Decentralized Wastewater Treatment System (DEWATS or DWTS) for Medium Strength Domestic Wastewater

A Thesis submitted in partial fulfilment of the requirements for the degree

of

Masters of Science

in

Environmental Engineering

By

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(2019)

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ACKNOWLEDGEMENTS

I'm really grateful to the universal Creator **Allah Almighty** for giving me the strength to complete this thesis along with a hectic job routine. Ample amount of respect and regard for **Hazrat Muhammad (SAWW)** who provided us with his Sunnah and by ensuing those principles, Alhamdulillah today I'm capable of doing the right things in my life.

I'm really thankful to my most competent supervisor **Dr. Sher Jamal Khan** who believed in me and gave me this opportunity to work under his supervision. It really is an honor for me to work under such great and competent person. Speaking of competent personalities, I would like to express my gratitude towards my GEC members, **Dr. Zeshan** and **Dr. Yousuf Jamal**. These great personalities are not just my GEC members but my colleagues too and they have provided a very friendly and competent environment for me to work. Each time I got stuck in in my research and needed a helping hand, they helped me out.

Besides my supervisor and GEC, I really appreciate the guidance received from all of my teachers specially **Dr. Imran Hashmi**, **Dr. Muhammad Arshad**, **Dr. Zeeshan Ali Khan** and **Engr. Rashid Iftikhar**. These great teachers consistently pushed me to my best efforts for completing this research.

I would like to extend my sincere gratitude towards **WaterAid Pakistan** and **NUST** for funding my research. Without their generous financial support, this research could not be accomplished.

The support shown by my friends could not be neglected during my research including **Bilal Ahmad**, **Sabtain Ali**, **Alia Aslam**, **Tabish Khan**, **Muhammad Adnan**, **Muhammad Ali**, **Surraya Malik**, **Aamir Shahzad** and rest of my MBR family. Besides these, I'm also indebted to **Aitzaz Ahsan Malik**, **Muhammad Ahmad** and **Muhammad Arslan**, my UG class fellows and my best buddies who constantly pushed me to complete my thesis in time. Special Thanks to **Mehwish Khalid** for helping me out with the microbial analysis.

Lastly, I would like to acknowledge my gracious **Father**, loving **Mother** and kindhearted **Sister** for their moral support and prayers.

Engr. Aamir Khan

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LIST OF ABBREVIATIONS

ABR	Anaerobic Baffled Reactor
AF	Anaerobic Filter
NEQs	National Environmental Quality Standards
WHO	World Health Organization
SDGs	Sustainable Development Goals
DEWATS	Decentralized Wastewater Treatment System
TMP	Trans-Membrane Pressure
COD	Chemical Oxygen Demand
TKN	Total Kjeldahl Nitrogen
ORP	Oxidation-Reduction Potential
HRT	Hydraulic Retention Time
SRT	Solids Retention Time
NUST	National University of Sciences & Technology, Islamabad
PVC	Poly Vinyl Chloride
MLSS	Mixed Liquor Suspended Solids
LMH	Liters per squared meter per hour (L/m^2-h)

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ABSTRACT

Wastewater collection and treatment has always been a challenge for developing countries because of lack of infrastructure for transport and treatment. Most of the areas in developing countries lack proper sanitation facilities and sewer network due to which centralized treatment become impossible. In such cases, Decentralized systems may be installed to treat the water on site. In this study, performance of Decentralized Wastewater Treatment System (DWTS) was studied. The lab scale DWTS comprised of Anaerobic Baffled Reactor followed by Anaerobic Filter and finally effluent was filtered through Woven Fiber Micro Filtration (WFMF) membrane. ABR was run at 3 different HRTs i.e. 8, 10 and 12 hours. During the whole run of around 8 months, the average COD removal efficiency in ABR was found to be 65, 68 and 72% at HRT of 8, 10 and 12 hours, respectively. AF was run with 4 different media i.e. Kaldnes K1, PVC pipe 25 mm length, PVC pipe 20 mm length and PVC pipe 15 mm length. The diameter of the PVC pipe was 15 mm. The average COD removal efficiencies in Kaldnes, PVC 25, PVC 20 and PVC 15 were found to be 86.5, 84, 85 and 86%, respectively. The membrane reactor was run with Microfiltration Woven Fiber (WFMF) membrane with nominal pore size of 1-3microns. The average COD and Turbidity removal efficiency shown by this whole system was 96 and 98%, respectively. This membrane system ran successfully for 37 days showing a gradual increase in TMP with the passage of time. On 37^{th} day of run, the membrane reached the TMP of 20kPa, an indication of a fouled membrane.

Chapter 1

INTRODUCTION

1.1 Background

The natural resources of the world are depleting at a very faster rate. Utilization of these resources by humans as well as other beings is at peak but replenishing those valuable assets is a task of least importance to us. One of the major resource of concern to an Environmentalist is Water. Although, we have plenty of water on our planet Earth but major portion of that water is not available for direct consumption. We are excessively extracting groundwater for all our needs and then dispose-off that utilized water directly without any treatment which again contributes to pollution of the surface water bodies. Around 80% of the water that we use for our routine activities become wastewater (Renuka et al., 2016).

Pakistan like many other developing countries face problems of water quality due to agricultural, industrial and domestic discharges. The per capita water availability is estimated at around 700 m^3 in the year 2025 which will be an alarming situation as this figure is far below the international standard of 1000 m^3/capita (Ali et al., 2018). Pakistan Environmental Protection Agency (EPA) has set standards for discharge of wastewater into different water bodies known as National Environmental Quality Standards (NEQs), which needs to be religiously followed by industrialists, municipal committees and other related organizations.

Municipal sewage is one of the major contributors towards polluting surface water bodies. Around 7.57×10^6 m^3 of municipal sewage is discharged daily into surface water bodies and only 10% of this gets treated in different treatment plants which again is an alarming situation (Ali et al., 2018).

To cope with this water scarcity issue, we need to focus our research on utilization of the generated wastewater for different activities so that the fresh water reservoirs could be saved and utilized sustainably. For wastewater treatment, different technologies are being used worldwide. They are broadly classified into Biological Treatment and Physico-Chemical Treatment. The Biological treatment technologies are more wide spread due to its efficiency in removal of organic contaminants. These Biological technologies are again classified into Aerobic Treatment and Anaerobic Treatment.

Aerobic technologies are the primitive ones and utilize oxygen for converting organic matter into CO_2 and H_2O. Whereas, Anaerobic technologies do not require oxygen for degradation of the organic matter present in incoming feed water streams and also produce biogas during organics degradation. The complete description of these processes is being explained in the next chapter.

Anaerobic Baffled Reactor (ABR) was introduced and developed by Bachmann and McCarty at Stanford University to treat high strength wastewater (Li et al., 2015). These reactors consists of a series of baffles for ensuring maximum contact of acclimatized sludge with the incoming feed and guaranteeing longer HRTs as well. The water while passing through these baffles up and down gets treated and biogas is generated in each compartment which can be collected by installing valves above each compartment. These reactors consist of 3 different zones namely sludge zone, fluidized zone and settling zone. The biggest advantage of these reactors is that they can be made completely under gravitational flow where land topography allows it which means no operational cost for treatment of wastewater. The only thing that need to be assured while installing it underground is the ground water table. These reactors also have longer SRTs which means lesser amount of sludge production and another big advantage is that the design is very simple, no complexities are involved.

The performance of these reactors is further strengthened by adding additional treatment units to this system. These systems include Settler, Anaerobic Filter and Wetlands. The settler is utilized before ABR to remove the excess sludge from incoming wastewater. The HRT of settler is usually 2 hours. Anaerobic filters are utilized after ABR for further biological degradation of the organic matter through attached growth process. In the end, the water is polished in constructed wetlands to further reduce organic load and minimize odor problems.

1.2 Problem statement

Pakistan, as discussed earlier is facing acute water shortage and the water that is available for potable use also is unfit for drinking because of the poor sanitation practices. There are many rural areas that don’t have a collection system for domestic or municipal wastewater due to which the wastewater flows in their streets and the same trickles down to groundwater reservoirs. The water supply networks are also not

monitored regularly due to which if there is some sort of leakage in the distribution line, the trickling wastewater will get added to, causing severe health issues.

Our target area for this research was Jatoi city in district Muzaffargarh of the southern Punjab. Upon inspection of the target area (Ward 12 of Jatoi City), it was found that there are severe sewage related issues faced by the locals as the existing collection system is broken intermittently for carrying domestic sewage away and definitely no treatment at all. The municipal wastewater of the area was tested at NUST Islamabad and was found highly contaminated as the average COD was found to be 500 mg/L.

The case is not just for Jatoi but there are other rural areas in Southern Punjab and KPK where the situation is similar. Their raw wastewater flows into their streets which pose serious health problems.

1.3 Objectives

- Establishment of lab scale ABR followed by AF and finally WFMF membrane system
- Optimization of HRT of ABR
- Comparison of treatment performance of conventional Kaldnes media with locally available PVC media
- Polishing the effluent of AF through Flat Sheet Woven Fiber Micro-Filtration (WFMF) membrane

1.4 Scope of the study

This research is focused on decentralized systems for domestic wastewater treatment. The system was run on synthetic wastewater of same composition as was collected from site where a full scale plant is to be constructed i.e. Ward No. 12 of Jatoi City.

Anaerobic Baffled Reactor (ABR) was first implied to treat the incoming wastewater at an HRT of 8, 10 and 12 hours. The treated water was then fed into 2 parallel Anaerobic Filters and finally polished through a Woven Fiber Micro-filtration (WFMF) flat sheet membrane.

In the 1st phase, treatment performance of Kaldnes media was compared with PVC media of 15 mm diameter and 25 mm length.

In the 2nd phase, treatment performance of 20 mm length of PVC was compared with 15 mm length to determine the optimized media size for use in full scale plant.

In the last phase, the influent to membrane unit and its permeate were tested for overall COD and Turbidity removal efficiencies. The TMP of the membrane system was regularly monitored to determine fouling rate of the membrane.

CHAPTER 2

LITERATURE REVIEW

2.1 Domestic wastewater challenges and production

Pakistan has recently become a water deficit nation due to exhausting ground and surface water reservoirs, predominant drought conditions and shifting of fresh water to more persistent domestic as well as industrial uses. Therefore, hunt for non-conventional water resources for irrigation and other uses has become area of prime focus. These other sources include wastewater which is already generated in ample amount across the country. At current, around 40.5 MAF of groundwater is being pumped yearly and around 36% of the groundwater is classified as highly saline and around 60-80% of the water as saline. The annual per capita water availability has decreased from 1,299m^3 to 1,100m^3 in 2006. It is further projected that the water availability will be decreased further leaving less than 700m^3 per capita by 2025 (Murtaza & Zia, 2012).

According to recent studies, the total annual volume of wastewater produced in Pakistan is 962,335 million gallons which include 674,009 million gallons from domestic and 288,326 million gallons from industrial facilities. The amount of wastewater discharged directly to the major rivers is 392,511 million gallons that include 316,740 million gallons of municipal sewage and 75,771 million gallons of industrial effluents (Murtaza & Zia, 2012). It has also been estimated that around 2,000 million gallons of untreated wastewater is being discharged into local surface water bodies every day. Though, there are some sewerage collection systems carrying the domestic discharge to the nearest water bodies but the collection is only 50% over the country and only 10% of the sewage gets treated before it gets discharged into water bodies (Martin, 2006).

