorbent regeneration, dumping, excess sludge production and high cost of adsorbent due to which application of this technology get restricted (Holkar et al., 2016).

2.2.3.3. Filtration

For water recovery and reuse, filtration methods like Ultrafiltration (UF), Nanofiltration (NF) and Reverse osmosis (RO) techniques can be implemented on textile wastewater. The condition which is important to consider is composition and temperature of textile wastewater before selecting filter and its permeability. By using membrane treatment, unfixed reactive dyes and supplementary compounds can be recovered. Application of membrane filtration brings capability of recovering unfixed dyes and supplementary compounds used in dyeing which simultaneously reduced the biological oxygen demand (BOD), Chemical Oxygen Demand and dyes from textile wastewater. But, membranes also have a significant production of wastes containing indigo dye which is not soluble in water and further treatment is required for starch (Koyuncu and Güney, 2013).

2.2.4. Membrane Bioreactors

Membrane technologies have gradually attained consideration to the conventional treatment processes of wastewater. The implementation of this technology not only facilitates effective removal efficiencies, but also permits water reuse and reuse of valued components (Fersi et al., 2005). In membrane bioreactor, biological process used for biological treatment and membranes are used for separation of solids from liquids. Biological process can be aerobic or anaerobic. Different features of aerobic and anaerobic MBRs are indexed in **Table** 2.

Table 2 Features of aerobic and anaerobic MBRs.(Jegatheesan et al., 2016)

Overall feature	Aerobic MBR	Anaerobic MBR
Permeate quality	Excellent	High
Organics removal	High	High
Footprint	Small	Small
Organic loading rate	High to moderate	High
Biomass retention	Total retention	Total retention
Sludge production	High to moderate	Low
Nutrient requirement	High	Low
Sensitivity to temperature	Low	Low to moderate
Energy requirement	High	Low
Bioenergy recovery	No	Yes

Numerous membrane separation processes have been combined with anaerobic treatment targeting increase in biomass retention in bioreactor and to enhance permeate quality. Anaerobic membrane bioreactor is hybrid process with combination of anaerobic process with low-pressure membranes for example Microfiltration and Ultra Filtration and that is a remarkable approach. However, these low pressure membranes used conventionally in anaerobic MBRs does not retain soluble organic carbon. Therefore, these MBRs are not effectively used for recovering energy and not efficient for energy recovery and cannot generates a high quality permeate .Additional research in An-MBR process has given rise to unique integration of anaerobic treatment with high retention membrane technologies such as Membrane Distillation (MD), Nano-filtration (NF), Reverse Osmosis (RO), and Forward Osmosis (FO). Between all these high retention membranes, Forward Osmosis is emerging as the most favorable for combination with anaerobic process due to its high removal efficiency and irreversible fouling (Ansari et al., 2017).

2.2.4.1.An-MBR configuration:

In two different configuration membrane is attached to bioreactor (a) Cross flow AnMBR (b) Submerged AnMBR (SAnMBR) (c) Side stream AnMBR (SSAnMBR) as shown in **Figure** 7 below.

a) Cross Flow AnMBR

In this case membrane module is placed outside the anaerobic bioreactor , supernatant of anaerobic digester and permeate flows in cross flow mode. This configuration is simple for membrane replacement and easy to clean.

b) Submerged AnMBR

In this configuration membrane is immersed inside the anaerobic bioreactor and no is energy required for pumping. To minimize fouling biogas is recirculated which cause shear on membrane surface.

c) Side stream AnMBR

In this configuration membrane module is placed outside the anaerobic, it increases pumping cost but it is less prone to fouling. (Watanabea et al., 2014).

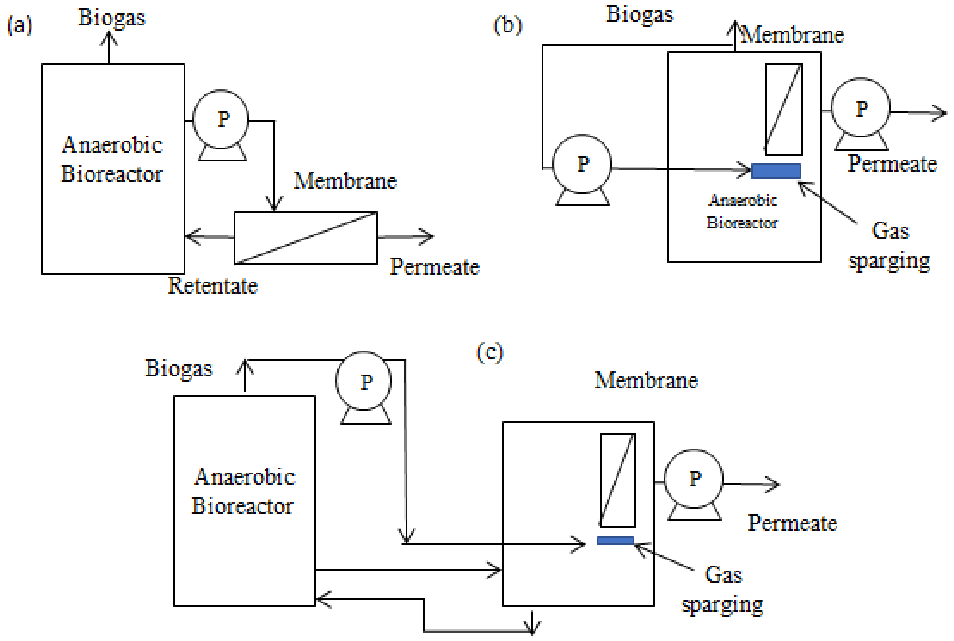

Figure 7 Configuration of AnMBR (a) Cross Flow AnMBR (b) Submerged AnMBR (c) Side Stream AnMBR

2.3. Forward Osmosis Membrane Bioreactor

2.3.1. Osmotic process

The transport of water across a semipermeable membrane from a region of lower solute concentration towards higher solute concentration is called osmosis. The solute concentration in both sides of membrane is the driving force for this process as it permits water to pass out of membrane, but retains the majority of solute molecules and ions. Osmotic pressure (π) is the pressure needed to stop the flow of solvent across highly selective membrane.

2.3.2. Classification of Osmotic Processes

Forward osmosis process uses the difference in osmotic pressure ($\Delta\pi$) through the membrane, instead of difference in hydraulic pressure as used in reverse osmosis, as the forcing drive for water transportation through the membrane. This process results in feed concentration and dilution of draw solution. Pressure Retarded Osmosis (PRO) can be considered as in-between process between Forward Osmosis and Reverse Osmosis, where pressure is applied in the opposite direction of the osmotic pressure gradient (like in Reverse Osmosis).But , the resultant water flux is also in the direction of the concentrated draw solution (just like happens in Forward Osmosis). The universal equation used for explaining water transportation in Forward Osmosis, Reverse Osmosis and Pressure Retarded Osmosis is:

$$Jw = A (\sigma\Delta\pi - \Delta P) \qquad (1)$$

Where

Jw = Water Flux

A = Water Permeability Constant of the membrane

σ = Reflection Coefficient

P = Applied pressure.

For Forward Osmosis process, applied pressure is zero; and for Reverse Osmosis, change in applied pressure is greater than change in osmotic pressure (i.e. $\Delta P>\Delta\pi$). The flux directions of the permeating water in FO, RO and PRO are demonstrated in **Figure** 8a.

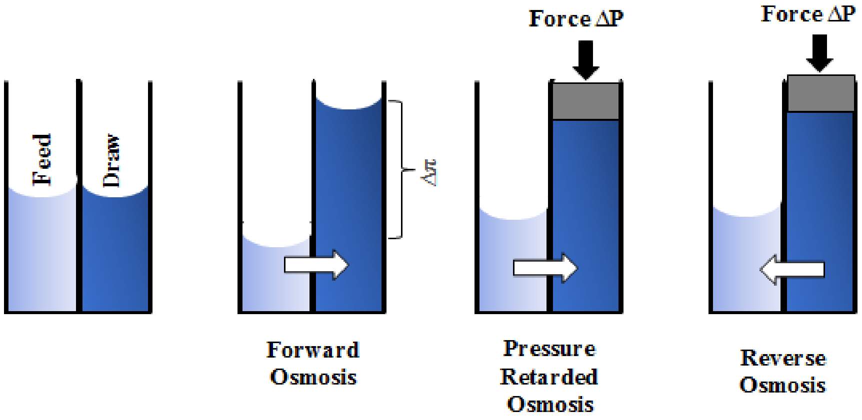

Figure 8a Direction of Flux in Osmotic Processes adapted from (Lee et al., 1981)

For all these osmotic processes , driving forces and flux directions were categorized in the early 1980s (Lee et al., 1981). The Forward Osmosis point, Reverse Osmosis and Pressure Retarded Osmosis region, as well as the reverse solute flux, are demonstrated in **Figure** 8b. Forward Osmosis takes place when the applied pressure difference is zero; Reverse Osmosis region is where the applied pressure difference is greater than the osmotic pressure difference and the region of Pressure Retarded Osmosis is where the osmotic pressure is greater than applied or hydraulic pressure.

