

Performance Evaluation of Anaerobic Fertilizer Driven Forward Osmosis MBR



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

Bachelors of Engineering

In

Environmental Engineering

Institute of Environmental Sciences and Engineering (IESE)

School of Civil and Environmental Engineering (SCEE)

National University of Sciences and Technology (NUST)

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**PERFORMANCE EVALUATION OF ANAEROBIC
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TO

OUR LOVING FAMILIES

AND

FRIENDS

It wouldn't have been possible without their prayers and constant support.

ACKNOWLEDGEMENT

We would like to first and foremost extend our thankfulness and countless gratitude to **Allah Almighty**, the most merciful and generous, for bestowing upon us His blessings and strengthening us to complete the entire study and dissertation.

We would like to express our sincere gratefulness to our respectable and highly dedicated supervisor **Engr. Nida Maqbool**, Institute of Environmental Sciences and Engineering, for her utmost interest and keen attention, worthy advices and guidance, constructive criticism and the encouragement and positivity throughout the course of our final year project. This research would not have been possible without her valued time that she was always most kind to spare for us, from her actively engaged schedule.

Our Co-Supervisor **Dr. Sher Jamal Khan**, Institute of Environmental Sciences and Engineering, has been a guiding light for us since the beginning of our project. We are extremely thankful for his skillful feedback, financial assistance and valuable suggestions throughout the course of the project.

We must also pay our utmost thanks to **Engr. Aamir Khan, Sir Basharat, and Sir Mamoon**, Institute of Environmental Sciences and Engineering, who were always supportive and provided the much-needed assistance in Lab work.

We are also highly grateful to **Mr. Adnan, Mr. Talha Bin Umeed, Ms. Suraiya and Ms. Alia**, Institute of Environmental Sciences and Engineering, for their assistance, knowledge and apparatus as senior Masters Students. We are also extremely humbled for the continued support and technical assistance from the staff of the Waste Water Lab and Chemistry lab.

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LIST OF ABBREVIATIONS:

FO	Forward Osmosis
FDFO	Fertilizer-Driven Forward Osmosis
LMH	Liters per Meter ² Hour
MAP	Mono Ammonium Phosphate
MF	Micro Filtration
UF	Ultra Filtration
NF	Nano Filtration
RO	Reverse Osmosis
DI	De Ionized water
SDG	Sustainable Development Goal
FAO	Food and Agriculture Organisation
IRIN	Integrated Regional Information Networks
PCRWR	Pakistan Council of Research and Water Resources

ABSTRACT:

Freshwater resources are getting scarcer globally and the consumption by the agricultural sector is approximately 70% of the available freshwater (Clay, 2004). Thus, we proposed a low-cost anaerobic fertilizer-driven forward osmosis membrane bioreactor (AnFDFOMBR) for treating high-strength industrial wastewater and supplying nutrient solution simultaneously (for fertigation), eliminating the need of Draw Solute recovery. Synthetic textile industry wastewater was used in the feed tank and 0.5M Mono Ammonium Phosphate Solution (MAP) was used as the Draw Solution, which was continuously diluted and recirculated. Acclimatized sludge was used in the Bio Tank that consisted of a module housing Cellulose Triacetate (CTA) Membrane (Effective Surface Area = 550cm²). The Chemical Oxygen Demand (COD) removal, Total Phosphates (TP) and Total Kjeldahl Nitrogen (TKN) concentrations were checked for daily draw solute samples. 91.8%, 71% and 43.8% removal was achieved of COD, TP and TKN respectively. However, the diluted Draw Solution at the end of the runs required more dilution in order to meet the standard nutrient requirements for crops and hydroponics.

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CHAPTER 1: INTRODUCTION

1.1 Global water crisis and the Sustainable Development Goals

Freshwater scarcity resulting from economic and population growth is becoming an increasingly critical global concern. Approximately, a total of 2.1 billion People worldwide lack the access to clean and safe drinking water (WHO, 2017). This issue is much more severe in arid regions that are exposed to a serious shortage of freshwater. Water is essential to human life: in households, agriculture, industry, energy and livestock. The amount of fresh water available for human consumption is limited. Out of the total water available on the earth, about more than 97% is salt water and less than 3% is fresh water. While just less than 1% of the freshwater is directly available for human consumption since more than two-thirds is frozen in the form of glaciers and polar ice caps. The remaining water is found as the finite groundwater.

It has been predicted that during the course of the next decades, water will continue to become the most strategic resource, particularly in arid and semi-arid areas of the world (Gohari, 2013). Water is highly interlinked to energy and together the two entities that govern our lifestyles and the development of the civilization. Owing to this close connection, water shortage is also expected to thereby increase the issues of the energy crisis (Gorijan, 2015).

Moreover, the seriousness of the current water crisis has caused the United Nations (UNDP, 2007) to state that water shortage, not a shortage of cultivable land, will prove to be the key limitation in improved agricultural production over the course of the next few decades. As an example, Australia, a land abundant country, is one of the leading foods producing country but due to droughts its agricultural output has decreased significantly (Gorijan, 2015).

The Sustainable Development Goals (SDGs) were a global call to action by the United Nations to end poverty, ensure peace and prosperity and protect the planet (UNDP, 2016). This study aims at the SDG No.6 and 7, “clean water and sanitation” and “affordable and clean energy”. The provision of clean water depends on its sustainable use and management. This research is conducted to provide an affordable and sustainable solution in line with SDG No.6 and 7.

1.2 Current situation in Pakistan

1.2.1 Water Crisis

In 1995, UNDP ranked Pakistan among countries that have the highest water potential per capita out of a total 130 nations and that it must drastically progress from its current water condition to combat the shortage and avoid any future occurrences. However, due to failure to make any improvements, Pakistan's ranking has significantly dropped to 162 out of 180 countries in 2014. (FAO)

Furthermore, availability of water in Pakistan does not ensure access to safe drinking water in ample amount. Pakistan's water quality ranking is 80th among a total of 122 countries (IRIN, 2011). Water in the piping systems of Pakistan is contaminated due to either leakages (with many types of microorganisms) or because of environmental conditions and inadequate treatment, demonstrating alarmingly high concentrations of arsenic and fluoride (PCRWR, 2012). Water that is extracted from the ground by means of hand pumps – the major source of water in villages and other rural areas – is typically brackish and saline water and unfit for human consumption such as drinking, cooking and other purposes. Moreover, estimates by the Pakistan Council of Research and Water Resources (PCRWR) state that approximately 50 percent of water supply in the cities is unfit for human consumption.

The scarcity of water and the poor quality has many implications. The estimates from the Government of Pakistan about diarrhea (a major water-related disease) demonstrate that it is responsible for 14 percent of sickness for children of ages five years or younger and for 7 percent of all illnesses in people aged five or older. (Roseman, 2012) The Pakistan Council of Research and Water Resources (PCRWR) estimates that out of all reported medical cases, 40 percent are water-related. Unsafe water has the most significant impacts on the rural and urban poor, who are the major victims of the diseases caused by water.

This study, therefore, aims to find a solution that helps improve the current water shortage in the country. Moreover, the product water must be safe for use.

1.2.2 Energy Crisis

Energy is a key element of socio-economic advancement and economic prosperity. The shortage of available electricity at affordable prices would mean prospects of developing the economy of a country are little and the quality of life of its people is low. In Pakistan, only 55% households have access to electricity (Harijan, 2015). 68% of the total population of the country are residents of the rural areas and the majority of this population does not have access to commercial energy and utilize biomass such as firewood, agricultural wastes and animal dung (Kiani, 2014). The demand for gas rises past the transmission capacity during winter months and major users primarily industries, power plants etc. are reduced in winter in order to supply to domestic, and small industries. Due to the energy shortage in the country several industries operating in the country have been forced to shut-down operations and that has negatively affected industrial output production and the livelihoods of many families (Asif, 2015).

Therefore, a feasible solution for sustainable water use in Pakistan must not be energy intensive.

1.2.3 Textile Wastewater

Industrial wastewater in Pakistan is dumped into water bodies with little or no treatment. This wastewater contains several toxic pollutants and heavy metals. The situation is alarming since the water treatment is minimal and can cause several diseases with environmental degradation. Major industries of Pakistan include cotton textile, cement, fertilizer, edible oil, sugar, steel, tobacco, chemicals, machinery, and food processing.

Textile industry is one of the largest manufacturing industries in Pakistan, which contributes 8.5% to the GDP of the country. These industries usually consume large amounts of water specifically in wet processing such as desizing, scouring, mercerizing, bleaching, dyeing, printing and finishing. Wastewaters produced by these processes have variable compositions of textile dyes, mineral oils, suspended solids and electrolytes. The most harmful amongst which is the dyeing effluent because of the presence of toxic and non-biodegradable dyes (Carmen & Daniela, 2010).

High percentages of dyes in the industries do not have a good fixation and are released directly into the wastewater streams (Mattioli et al., 2005). The presence of even small quantity of dyes impacts the environment by increasing the toxicity, COD and BOD levels significantly and is

damaging to human health, aquatic life and is also esthetically displeasing (Chequer et al., 2013; Kant, 2012; Shaikh & Engineering, 2009). These complex contaminants actually hamper plant growth by adding color to the wastewaters, which are extremely difficult to degrade through conventional treatment technologies as ozonation, bleaching, activated sludge and electrochemical method (Eng et al., 2014).

1.2.4 Consumption by Agricultural Sector

Out of available resources, only 3 percent out of Pakistan's available fresh water is used for domestic consumption and drinking (Asif, 2015). The agricultural sector consumes 96 percent of all fresh water available (Nawab, 2017). The focus of the water debate in Pakistan is more towards water required for agriculture compared to water required for domestic consumption (Khosro, 2013). It is assessed that surface water succeeds to meet only 75-80 percent of water requirements for agriculture. Consequently, groundwater is merely seen as a substitute water source for crops and agricultural production. (Roseman, 2012)

The high demand of water for agricultural purposes is an opportunity for sustainable water management. This study aims to explore this potential in an attempt to tackle the water shortage in the country.

1.2.5 Concept of fertigation

One way to meet the water demand for crops is using 'fertigation'. Fertigation is an amalgamation of 'fertilizer' and 'irrigation.' It is the addition of nutrients, fertilizers, soil amendments, and other water-soluble products into the irrigation system (Kafkafi, 2013). It is the process of providing mineral fertilizers to crops along with the application of irrigation water. This term was first coined in the USA when anhydrous ammonia was bubbled into irrigation water. Later, other types of fertilizers were applied through a sprinkler system. Fertigation is most widely used in drip irrigation.

The idea of irrigation with dissolved fertilizers and nutrients goes back to Roman ages, when sewage water of the city was used for irrigation. In the early 1930s, farmers in the Jordan Valley were reported to use jute bags of ammonium sulfate ((NH₄)₂SO₄) fertilizer at the entrance of

canals. In modern applications, it is used for hydroponics applications. Nutrient injection system makes the growth more effective for hydroponics.