2.2 Domestic wastewater composition

Domestic wastewater usually contain 99.9% water and only 0.1% of solids which include microbes, organic and inorganic suspended plus dissolved solids (Sperling, 2007). Out of these solids, around 30% are inorganic solids while the rest of 70% are organic solids that include Proteins, Carbohydrates and Fats.

Table 1: Typical Range of Pollutants in Raw Domestic Sewage

S. No.	Parameter	Concentration Range (mg/L)
1.	Total Suspended Solids	100 - 350
2.	Biological Oxygen Demand	100 - 400
3.	Chemical Oxygen Demand	250 - 1000
4.	Total Phosphorus	4 - 15
5.	Total Nitrogen	20 - 100

(Dionisi, 2017)

Municipal Wastewater is generally classified into 4 categories.

- **Gray Water**

  Wastewater from kitchen and bathroom sinks. It also include washing water from laundry but do not include urine or faeces.

- **Black Water**

  Water from toilets with urine and faeces

- **Yellow Water**

  Wastewater from separate toilets having only urine

- **Brown Water**

  Wastewater containing only faeces. Very difficult to collect as urine and faeces are usually discharged together.

2.3 Strength of wastewater

The composition of typical municipal wastewater is shown where concentrated wastewater represent cases with very less water consumption. Diluted wastewater on the other hand represents high water consumption. Stormwater if added will further dilute the wastewater as stormwater has lesser pollutant concentrations.

Table 2: Typical composition of Raw Municipal wastewater with minor contributions of Industrial effluents

S. No.	Parameter	Concentration Range for different Strengths (mg/L)		
		Low	**Medium**	**High**
1.	Total COD	500	750	1200
2.	Particulate COD	300	450	720
3.	Soluble COD	200	300	480
4.	BOD	230	350	560
5.	Total Nitrogen	30	60	100
6.	Ammonia Nitrogen	20	45	75
7.	VFAs	10	30	80
8.	Total Phosphorus	6	15	25
9.	Orthophosphates-P	4	10	15
10.	TSS	250	400	600
11.	VSS	200	320	480

(Henze & Comeau, 2008)

2.4 Impact of wastewater on surface and ground water

Raw wastewater usually contain many substances that can cause severe pollution in the environment and can also become a cause in the negative effects on human health. There are two different types of contaminant effect that you may encounter while dealing with wastewater. The first one is Toxicity, which is the poisoning caused by metals and organic pollutants and the second one is oxygen depletion.

As the untreated water contain different pathogenic micro-organisms, these waters when percolate down to the groundwater becomes part of it and thus polluting the whole aquifer. People when use such polluted water become affected by different water borne diseases caused by those microbes. The organics and heavy metals on the other hand also cause severe health problems like neural disorders, disorders of the digestive system etc.

The organics in the wastewater are a major cause of DO depletion in receiving surface water bodies and the reduced concentration of DO causes death of aquatic life like fishes.

2.5 Wastewater management strategies

Wastewater Management Strategies can be classified into Centralized and Decentralized systems

2.5.1 Centralized system

Centralized wastewater management contains a single centralized collection system of sewer lines which collect all the wastewater from houses, commercial structures & areas, industrial plants and establishments, recreational areas and transports it to a centralized wastewater treatment plant located in an off-site location away from the settlements and disposes of the treated water far away from the point of origin. Thus, this system is also called as Off-site management system.

The centralized strategy has been proved to be the conventional wastewater management approach in the past. It was proven to be relatively efficient in wastewater collection, treatment and pollution control. However, such conventional systems, and especially the collection system and the energy intensive treatment technologies used in such system, need skilled labor, large quantities of capital, and steady socio-economic situations. Huge amount of capital is required for constructing large network of sewers for collection and transport of all sewage to an off-site location. All these make it problematic and in many cases not beneficial, particularly in low population density areas to apply this strategy for wastewater treatment (Hophmayer-Tokich, 2006).

2.5.2 Decentralized system

Decentralized wastewater management is a system in which sewage is collected, treated and disposed or reused near the point of generation. It is hence referred to as on-site management of wastewater. The attention in such technology was redeveloped as it became obvious that the centralized system is not practicable in many regions, or simply not cost-effective alternative in some cases. Due to its high construction cost and complexity of construction, operation and maintenance, or the fact that they need high water consumption, centralized systems may be less appropriate for areas such as

low-income areas and rural areas with low population density, water-scarce areas and areas with unreliable water supply system (Hophmayer-Tokich, 2006).

The decentralized approach can be easily applied on different scales. It can be applied for/to

- Individual households
- A cluster of households
- A neighborhood
- Public facilities
- Commercial area
- Industrial parks
- Small portions of large communities

2.6 Technologies for wastewater treatment

For wastewater treatment, different technologies are implemented worldwide. These technologies are classified into Physical methods, Chemical methods and Biological methods.

2.6.1 Physical methods

In these methods, there isn't any chemical change in the properties of the pollutants. The contaminants are separated using mechanical structures and in some cases with the help of gravity. These include

2.6.1.1 Screening

A screen is a device with small pre-determined openings of uniform size which is used to help retain the particles that are larger in size than the screen opening size. Sometimes, combination of screens are used which include installing a Bar screen first and then a Mesh screen of smaller opening size.

2.6.1.2 Communitors

These are the devices that are used to only shred the solid particles. When installed before a screen they can shred the solids to a size of 6 to 20 mm.

2.6.1.3 Grit chambers

Grit means solids like sand, gravel, heavy solids or cinders having specific gravity greater than the organic solids. Grit can be thus easily removed from wastewater influents with very little hydraulic retention time, usually few minutes.

2.6.1.4 Primary clarifiers

These are the sedimentation tanks used for removal of inorganic larger solids through gravitational force. Wastewater is fed into Primary clarifier or sedimentation tank where it is kept undisturbed for approximately 1.5-2hrs. During this time, the solids are settled down and the suspended organics move out with the supernatant for further treatment.

2.6.2 Biological processes

There are usually four different type of contaminants in wastewater bodies. These are soluble organic matter, insoluble organic matter, soluble inorganic matter and insoluble inorganic matter. The insoluble inorganic matter is usually removed by preliminary physical treatment because microbes are unable to transform such matter.

In biological processes, microbes are responsible for degradation and breakdown of different organic pollutants with the help of nutrient uptake. The microbes use the organics as a food source, converting a portion of the carbon matter into new biomass and the remainder of the carbon into carbon dioxide. The CO_2 thus produced is released as a gas and the biomass produced is removed by liquid-solid separation, leaving the wastewater free from the original organic matter. This process can either occur in the absence of oxygen or in the presence of oxygen. If oxygen is used by microbes for conversion, the process is called aerobic digestion and if no oxygen is involved, the process is termed anaerobic digestion. Anaerobic processes are practicable when the strength of wastewater is insignificantly high (C. P. Leslie Grady et al., 2011).

At low concentrations, carbon adsorption is more economical, although biochemical operations are used for treatment of polluted groundwater that contain less than 50 mg/L of COD. Even though, they must be followed by aerobic cultures to provide an effluent appropriate for discharge. Anaerobic cultures are often used for high strength wastewaters because they do not require oxygen, give less biomass because of longer SRT and produce methane gas as a usable product. If the COD concentration is above

50,000 mg/L, then evaporation and incineration may be more cost-effective. Anaerobic cultures are also used to treat wastewaters of modest strength (down to about 1000 mg/L as COD), and have been proposed for use with dilute wastewaters as well (C. P. Leslie Grady et al., 2011).

2.6.2.1 Aerobic treatment technologies

Aerobic cultures of microbes are mainly suitable for the removal of organic matter in the concentration range between 50 and 4000 mg/L as biodegradable chemical oxygen demand. The utmost significant characteristic of the environment in which microbes grow is the terminal electron acceptor they remove as they oxidize chemicals to obtain energy. There are 3 major sorts of electron acceptors: oxygen, inorganic compounds and organic compounds. If dissolved oxygen is supplied in adequate quantity, the environment is considered to be aerobic. Growth is generally most effective in this environment and the amount of biomass formed per unit of waste stabilized is high.

Wastewater treatment in bioreactors fall into 2 major types, depending upon the way in which microorganisms grow in them: suspended in the liquid or attached to a solid support. When suspended growth cultures are used, adequate mixing is needed to keep the activated biomass in suspension and some form of physical unit operation, such as sedimentation or membrane filtration is required to remove the excess biomass from the treated effluent preceding discharge. In dissimilarity, attached growth cultures grow as a biofilm on a solid support and the water being treated flows past the media. However, because micro-organisms can slough from the support media, a physical unit operation is usually required prior to discharge.

2.6.2.1.1 Activated Sludge Process (ASP)

In this process, adequate concentration of active biomass need to be maintained in the oxidation ditch or aeration basin for microbes to degrade organic pollutants. There are 4 important factors common to all activated sludge processes.

1) A flocculent slurry of microbes (mixed liquor suspended solids or MLSS) is used to remove soluble and particulate organic matter from the influent stream

2) Liquid-solid separation is adopted to remove the MLSS from the process flow stream, producing effluent having lower suspended solids concentration

3) Concentrated solids are recycled back from the liquid-solid separator (clarifier/sedimentation tank) back to the bioreactor and is known as Returned Activated Sludge (RAS)

4) Excess solids are wasted to control the solids retention time (SRT) to a favorable value. These solids are known as Waste Activated Sludge (WAS)

The term MLSS is used to signify the microbial slurry as it is a mixture of microorganisms, undegraded particulate substrate and inert solids (C. P. Leslie Grady et al., 2011).

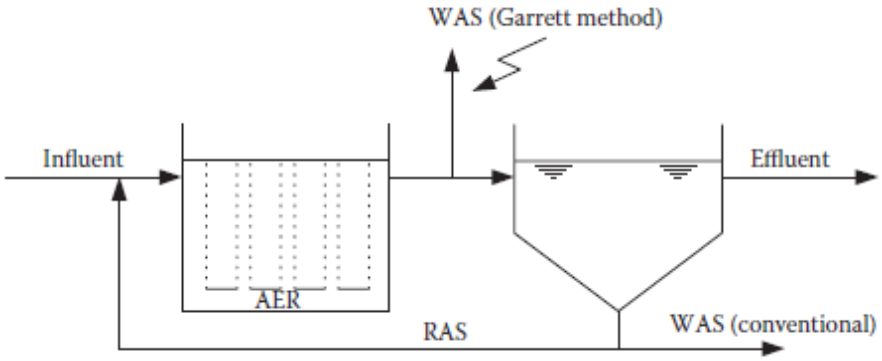

Figure 2-1: Activated Sludge System

2.6.2.1.2 Trickling Filters

Operation of Trickling filters involve spraying wastewater over solid media such as plastic, rock, or redwood slats. As the wastewater trickles down over the surface of the media, a layer of microorganisms develops on the surface of the media known as Bio-film. This growth is noticeable as a shiny slime layer. As wastewater passes over this slimy layer, the organic matter gets absorbed. The organic matter is utilized for food by the microorganisms. At the same time, air passing through the open spaces in the trickling filter transfers oxygen. This oxygen is then transported to the slime to keep the outer layer aerobic. As the microbes use the food and oxygen, they produce more organisms, sulfates, carbon dioxide, nitrates and other stable by-products. These constituents are then discarded from the slime back into the wastewater flow and are transported out of the filter (Spellman, 2009).