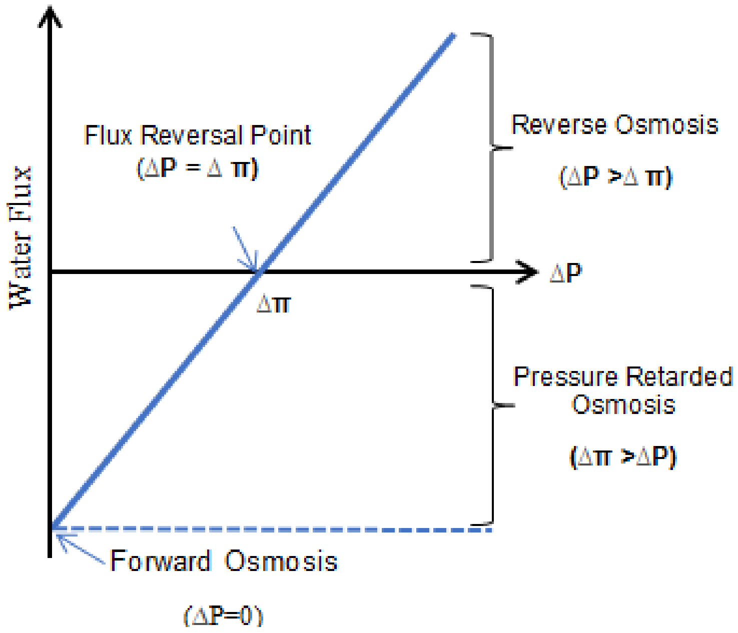

Figure 8b Magnitude of Water flux as Function of Applied Pressure for FO, RO and PRO Processes (Lee et al., 1981).

2.3.3. Advantages of forward osmosis

Potential advantages of Forward Osmosis over other technologies enhance its use for treating complex waters. In Forward osmosis process no hydraulic pressure is required since it is based on natural osmotic pressure, due to this neither energy contribution is required nor high strength material is required. Due to mean pore radius of 0.25- 0.37 nm of Forward osmosis membrane, it rejects all substances, microorganisms and emerging substances and it reveals efficient salt rejections and contrasting from conventional treatment technologies, effectively removes total dissolved solids . It can be used for dewatering or concentration of anaerobic digester sludge, this process is uncomplicated, eco-friendly and higher in effectiveness than conventional dewatering technologies. Forward Osmosis has shown outstanding process in terms of robustness, consistency and permeates water quality of highly polluted waters. (Lutchmiah et al., 2014).

FO process has exhibited manageability and suitability due to:

a) Scalability of the membrane system

b) Decreased fouling tendency (Achilli et al., 2009) and simple cleaning as compared to RO (Lutchmiah et al., 2014).

2.3.4. Osmotic Membrane Bioreactor (OMBR)

When in membrane bioreactor forward osmosis membrane is utilized then this MBR termed as Osmotic Membrane Bioreactor. In an Osmotic Membrane Bioreactor, wastewater is fed into bioreactor. Draw solution (DS) have lower water chemical potential due to high concentration of solute, water molecules diffuse from bioreactor to draw solution across semipermeable membrane through natural osmosis process .The FO membrane acts as an obstacle to solute transport and provide high retention of the pollutants in the wastewater side. The diluted draw solution is directed to a regeneration process (e.g., distillation or reverse osmosis) which generates best quality product water by reconcentrating the draw solution. Therefore, in most wastewater treatment applications, Forward Osmosis is not the final process but rather a high-level pretreatment step before a final reconcentration process (Achilli et al., 2009).

Due to expensive recovery process of diluted DS, recently fertilizer-driven forward osmosis membrane bioreactors (FDFOMBR) has gained attention as diluted draw that can be used for irrigation directly (Phuntsho et al., 2011; Phuntsho et al., 2012). Instead of different organic and inorganic draw solutes, FDFO process utilizes fertilizers as an osmotic agent and gets diluted during the operation. Moreover, for treatment of high strength wastewater generally anaerobic membrane bioreactor (AnMBR) has been used (Gu et al., 2015). Advantages of AnMBR involve less sludge production and less energy requirement, as there is no aeration required like in aerobic membrane bioreactors (AMBR) (Lin et al., 2013). The concept of hybrid OMBR and AnMBR has been investigated by several researchers for overcoming issues related to pressure driven membrane processes (Ansari et al., 2015; Chen et al., 2014; Gu et al., 2015; Kim et al., 2016; Wang et al., 2017). Previous studies on OMBR merely focused on domestic wastewater treatment (Chen et al., 2014; Li et al., 2018; Tang and Ng, 2014; Wang et al., 2017). However anaerobic fertilizer driven osmotic membrane bioreactor (AnFDOMBR) has not been investigated for the treatment of textile wastewater.

In this study, feasilbility of AnFDOMBR was evaluated for treating high strength textile wastewater and to check whether it is possible or not to attain required fertilizer DS dilution with textile feed.

Chapter 3

Material and Methods

3.1. Experimental Setup

The schematic diagram of laboratory scale AnFDOMBR is shown in **Figure** 9 and pictorial view of lab scale setup is shown in **Figure** 10. A bench scale anaerobic fertilizer driven osmotic membrane bioreactor having an effective volume of 3.6 liters was used in study. A magnetic stirrer (PC-420D, Corning, USA) was set up at the base of bio-tank (600 rpm) to develop the shear force on the membrane surface to reduce membrane fouling. Mono ammonium phosphate (MAP) as draw solution (DS) was continuously circulated during the operation with a flow rate of 500 ml/min.

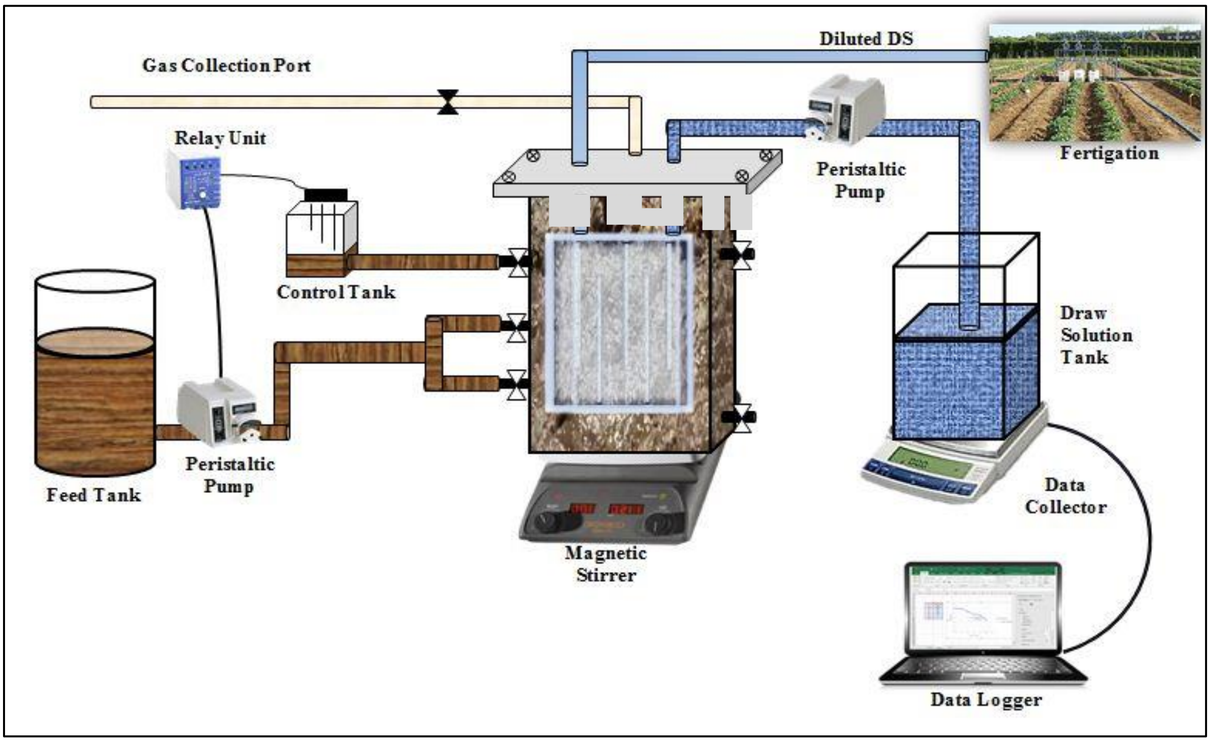

Figure 9 Schematic diagram of bench scale Anaerobic Fertilizer Driven Osmotic Membrane Bioreactor

Draw solution tank was set on a top loading balance (UX6200H, Shimadzu, Japan) linked with computer to ascertain the flux of FO membrane with time. A peristaltic pump (BT 300-2J, Longer, China) connected with relay unit (LLC- 101-X, Micro Max, Iran) was fitted to serve the bioreactor. Conductivity of draw solution and bioreactor was measured daily, using conductivity meter (CON 110, OAKTON, Australia) by taking samples from both tanks.