Fertigation offers some specific benefits over broadcast and band fertilization: (1) a regular supply of nutrients minimizes fluctuation of composition of nutrients in soil; (2) there is effective use and accurate application of fertilizers according to the standard nutrient requirements of the crop; (3) nutrients are applied all through the soil volume; (4) fertilizers can be applied to the soil during times when soil or crop conditions would otherwise forbid entrance into the field with traditional machinery. (Kafkafi, 2013)

1.3 Significance of the study

Global water scarcity is major issue faced worldwide, which therefore requires timely attention on research to find out new and robust approaches to purify water using low energy and cost. If low cost wastewater treatment technologies are made accessible, it can make a radical impact on the agriculture sector especially for countries like Pakistan where agriculture and textile industry sectors are providing major contribution for economic development of the country.

1.4 Objectives of the study

Due to rapid industrialization and increasing water scarcity, the industrial wastewater is required to be treated by means of a low cost and environment-friendly technology that produces high-quality effluent.

Therefore, our objectives include;

- Phase 1: Establishment of Anaerobic Fertilizer Driven Forward Osmosis Treatment Process to treat high strength industrial wastewater at lab scale at IESE-NUST
- Phase 2: Evaluation of the performance in terms of organics and nutrients removal

CHAPTER 2: LITERATURE REVIEW

2.1 Types of dyes present and other pollutants in textile wastewater effluent

The types of chemicals and dyes used in the textile are of different characteristics and composition depending upon the application and type of fabrics.

The dyes can be categorized into direct dyes, reactive dyes, basic dyes, acidic dyes, Azo dyes, Dispersed dyes, vat dyes and sulfur dyes. Other than dyes there are many other pollutants present in wastewater stream including heavy metals Hg, Fe, As, Pb many other chlorinated compound, trace organic substances, silicates, chlorides, nitrates of different elements. The composition of Wastewater stream produced at the end of different unit processes of textile industry is being tabulated in fig 3. (Ghaly et al, 2014)

Table 1: The composition of Wastewater stream produced at the end of different unit processes of textile industry

Process	Possible Pollutants	Nature of Effluent
Desizing	Starch, glucose, PVA, resins, fats and waxes do not exert a high BOD.	Very small volume, high BOD (30-50% of total), PVA.
Kiering	Caustic soda, waxes, soda ash, sodium silicate and fragments of cloth.	Very small, strongly alkaline, dark colour, high BOD values (30% of total)
Bleaching	Hypochlorite, chlorine, caustic soda, hydrogen peroxide, acids.	Small volume, strongly alkaline, low BOD (5% of total)
Mercerizing	Caustic soda	Small volume, strongly alkaline, low BOD (Less than 1% of total)
Dyeing	Dye stuff, mordant and reducing agents like sulphides, acetic acids and soap	Large volume, strongly coloured, fairly high BOD (6% of total)
Printing	Dye, starch, gum oil, china clay, mordants, acids and metallic salts	Very small volume, oily appearances, fairly high BOD.
Finishing	Traces of starch, tallow, salts, special finishes, etc.	Very small volume, less alkaline, low BOD.

2.1.1 Azo dyes

Azo dyes are used in textile industries and leather tanneries to treat leather articles and textile goods. Azo dyes are usually insoluble in water and many other solvents.

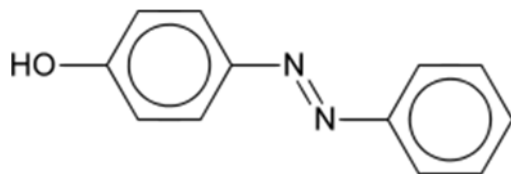


Fig .1: An Orange Azo dye

2.1.2 Disperse Dyes

These dyes are less soluble in the water at room temperature due to their non-ionic nature and have affinity for some hydrophobic fibers such as nylons and polyesters. A fine aqueous dispersion is prepared to apply these dyes to hydrophobic fibers such as polyacrylonitrile, polyamide, polyester, and polypropylene.

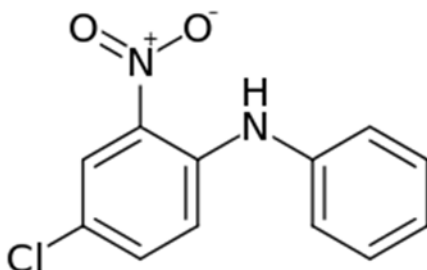


Fig.2: Disperse Yellow 26

2.1.3 Reactive Dyes

The reactive dyes have the ability to form the covalent bond with the substrate molecules that are needed to be colored. The reactive dyes form chemical bond with the main components of cotton fibers. Reactive dyes are highly colored substances that are organic in nature and are used for tinting textiles.

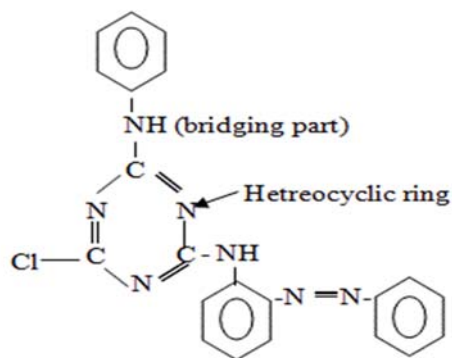


Fig 3: Chemical structure of reactive dyes

2.1.4 Direct Dyes

Direct dyes are called Substantive Dyes also. These dyes are soluble in water and have an affinity for fibers. Direct dyes are taken up directly by the fibers. Direct dyes can be easily applied and have less cost.

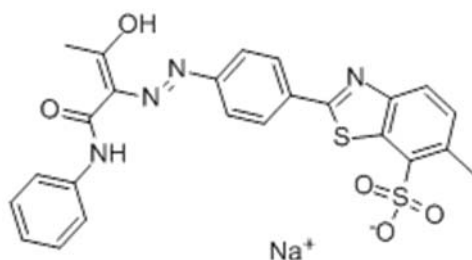


Fig.4: Direct Pure Yellow 5G

2.2 Existing treatment technologies for textile wastewater treatment

Textile industries generate wastewater from different processes. The wastewater resulting from all processes vary in composition due to different processes in the industry different machinery and products. Specifications of textile industry wastewater have great importance for the separate treatment of different process streams. Textile industries use a huge quantities of chemicals and large quantities of water. Caustic soda and Detergents are used to remove waxes, oils and dirt. Bleach enhances whiteness and brightness of fabrics. Most important Dyes and fixing agents that are used to provide brilliant colors according to market demands. Sizing agents

are used to enhance weaving. Oils are used to increase knitting and spinning. Glues and Latex are utilized as binders. A variety stain release chemicals and softeners are also used. Most chemicals in these become part of the industrial product and the remaining chemicals removed from the fabric and become part of effluent stream. The characteristics of effluent produced in different processes of textile industry are in table.

Table 2: Wastewater Analysis of Textile Industry

Process	Effluent
Singering Desizing	High BOD, high TS (Total Solids), neutral pH
Scouring	High BOD, high TS, high alkalinity, high temperature
Bleaching	High BOD, high TS, alkaline wastewater
Mercerizing	
Heat-setting	Low BOD, low solids, alkaline wastewater
Dyeing, Printing & Finishing	Wasted dyes, high BOD, COD, solids neutral to alkaline wastewater

The conventional treatment methods for textile effluent treatment are:

1. Biological Treatment methods (Activated Sludge Processes, Trickling filters, Aerated Lagoons, Oxidation Pond etc.)
2. Chemical Treatment methods
3. Physical Treatment methods

2.2.1 Biological Treatment

Biological wastewater treatment methods play important role in wastewater treatment systems and are able to treat wastewater streams coming from textile industries. Biological treatment processes are usually used as Secondary Treatment processes which are used to remove many contaminants that remain after primary treatment of wastewater stream. Micro-organisms are used to degrade the contaminants in biological wastewater treatment process. The treatment depends on bacteria, rotifers algae, nematodes, protozoa, fungi to convert unstable organic contaminants using cellular processes to stable inorganic species.

The efficiency of Biological treatment process depends upon the maintenance and development of suitable, active, mixed microbial species population in the biological treatment system. The microbial species are present as either a fixed film attached to a support medium as in the

trickling filters and rotating biological contactors, or in activated sludge processes as suspended growth and anaerobic digestion. Waste matter acts as source of food for micro-organisms in all biological treatment systems. During their life processes, these microorganisms use some of the organic matter in order to build new cell materials and then get the energy from their formation.

Biological treatment is considered as most economical technology as compared to the chemical and physical techniques because involves no chemical addition and less mechanical cost. These biodegradation techniques include microbial degradation, fungal decolorization and adsorption by living or dead microbial species and bioremediation systems. Biological treatment requires considerable land area, long treatment times and large amounts of sludge produced at the bottom of the tanks that need to be disposed of. (Robinson, McMullan, Marchant, & Nigam, 2001)

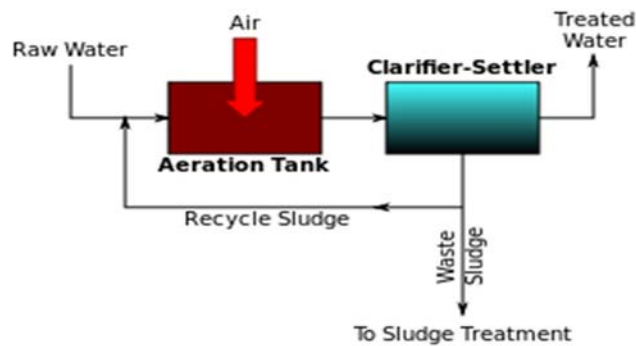


Fig.5: A generalized, schematic diagram of an activated sludge process

2.2.2 Chemical Treatment

Industrial wastewater is treated by a series of chemical processes to enhance the water quality. The chemical processes during which chemical reactions are taken place are known as chemical unit processes, and are used with different biological and physical processes to meet water standards. Chemical cleaning processes include chemical precipitation, chemical coagulation, chemical neutralization, chemical stabilization, chemical oxidation and advanced oxidation and ion exchange which can be applied to wastewater during cleaning.

Textile wastewater can be treated by the electrochemical methods using metal electrodes applying electric voltage. All these chemical methods are often cost intensive although the chemical but the sludge produced during these process may contain hazardous inorganic chemicals and heavy metals the disposal of which is another problem. Excessive chemical utilization may cause the secondary pollution. The cost of electrical energy and the consumption of chemical reagents are major problems.

2.2.3 Physical Treatment

Physical methods are the processes where no gross biological or chemical changes are carried out and physical processes are used to treat the wastewater. Physical treatment of textile industrial wastewater includes adsorption and absorption by activated carbon, Silica gel and the other materials like natural clay, rice hulls, corn cobs etc., for the removal of dyes is advantageous due to their availability and cost effectiveness (Robinson, McMullan, 2000).

Electrokinetic coagulation is an economically viable and cost effective method of dye removal. In which ferric chloride and ferrous sulphate are used to remove direct dyes from wastewaters (Mishra and Tripathy, 1993). A large amount of sludge is produced that results in high disposal costs of sludge (Gahr et al. 1995). Irradiation is another physical process to treat the wastewater stream of textile industry. The dye containing wastewater is treated in dual tube bubbling reactor to remove dyes. This method is not very effective on industrial scale but has shown good results on lab scale (Hosono et al., 1993).