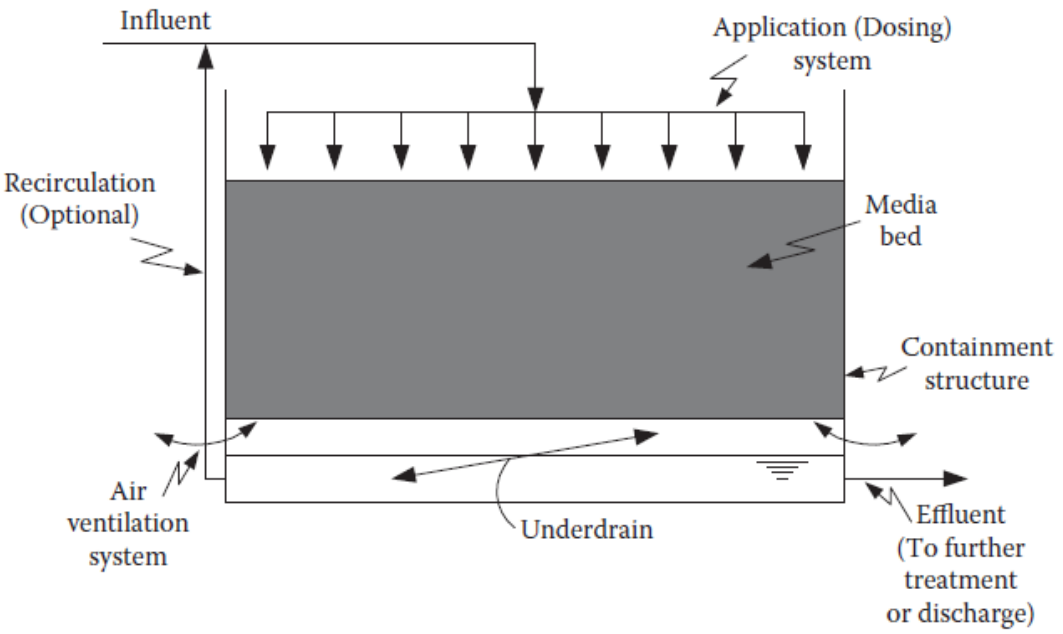

Figure 2-2: Trickling Filter (C. P. Leslie Grady et al., 2011)

2.6.2.1.3 Rotating Biological Contactors (RBCS)

RBCs consists of a series of circular plastic disks mounted side by side and closely set apart. They are typically around 11.5 ft in diameter, attached to a horizontal rotating shaft, approximately 40% submerged in a reactor that contains the influent to be treated. As the shaft rotates, the attached biomass on the surface of the disks moves into and out of the wastewater. While submerged in the wastewater, the microbes absorb organics from the feed water and when they are rotated out of the wastewater and comes into air, they are supplied with desirable oxygen for aerobic decomposition. As the slime returns to the wastewater, excess solids and waste products are stripped off the RBC media as sloughings. These sloughings are transported with the wastewater flow to a sedimentation tank for removal (Spellman, 2009).

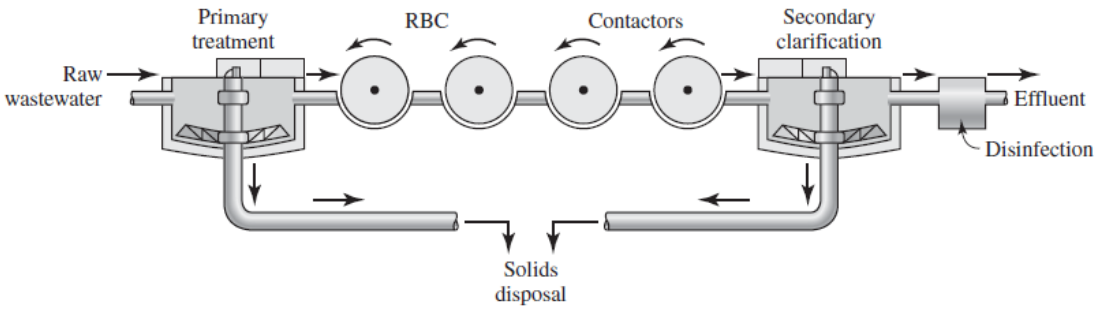

Figure 2-3: Rotating Biological Contactors (Davis, 2010)

2.6.2.2 Anaerobic treatment technologies

The anaerobic treatment of wastewater and sludge involves three distinct stages. In the first stage, complex waste components, including fats, proteins, and polysaccharides, are hydrolyzed to their smaller sub-components or subunits. This process is accomplished by a heterogeneous group of facultative and anaerobic bacteria. The bacteria then subject the products of hydrolysis (triglycerides, fatty acids, amino acids and sugars) to fermentation leading to the formation of simple organic compounds and hydrogen production in a process called acidogenesis or acetogenesis. The organic compounds formed are mainly short-chain volatile acids and alcohols. The second stage is acid fermentation. In this stage, organic matter is simply converted to organic acids, alcohols and new bacterial cells, so that slight stabilization of BOD or COD is realized. In the third stage, the end products of the 2^{nd} stage are converted to gases (mainly methane and carbon dioxide) by several species of strictly anaerobic bacteria. The primary acid produced during the acid fermentation stage is acetic acid (Davis, 2010).

The microbes accountable for hydrolysis and acid fermentation include facultative and obligate anaerobic bacteria. Examples of genera found in anaerobic reactors include *Clostridium, Corynebacterium, Actinomyces, Staphylococcus* and *Escherichia*.

The microorganisms accountable for methane fermentation are strictly obligate anaerobes. Examples of genera found in digesters include *Methanosarcina, Methanothrix, Methanococcus, Methanobacterium* and *Methanobacillus*. The first two genera are able to utilize acetate for production of methane and carbon dioxide. The others oxidize hydrogen with carbon dioxide as the electron acceptor (Davis, 2010).

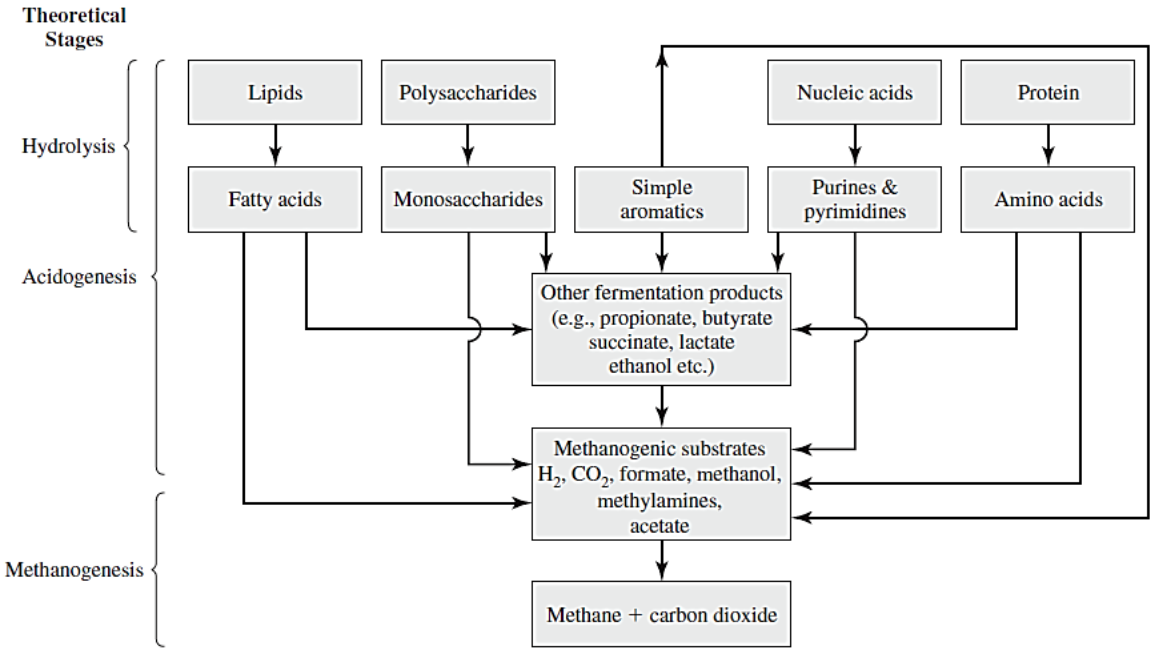

Figure 2-4: Stages of Anaerobic Digestion (Davis, 2010)

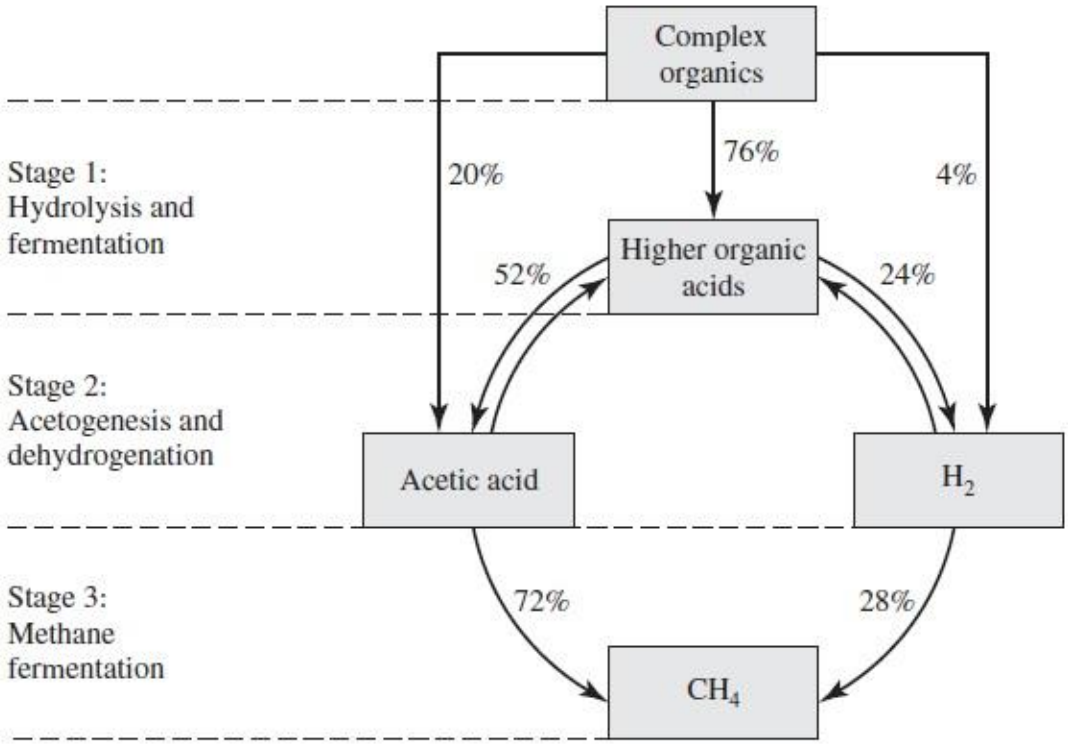

Figure 2-5: Anaerobic Digestion process with Energy flow (Davis, 2010)

2.6.2.2.1 Up-flow Anaerobic Sludge Blanket (UASB) reactor

The UASB process uses biomass suspended in the reactor, but the gas-liquid-solids separation system is vital with the bioreactor. More importantly, the conditions developed in the bioreactor can result in the formation of large, dense, easily settleable particles called granules, which allow high concentrations of suspended solids in the range of 20 to 30 g/L as VSS to be accumulated (C. P. Leslie Grady et al., 2011).