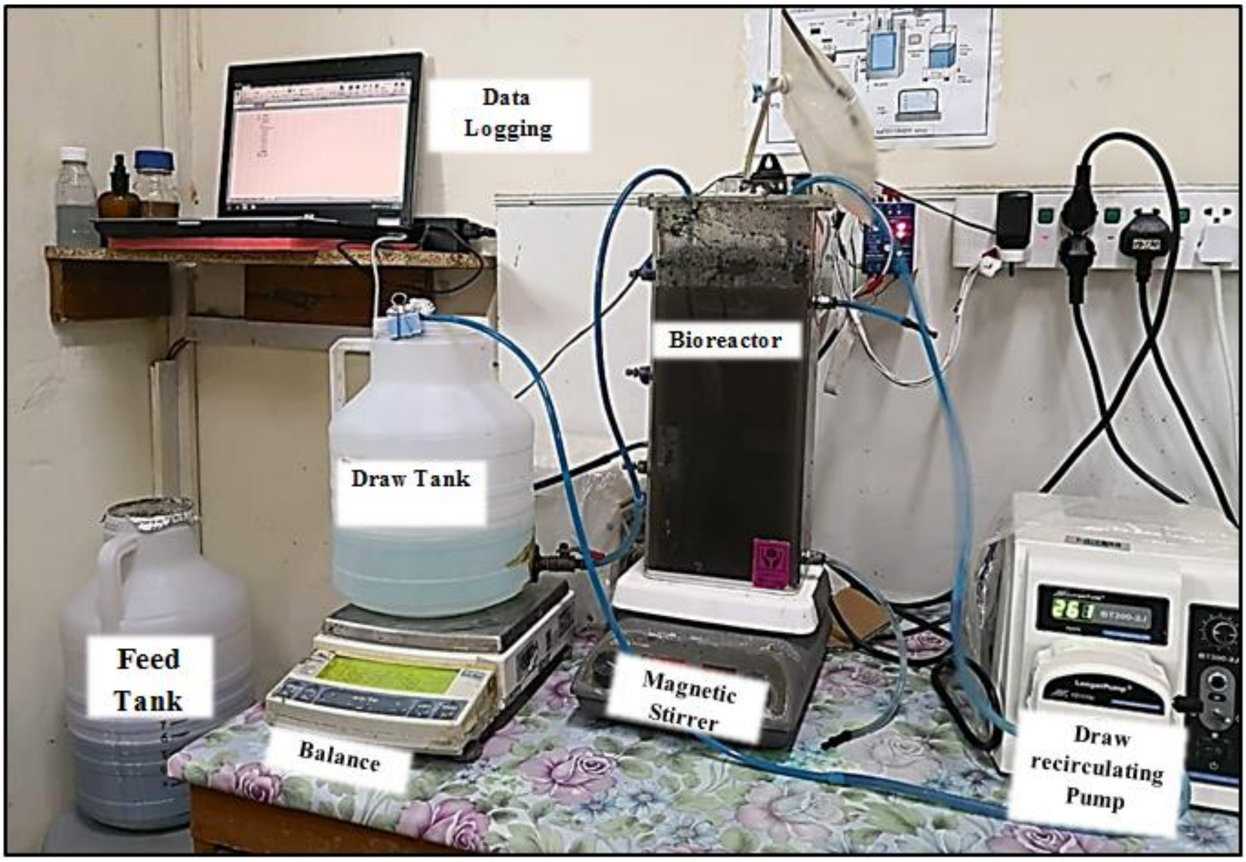

Figure 10 Pictorial View of Lab scale AnFDOMBR Setup

During operational period, the AnFDOMBR was operated at ambient temperature of 27 ± 4 °C. Hydraulic retention time (HRT) of the system was 16 hours initially and it increased due to the flux drop of forward osmosis membrane. Anaerobic seed sludge was taken from anaerobic continuously stirred tank reactor (CSTR) at Water & WAstewater laboratory of Institute of Environmental Sciences & Engineering (IESE-SCEE),NUST. Sludge was acclimatized for two months before starting AnFDOMBR. During operation of AnFDOMBR sludge retention time (SRT) of the system was 60 days.

3.2. Membrane and Membrane Module

FO module with effective membrane area of 400 cm^2 was submerged in anaerobic bioreactor. Membrane was attached on both sides of module to make closed channel for DS. To enhance the contact time of DS within the module, 5 baffles were provided each having a thickness of 0.6 cm. The cellulose triacetate (CTA) forward osmosis membrane (Hydration Technologies Inc.,USA) had an orientation of active layer facing the feed side; as this orientation is less susceptible to fouling and scaling (Tang et al., 2010). The details of membrane are described in **Table** 3.

Table 3 Details of CTA-FO membrane (HTI)

Name of membrane	Pure water permeability coefficient (A – LMH/bar)	Solute permeability coefficient (B – LMH)	Structural parameter of support layer (S - µm)
Cellulose triacetate (CTA – HTI)	1.17	0.98	473

3.3. Synthetic textile wastewater & draw solution

The Synthetic textile wastewater was used as the feed of AnFDOMBR with C: N: P ratio of 100:10:1 and recipe of synthetic wastewater have been summarized in **Table** 4. The pH and conductivity of synthetic wastewater were 7 ± 0.2 and 3.4 ± 0.4 mS/cm, respectively.

Table 4 Synthetic Feed Composition

Chemicals	**Chemical formula**	**Concentration (mg/l)**
Glucose	$C_6H_{12}O_6$	3000
Sodium Bicarbonate	$NaHCO_3$	500
Ammonium Chloride	NH_4Cl	1146
Potassium Dihydrogen Phosphate	KH_2PO_4	143.1
Calcium Chloride	$CaCl_2$	29.19
Magnesium Sulphate	$MgSO_4 \cdot 7H_2O$	9.73
Iron Chloride	$FeCl_3$	1

Chemicals	Chemical formula	Concentration (mg/l)
Cobalt Chloride	$CoCl_2$	0.1
Zinc Chloride	$ZnCl_2$	0.1
Methylene Blue[2]	$C_{16}H_{18}N_3SCl \cdot 3H_2O$	5
Cibacron Blue P-3RGR[3]	$C_{32}H_{23}C_{l}N_7Na_3O_{11}S_3$	5
Cibracon Yellow C-R-01[4]	$C_{25}H_{15}C_{l3}N_9Na_3O_{10}S_3$	5

Kim et al. (2016) reported that mono ammonium phosphate (MAP) had less salinity buildup due to less reverse solute flux and relatively higher water flux than other fertilizers. Also MAP has less inhibition to microbial activity in anaerobic treatment. For these reasons MAP was selected as a draw solution to investigate its performance in the AnFDOMBR at 1M concentration.

3.4. Analytical methods

Analyses were performed in accordance with Standard Methods. For treatment analysis Chemical Oxygen Demand (COD) and for analysis of dilution of nutrients Total phosphorus (TP) and Ammonium-N (NH_4^+-N) were analyzed. Effect of salinity buildup on sludge characteristic was monitored by analyzing mix liquor suspended solids (MLSS), mix liquor volatile suspended solids (MLVSS), soluble microbial products (SMP), extra cellular polymeric substances (EPS), particle size distribution (PSD) and capillary suction time (CST) (APHA, 2012).

Particle size analyzer (LA-300, HORIBA, Japan) was used to measure mean particle size of sludge samples. To analyze the filterability and condition of sludge, capillary suction time (CST) test was performed by using CST apparatus (304B-CST, Triton, Canada). After

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[2] Bhattacharyya, K.G., Sharma, A., 2005. Kinetics and thermodynamics of methylene blue adsorption on neem (Azadirachta indica) leaf powder. Dyes and pigments 65, 51-59.

[3] Lemlikchi, W., Sharrock, P., Mecherri, M., Fiallo, M., Nzihou, A., 2012. Treatment of Textile Waste Waters by Hydroxyapatite Co-Precipitation with Adsorbent Regeneration and Reuse. Waste and biomass valorization 3, 75-79.

[4] Wang, G.-W., Zhuang, L.-H., Sun, J., Zheng, C.-L., 2014. Salt-free dyeing of ramie fabric with an amino-terminated hyperbranched polymer. Cellulose 21, 3725-3736.

placing sample in funnel on standard filter paper through suction water was filtered from sludge and sludge characteristics defines the rate at which water permeates through filter. Water front take time to travel between these two electrodes this time taken is termed as capillary suction time.