Membrane filtration is used to concentrate, clarify and to separate dye effectively from wastewater effluent (Mishra and Tripathy, 1993; Xu and Lebrun, 1999). Membranes have some special features which make them better from other methods for example membranes show resistance to high temperature, microbial attack and an adverse chemical environment. The problems associated with membrane separation are removal of concentrated residue (sludge), and high capital and initial cost and the possibility of fouling and clogging. Membrane filtration is suitable for water recycling and reuse in the textile industries having relatively low concentration of dyes so usually used in the tertiary treatment steps (Nigam et al., 2014).

UF and MF membranes are used to remove colloids, suspended particles, and biological extra cellular polymeric substances at comparatively low pressure. Therefore membrane processes are used as standard unit operations (Bruggen et al, 2013). Membrane process usually Nano filtration and reverse Osmosis is commonly used as textile wastewater treatment (Ong et al., 2014) that show high fluxes and good removal efficiencies.

2.3 Forward Osmosis

Forward osmosis (FO) is the process in which solvent moves from less concentrated solution to the solution which has higher concentration of solute through a semi-permeable membrane under osmotic pressure. It is a naturally driven process.

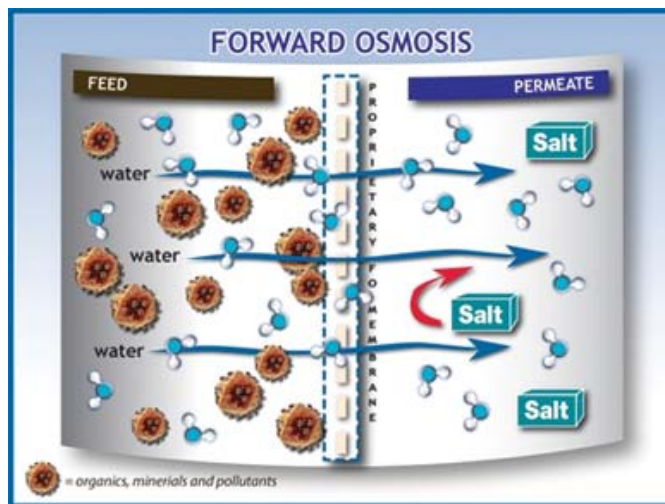


Fig 6: Forward osmosis

The simplest equation describing the relationship between the relationship between hydraulic and osmotic pressures and water flux can be described as

$$J_w = A (\Delta\pi - \Delta P)$$

- **J_w** is representing water flux across the two solutions
- **A** represents the hydraulic permeability of the membrane,
- **Δπ** is the difference of osmotic pressures on feed and draw solution sides
- **ΔP** represents the difference in hydrostatic pressures

Forward osmosis is being utilized from decades in different water treatment and wastewater treatment applications like Emergency drinks (hydration bags), Desalination, Brine concentration, Landfill leachate treatment, Feed water softening, Osmotic power generation, Evaporative cooling tower – make-up water.

FO depends on difference of osmotic pressure across the semipermeable membrane to extract purified water from the feed solution; FO is usually used as “pre-treatment” process before reverse osmosis to minimize the load on RO membranes and to get high quality permeates. (Lutchmiah et al, 2014)

Forward osmosis has high rejection capacity and low fouling membrane trends. Forward Osmosis is usually coupled with physical and biological water treatment processes to get the best results. Such as FORO, OMBR-RO, FOMD, FONF and integrated with seawater desalination enhances FO performance and makes it economically and commercially suitable. FO technology is becoming as a potential, reliable, and cost competitive alternative to meet the existing water quality standards.

2.4 Membrane Bioreactor

Membrane Bioreactors are wastewater treatment processes in which a semipermeable membrane is used along with some biological treatment process either aerobic or anaerobic (JUDD et al, 2011). Microfiltration or ultrafiltration membrane usually coupled with suspended growth bioreactors can be aerobic or anaerobic. MBR systems are widely used for industrial and municipal wastewater treatment with plant sizes up to 80'000 population equivalents (BEDDOW et al, 2010). MBR is a technical solution for the wastewater treatment so it needs expertise and skilled people to operate. Old treatment plants can be upgraded by MBR to increase treatment efficiency. Some Advantages of MBR systems are

- High effluent quality.
- Lesser production of sludge (Champan et al.)
- Ability to deal with High loading rates (Persson& Larsson, 2004)

- Tertiary filtration processes and Secondary clarifiers can be replaced by MBR system, so the footprint of plant is reduced. In some cases, footprint is reduced further because the processes such as digestion in digesters or UV disinfection can be reduced or eliminated. (Chapman et al).

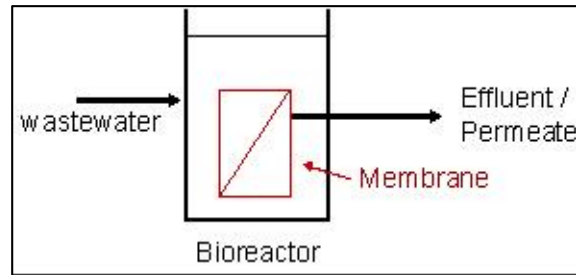


Fig. 7: Membrane Bioreactor systematic diagram

2.5 Forward Osmosis Membrane Bioreactor (FOMBR)

In this system a forward osmosis (FO) membrane module is submerged into a bioreactor. Water is transported naturally through the process of osmosis from the mixed liquor across a semi-permeable forward Osmosis dense membrane following the solution diffusion model of transportation into a draw solution (DS) which has higher osmotic pressure than the feed solution. Experiments have done with CTA (cellulose triacetate) flat-sheet Forward Osmosis membrane have shown high forward flux and lesser reverse transport of solutes from the Draw Solution into the mixed liquor side. (Achilli et al. 2009)

It was found in the FOMBR system the FO membrane can reject 98% of organic carbon and higher percentage of ammonium-nitrogen upto 90%; suggesting a better compatibility of the FOMBR systems are the better combination with downstream RO systems than conventional MBR systems in which UF or MF (porous membranes) are used. (Luo et al, 2017)

In AnFOMBR (Anaerobic Forward osmosis MBR) the suspended biomass is Anaerobic in nature which has many advantages over conventional MBR system.

- AnFOMBR produces stable flux.

- It can work with high organic loading rates.
- The production of sludge is very low. (Chen L. et al, 2014)
- Biodegradation of organic dyes and organic matter produces the methane gas which can be stored and utilized as fuel.(Tang MK et al, 2014)
- Less membrane fouling than other membrane processes. (Nguyen et al, 2015)
- High Quality Permeate.

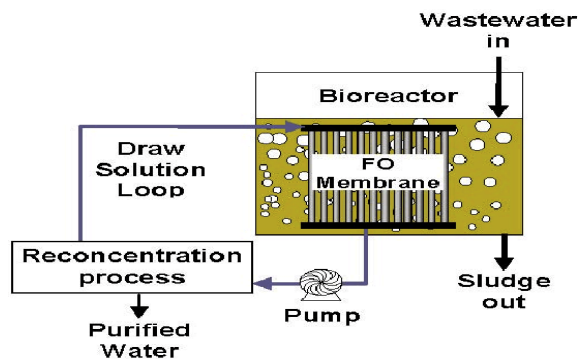


Fig.8: Forward osmosis membrane Bioreactor

2.6 Selection of membrane

Cellulose triacetate membrane is by made phase inversion technique has been selected for the present study due to following specifications.

- CTA membranes are highly hydrophilic membranes so able to produces the higher forward water flux.(Philip et al., 2010)
- Less concentration polarization and fouling as compared to other dense membranes.
- Higher rejection of pollutants and contaminants including extra cellular polymeric substances, divalent molecules and biological molecules. (Song et al., 2011)
- Good mechanical strength and thermal resistance.
- Readily available and low cost.
- Resistive to chlorine actions.
- High compaction tendency(Achilli et al, 2010)

2.7 Selection of Draw Solute

Mono Ammonium Phosphate was selected as draw solute for the present study by considering its fertigation applications and following characteristics which were found in literature.

- MAP shows the lower reverse salt flux.
- High forward flux and methane production.(Kim et al., 2016)
- Completely soluble at room temperature to make highly concentrated draw solutions. Highly soluble in water easy uptake by plants. (Phuntsho, Kyong, Hong, Lee, & Vigneswaran, 2011)
- Hygroscopic in nature having affinity with water. (Phuntsho et al., 2015)
- No need of recovery as it is used to fertigate the crops.
- MAP is a good fertilizer and it is a widely used as a source of Phosphorus and Nitrogen.
- Less molecular weight as compared to many other fertilizers.
- Easily available commercially.
- The draw solution made is less viscous.

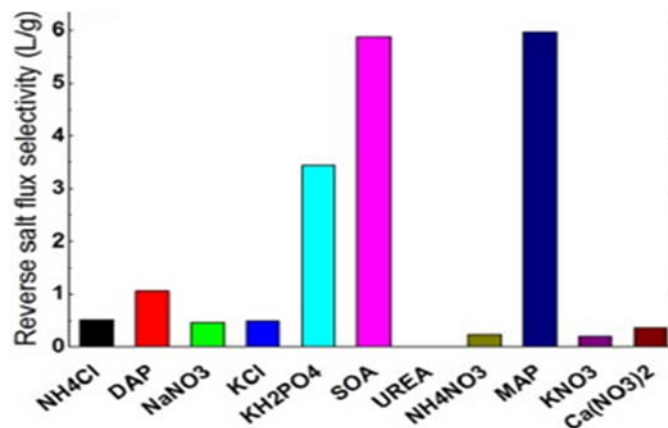


Fig. 9: Reverse salt flux selectivity of different draw solutes (Kim et al., 2016)

Our purpose is to fertigate the crop after treating the textile industrial wastewater so MAP is highly recommended as it contains the both essential nutrients nitrogen and phosphorus. Mono ammonium phosphate (MAP) is a widely used source of P and N.

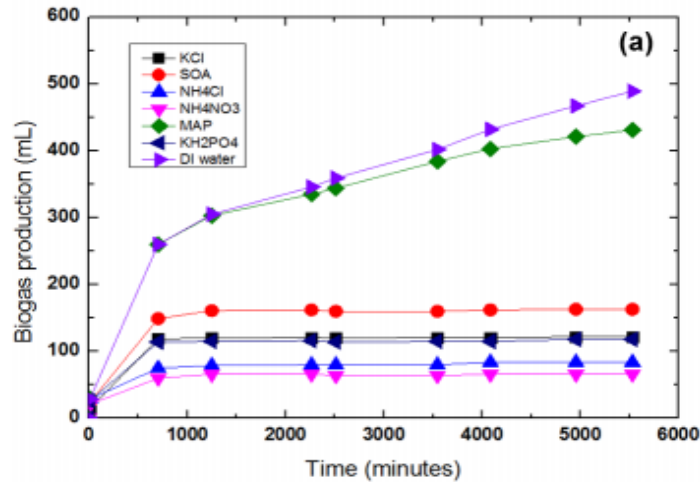


Fig. 10: MAP Biogas production compared to other salts

2.8 Fertigation and Dilution Requirements

MAP which is being used as draw solute is a fertilizer and product water is being utilized for fertigation purposes. Fertigation is a process that combines irrigation and fertilization by injection of soil amendments, required fertilizers and soluble products into an irrigation system which are required by plants. This method is usually common in extensive agriculture and horticulture. It is also used for landscaping due to the simplicity of the dispenser unit and increasing reliability (Mali et al, 2016). Fertigation systems have the ability to add the required amounts of fertilizers according to the plants nutrient requirements.