In this process, wastewater influent enters from the bottom of the reactor through a uniform distribution system that is intended to provide relatively uniform flow across the cross section. A thick slurry of granules is formed in the bottom of the bioreactor, and the combined effect of the influent distribution and gas production results in mixing of the influent with the dense slurry of granules. Treatment or degradation of organics occurs within the dense blanket of sludge granules. For some wastewaters, a less dense flocculent sludge also develops which accumulates on the top of blanket of granules provided the up-flow velocity is insufficient to transport it. Other wastewaters contain suspended solids that are not captured in the granular sludge and these solids also accumulates as a flocculent sludge blanket over the granules. Treated effluent exits the granular and flocculent sludge zones and flows towards top into the gas-liquid-solids separator. The top of the reactor consists of a gas collection hood with a settler section overhead. Gas bubbles and the upward flowing liquid cause the granular and flocculent solids (mostly small granules) to rise through the bioreactor and enter the gas-liquid-solids separator. Gas separation occurs in the hood, thereby letting some of these suspended solids to return directly to the sludge blanket. Gas is collected in the upper inverted V section of the hood and is removed from the reactor. Liquid with some entrained solids flows out of the hood into the settler where liquid-solids separation occurs. Clarified effluent overflows the weirs while separated solids settle back into the reaction zone (C. P. Leslie Grady et al., 2011).

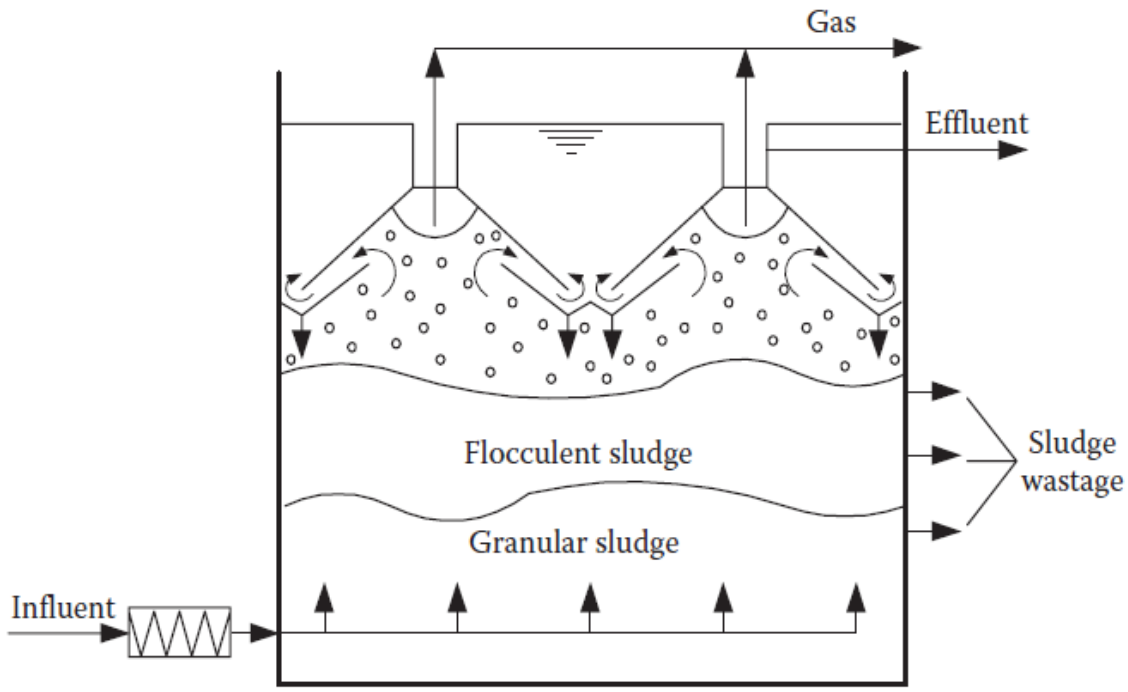

Figure 2-6: Upflow Anaerobic Sludge Blanket Reactor

2.6.2.2.2 Anaerobic Baffled Reactor

Anaerobic Baffled Reactors (ABRs) have a very simple design, low capital and operating costs and provide better retention of sludge/solids in response to load variations than a single up-flow anaerobic sludge blanket (UASB) reactor. ABRs often described as a series of up-flow anaerobic sludge blanket reactors (UASBs), direct influent through sequential cells/compartments under up-flow and down-flow conditions, each time passing through a blanket of sludge. Such configuration allows a naturally occurring spatial separation of the microbes that perform the sequential steps of hydrolysis, acidogenesis and methanogenesis in the transformation of complex organics to methane. Solids are well retained in the system, providing additional time for hydrolysis, which make it an excellent technology for colder climate anaerobic treatment and eliminating the need for primary clarification (Hahn & Figueroa, 2015).

Several microbial communities may get developed within each cell/compartment of the ABR due to its unique design. Generally, acidogenic bacteria will tend to dominate in the initial compartments of the ABR where substrate/organic concentrations are higher, whereas methanogenic bacteria will dominate the later compartments of the reactor. The development of these microbial communities are also dependent on the type and

amount of substrate available along with the pH and temperature of the system (Barber & Stuckey, 1999)

Previous studies related to hydrodynamics of ABR showed that low dead space (7 to 30%) occurred in ABR. This fraction of dead space of ABR was much lower than that of other currently used alternative high-rate anaerobic treatment systems like anaerobic filter (AF) and the up-flow anaerobic sludge blanket (UASB) reactor (Sarathai et al., 2010).

Recirculation of water may negatively impact the treatment performance of ABR as the reactor tends to become a completely stirred system and the benefits from phase separation are partially lost. More Intense mixing conditions also increase loss of solids, break the microstructures of microorganisms arranged in symbiotic relationships, as well as increase the amount of dead spaces within reactors. Nachaiyasit in 1995 observed a decrease in gas production rate in ABR when the rate of recirculation was increased (Vuitik et al., 2019).

Zabihollah Yousefi and his co-workers in 2018 studied the performance of ABR followed by AF for slaughter house wastewater treatment and found that at OLR of 7 & 10 kg COD/m^3/d and HRT of 18 hours had removal efficiencies of 83.29% and 85.79%, respectively. Whereas, AF reactor at OLR of 0.981, 0.576 & 0.561 kg COD/m^3/d and HRT of 36 hours, had removal efficiency of 79.39%, 74.09% and 63.14%, respectively. Concluding that the optimum HRT and OLR for ABR were 24 hours and 7 kg COD/m^3/d and for AF, HRT of 36 hours and OLR of 1 kg COD/m^3/d (Yousefi et al., 2018).

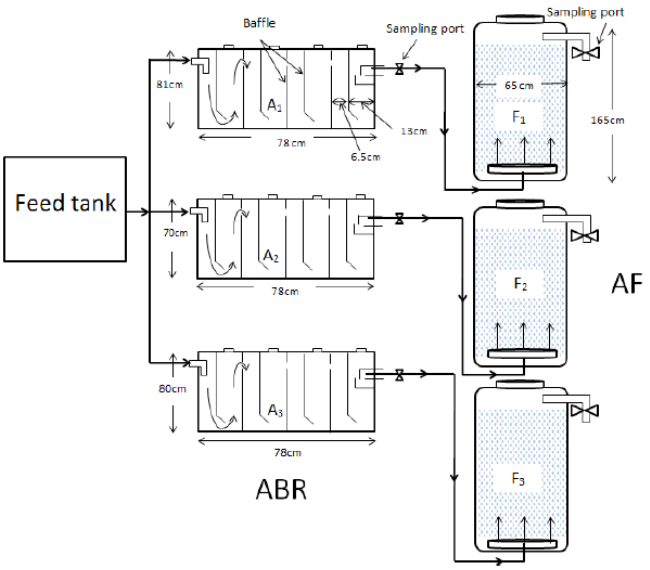

Figure 2-7: Experimental Setup used in the study of (Yousefi et al., 2018)

Another study conducted in 2015 for biologically enhanced treatment of raw municipal wastewater consisted of an ABR comprising of four sequential cells, each 0.457 m^2 and 1.22 m tall, with a total working volume of 869 L was used to treat raw municipal wastewater in 2015. The setup was operated at 12 hour HRT and the removal efficiency of TSS and BOD_5 were found to be 83 ± 10% and 47 ± 15%, respectively (Hahn & Figueroa, 2015).

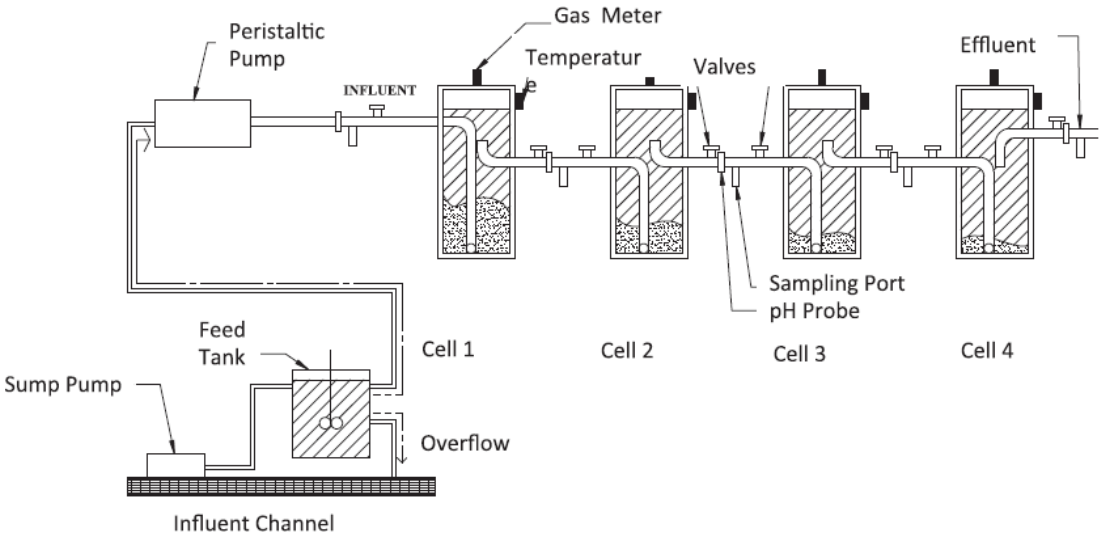

Figure 2-8: ABR used in the study of (Hahn & Figueroa, 2015)

The performance of ABR is further improved through additional polishing technique like MBR. In 2015, Chavalit Ratanatamskul and his co-workers studied biological nutrient removal for high-rise building wastewater recycling in ABR-MBR system. The total operational HRT of the ABR-MBR system was 3 hours (2 hour for ABR and 1 hour for aerobic MBR). The average permeate flux of the membrane during the study was kept at 30 L/m^2-h. The installed ABR-MBR system removed more than 90% of COD, total nitrogen, and total phosphorus from building wastewater at total operating HRT of only 3 hours (Ratanatamskul et al., 2015).