3.5. Extra polymeric substance (EPS) extraction and quantification:

For extraction of extracellular polymeric substances (EPS) from the sludge, cation exchange resin (CER) method was used (Frølund et al., 1996). Carbohydrates and proteins concentrations were used to measure SMP and EPS. Calibration curve for protein (PN) was established by utilizing different concentrations of bovine serum albumin (BSA). The concentration of protein (PN) was measured by following Lowry method in which Folin-Ciocalteu’s phenol reagent was used to prepare solution and absorption was measured at the wavelength of 750 nm by using spectrophotometer (T60-UV/VIS, PG Instrument, Britain) (Lowry et al., 1951). Dubois method (Phenol-Sulfuric Acid) was used to quantify concentration of carbohydrates, absorption of solution was measured at wavelength of 470 nm (Dubois et al., 1956). Analytical grade glucose was used to develop calibration curves of carbohydrates.

3.6. Cleaning Protocol

Membrane after each cycle of 8 days was cleaned with tap water manually and then hydraulic flushing of membrane was undertaken by changing feed and DS with deionized (DI) water and circulating it for 30 minutes. Backwashing of membrane was also conducted osmotically in which the textile feed solution was switched with 1M sodium chloride and fertilizer DS with DI water to establish a opposite water flux (Kim et al., 2017b). After cleaning membrane, the textile feed and fertilizer draw solute were turn back to the setup.

Chapter 4

Results and Discussion

4. Flux performance and salinity buildup

System was operated by using 1 M MAP as draw solution and results are shown in **Figure** . **11**. In all six test cycles, flux declined with time throughout the operation. After completion of each cycle, sticky gel layer was observed at the surface of membrane on feed side (active layer) as illustrated in **Figure** . 12. Reason behind the formation of this fouling layer was enriched production of soluble microbial products (SMP), due to an enhanced salts accumulation on feed solution (FS) side (Zhang et al., 2014).

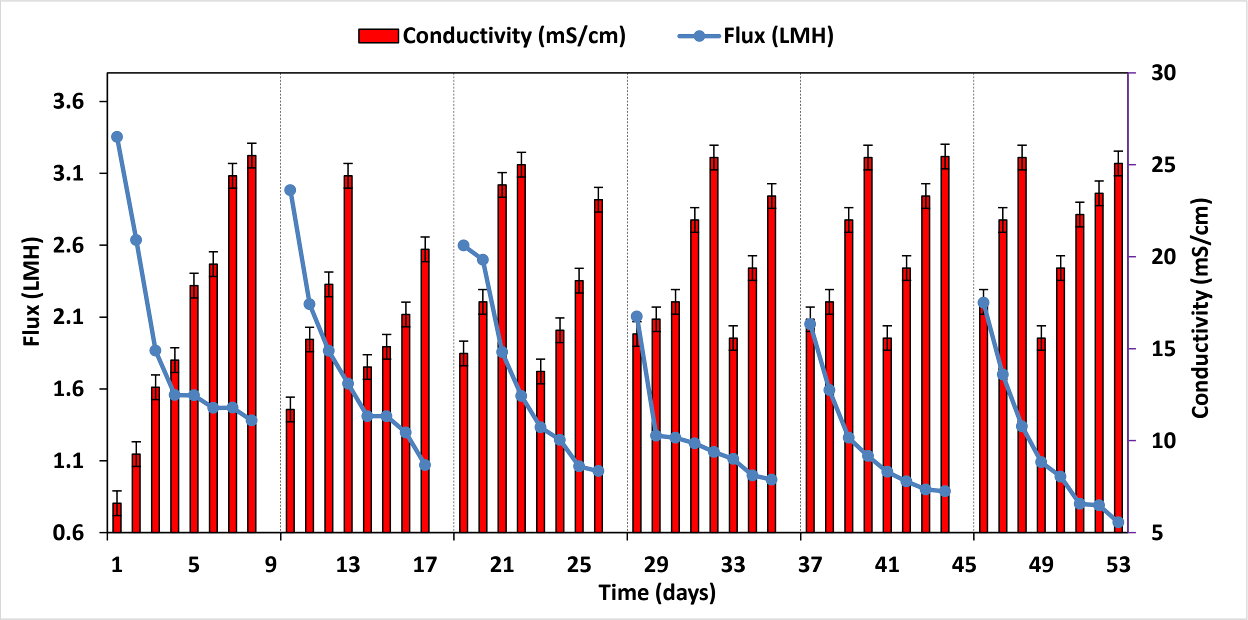
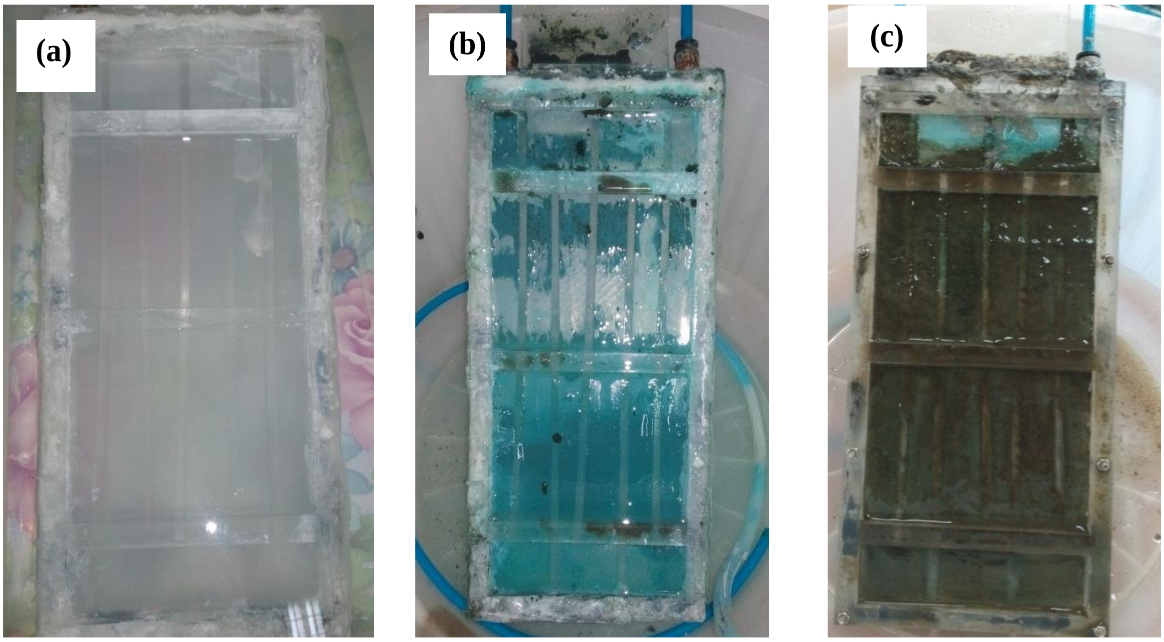


Figure 11: Flux performance and conductivity vs. time using 1M MAP concentration

Initial flux through membrane was not restored after osmotic backwashing. Pictures of the physically clean membrane and osmotically clean membrane after first cycle and last cycle on instant removal from the bioreactor were shown in **Figure** . 13 & 14._Perhaps, the initial flux in the first cycle was 3.4 LMH, which reduced to 1.4 LMH over 8 days of continuous operation. Though, the initial flux in the sixth cycle was only 2.2 LMH, which reduced to only 0.7 LMH after completion of the cycle. Flux for every next cycle got more and more declined instead of stabilizing. The regression of flux can be described by the membrane fouling and the corresponding decrease in the osmotic pressure. Throughout the AnFDOMBR operation, the concentrated fertilizer draw solution was gradually diluted, and the conductivity of the bioreactor was progressively raised. These two progressions bring about the rapid decline of the transmembrane pressure difference, i.e., the decrease in driving force (Aftab et al., 2015; Li et al., 2018).