Some advantages of fertigation are being given

- reduces water consumption
- Reduces soil erosion
- minimizes the risk of the roots contracting soil-borne diseases
- increases the nutrients absorbed by the plants
- reduces the amount of fertilizer used
- Controls the precise time and rate of fertilizers being released. (Kant et al, 2011)

The growth of the plants consist upon three different stages during their life period, the allowable concentration of nutrient for fertigation varies in the range of 120–200 mg/L of Nitrogen, 40–50

mg/L of Phosphorus and 180–300 mg/L of Potassium. The nutrient concentrations in the final product stream from the fertilizer driven forward osmosis desalination must meet the water standards for the direct application of fertigation. The higher concentration of the nutrients more than the requirements in the draw can increase salinity of soil and can cause toxicity in the plants and also economic losses. Also the higher concentration of nutrients can cause the eutrophication in fresh water bodies when nutrients reach water bodies with fertigation runoff water. (Kim et al., 2013)

Table 3: Acceptable nutrient concentrations for direct fertigation of tomato plants (Phuntsho et al., 2013)

Physiological Stages	N (mg/L)	P (mg/L)	K (mg/L)
Planting and establishment	120–150	40–50	180–220
Flowering	150–180	40–50	220–270
Ripening and harvest	180–200	40–50	270–300

CHAPTER 3: MATERIALS AND METHODS

3.1 Materials

Our setup consisted of a Feed Solution Reservoir (FS) and a Draw Solution reservoir (DS). The Feed Solution was stored in a tank of 6 Liters was pumped into Anaerobic Bio Tank that contained the Membrane Module. The Anaerobic bio tank had a volume of 5000cm^3 . The Membrane was a flat sheet CTA membrane that was prepared to have an effective area of 550 cm^2 . The Draw solution was kept in a tank having a volume of 6 liters. The flowrates of both the Draw solution and the Feed solution were maintained at 5.5ml/min using 2 pumps. Longer Man Peristaltic Pumps were used. The flux was calculated by placing the draw Tank on a (Shimzadu) Mass balance and recording the changes in the readings. To record these changes against time automatically, a data logger was connected to the mass balance and the mass reading was recorded in an Excel spreadsheet

3.1.1 Feed solution

The Feed solution was a synthetic wastewater that was prepared using previous research done on real textile wastewater from Kohinoor textile mills.

The recipe for the synthetic wastewater is shown in the table below:

Table 4: Synthetic Wastewater Composition

Chemicals	Composition	Chemicals	Composition
Hydrated D-Glucose	3000 mg/L	Zinc Chloride (ZnCl_2)	0.1 mg/L
Ammonium Chloride (NH_4Cl)	1146 mg/L	Nickel Chloride (NiCl_2)	0.1 mg/L
Calcium Chloride (CaCl_2)	29.19 mg/L	Cibracon Yellow ($\text{C}_{25}\text{H}_{15}\text{Cl}_3\text{N}_9\text{Na}_3\text{O}_{10}\text{S}_3$)	20 ml/L

Magnesium sulfate Heptahydrate (MgSO ₄ ·7H ₂ O)	9.73 mg/L	Cibracon Blue (C ₂₉ H ₂₀ ClN ₇ O ₁₁ S ₃)	20 ml/L
Ferrous chloride (FeCl ₂)	1 mg/L	Methylene Blue (C ₁₆ H ₁₈ ClN ₃ S)	20 ml/L
Cobalt chloride (CoCl ₂)	0.1 mg/L	Sodium Bicarbonate (NaHCO ₃)	500 mg/L

3 types of reactive dyes were also added to the synthetic wastewater so to assess our treatment method's dye removal capacity. They were Cibracon Blue, Cibracon Yellow and Methylene Blue each with a concentration of 20ml/L. The dyes used were typical in textile industry's wastewaters.

3.1.2 Draw Solution

In the research project, the fertilizer selected for the synthesis of Draw Solution was Mono Ammonium Phosphate (MAP). Reagent grade MAP (Sigma Aldrich) was used and the molarity of the solution was varied from 0.25 molar to 0.5 molar. The draw solution was prepared by weighing out the reagent grade MAP in a beaker and dissolving it in deionized water. Then adding in appropriate amount of deionized water. The draw solution was stored and capped in 6-liter plastic tanks.

3.1.3 Anaerobic Biotank

The Biotank consisted of acclimatized sludge that was acquired from a lab scale CSTR in the wastewater lab. The BioTank was sealed and strict Anaerobic conditions were maintained in the tank. The tank was fabricated of acrylic and had a length of 14 cm, a width of 10 cm, and a height of 35 cm, which gave a volume of 4900cm³. The initial mlss and mlvss of the sludge was found to be 4.5 and 3.5, respectively.

3.1.4 Control Tank

In the water relay tank, a fixed head of the fs is maintained. It has 3 sensors, they function together to regulate and maintain the head of water in the BioTank. The relay is connected to the peristaltic pump that is connected to the fs tank, which varies the flowrate according to the water head in the BioTank.

3.1.5 Membrane module:

The membrane employed in this study was obtained by HTI-Hydration Technology Innovations (USA); The Cellulose Triacetate Membrane (FO membranes) fixed in a woven polyester mesh. The membrane specifications are as follows.

- The Pure water permeability coefficient of the CTA membrane is 1.17 LMH/bar.
- The Solute permeability coefficient is 0.98 LMH.
- The Structural parameter of the CTA support layer is 473 S - μm

The membrane was set in a plate and frame housing, and the Active Layer facing Feed Side configuration.

3.2 Process Flow:

The setup was produced keeping in mind to cover all aspects of our study and for it to be simple, cohesive and concise.

Feed solution (FS) and Draw solution (DS) were kept stored and capped plastic containers (6 liters) each to prevent the reduction of COD and the contamination of the solutions.

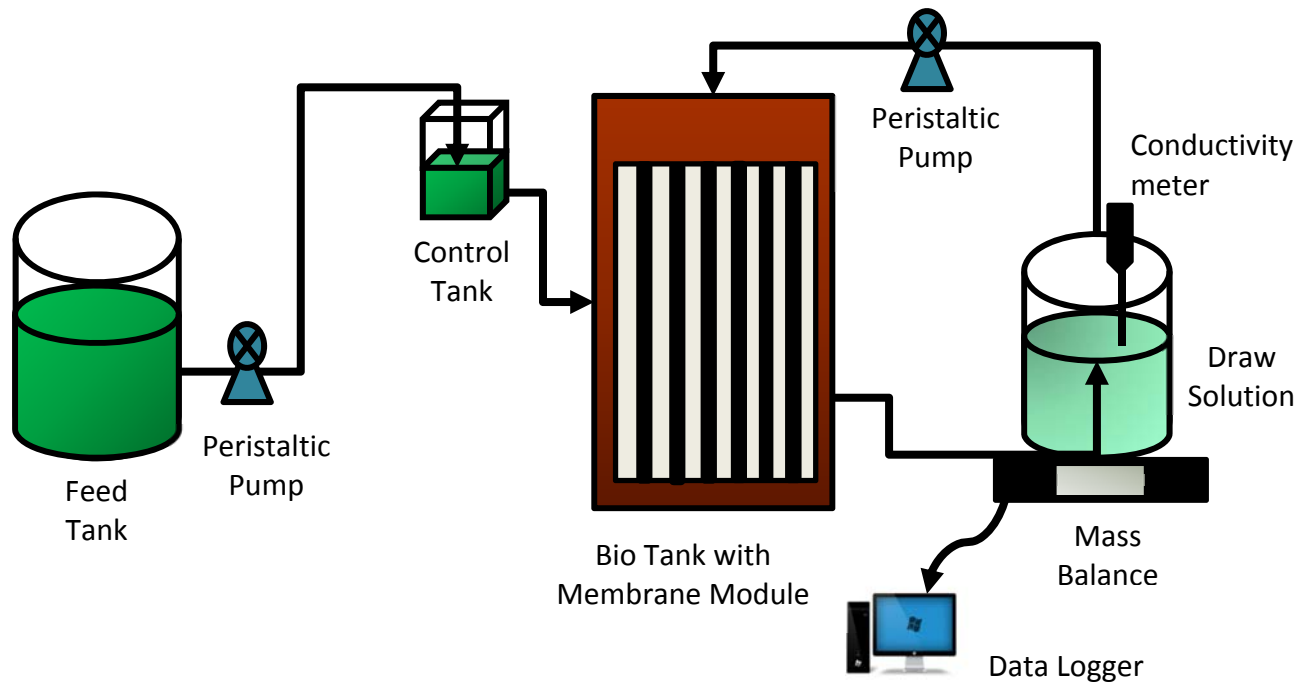


Figure 11: Process flow diagram of Experimental Setup

In the bottom of the plastic tanks a hole was made and the FS and DS were connected by a plastic pipe to the peristaltic pump (Longer Pump BT300-2J), and the pumps permitted the exact flowrate for relatively small discharge values.

A baffled plate and frame Membrane Module of active area 550 cm^2 was designed, fabricated and submerged in the BioTank. The CTA membrane placed within this housing had the Active side facing the Feed Stream thus maintaining the FO mode in all the experiments conducted.

The BioTank was placed on a magnetic stirrer (PC 420D CORNING) to provide appropriate agitation to the microbial consortium so the biomass acts on all the wastewater instead of settling in the bottom of the tank.

A mass balance (UW6200H SHIMADZU) was kept under the Draw tank to record the gain in mass of the Feed tank which was used to calculate the permeate flux and the decrease in flux over time. This scale was connected to the Data Logger which plotted these periodic readings into a spreadsheet program.