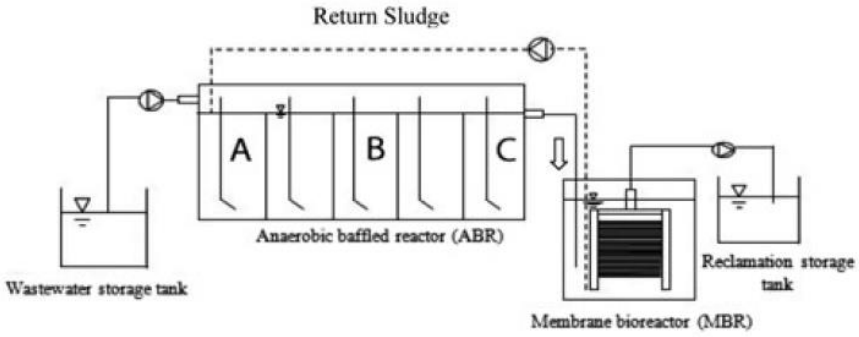

Figure 2-9: Experimental Setup for (Ratanatamskul et al., 2015)

Table 3: Experimental Results for the study

Parameter	Influent	Effluent	Removal (%)
Suspended Solids (mg/L)	72.3±12.7	0.12±0.63	99.1±0.15
COD (mg/L)	211 ± 14	7.63 ± 1.4	96.4 ± 0.69
TKN (mg/L)	56.7 ± 3.56	2.7 ± 0.24	95.2 ± 0.58
Nitrite (mg/L)	0.009 ± 0.004	0.054 ± 0.004	
Nitrate (mg/L)	0.25 ± 0.071	0.67 ± 0.023	
TP (mg/L)	5.26 ± 0.41	0.44 ± 0.003	91.4 ± 0.86

(Ratanatamskul et al., 2015)

Advantages of the ABR

- The major advantage of the ABR is that it can separate the various stages of anaerobic digestion process, specifically acidogenesis and methanogenesis, longitudinally down the ABR
- Highly efficient in treating medium strength soluble organic waste
- Simple design and inexpensive construction
- Long solid retention time (SRT), resulting in less sludge production

- Minimal clogging
- No mechanical mixing required

Disadvantages of the ABR

- Long start-up periods
- Further treatment of effluent due to reduced nutrient removal

2.6.2.2.3 Up-flow Anaerobic Filter

Anaerobic filter system use up-flow bioreactors that are filled with support media. The packing material is the same as that used with aerobic attached growth processes. The specific surface area is usually 100 m^2/m^3 with a void volume of 90 to 95%. The existence of packing media allows the growth of some attached biomass, but the principal role of the media is to retain suspended growth (C. P. Leslie Grady et al., 2011).

Influent wastewater is fed from bottom and distributed across the bioreactor cross section. It flows upward through the media to the top. Treatment occurs as a result of the suspended and fixed biomass retained by the filter media. Effluent exits from the top of the media section and is collected for discharge. Gas is collected under the bioreactor cover and is conveyed to subsequent use. Hydraulic retention times between 0.5 and 4 days are typical in such filters, along with volumetric organic load in the range of 5 to 15 kg COD/m^3·day (C. P. Leslie Grady et al., 2011).

The performance of AF was investigated in 2018 using 3 identical polyethylene (PE) cylinders as AF reactors (65 cm inner diameter and 165 cm height) with 300 L effective volume. Influent wastewater entered each reactor from the bottom (under the bed support media) through a T-inlet. Two-thirds of the reactor (around 100 cm) was filled with polyvinyl chloride (PVC) corrugated tubes (3 cm height and 1.5 cm in diameter). The reactor packing medium provided a specific surface area of 250 m^2/m^3 and a porosity of almost 93% for biomass attachment, thus, making the working volume of AF reactors 280 L. Their results showed that at an OLR of 1 kg COD/m^3/d, AF removed 68.83%, 78.46% and 79.39% of COD at HRT of 18, 24 and 36 hours, respectively. Similarly, the reactor showed removal efficiency of 59.2%, 70.97% and 74.09% at OLR of 0.760 kg COD/m^3/d and HRT of 18, 24 and 36 hours, respectively (Yousefi et al., 2018).

2.7 Wastewater Reuse Standards

Table 4: Wastewater Reuse Standards

Parameter	**US-EPA**	**US-FAO**	**KSA-MMRA/MWE**	**Jordanian Standards**	**Kuwait Wastewater Reuse Standards**
Turbidity (NTU)	≤ 2	< 2	< 5	< 10	----
BOD5 (mg/L)	≤ 30	< 30	10	< 30	< 20
COD (mg/L)	----	----	50	< 100	< 100
pH	6 - 9	6.5 – 8.4	6 – 8.4	6 - 9	6.5 – 8.5
Residual Chlorine (mg/L)	1	----	0.5	----	0.5 - 1

US-EPA: United States Environmental Protection Agency

US-FAO: United States Food and Agriculture Organization

KSA-MMRA: Kingdom of Saudi Arabia, Ministry of Municipal Regulatory Authority

KSA-MWE: Kingdom of Saudi Arabia, Ministry of Water and Environment

CHAPTER 3

MATERIALS & METHODS

The complete reactor setup and methodology for carrying out different tests are being explained in detail in this chapter.

3.1 Lab scale setup

A combination of 3 units were utilized for carrying out this research. These consisted of Anaerobic Baffled Reactor, Anaerobic Filter and Membrane Tank.

3.1.1 Anaerobic Baffled Reactor design

A lab scale ABR was fabricated from acrylic sheet having 6mm thickness. The reactor consisted of 6 chambers and on top of each chamber a gas outlet with 6mm diameter valve was provided. Sampling ports (6mm diameter valve) at each chamber of ABR were also installed to check the performance of individual compartment.

The total volume of the fabricated ABR was 23 liters with effective volume of 21 liters. The remaining 2 liters accounted for free board provided at top.

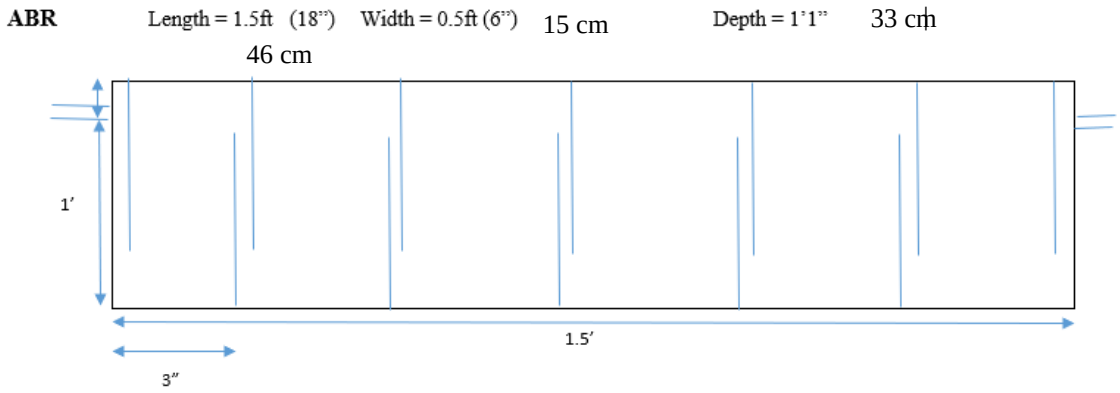

Figure 3-1: Lab Scale ABR Design

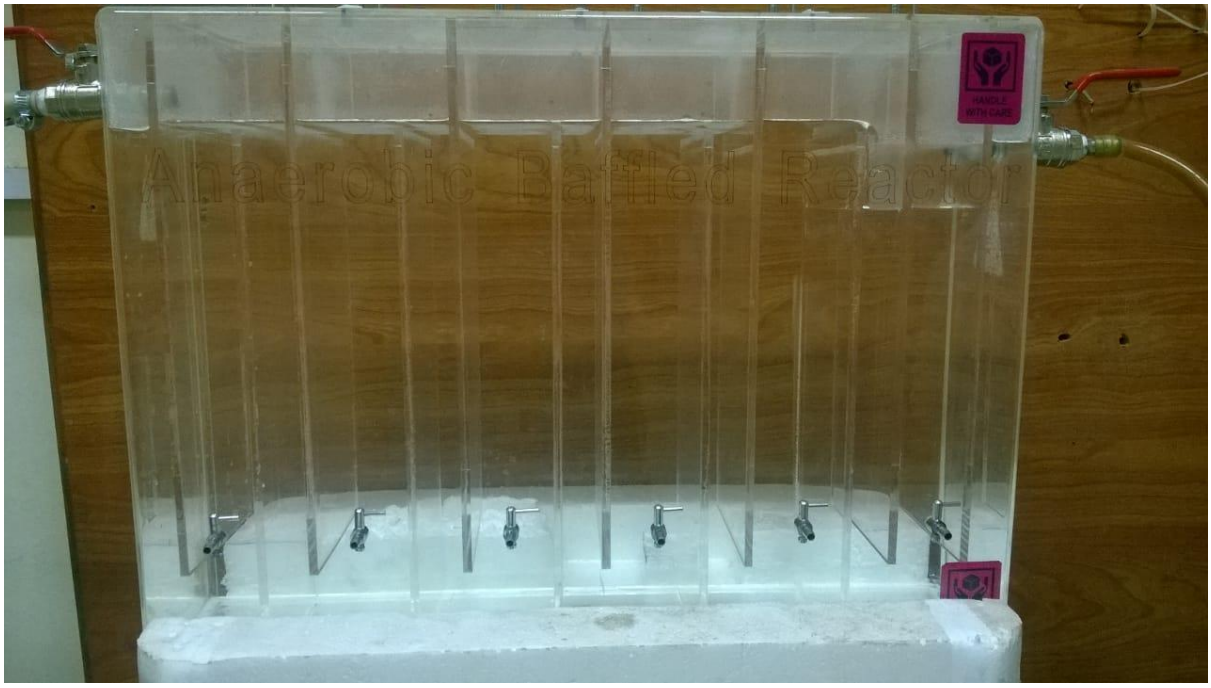

Figure 3-2: Fabricated ABR for Lab scale study

3.1.2 Anaerobic Filter design

The anaerobic filter used in the study was also fabricated from the acrylic sheet having 6 mm thickness. The total depth of the system was 35.5 cm (14 inches) with 30.5 cm (12 inches) of media depth. The media used in the study was the conventional Kaldnes K1 media and locally available PVC corrugated tubes with a diameter of 15 mm (Pipes used for collection of distilled water in ACs). The PVC media was cut into pieces of different length i.e. 25 mm, 20 mm and 15 mm to study the performance of AF. All these media were filled in individual filters and the performance of all media was compared i.e. Kaldnes, PVC 25 mm, PVC 20 mm and PVC 15 mm. To prevent the media from fluidization, a mesh with pore size of 12mm was installed at the bottom of media bed and 2.54 cm or 1 inch above AF reactor bed. Similarly, another mesh with pore size of 6mm was installed at the top of the media bed and just below effluent line to prevent media fluidization and sludge escape into the effluent line. The total volume of the reactor was 16.5 liters with effective volume of 15.33 liters.