Figure 12:Membrane pictures (a) clean membrane (b) Fouled membrane just after completion of First Run (c) Fouled membrane just after completion of Last Run



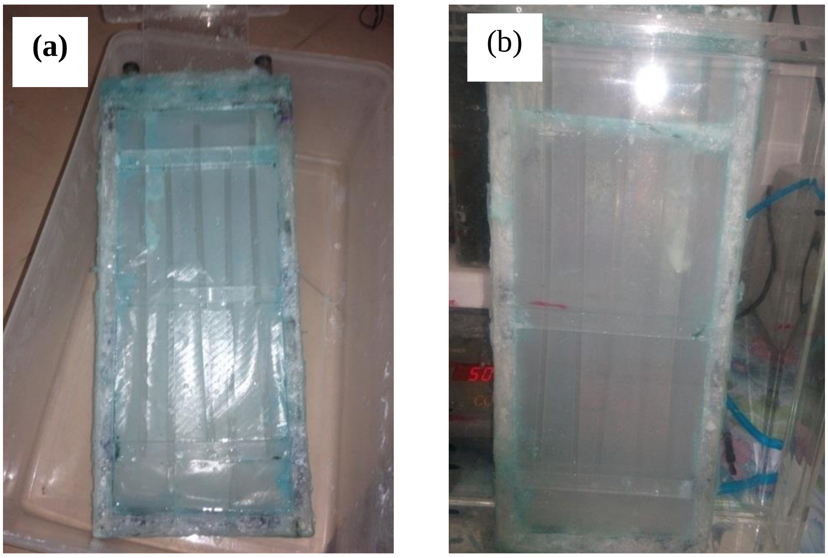


Figure 13:Membrane Cleaning Pictures after Run 1 (a) Membrane Clean with Tap Water (b) Osmotically Clean Membrane

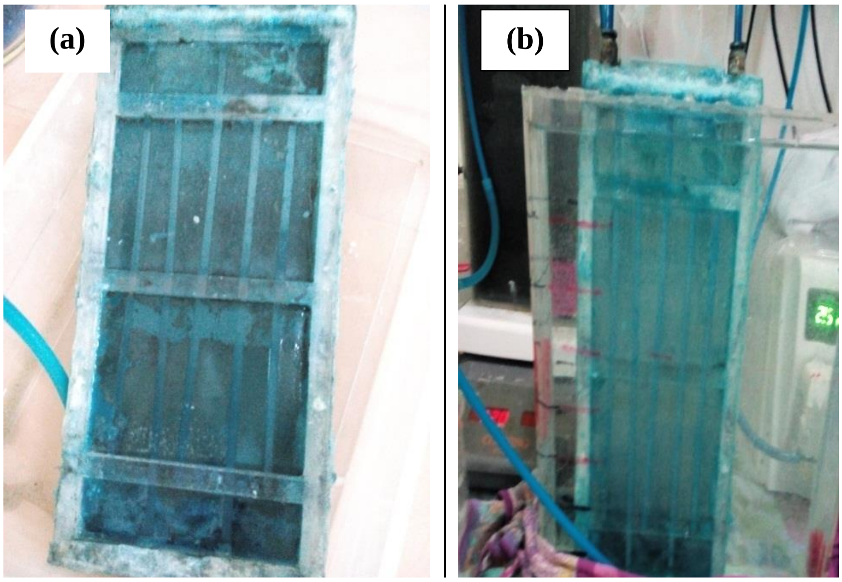


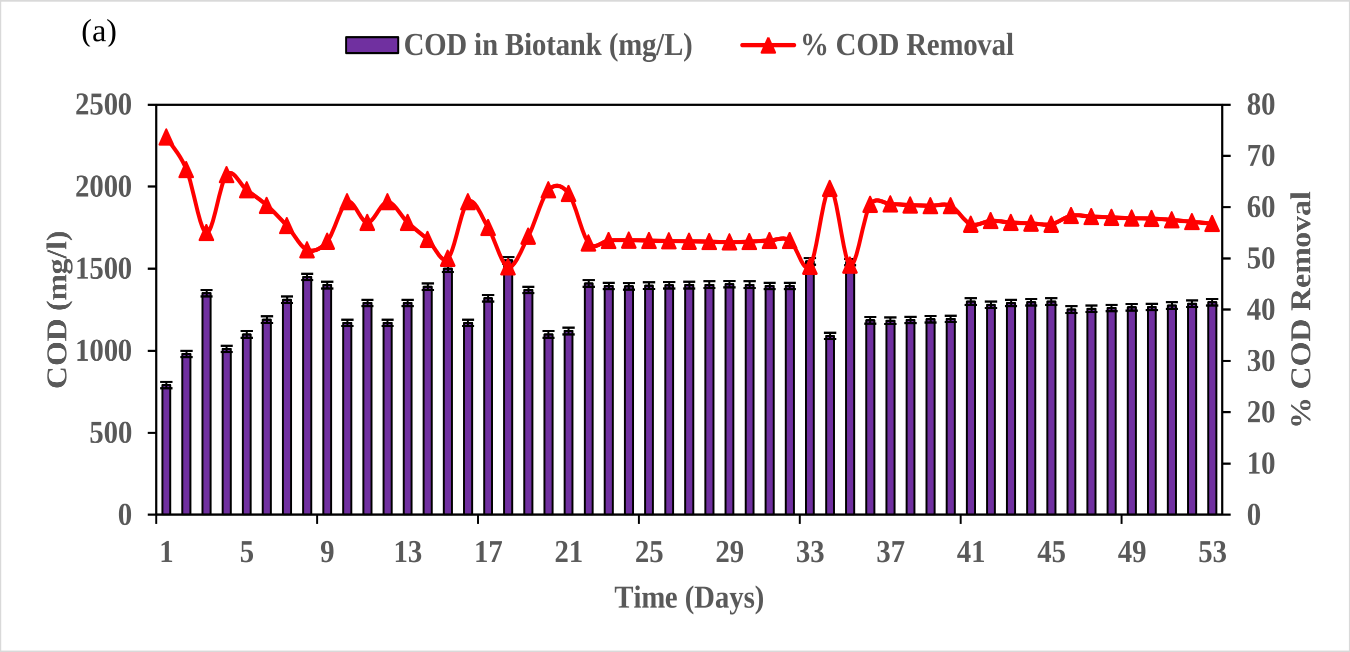
Figure 14:Membrane Cleaning Pictures after Last run (a) Membrane Clean with Tap Water (b) Osmotically Clean Membrane

Flux and conductivity does not show repeated trends .The rise of conductivity was probably due to buildup of salts from the textile feed as well as the solutes diffused in reverse direction through the membrane from the draw solution side into the bioreactor (Lay et al., 2011). Conductivity of bioreactor was maintained up to 25 mS/cm by performing sedimentation (1 h) every day to settle sludge and resultant supernatant was decanted to control the salt accumulation in the reactor. After 22 hours of operation, the diluted fertilizer was replaced with concentrated draw solution manually. Result shows that, 1 kg of fertilizer extracted approximately 10.5 liters water with an average flux ranging from 1.9 to 1.1 LMH during whole operational period.

4.1. Reactor performance

4.1.1. Chemical oxygen demand (COD) removal

During the whole operational period, samples of feed, supernatant from bioreactor and permeate from draw tank were collected on daily basis for examination. In the supernatant of anaerobic bioreactor, COD removal efficiency was 57 ± 5 % against feed solution (COD ≈ 3000 mg/L) as shown in **Figure** . 15 (a). Though, in permeate the total COD removal was about 91 ± 4% is shown in **Figure** . 15 (b). It happened due to magnificent removal potential of the FO membrane. It is also worth mentioning that the FO rejection was steady during the operation due to high rejection of semi permeable FO membrane. Maximum removal of COD in bioreactor was 74% initially which was reduced to 53 % at the end of operating period. The activity of microorganisms decreased substantially by the biodegradation and harmfulness of dye molecules and intermediates, the generation of lethal intermediates and representative pollutants may have slowed down the production of microorganisms in the bioreactor as shown in **Figure** 17. Additionally, an influence to this inhibition from the buildup in salinity cannot be excluded as well (Li et al., 2018).



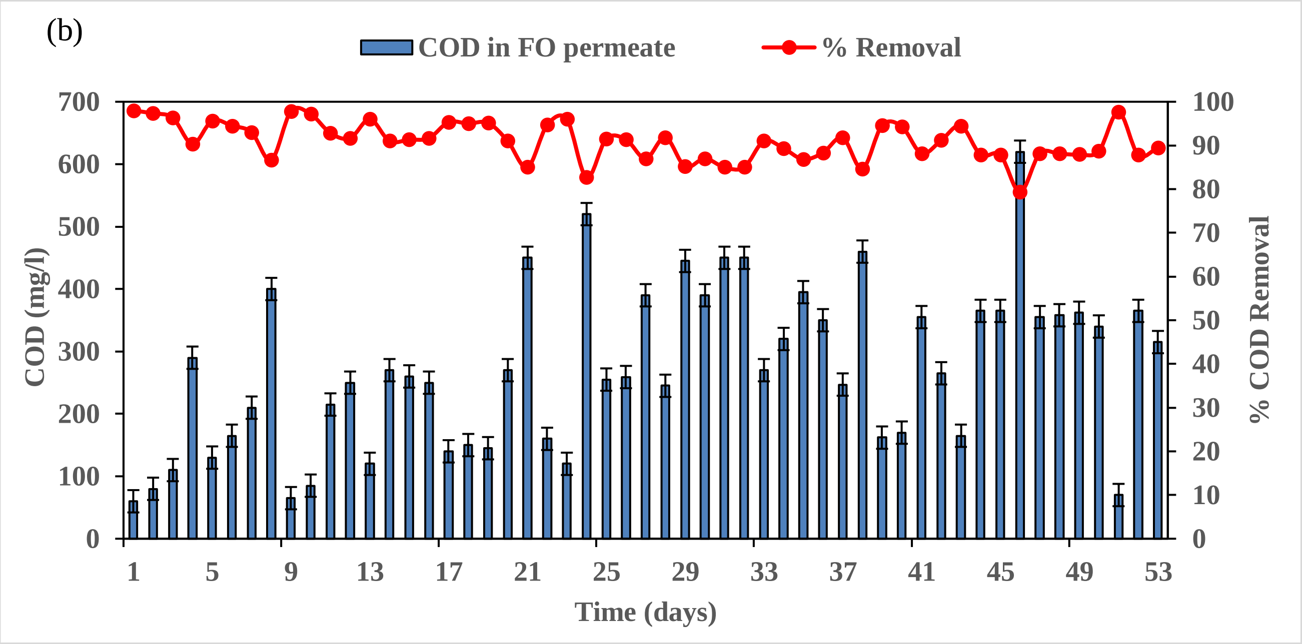


Figure 15: (a) Variation of COD in bioreactor and (b) FO permeate and corresponding removal efficiencies