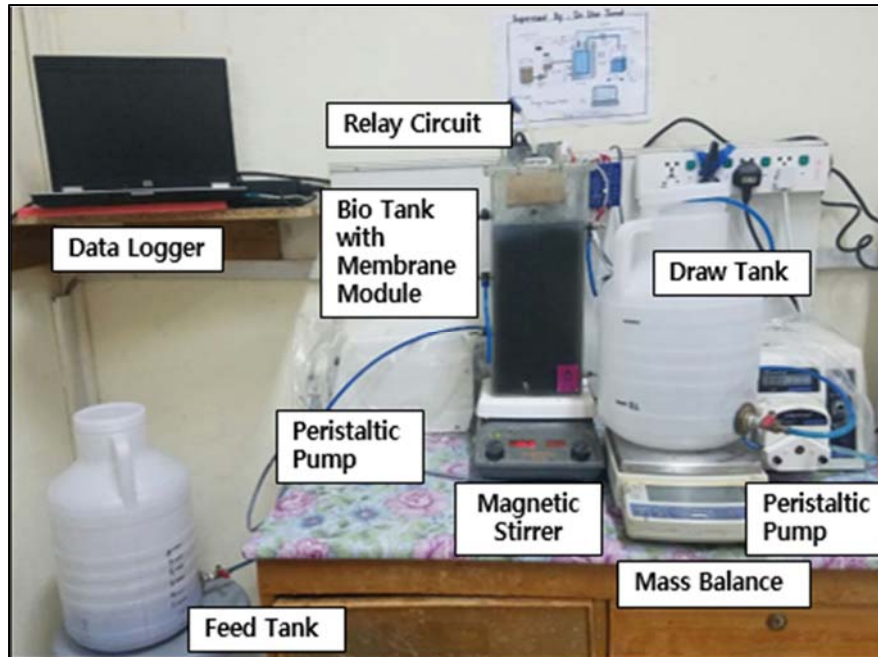


Figure 12: Experimental Lab Setup

3.3 Equipment used

In order to record and interpret the parameters required for the conclusion of results, different techniques were employed to extract this information in a comprehensive manner. The equipment used include:

3.3.1 Laboratory Oven (BOEKEL Scientific)

The oven was used for the sludge characterization tests, namely the mlss and the mlvss and also for the preparation of the reagents. The oven was used to heat up the retentate left on the Whatman filter paper to 105 °C so that the suspended solids only are left. The oven was a versatile and a highly useful tool that allowed us to conduct our tests safely and with great precision.

3.3.2 Benchtop Muffle Furnace (ThermoFisher)

The Muffle Furnace can be used to achieve temperatures in the range of 500 °C to 1700 °C safely in the laboratory. It allowed us to conduct the mlvss test, which requires for the retentate to be volatilized at a temperature of 550 °C so that only the microbes or the biomass is left. And then the sample is weighed.

3.3.3 Mass Balance (UW6200H SHIMADZU)

The mass balance used during the experimental runs allowed for very accurate readings as well as the permission to connect it to a computer system and record readings at a fix time interval specified by the user. These values for change in mass per unit time were used to derive the water flux and multiple values over each run duration were used to form graphs depicting the trend lines for these fluxes.

3.3.4 UV-Spectrophotometer (Model T-60U range 190-1100nm)

The principal mechanism for the equipment is the direct relation between the concentration of the specific particle and the quantified amount of light that has been absorbed by that particle at a specific wavelength that is unique for different types of particles. Light at only a certain characteristic wavelength is absorbed and this absorbance is proportional to the concentration of the particles in solution.

3.3.5 Conductivity meter (inoLab ph/conductivity 720)

Conductivity, defined as the ability of an electrolyte solution to conduct electricity, was a major principle used to determine the amount of fertilizer that had travelled from the Draw stream into the Feed stream. As the fertilizer is fundamentally a salt that forms ions in a solution, these ionic solutions form electrolytes. The concentration of these ions present in the solvent is directly proportional to the conductivity of this solution. After the completion of each baseline run where the feed stream was pure water, the conductivity of distilled water which had been determined prior to the experiment was subtracted from the conductivity after the completion of the run. As the only constituent of this solution was fertilizer from the Draw stream, the conductivity value was an accurate representation of the fertilizer concentration in the Feed stream. Conversion to attain values from micro Siemens to units of concentration was performed to depict greater clarity.

3.3.6 Total Nitrogen (Total Kjeldahl Nitrogen Apparatus)

These experiments were performed to assess the final amount of Nitrogen present in the Draw Stream after each run. This value of nitrogen has to be assessed as some salt permeates towards the Feed side during experiment, this is referred as the reverse solute flux (RSF). TKN values also showed the extent to which the nitrogen has been diluted as this was used to further understand

how much dilution would be required in order to make this draw stream water adequate for direct application to plant and crops.

3.3.7 Chemical Oxygen Demand (COD) Reactor (Hach)

The COD reactor equipment can digest up to 25 samples at one time. Each test set-up is a self-contained vial. The vials are placed in the reactor for two-hour digestion time. The COD can be digested completely in 30 minutes to 2 hours at 150 °C, the normal operating temperature. Then the titrations would be carried out.

3.4 Reactor Design:

After consultation with our respectable supervisors and by conducting intensive literature review we came to the conclusion of setting the HRT at 12 hours. And the flux through the membrane was required to be 6LMH.

The Working Volume of reactor was assumed to be 4 Liter so;

Hence the Flow is calculated to be 0.33 l/h

And, the Membrane Effective Area = $\text{Flow} / \text{Flux} = 0.33 \text{ l} / \frac{6 \text{ l}}{\text{m}^2 \cdot \text{h}} = 0.055 \text{ m}^2 = 550 \text{ cm}^2$

The Area of Single Membrane is $550/2 = 275 \text{ cm}^2$

Length of Membrane = 25 cm

Width of the Membrane = 11cm

The Factor of Safety for reactor volume was kept 25 %

Volume of reactor = $4 + 4 \times 0.25 = 5 \text{ Liter} = 5000 \text{ cm}^3$

Width of Membrane = 11 cm

Length of Reactor = 14 cm

Width of reactor = 10 cm

Height of Reactor = 35 cm

In the study, an optimum cross flow velocity was maintained keeping in mind that a very low velocity will result in thicker boundary layer formation which will aid concentration polarization. A fall in permeate flux will result due to this concentration polarization phenomenon. If the cross-flow velocity is kept at an inadequately high value than the chances of physical damage to the membrane itself are high.

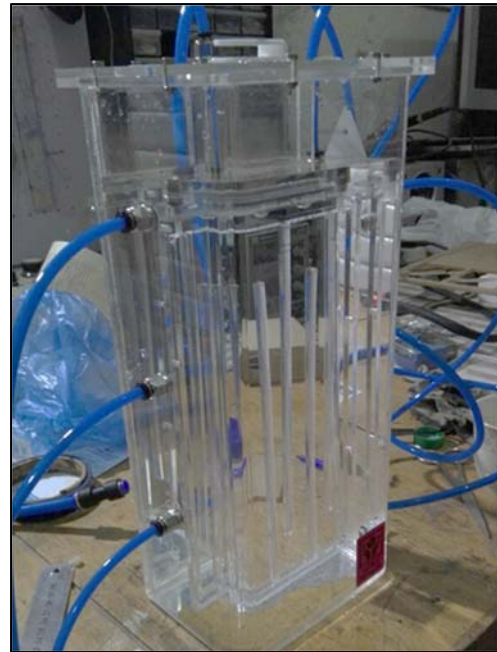
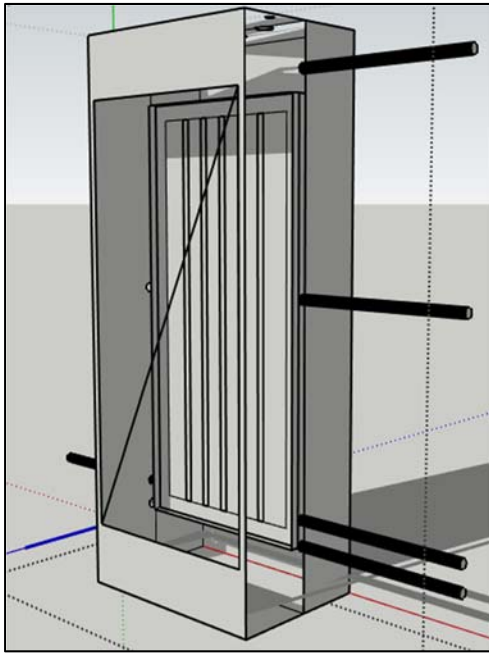


Figure 13: The reactor design as shown on AutoCAD software
Figure 14: The fabricated Acrylic reactor based on the design showed on the left

3.4.1 Operating Conditions

The operation conditions were maintained and monitored throughout the course of the experiment so to ensure the authenticity of the research.

- Temperature = $24 \pm 1^\circ\text{C}$
- Cross Flow velocity = 7.275 cm/s
- Run Time = 10 Hours
- Membrane Forward Flushing = 1 Hour
- Feed Volume = 6 Liters

- Draw Volume = 6 Liters

3.5 Experiment Procedure

The sequence of each individual experiment followed the basic steps of

1. Draw Solution Preparation
2. Synthetic Feed Wastewater Preparation
3. Switching on setup
4. Measuring conductivity of draw solution and Biotank
5. Calculating flux using Mass balance values

The draw solution was prepared in deionized water of reagent grade MAP. To prepare a 1 liter solution of 0.25 molar; 28.75 grams of MAP was taken and dissolved in DI water. Then the solution was made up to 1 liter by adding more DI water.

Likewise, to prepare a solution of 0.5molar MAP 57 grams of MAP was dissolved in DI water and the solution was made up to 1 liter.

The synthetic feed was prepared according to the recipe.

The conductivity of the draw solution was measured at time intervals so that the comparison of the conductivity of the DS and the flux generated by it can be drawn.

The Experimental flux (L/m²/h-LMH) was calculated by $J_w = \frac{\Delta m}{\Delta t} \frac{1}{Am}$

Where Δm is the mass change, Δt is the test time duration which is 10 h and Am is the effective membrane area which is 0.055m².

3.5.1 Baseline experiments

To assess the flux performance of the CTA Membrane, baseline experimental runs were performed with MAP DS as a general procedure to verify the specifications of the commercial grade membrane. For all baseline experimentation, the feed stream is always kept as distilled water and the concentration of salt in the Draw Stream may be varied.

3.5.2 Pure water flux for Mono Ammonium Phosphate (MAP)

To establish the baseline water and define a reference value for comparative reasons, controlled 10 hour runs were performed by using Distilled water as a feed stream and different molarities of MAP as draw stream, i.e. 0.25M or 0.5M.

3.5.3 Performance evaluation of AnfdFO MBR process with Synthetic wastewater and Dyes.

Through the consultation of published literature and the wastewater characteristics report of the textile manufacturing industry; Kohinoor Textile Industry. Limited, concentration of textile dyes was specified at 20 ml/L for Cibracon Yellow, Cibracon Blue and Methylene Blue.

After the synthetic wastewater feed was introduced the DS molarity was initially kept at 0.25 molar. However, the calculate dflux was coming out to be very low.

Hence the molarity of the ds was increased to 0.5 molar. And the system started running smoothly.

3.5.4 COD Tests

The sample of ds was collected after each subsequent run of 10 hours, and the COD of the sample was calculated by using the 'Standard APHA methods, 2005' for COD determination. The COD of all the samples was recorded and carefully studied to check for any irregularities that would indicate an abnormality in the system. The percentage removal of COD was plotted against time. The initial COD of the synthetic wastewater feed was 3000 mg/l.