AF	Length = 12”	30.5 cm	Width = 6”	15.24 cm	Depth = 14” 35.5 cm

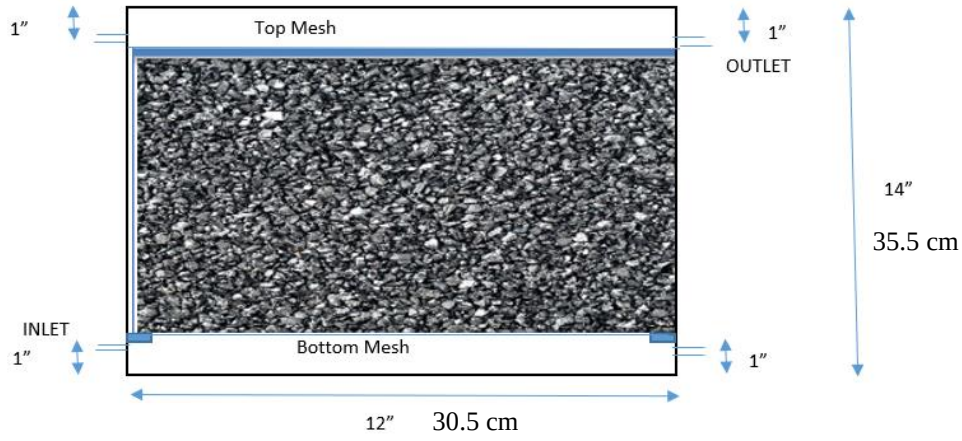

Figure 3-3: Lab Scale AF Design

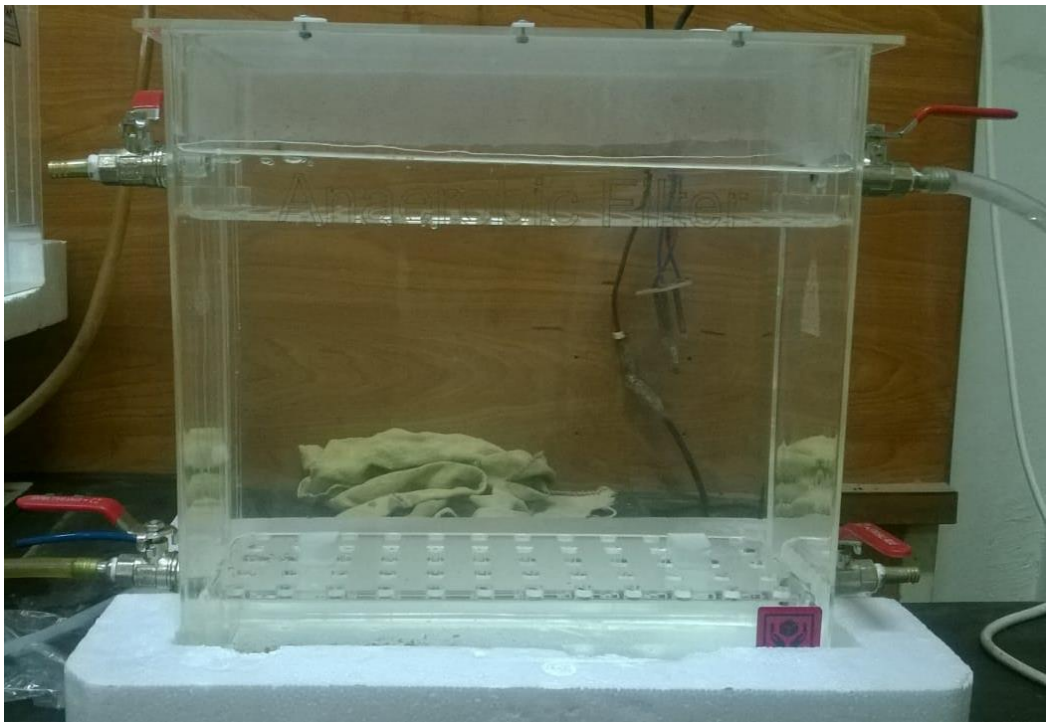

Figure 3-4: Fabricated AF for Lab scale study

Figure 3-5: Kaldnes media used in the study

Figure 3-6: PVC media used in the study

3.1.3 Membrane Tank and Module design

A 5 liter tank was fabricated from the acrylic sheet used for ABR and AF. Water was fed into the tank from top and an effluent port was provided 2.54 cm or 1 inch below for excess water discharge. A port at the bottom was also provided to collect sample from the tank.

Membrane module was fabricated from HDPE material having 1cm thickness, 15cm length and 18cm width. Two ports were provided at top for collection of permeate. The membrane was pasted on front and back side of the module using CLEAR glue.

The membrane used was a Woven Fiber Micro Filtration membrane with pore size of 1-3 µm. The surface area was 0.044 m^2 and operational flux was maintained at 6 LMH with the help of a peristaltic pump.

Table 5: Membrane Specifications

Membrane Specifications	
Pore Size	1-3 µm
Operational Flux	6 LMH
Max allowed TMP	200 mbar
Surface Area	0.044m^2
Operation Mode	Dead End – Outside IN
Configuration	2 sheets pasted on both sides of module

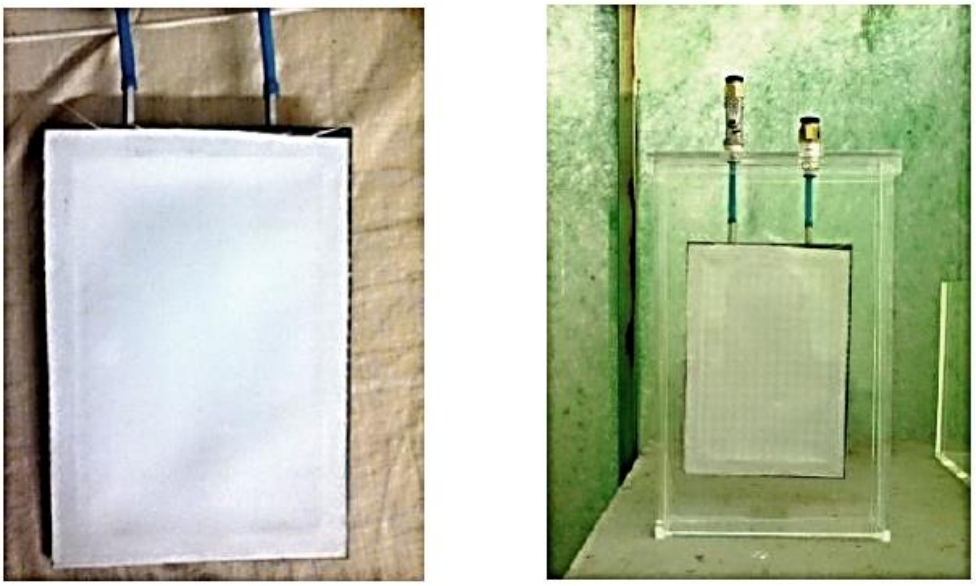

Figure 3-7: Membrane Module

Figure 3-9: Membrane Tank

Figure 3-8: Membrane Glue

3.2 Process flow

Wastewater from a 100 liter Plastic Tank was fed into the ABR through a peristaltic pump (Longer Precision Measuring instrument BT 300-2J, China). The feed water came down, came into contact with bottom sludge and rose up to go into the second chamber. The pattern was followed for all six chambers and finally the treated water was collected at designated effluent port from ABR. From there, the water was pumped into AF from bottom. The water in AF started to rise, came into contact with the biofilm attached on filter media and was finally collected at top. The treated water from AF was then fed into M-Tank. The membrane module was submerged into the M-Tank. Permeate was collected from module through a peristaltic pump. A water trapper and a TMP meter (Data Logging Manometer, Sper Scientific USA, 15 psi) was installed with the permeate line to check the TMP across membrane.

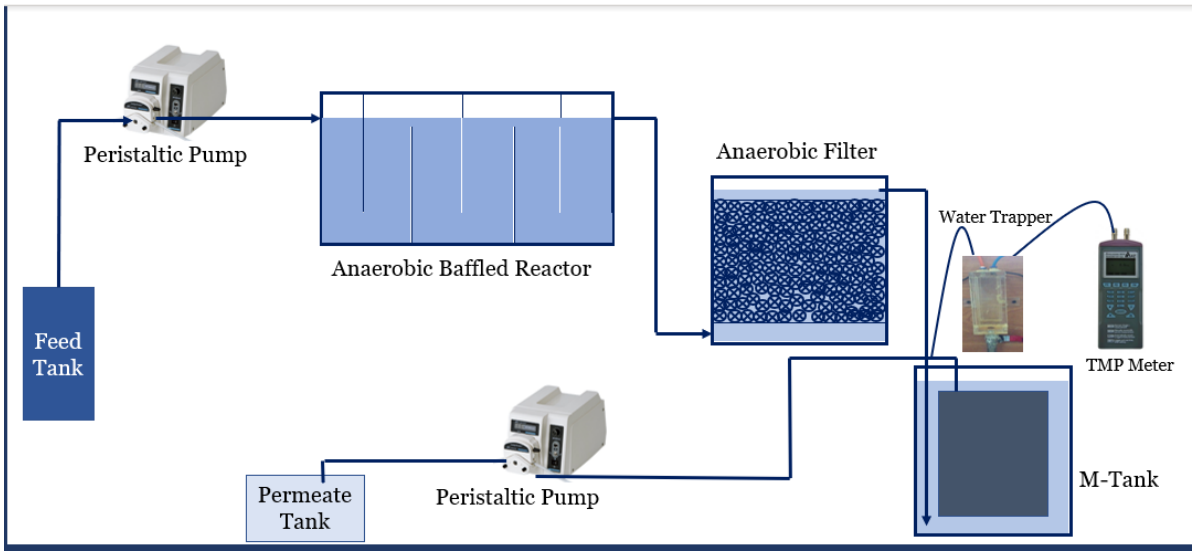

Figure 3-10: Process Flow Diagram

Figure 3-11: Experimental Setup

3.3 Equipment used in the study

- Peristaltic Pump (Longer Precision Measuring Instrument China, BT300-2J)
- TMP Meter (Data Logging Manometer 15psi, Sper Scientific USA)
- COD Thermo-reactor (Velp ECO-25)

- pH meter (Eutech pH700, USA)
- ORP meter (Hanna HI83141, USA)
- UV-Vis Spectrophotometer (Specord 200, Analytik Jena, Germany)
- TKN Analyzer (DK-6 Digestor, SMS Scrubber, JP Water Pump & UDK-149 Distillation unit, Velp Scientifica Italy)

3.4 Seeding

Seed sludge was used for start-up of the reactor. The sludge was collected from bottom of the wetlands installed near ISRA apartments in the NUST H-12 campus. Each compartment of the ABR was fed with approximately 10% by volume of seed sludge. The anaerobic sludge obtained from the wetlands had an ORP of -410 mV.

3.5 Synthetic wastewater

The study was basically conducted for treatment of domestic wastewater of Ward 12 Jatoi City of District Muzaffargarh. But it was not possible to bring samples on regular basis so a synthetic feed of exactly the same wastewater composition was prepared in the lab for optimization of the system.

The average concentration of the real wastewater was 520 mg/L of COD, 35 mg/L of TKN and 14 mg/L of TP. Keeping in view the mentioned pollutants, synthetic wastewater was prepared twice a week. For COD, commercial grade glucose was used. For Phosphorus, Potassium Dihydrogen Phosphate was used. For Nitrogen, Ammonium Chloride was used and the rest of the micro-nutrients were added as per requirements. For maintaining pH in the range of 6.8 to 7.2, Sodium Bicarbonate was added in the feed. The composition of the wastewater is reflected in the following table

Table 6: Synthetic Wastewater Recipe

Chemicals	**Concentration (mg/L)**
Dextrose ($C_6H_{12}O_6$)	520.00
Ammonium chloride (NH_4Cl)	133.66
Potassium di hydrogen phosphate (KH_2PO_4)	17.15
Calcium chloride ($CaCl_2$)	4.87

Magnesium sulfate ($MgSO_4$)	4.87
Ferric chloride ($FeCl_3$)	0.50
Sodium hydrogen carbonate ($NaHCO_3$)	80.00
Cobalt chloride ($CoCl_2$)	0.05
Zinc chloride ($ZnCl_2$)	0.05
Nickle chloride ($NiCl_2$)	0.05

It was first determined from lab testing that how much of a specific salt/sugar will contribute towards specific pollutant. For example,

- 1.02 mg/L of Glucose contributed to 1 mg/L of COD
- 3.819 mg/L of Ammonium Chloride contributed to 1 mg/L of TKN
- 1.43 mg/L of Potassium Dihydrogen Phosphate contributed to 1 mg/L of TP

3.6 Experimental runs

This research was carried out in 3 different phases for optimization of HRT as well as media selection. During all phases, the anaerobic conditions of the system were ensured through regular monitoring of pH and ORP.