4.1.2.Dilution of ammonium nitrogen and total phosphorus in fertilizer

Fertilizer driven forward osmosis (FDFO) has obtained intensified consideration since the resulted dilute solution of fertilizer can be applied for irrigation and consequently the regeneration of diluted DS is not needed, which is energy extensive process (Phuntsho et al., 2011). Dilution of nutrients occurred during each cycle is shown in **Figure** . 16. Greater the flux, more dilution of nutrients occurred and vice versa. Average fertilizer nutrients concentration (NH_4^+-N and total phosphorus) in diluted draw were 4 g/L and 13 g/L respectively which further required considerable dilution to meet nutrients criteria for fertigation (Phuntsho et al., 2013).

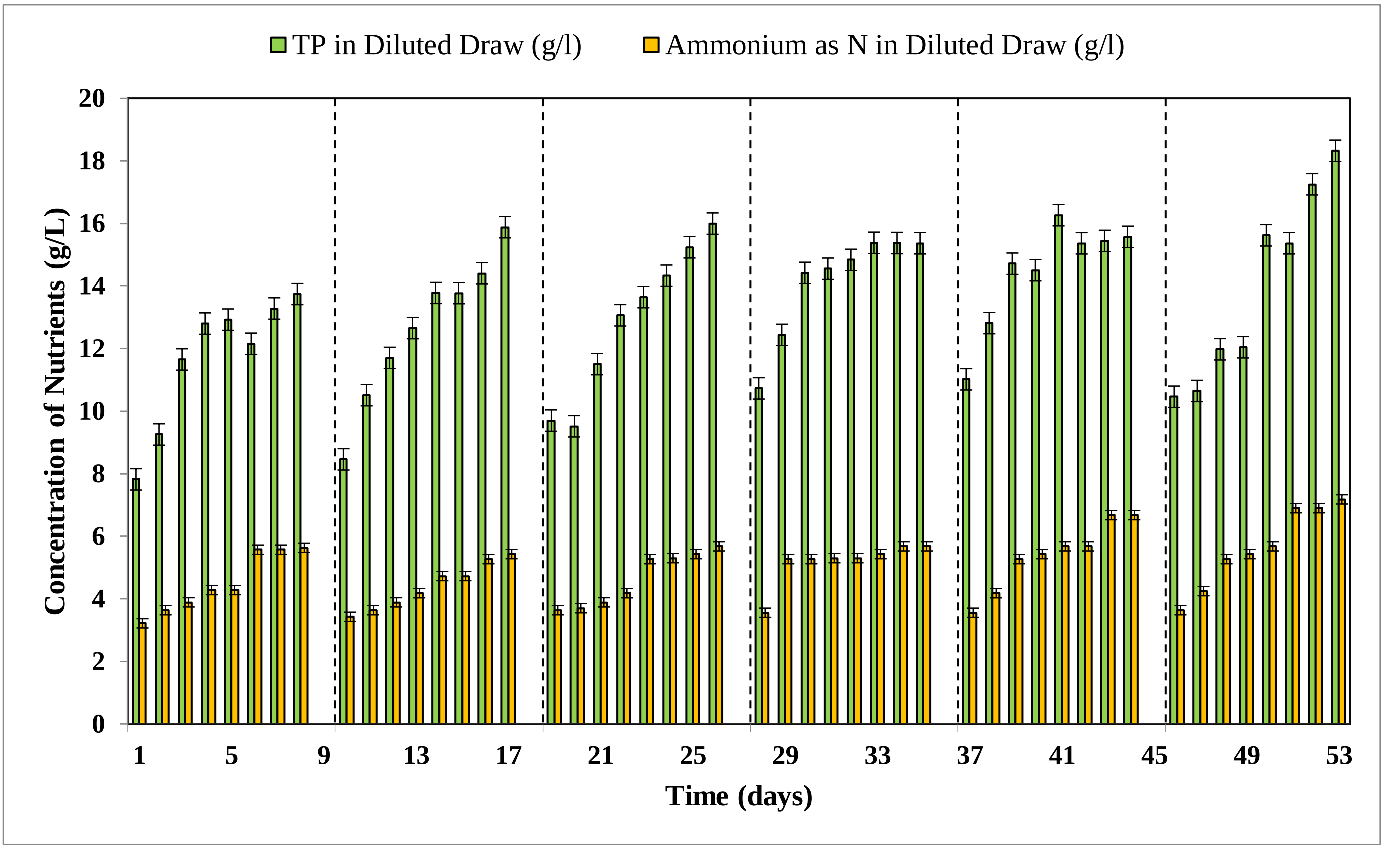


Figure 16: (a) Concentration of total phosphorus and concentration of ammonium nitrogen in diluted draw vs. time

4.2. Sludge characteristics

4.2.1. MLSS and MLVSS behavior during AnFDOMBR operation

MLSS and MLVSS were measured to evaluate the effect of MAP draw solution on bacterial consortium. During the operational period, MLSS and MLVSS were reduced to 2.5 g/L and 1.3 g/L from initial value of 5.9 g/L and 4.9 g/L respectively as shown in **Figure** 17.

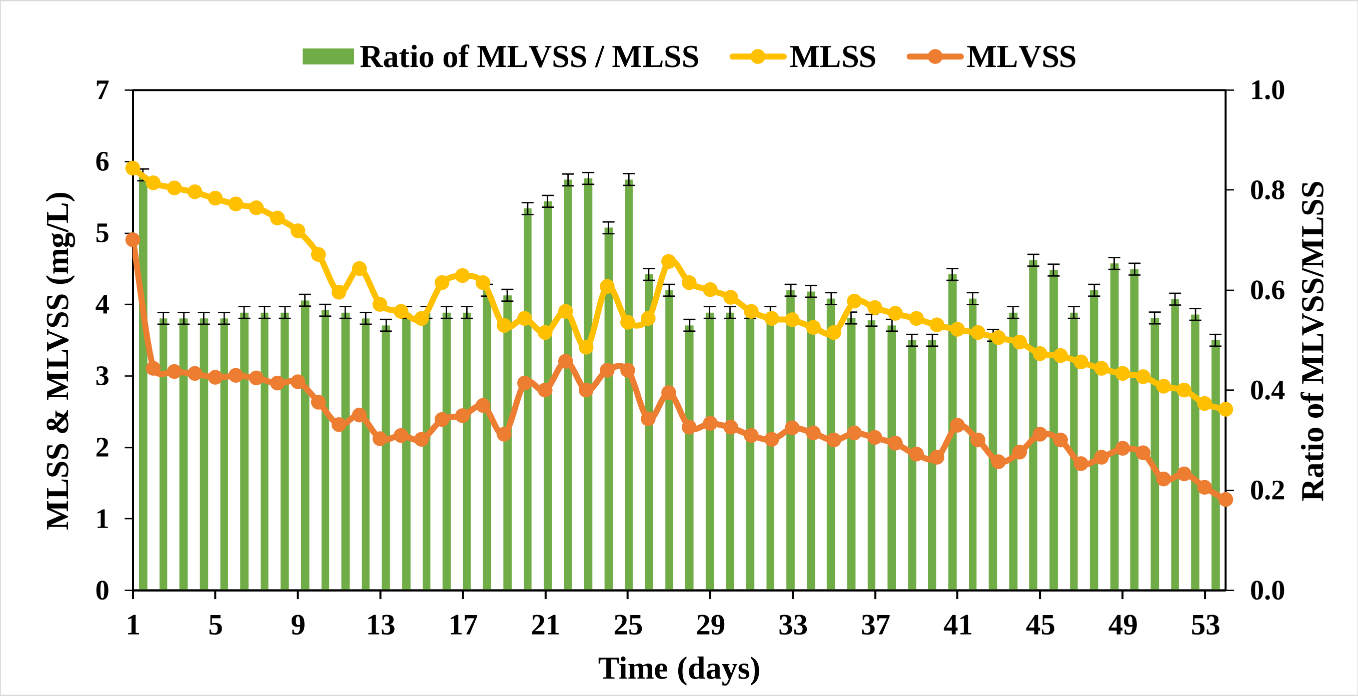


Figure 17:Variations of MLSS & MLVSS during operating period

These findings point out that microbe in the bioreactor were deteriorated by forward osmosis process, uniform with preceding investigation (Kim et al., 2017a).