3.5.5 TP Tests

The aim of the study was to use the product water for fertigation and for minimal dilution of the ds before being applied to the fields. As phosphorous is an important element for the growth of the crops, the NPK ratio of common crops such as tomato was studied and a relation was drawn between the final TP in ds and the phosphorous requirement of the plants.

Hence, the dilution factor was determined.

The robustness of the system was quantified by the percentage of TP, and they were plotted on a graph against time.

3.5.6 TKN Tests

Like TP, nitrogen is crucial for plants and the concentration in the ds and the requirement by tomatoes was compared to define the dilution factor.

The action of the Anaerobic process and the FO membrane resulted in substantial removal of TKN, and the percentage removal of TKN was plotted on a graph against days.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Water flux at different molar concentrations of draw solution:

4.1.1 Run with 0.25M Draw solution and Distilled water

Equipment is run at different molar concentration with distilled water and synthetic waste water respectively to calculate water flux. First of all it is run with 0.25M draw solution of Mono Ammonium Phosphate (MAP) with Distilled water. The results are depicted in figure 15.

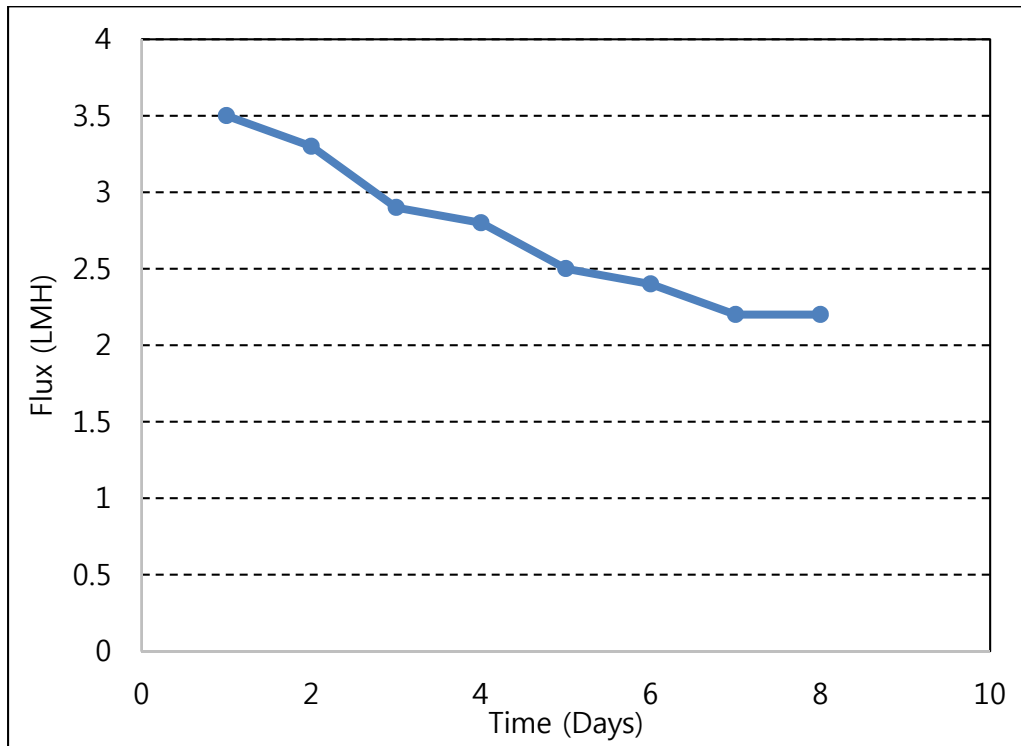


Fig 15: Flux against time graph for distilled water and 0.25M DS

The equipment was run for continuously 8 days and the flux was measured. As it can be seen that the flux is decreasing with every passing day this is due to the fact that the concentration difference between the feed tank and the draw tank decreases with time so as a result the flux also decreases. Our initial value was 3.5 LMH on the first day and after approximately 8 days the flux reduced to about 2.3 LMH (approx). As the equipment is run with distilled water so there is no membrane clogging contributing to the decrease in flux.

In order to get the better the flux results the equipment was then run with 0.25M draw solution of Mono Ammonium Phosphate (MAP) and synthetic waste water whose composition is near to that of Textile waste water. The figure 16 depicts the results:

4.1.2 Run with 0.25M Draw solution and Synthetic Waste Water

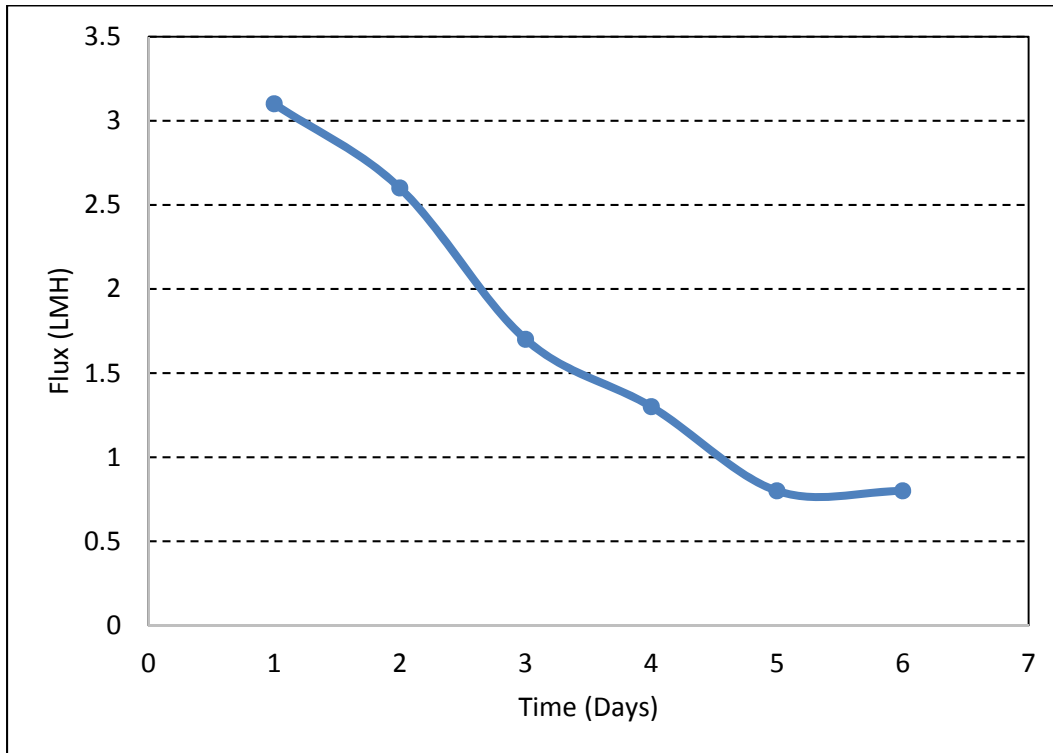


Fig 16: Flux against time graph for synthetic water and 0.25M DS

As it can be seen the initial value of flux with 0.25M draw solution and synthetic waste water which is nearly 3.2 LMH is less than that of 0.25M draw solution and distilled water. The final value which is also very low is nearly 0.7 LMH. This is due to the fact that as we are using synthetic waste water so along with decrease in concentration difference between feed side and draw side, the membrane clogging is also playing its role in decreasing the flux.

The major difference between the run with synthetic waste water and distilled water is the membrane clogging as distilled water doesn't contain any such particles so membrane clogging won't be any such factor in decreasing the flux but in case of synthetic waste water it plays a role.

4.1.3 Run with 0.5M Draw solution and Distilled water

The equipment is again run but this time with 0.5 Molar concentration of Mono Ammonium Phosphate (MAP) with distilled water in order to see the effect of change in molar concentration on the water flux.

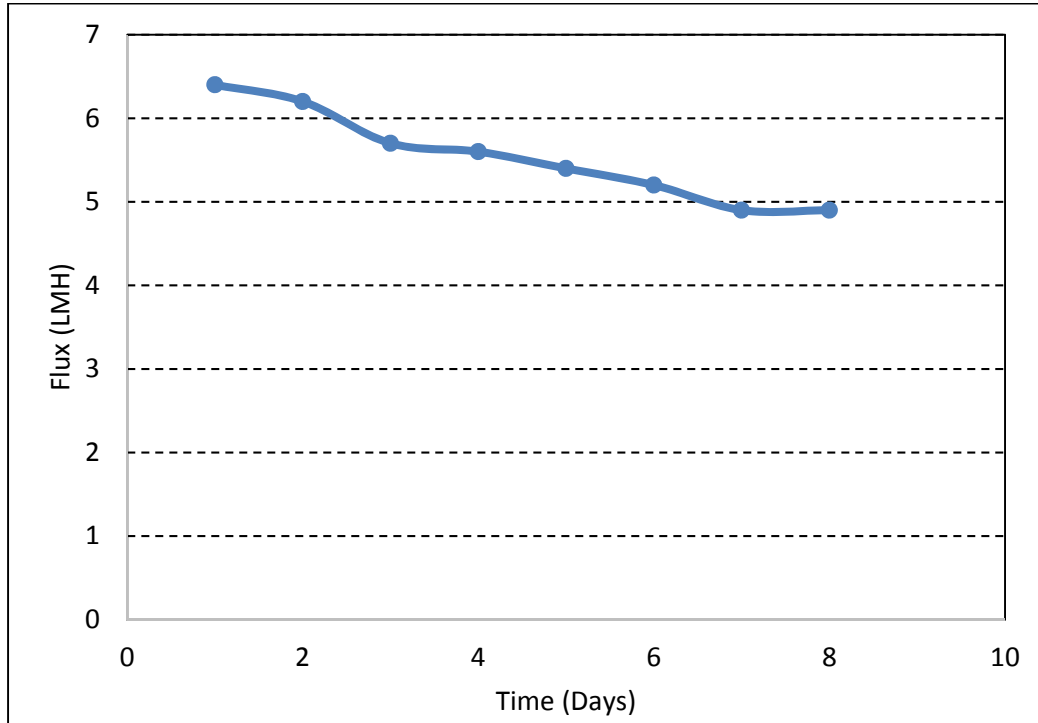


Fig 17: Flux against time graph for distilled water and 0.5M DS

The equipment again was run for nearly 8 days continuously with 0.5M concentration of Draw solution and distilled water and the flux trend was recorded which is shown in figure 18. The graph shows that the initial flux which is approximately 6.5 LMH is higher than that of run with 0.25M draw solution. This change in flux value is because as the molarity of the draw solution is increased from 0.25 to 0.5 molar so as a result the concentration difference between the feed side and draw side also increased resulting increase in initial flux. The other flux decreasing phenomena is again the same as the run is with 0.5M draw solution and distilled waste water so the decrease in flux will be solely due to decrease in concentration of difference with the passage of time. As a result the final value is 4.9 LMH.