3.6.1 Phase I

This phase consisted of around 3 months with 2 months of prior acclimatization. The setup was started in August 2018. It was run for 2 months on feed water for acclimatization. Once the system was acclimatized, it was run for another 3 months i.e. October 2018 to December 2018 for obtaining results. The ABR was run at 3 different HRTs of 8, 10 and 12 hours whereas the AF was run at fixed HRT of 12 hours. 2 parallel AF reactors were utilized for saving time. One reactor was filled with Kaldnes K1 media and the other one was filled with PVC media having 25 mm length.

In the start, the ABR was seeded with anaerobic sludge in such a way that each chamber was fed with approximately 10% by volume of seed anaerobic sludge. Then the system was purged with Nitrogen gas to remove oxygen from the system and finally the feed flow was started.

3.6.2 Phase II

After the first phase was over by end of December, the media from AF reactors was removed. Now, one of the AF reactor was filled with 20 mm length PVC media and another one with length of 15 mm PVC media. The HRT of the ABR and AF were the same as that of Phase I. This phase was continued from January 2019 till May 2019.

For saving time and early acclimatization, the 15 mm and 20 mm media were prepared one month before the start of Phase II and dipped into anaerobic sludge to ensure biofilm formation on the media.

3.6.3 Phase III

This phase consisted of polishing technique. The effluent from AF reactor was fed into the Membrane tank. Membrane module submerged in the reactor having surface area of 0.044m^2 was operated at a flux of 6 LMH and the permeate was collected through a peristaltic pump. A temperature sensor was installed in the reactor for monitoring of water temperature. Water trapper and TMP meter was installed with the permeate line to monitor the TMP across membrane. Duration of this phase was 2 months. i.e. April 2019 to May 2019.

3.6.4 Membrane Resistance and Cleaning

After filtration run, when the membrane become fouled, the module was removed from the reactor and was placed in clear tap water and water was filtered through it for 1 hour. The TMP noted during this hour gave us Total Resistance (Rt). The cake layer developed on the membrane surface was then washed out with the help of soft tooth brush and a detergent. This step is known as Physical cleaning. Then the membrane was again kept in tap water and water was filtered through it for 1 hour. The TMP during this filtration gave us the resistance caused by membrane and pore clogging. Thus subtracting this resistance from the total gave us Resistance due to cake layer (Rc). Then for Chemical cleaning, the module was kept for 24 hours soaked in a solution of 5% NaOH and 1% NaOCl for removal of organic pollutants. After removing from the solution, the module was then dipped for 24 hours into a solution of 1% HCl for removal of inorganic pollutants. Again the module was dipped into tap water and water was filtered for 1 hour to determine the resistance caused by pore clogging. The TMP gave resistance due to membrane or intrinsic membrane resistance (Rm) and when this

resistance was subtracted from the previous resistance, we got Resistance due to pore clogging (Rp). After this, the membrane was again dipped into NaOH and NaOCl solution (used for cleaning) and this solution was filtered through this membrane for almost 1 hour at a flux of 6 LMH.

During the resistance analysis, the total hydraulic resistance (Rt) was calculated using the following equation.

$$Rt = \Delta P / \mu J$$

The resistance-in-series model was applied to estimate the filtration characteristics using the following equations.

$$Rt = Rm + Rc + Rp$$

$$Rc = Rt - (Rm + Rp)$$

$$Rp = Rt – (Rc + Rm)$$

Where;

J = operational flux ($L/m^2.h$) or LMH,

ΔP = TMP (kPa),

μ = viscosity of permeate or Tap water (Pa.s), it can be determined from the already available table of viscosity on internet at specific temperature

Rt = total hydraulic resistance (m^{-1}),

Rm = intrinsic membrane resistance (m^{-1}),

Rc = reversible cake resistance created by the cake layer (m^{-1}),

Rp = irreversible fouling caused by adsorption of dissolved / colloidal onto the surface of membrane and also into the pores (m^{-1})

3.7 Analytical methods & their removal mechanisms

A total of 5 parameters were regularly monitored to check the performance of the system. COD, TKN and TP were determined to check the removal efficiency of the

system while pH and ORP were used to maintain the system in an anaerobic state at all times.

3.7.1 Chemical Oxygen Demand (COD)

The Chemical Oxygen Demand (COD) is a very important parameter for finding the strength of wastewater. It is based on this principle that all organic substances except few can be converted into CO_2 and H_2O in the presence of an oxidizing agent in the presence of an acidic medium. The fact that COD is always greater than BOD is because that during COD test all the organic matter is converted into H_2O and CO_2 regardless of the bacterial assimilation of the readily biodegradable matter. The COD values will be much higher if the feed contains more biologically resistible matter.

$$a\ C_6H_{12}O_6 + b\ O_2 \text{ ------> } x\ CO_2 + y\ H_2O$$

where, a, b, x and y are stoichiometric constants.

The method used for COD determination is termed as Closed Reflux method. In this method, a known amount of sample is mixed with known amount of Potassium Dichromate which acts as an oxidizing agent. We can use other oxidizing agents as well like Potassium Permanganate, Potassium Iodate, Ceric Sulfate but Dichromate is better as it is capable for oxidizing a wide variety of pollutants to CO_2 and H_2O completely. The remaining dichromate at the end of digestion is then determined using a titrant. In order to oxidize the organic matter, a strong acidic medium is required under high temperature for which Sulfuric Acid (H_2SO_4) is used. At elevated temperatures, volatile organic contaminants tend to escape due to which we close the end of the tubes in digestion, this is the reason that this method is known as Closed-Reflux method. There are also some contaminants like low molecular weight fatty acids that are not oxidized unless some catalyst is used and for this purpose, Silver Sulfate is used along with sulfuric acid during digestion of the sample. Certain inorganics like Chlorides may also get oxidized during digestion which will result in higher COD. Mercuric Sulfate is thus used during digestion process to overcome the interference caused by chlorides in the sample. Hg ions will combine with chloride ions to form Mercuric Chloride (Sawyer et al., 2003).

Chemicals, Glassware & Equipment required for COD analysis

- Concentrated Sulfuric Acid (98%)
- Silver Sulfate
- Potassium Dichromate
- Mercuric Sulfate
- Ferroin Indicator (1,10-Phenanthroline iron(II) sulfate)
- Ferrous Ammonium Sulfate
- Potassium Hydrogen Phthalate (for calibration)
- 15mL Autoclavable Digestion vial (preferably Hach standard vial)
- 10mL Glass Pipette
- Pipette Filler
- 10mL Beakers for preparing dilutions (if required)
- 25mL Titration Flask
- Reagent Bottles for storing different reagents and titrant
- 50mL Burette
- COD Reactor/Digestor

Reagents preparation

Sulfuric acid reagent

Add 5.5g of Silver Sulfate in 500mL conc. Sulfuric Acid and mix for approximately 2 hours.

Standard potassium dichromate reagent (0.1N)

Add 4.913g of pre-dried Potassium Dichromate and 33.3g of Mercuric Sulfate to 167mL conc. Sulfuric Acid and make up the volume to 1litre using distilled water.

The reagent prepared has a normality of 0.1 because Potassium Dichromate has equivalent weight of 49.13g.

Ferroin indicator

Dilute the concentrated indicator in a separate bottle at a ratio of 1:1

Ferrous Ammonium Sulfate (FAS) titrant (0.1N)

Add 39.2g of Ferrous Ammonium Sulfate to 500mL distilled water. Add 20mL conc. Sulfuric Acid and finally make up the volume to 1litre using distilled water

Dilute the prepared solution again at a ratio of 1:5 using distilled water. The prepared titrant will now be approximately 0.02N. But we will determine its exact normality from the blank used in the test using the relation

$$N_1.V_1 \text{ (Std. Potassium Dichromate Reagent)} = N_2.V_2 \text{ (FAS)}$$

Procedure

- Take 2.5 mL of sample in the digestion vial and 2.5mL distilled water in another (If the sample approx. COD is greater than 450mg/L, dilute the sample accordingly because if you don't, all the dichromate would be utilized by organic matter and there'll be no dichromate left in the end for titration)
- Add 1.5 mL Standard Potassium Dichromate to the vials using pipette
- Add 3.5 mL Sulfuric Acid Reagent to both vials using pipette or automatic dispenser (The tubes will become hot because of exothermic reaction. So it's better to use proper gloves)
- Now tightly cap the vials so that fumes can't escape the vial during digestion.
- Place the vials in COD digester. Set temperature at $150^{O}C$ and timer for 2 hours.
- After 2 hours of digestion, remove the vials from the digester and place them in test tube rack.
- If the contents in the sample vial has turned GREEN, you can't proceed further. You need to dilute the sample and run the test again.
- If the color is still yellowish, pour the contents into titration flask and add 2-3 drops of Ferroin indicator
- Now start adding FAS from burette drop wise to it. A blue color will be achieved which is due to Cr^{+3} ions but ignore this and continue adding titrant until a reddish brown color is achieved. This is the end point of titration. All the remaining potassium dichromate is now titrated

Calculate COD from given relation

$$COD\ \left(\frac{mg}{L}\right) = \frac{(B-A)xNx8000}{mL\ of\ sample}$$

Where, B = volume of FAS used for Blank

A = volume of FAS used for Sample

8000 = "8" is equivalent weight of Oxygen in grams and "1000" is for converting mL in denominator into L

N = Normality of FAS calculated from Blank i.e.

$$N_1.V_1 \text{ (Std. Potassium Dichromate Reagent)} = N_2.V_2 \text{ (FAS)}$$

Here, N_1 is 0.1N, V_1 is 1.5mL, V_2 is the volume of FAS used to titrate the Blank

3.7.2 Total Kjeldahl Nitrogen (TKN)

Nitrogen is very important to determine in water samples as it is an essential nutrient for growth of different microbes. Nitrogen exists in 4 different forms in wastewater bodies. These are Organic Nitrogen, Ammonia Nitrogen, Nitrates and Nitrites. Nitrates & Nitrites collectively are known as Inorganic Nitrogen whereas Organic and Ammonia nitrogen together are known as Kjeldahl Nitrogen.

Nitrogen from atmosphere is converted into protein by nitrogen fixing bacteria i.e. photosynthetic cyanobacteria which are quietly similar to algae. In addition to this, nitrogen in the form of ammonia and ammonium is also applied to soil as a nutrient for plants. Mostly, urea is used as it release ammonia gradually. Humans on the other hand can't utilize atmospheric nitrogen for protein synthesis so they take it in prepared from through plants. These nitrogen compounds then can escape human body in urine in the form of urea. When microbes comes into contact with this urea, ammonia is released. This ammonia is utilized by Nitrosomonas or Nitrifying bacteria to produce Nitrites. These in turn is oxidized by Nitrobacter into Nitrates. Nitrates serves as fertilizer for plants but these nitrates and nitrites are reduced in anaerobic conditions through a process called Denitrification.

TKN determination principle

Ammonia and Organic Nitrogen is determined through distillation method. The sample when heated can liberate ammonia which can then be captured in an acidic solution but the organic nitrogen doesn't escape in the form of ammonia. Therefore, concentrated sulfuric acid is used to destroy the organic portion thus freeing the nitrogen as ammonia at temperatures of around 360^{O}C. A salt-copper mixture is also used to target the

resistant organic matter usually Potassium Sulfate and Copper Sulfate is used. During digestion, excess water is evaporated and concentrated sulfuric acid attacks the organic matter. As the sulfuric acid reaches its boiling point, white fumes are formed in the digestion tube. The mixture will turn black if enough organic matter is present due to the dehydrating action of the sulfuric acid. After complete destruction of organic matter, solution will turn clear. The excess sulfuric acid can be neutralized using diluted NaOH and phenolphthalein indicator. The sample is then distilled to liberate ammonia.