4.2.2. Soluble microbial products (SMP) and extracellular polymeric substances (EPS) extraction and measurement

During constant operation of AnFDOMBR, samples of sludge were taken from the anaerobic bio-tank after every 13 days, for analyzing the soluble microbial product (SMP) and extracellular polymeric substance (EPS) constituents, as shown in **Figure** 18 and **Figure**

19. Usually, anaerobic membrane bioreactor has greater propensity to foul than aerobic membrane bioreactor. Anaerobic MBR demands a prolonged sludge retention time which possibly lead to severer internal pore blocking probably due to elevated concentrations of responsible foulants such as protein and carbohydrate molecules in soluble microbial products (Jegatheesan et al., 2016). In anaerobic MBR, SMPs was reported to be as high as 500% more than aerobic MBR under similar conditions (Martin-Garcia et al., 2011).

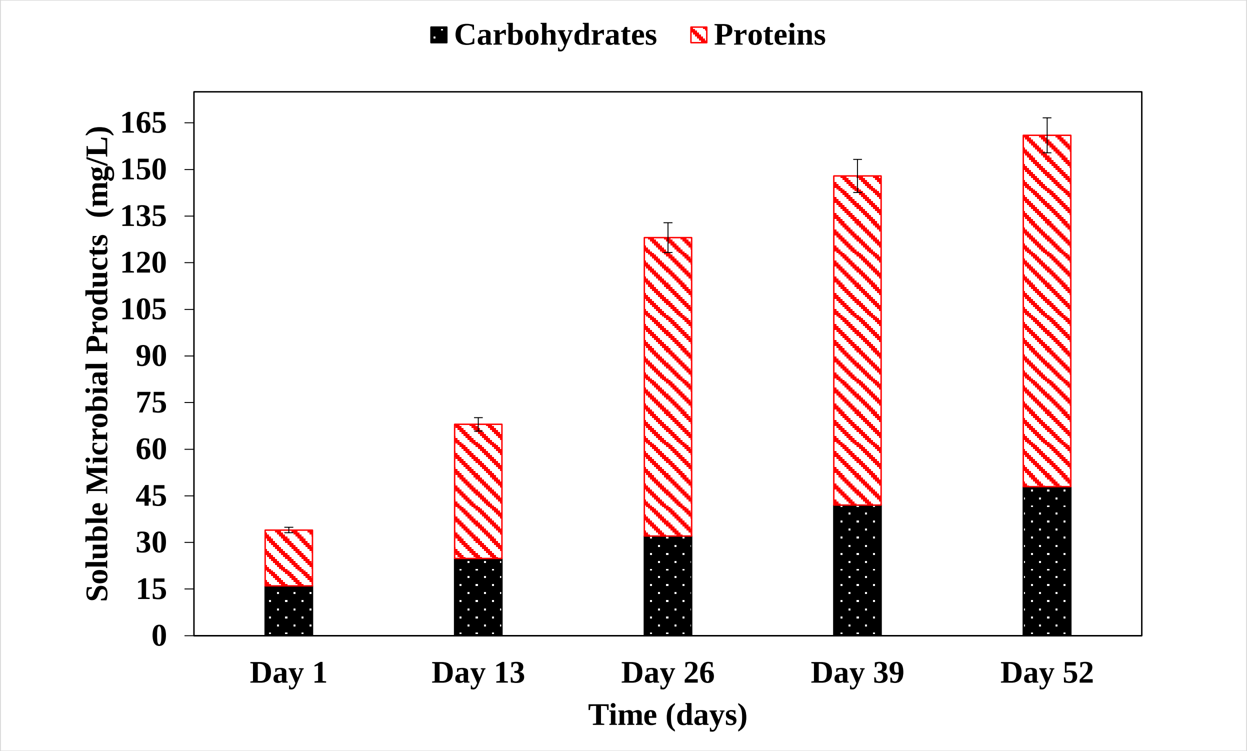


Figure 18: Variation in trend of SMP during whole operating period

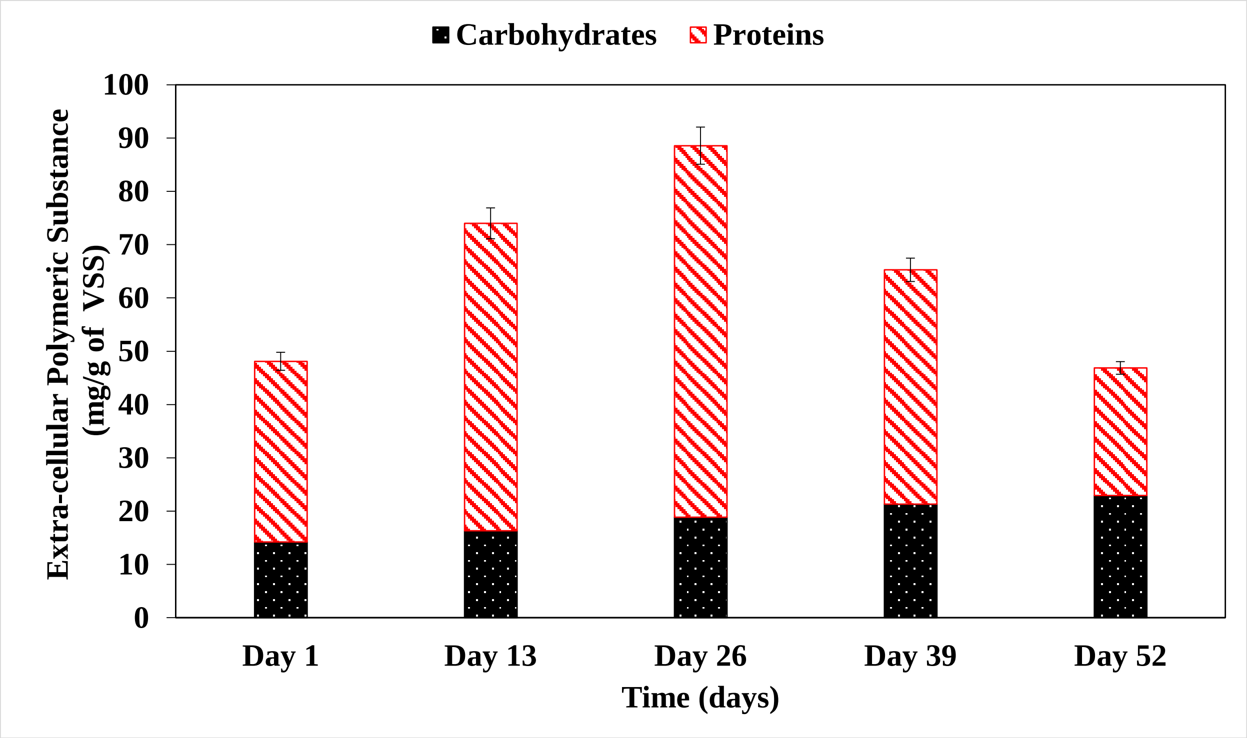


Figure 19: Variation in trend of EPS during whole operating period

In pressure-driven membrane processes, SMP and EPS are counted to be the main reasons of membrane fouling (Wang and Li, 2008). These compounds are constituted mostly of carbohydrates and proteins. In this study, SMP was the sum of proteins and carbohydrates in the supernatant of bioreactor and EPS was the sum of these two in the sludge. Meanwhile the EPS and SMP contents show a comparable ascending trend in the first 26 days, both go up with time. For example, EPS content increased from 48 mg/g VSS to 89 mg/g VSS in the first 26 days and SMP also raised from 34 mg/L to 128 mg/L, respectively. But EPS concentration did not increase in the remaining 26 days of the experiment. Though, the SMP concentration tended to increase, although with a slight intensity as shown in **Figure** 18.

Results of EPS are consistent with previous studies that at longer SRTs, concentrations of bound EPS became considerably lower, primarily due to decline in proteins content (PS) of EPS. Probably, this decline is due to proteins biodegradation, which became more significant during longer SRTs. Lower EPS concentrations were found after 26 days of operation which may be due to lower production and biodegradation of proteins (Faust, 2014). Prolonged SRT enhanced the concentrations of proteins and carbohydrate in SMP which ultimately speeds up the membrane fouling (Huang et al., 2011). Laspidou and Rittmann (2002) found that, SMPs are biomass-associated products (BAPs) and substrate utilization-associated products (UAPs). BAP´s are produced from biomass during decay but UAPs are generated directly from substrate utilization. In our study, increase in SMPs could possibly due to degradation of MLVSS because of salinity buildup, which causes excessive production of BAPs.