4.1.4 Run with 0.5M Draw solution and Synthetic Waste water

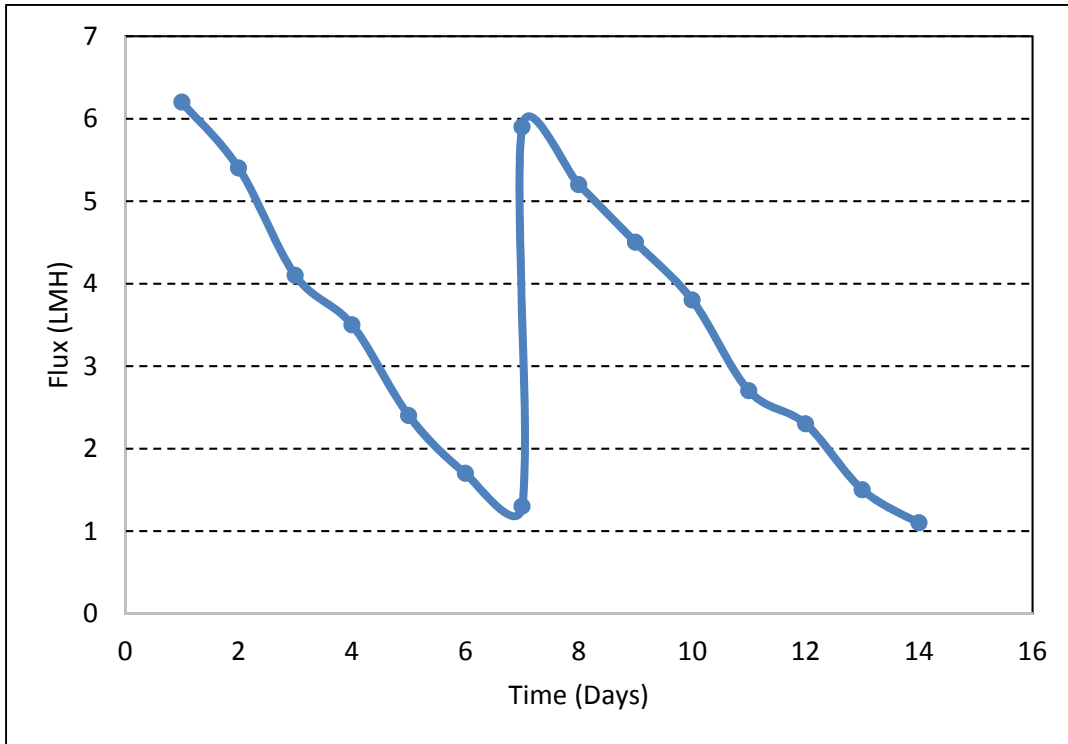


Fig 18: Flux against time graph for synthetic water and 0.5M DS

When the equipment was run with 0.5M concentration of Mono Ammonium Phosphate with synthetic waste water, the flux initial value was 6.3 LMH (approx.) but after 7 days of running the flux was decreased to the minimum possible value 1.2 LMH (approx.). This condition happened due to decrease in difference in concentration gradient between the draw side and feed side and also due to membrane clogging as it involved synthetic waste water too. So this was the first run after the equipment was stop for the physical and chemical cleaning of the membrane.

The second run started from 8th day after physical and chemical cleaning and the initial flux was approximately equal to that of the first run. After 7 days of continuous running it again clogged and required physical and chemical cleaning.

4.2 Water Conductivity at different molar concentration of draw solution

Measuring conductivity of water is an important parameter, as it tells us about the number of dilutions required to meet a specific plant requirement, moreover as the conductivity decreases the concentration of nutrients also decreases to the required value need by plants.

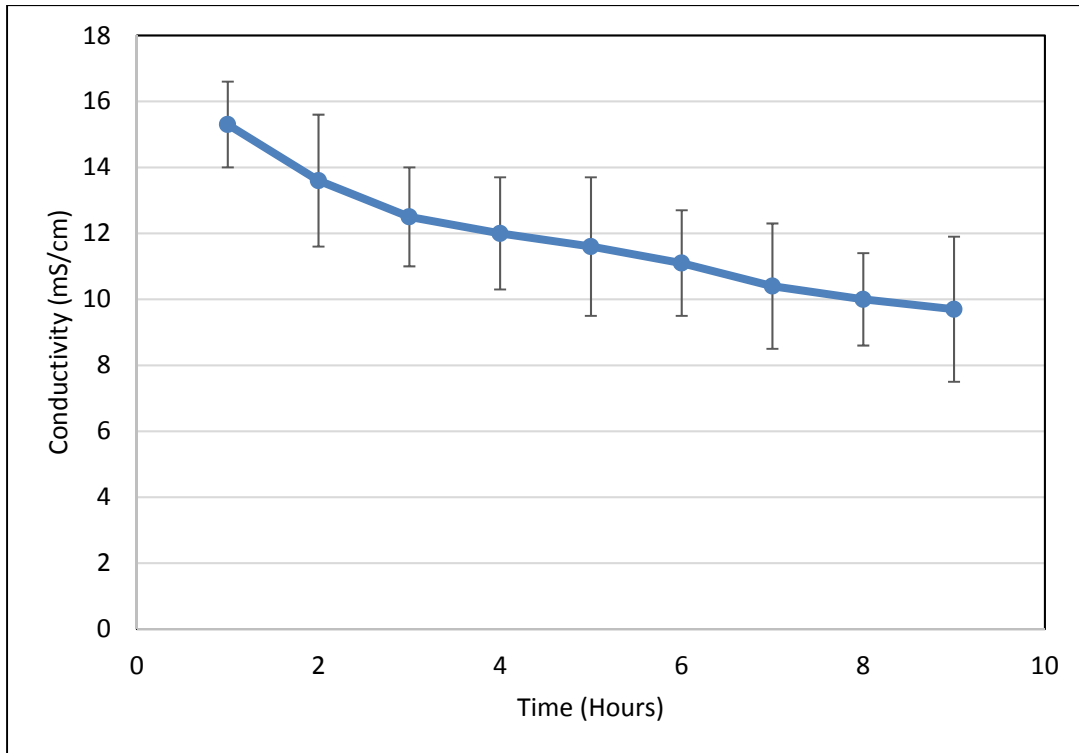


Fig 19: Conductivity against time graph for synthetic water and 0.25M DS

The graph above shows that the conductivity of water at 0.25 M concentration decreases with the passage of hours. The initial value was 15.7 mS/cm (approx.) and it was decreased to 11.9 mS/cm (approx.). The decrease in conductivity is due to decrease in the concentration of conductive ions per unit volume of water.

The conductivity at 0.5M draw solution of Mono Ammonium Phosphate with synthetic waste water was also measured which is depicted in Figure 20.

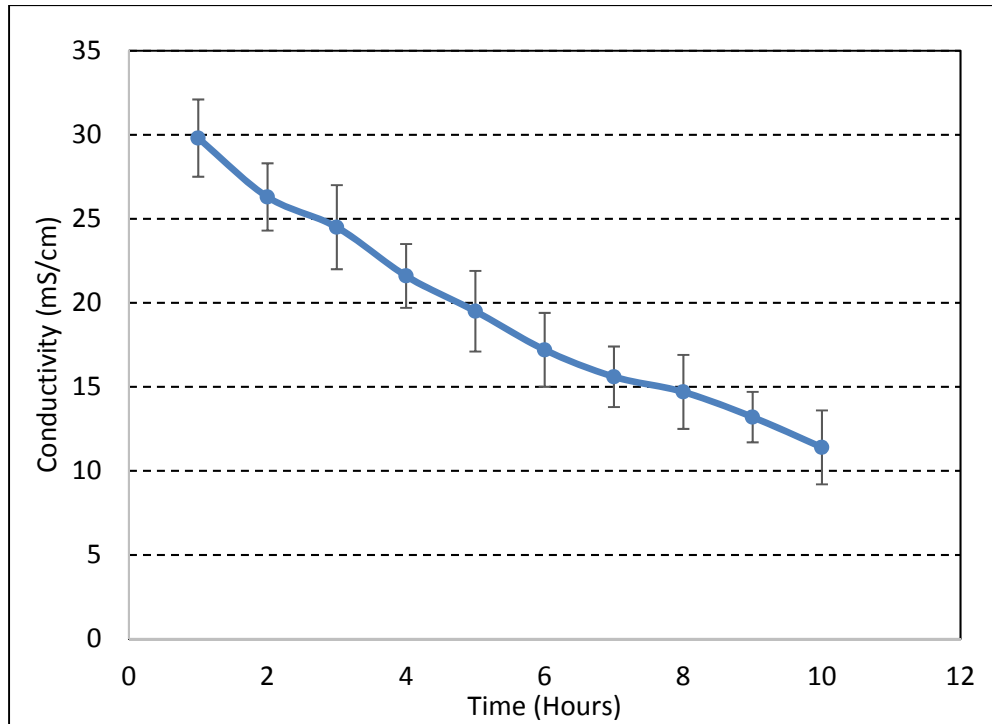


Fig 20: Conductivity against time graph for synthetic water and 0.5M DS

The figure 20 shows that initial value of conductivity is 30 mS/cm which is greater than that of 0.25M draw solution concentration because the number of conductive ions per unit volume of water was higher. With the passage of hours the conductivity was decreased drastically. So the final value is 11 mS/cm (approx.) which shows that the decrease in conductivity can be achieved greatly with higher molar concentration as the number of conductive ions per unit volume of water is decreased.

4.3 Effect on Chemical Oxygen Demand (COD) removal

Chemical Oxygen Demand or COD is a measurement of the oxygen required to oxidize soluble and particulate organic matter in water. Chemical Oxygen Demand is an important water quality parameter because, similar to BOD, it provides an index to assess the effect discharged wastewater will have on the receiving environment. Higher COD levels mean a greater amount of oxidizable organic material in the sample, which will reduce dissolved oxygen (DO) levels. So the removal of COD can be seen from the figure 21.