4.2.3. Particle size distribution and sludge filterability

Samples were taken at start, mid and end of OMBR operating period for analyzing the particle size distribution (PSD) in order to investigate the effect of salinity buildup on flocs size. During whole operation period, average particle size values decreased from 10.37 µm to 3.30 µm as shown in **Figure** 20. Siddique et al. (2018) also described that buildup of salinity has an adverse effect on flocs stability and sludge flocculation. Increase in saline stress results in an increase in the production of SMP and EPS, which may depreciate the settle-ability and flocculation properties of mixed liquor suspended solids (Zhang et al., 2014). Like other inorganic DS MAP showed an increased in capillary suction time (CST) during operational duration which exhibits drop in the filterability of sludge. The increase in CST is due to an increase of fine size particles (Siddique et al., 2018). In this context, Coackley and Allos (1962) divided sludge into various size ranges and found that filterability of sludge reduced with decreasing particle size.

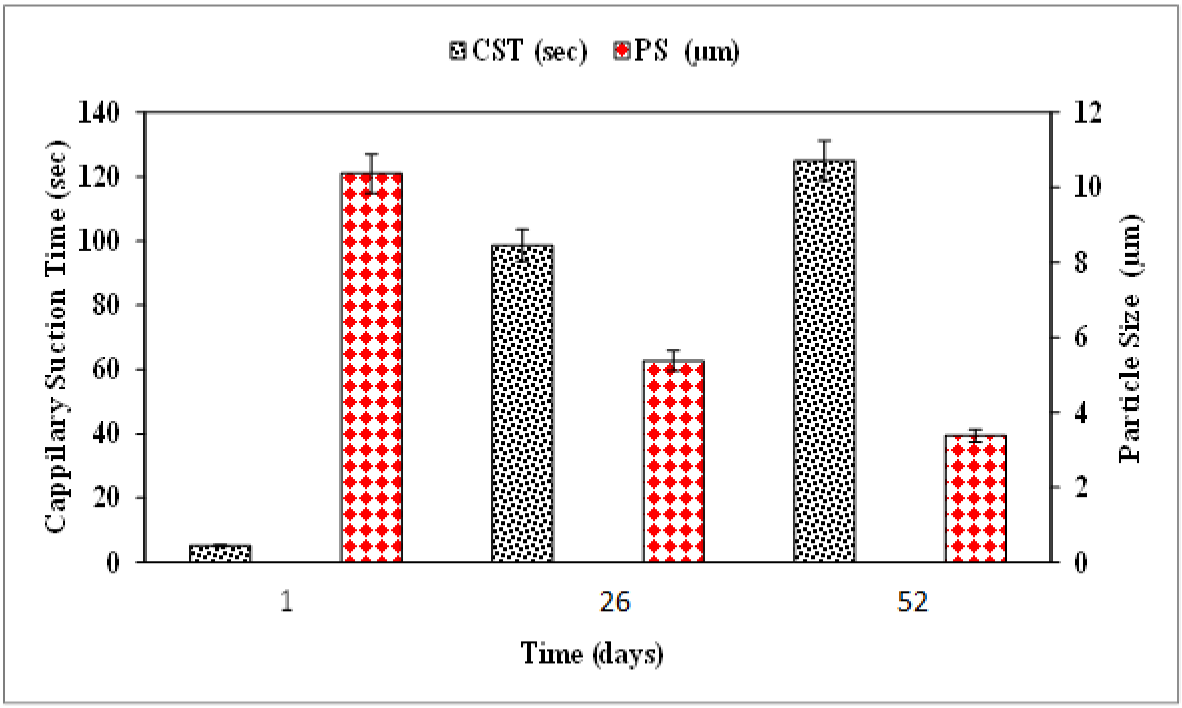


Figure 20: Particle size distribution and sludge filterability

4.2.4. Color removal

Samples from bioreactor and draw tank were taken to check color removal efficiency of AnFDFOMBR against synthetic textile wastewater (feed). Results revealed that, average color removal occurred during biological process was 44 ± 6% and it follows downward trend due to reverse solute flux and feed salts accumulation; which causes degradation of microorganism as discussed earlier as shown in **Figure** 21. Initially osmotic membrane bioreactor showed an excellent removal of dyes (up to 91 ± 2%), showing an effective rejection of chromophoric groups of dye molecules and byproducts. In last two cycles the removal efficiency of dyes decreases up to 88%, which might be due to membrane impairment.

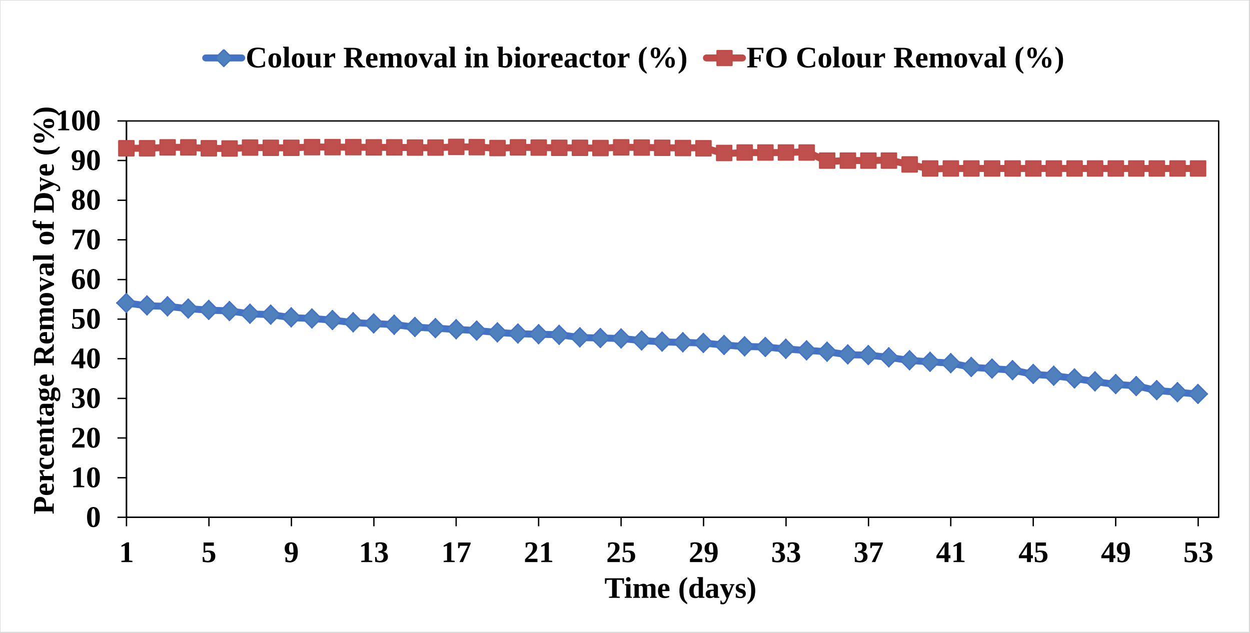


Figure 21:Removal of dyes during operating period

Conclusion & Recommendations

5.1. Conclusion

This study evaluated the flux performance of AnFDOMBR and the effect of reverse solute flux (RSF) on sludge characteristics for textile wastewater. The overall performance of AnFDOMBR in term of COD removal (i.e. 91±4%), color removal (i.e. 91±2%) and permeate quality was excellent. But biological performance of AnFDOMBR was inhibited due to high-retention property of the FO membrane. Shorter cycle of 8 days was investigated which helps in mitigating salinity of biotank. Prolonged SRT enhanced the concentrations of proteins and carbohydrate in SMP which ultimately speeds up the membrane fouling. However the filterability of sludge was also decreases due to increase in Capillary suction time (CST). Speedy membrane fouling and declined filterability of sludge causes drop in FO flux. Results revealed that final nutrients concentration would exceed the required limit. In order to keep final nutrients concentration low AnFDOMBR setup could be integrated with nano-filtration (NF) process as pretreatment to reduce the salinity of feed water or by using NF as a post treatment. Another optimum solution is to use AnFDOMBR setup with wastewater effluent treatment through multiple FO stages, although accomplishing textile effluent treatment and nutrient dilution simultaneously.

5.2. Recommendation

- Further investigation by using different fertilizer draw solutes for the treatment of textile wastewater may be performed.
- Different blends of the fertilizers may be used in AnFDOMBR for the treatment of textile wastewater.
- Future studies can be done on mitigating salinity buildup in bioreactor by coupling MF/UF membrane with AnFDOMBR.

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