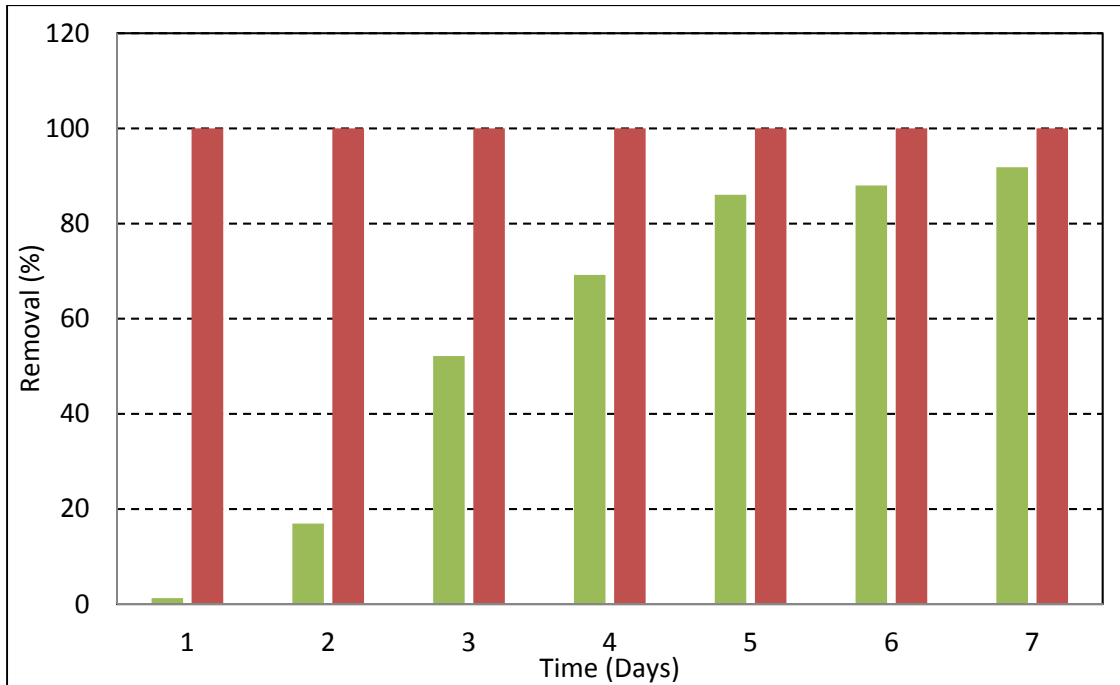


Fig 21: Percentage COD removal against time graph

The average influent COD was 3000 mg/l but with the passage of days (after 7 days) the COD was decreased to 91.8 and the final value was 246 mg/l which is suitable for fertigation. The value of COD was decreased as the anaerobic bacteria present in the sludge in membrane bio reactor utilized the organic matter present in the waste water.

4.4 Effect on Total phosphorous (TP) Concentration

The total phosphorous concentration test is also important as it tells about the dilutions needed to bring the phosphorous level to a suitable range in draw solution so that a particular plant can use it when applied. The figure 22 below shows:

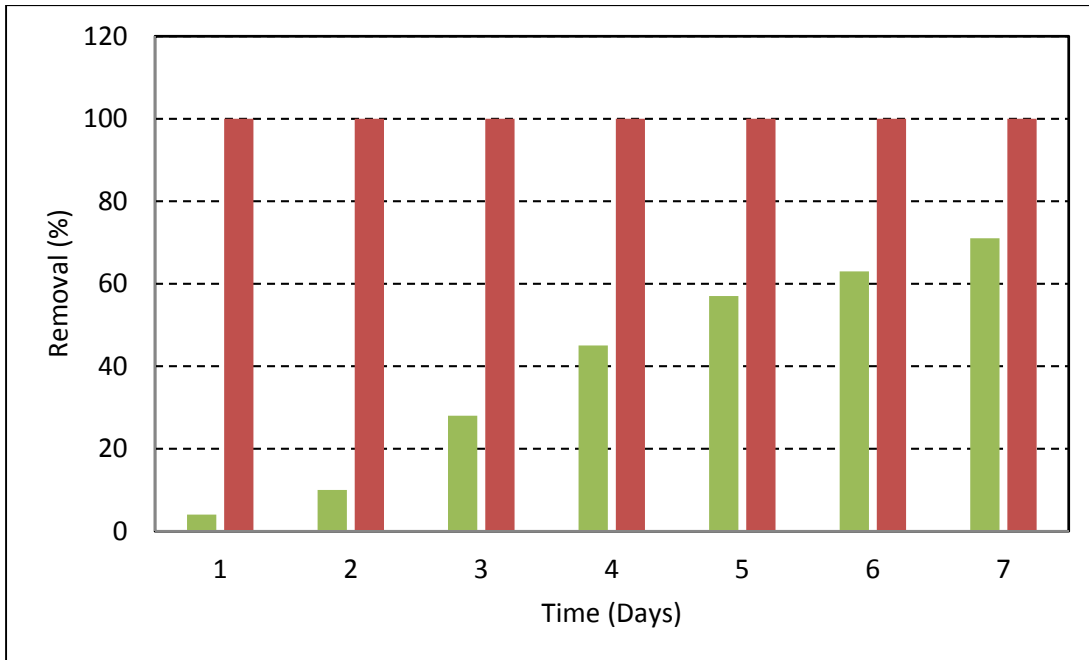


Fig 22: Percentage TP removal against time graph

The graph shows that initial value of TP was 865.4 mg/l but after further dilutions its final value was 70.9 mg/l which is in a suitable range in a draw solution to be applied for fertigation. It should be kept in mind that this phosphorous concentration is the concentration present in the draw side as draw solute (Mono Ammonium Phosphate) contains phosphorous content. No phosphorous is coming from the feed side.

4.5 Effect on Total Kjeldahl Nitrogen (TKN) Concentration

It is the concentration of organic nitrogen and ammonia present. It is very important to give nutrients and ammonia to plants in a suitable for their proper growth and strength. So:

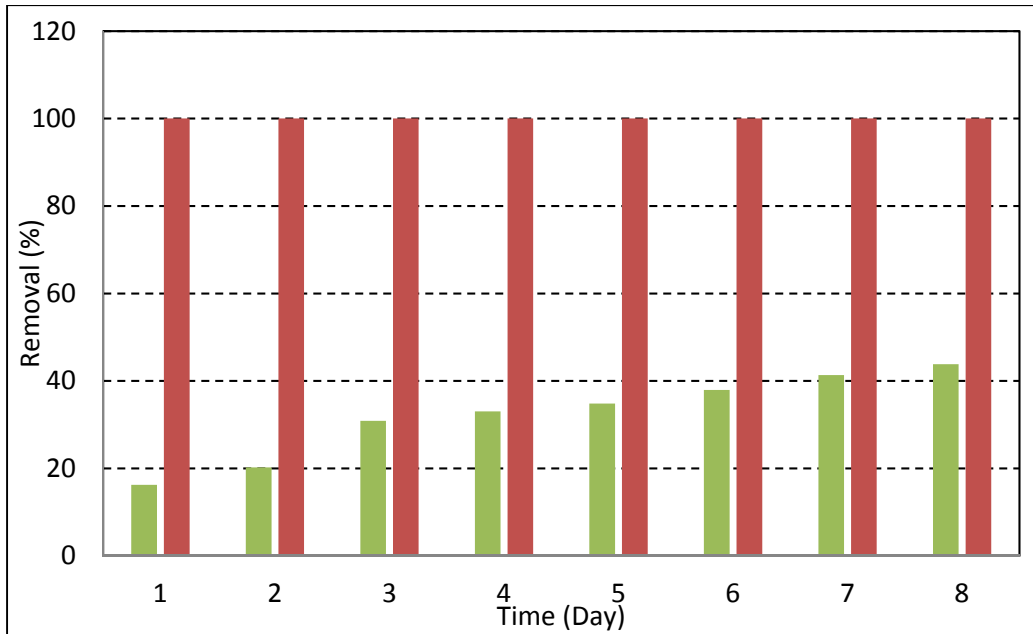


Fig 23: Percentage TKN removal against time graph

This graph shows that initial value was 615.6 mg/l but after 8 days due to dilutions the final value was 345.8 mg/l. it is very important to keep in mind that this is also the Total Kjeldahl Nitrogen present in Draw solution which is needed to be diluted to keep in suitable range so that it can be applied for respective fertigation.

Although high removal rates can be achieved during long-term operations, the final diluted DS still require dilution before meeting the nutrient standard requirements for hydroponics. Where hydroponics are the method of growing plants without soil using mineral nutrient solution in a water solvent.

Table 5: Dilution requirements for crops/fruit fields

Crops/Fruit Fields	Nitrogen Requirement (mg/L)	Dilution Required (Times)
Hydroponic Tomato	190	1.82
Rice	168.28	2.05
Potato	131.75	2.62
Cucumber	200	1.73
Pepper	190	1.82
Strawberry	50	6.916
Melon	200	1.73

4.6 Effect on Color Removal

It has been observed that after the run an approximate 100 percent color removal was obtained as the dyes used in this process are Cibracon Yellow and Methylene Blue which are eco-friendly and organic dyes. These dyes are consumed by anaerobic bacteria present in sludge present in Membrane Bio-Reactor and also as the molecules of these dyes are greater than that of the diameter allows passing through the membrane so as a result they are retained back, giving colorless water. The figure shows the change in color.

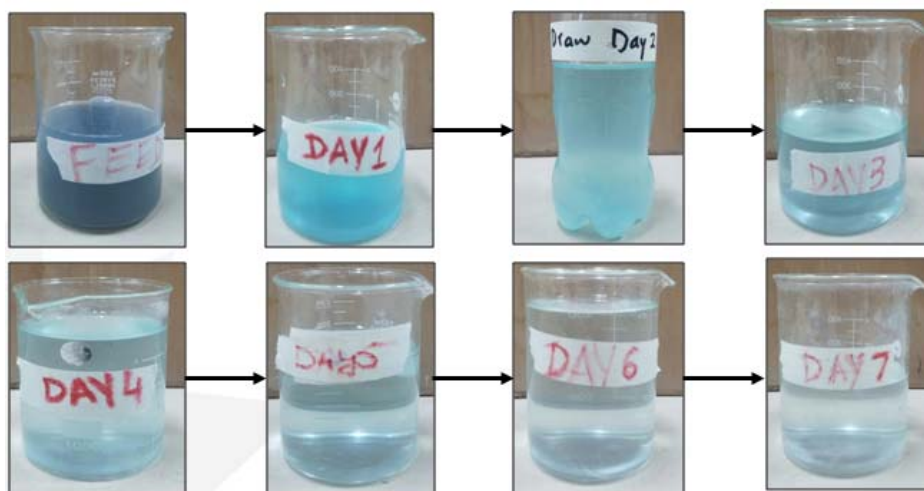


Figure 24: Gradual color removal over the 7-day run

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study investigated the potential of the FDFO process coupled with Anaerobic MBR to achieve simultaneous water treatment of high-strength industrial wastewater and sustainable agricultural application via fertigation techniques. The 0.5M Ammonium Phosphate Draw Solution resulted in considerable flux however the flux declined over the period of days. The membrane required chemical and physical cleaning to increase flux. The performance of the system was measured in terms of COD Removal, TP and TKN concentrations. The final concentrations were compared to standard requirements for crops and hydroponics. However, the diluted Draw Solution at the end of the run of experiments required more dilution in order to meet the standard nutrient requirements for crops and hydroponics.

5.2 Recommendations

Anaerobic FDFO MBR is a favorable novel technology for wastewater recovery. Forward Osmosis has the potential to treat textile wastewater; as this experimentation showed high removal rates of COD, TKN and TP using MAP fertilizer as draw solutions and making water reusable for direct fertigation. Fertilizers showed good performance in terms of high Flux and low RSF. Limitations lie in the further dilution of Fertilizer for direct application, and concentrating the feed stream. For that, here are some prospects to be researched in future:

- Use of blended fertilizers as DS
- Trying treated FDFO water for hydroponics and testing plant growth dynamics
- Development in FO Membrane technologies
- Identification of efficient draw solutes with economic feasibility
- Upscaling Laboratory Scale to Pilot Scale Plant
- Research about Concentrate management techniques

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