All the nitrogen that exist as ammonium is considered as ammonia nitrogen. Now when this ammonia is released during distillation, the liberated H^+ ions will tend to decrease pH of the solution due to which NaOH is added to the sample for utilizing these H^+ ions. The ammonia liberated is condensed into an acidic medium usually boric acid. The acidic medium will reduce the ammonia back into ammonium and thus it can't vaporize or escape. After all the ammonia has been condensed, the sample is titrated against Sulfuric Acid using mixed indicator (Sawyer et al., 2003).

Manual method

Chemicals, Glassware & Equipment required for TKN analysis

- Sulfuric Acid (98%)
- Potassium Sulfate
- Red Mercuric Oxide
- Sodium Hydroxide
- Sodium Thiosulphate Pentahydrate
- Phenolphthalein
- Boric Acid
- Methyl Red
- Methyl Blue
- Ethanol 95%
- 250 mL Beakers
- 500mL Flasks
- 800mL Kjeldahl Flask or any Round bottom flask that can withstand temperature upto $400^{O}C$
- Bulb Condenser

- Liebig Condenser
- Pipette with filler
- 50mL Burette
- Hot Plate

Reagents preparation

Digestion reagent

Mix 134 grams of potassium sulphate in 650 ml of distilled water. Then add 200 ml concentrated sulfuric acid and put it into stirring. Side by side mix 2 grams of red mercuric oxide in 25 ml of 6N sulfuric acid. Mix both of these solutions together and dilute to 1000 ml. Make sure the temperature is below $14^{O}C$ to prevent any formation of crystals

Mixed indicator

Dissolve 0.2 grams of methylene red in 100 ml of 95% Ethanol. Dissolve 0.1 grams of methylene blue in 50 ml of Ethanol. Mix the two solutions together.

Indicating boric acid

Add 20grams of Boric Acid to 500mL distilled water. Mix it and add 10mL mixed indicator into it. Dilute to 1000mL.

Stock ammonia

Dissolve 38.19 mg of Ammonium Chloride in 500 mL distilled water and dilute to 1000 mL. The resultant solution will have ammonia concentration of 10 mg/L

Phenolphthalein indicator

Dissolve 1g Phenolphthalein in 100 mL of 95% Ethanol

Sodium hydroxide & Sodium thiosulphate solution

Dissolve 500 grams of NaOH and 25 grams of $Na_2S_2O_3.5H_2O$ into 300 mL distilled water. Dilute to 1000 mL

0.1N NaOH

Dissolve 4 g of NaOH pellets in 500 mL distilled water. Dilute to 1000 mL

Standard 0.02N H_2SO_4

First prepare a 0.1N solution by adding 2.7 mL of concentrated sulfuric acid to 500 mL distilled water and diluting it to 1000 mL. Now take 200 mL of this 0.1N solution and dilute it to 1000 mL

Calculations:

$$N_1V_1 = N_2V_2$$

$$0.1N * V1 = 0.02N * 1000mL$$

$$V1 = 200mL$$

Procedure

- Take 100 ml of sample in a 500 ml volumetric flask and dilute it to 300 ml with distilled water.
- Add 50 ml of digestion reagent
- Heat it inside a fume hood at 360 to 400^{O}C. The solution should turn clear in about an hour, keep the digestion going for another 20 minutes. Alternatively, heat until the final volume is approximately 100mL.
- Cool the flask and dilute the solution to 300 ml with distilled water
- Adjust the pH using 0.1N NaOH and phenolphthalein as indicator
- Put the mixture into the Kjeldahl flask and connect it to the condenser. Allow the cooling water to circulate through the Liebig condenser for vapors condensation.
- Add 50 ml of NaOH-$Na_2S_2O_3$ into the Kjeldahl flask. Quickly connect the bulb condenser to the flask so that ammonia loss can be prevented.
- Put 50 ml indicating boric acid in a 250mL beaker below the Liebig condenser end where the condensate is collected. Make sure the bottom of the condenser is dipped in boric acid solution.
- Start the heating of the Kjeldahl apparatus at maximum heat and wait until the total volume of indicating boric acid solution placed below reaches 200 ml.
- As distillation is started, the color of the boric acid solution will start changing from purple to green.

- In the end, take the mixed indicator solution and titrate it against 0.02N Sulfuric acid until the first pink shade appears.
- Repeat the same procedure with 100 ml of blank.
- Calculate TKN using the following equation
- $TKN\ (^{mg}/_{L}) = \frac{(A-B)x\ 280}{mL\ of\ sample}$
- Where A is volume of H_2SO_4 used for sample, B is volume of H_2SO_4 used for blank, 280: 0.02N * 1000ml/l * 14g/mol

Automatic method using VELP Digestor (DK-6) & Distillation unit (UDK-149)

Chemicals, Glassware & Equipment required for TKN analysis

- 50mL Burette
- 250mL Titration Flasks
- Sulfuric Acid (98%)
- Potassium Sulfate
- Copper Sulfate Pentahydrate
- Sodium Hydroxide
- Boric Acid
- Methylene Blue
- Methylene Red
- Ethanol 95%
- Velp Digestion Tubes
- Velp DK-6 Digestor
- SMS Scrubber
- JP Water Pump
- Velp UDK-149 Distillation Unit

Reagents preparation

4% Boric acid solution

Dissolve 40 grams of Boric Acid into 600 mL distilled water. Shake well and dilute to 1000 mL.

30% NAOH solution for distillation

Dissolve 300 grams of NaOH in 500 mL distilled water. Dilute to 1000 mL.

15%NAOH solution for scrubber

Dissolve 150 grams of NaOH in 500 mL distilled water. Dilute to 1000 mL.

Standard 0.02N H_2SO_4

First prepare a 0.1N solution by adding 2.7 mL of concentrated sulfuric acid to 500mL distilled water and diluting it to 1000 mL. Now take 200 mL of this 0.1N solution and dilute it to 1000 mL

Calculations:

$$N1V1 = N2V2$$

$$0.1N * V1 = 0.02N * 1000mL$$

$$V1 = 200mL$$

Mixed indicator

Dissolve 0.2 grams of methylene red in 100 ml of 95% Ethanol. Dissolve 0.1 grams of methylene blue in 50 ml of Ethanol. Mix the two solutions together.

Procedure

- Take 20mL sample into Velp Digestion tube
- Add 2 tablets of Kjtabs VCM A00000274
- If you don't have Kjeldahl tabs, then add 7g Potassium Sulfate and 0.2g Copper Sulfate
- Add 20mL concentrated sulfuric acid into it
- Turn ON the DK-6 digestor
- Put the Steel cap on tubes and turn ON the JP Recirculating pump

- Heat the tubes at 160^{O}C for 30min, then at 260^{O}C for 30min, then at 360^{O}C for 30min and finally at 420^{O}C for 30min.
- Wait for digestion to complete
- Turn ON the UDK-149 distillation unit and wait for Pre-Heating to complete.
- Put the digested sample tube in distillation unit.
- Add 40mL distilled water and 25mL 30% NaOH into the tube by selecting the appropriate program
- Add 25mL 4% Boric Acid in titration flask and put it at the end of distillation pipe.
- Start the distillation process at 50% steam power for 5min.
- After distillation is complete, take the flask, add 0.5mL Mixed Indicator and titrate against 0.02N H_2SO_4.
- Calculate TKN using the following equation

$$TKN\ (mg/L) = ((A-B) \times 280)/(mL\ of\ sample)$$

Where A is volume of H2SO4 used for sample, B is volume of H2SO4 used for blank, 280: 0.02N * 1000ml/l * 14g/mol

3.7.3 Total Phosphorus (TP)

Phosphorus exists in wastewater bodies in 3 different forms. Orthophosphates, Polyphosphates (Ortho and Poly are collectively termed as Inorganic Phosphates) and Organic Phosphorus. Phosphorus in different forms comes into wastewater either from agricultural run offs or from human discharges. In human urine, it results from metabolic breakdown of different proteins and nucleic acids. Besides these, detergents used in households are a major contributor to high polyphosphates concentration in domestic wastewaters. These all forms of phosphorus are used for reproduction and synthesis of new cells by bacteria in biological processes. The incoming phosphorus thus gets assimilated in microbial cells and such microbes are called Phosphorus Accumulating Organisms or PAOs.

Orthophosphates can be directly quantified through colorimetric or spectrophotometric method. The concentration of orthophosphates can be determined through absorbance of light when a molybdate solution is added to it. When ammonium molybdate is added to water containing orthophosphates, it forms a molybdo-phosphate complex which is

yellow in color. The greater the orthophosphates, the darker the yellow color. But in some cases when concentration is quite low, the color may not develop properly and thus a vanadate solution is used in addition to molybdate to form a much more intense yellow color.

For determination of Polyphosphates, the sample needs to be hydrolyzed first so that the polyphosphates may be converted into orthophosphates and then the same procedure may be utilized for determination. The acid used for this purpose is a 1:3 Sulfuric acid solution. After acid digestion, the phosphates determined would be called total inorganic phosphates. If interested in quantification of polyphosphates alone, you have to determine the orthophosphates side by side.

Polyphosphates = Total Inorganic Phosphates - Orthophosphates

For quantification of Organically bound phosphorus, digestion is used. This digestion step will help breakdown the complex matter into phosphates. Usually Persulfate, Perchloric acid or Sulfuric-Nitric acid mixture are used for this digestion. The most common and least hazardous one is Persulfate digestion method. The phosphorus determined in this step will be Total Phosphorus and thus the inorganic fraction must be subtracted from this to obtain Organic Phosphorus (Sawyer et al., 2003).

Organic Phosphorus = Total – Inorganic

Chemicals, Glassware & Equipment required

- 250mL flasks for Digestion
- 10mL Pipette with Filler
- 1000mL Reagent Bottles
- 25mL flasks
- Distilled Water
- Potassium Dihydrogen Phosphate
- Hydrochloric Acid
- Phenolphthalein
- Sodium Hydroxide
- Ammonium Persulfate
- Sulfuric Acid (98%)
- Ammonium Molybdate - 4 Hydrate

- Ammonium Metavanadate
- Quartz Cuvette (Minimum One if you are using Single Beam Spectrophotometer and Two if you are using Double Beam)
- UV/Vis Spectrophotometer
- Hot Plate for Digestion

Reagents preparation

Phosphate stock solution

Dissolve 220 mg of anhydrous potassium dihydrogen phosphate in distilled water and dilute to 1000 mL

Concentration of P in KH_2PO_4 = $\frac{31}{39+2+31+64} x\ 220$

P concentration = 50 mg/L

Sulfuric acid digestion solution

Carefully add 300 mL concentrated Sulfuric acid to approximately 600 mL distilled water and dilute to 1 L with distilled water

Ammonium molybdate-metavanadate solution

Dissolve 2.5 g of Ammonium Molybdate in 30mL distilled water. Side by Side, Dissolve 0.125 g of Ammonium Metavanadate by heating to boiling in 30 mL of distilled water. Cool and add 33 mL of conc. HCl. Cool the solution to room temperature. Now mix the two solutions and dilute to 100 mL

1N Sodium hydroxide solution

Dissolve 40 g of NaOH in 500 mL distilled water. Dilute to 1000 mL

Phenolphthalein indicator

Dissolve 1g Phenolphthalein in 100 mL of 95% Ethanol

Method for analysis

- Prepare Phosphorus concentrations of 5 mg/L, 10 mg/L, 15 mg/L and 20 mg/L from the stock Phosphate solution
- Take 50 mL of each standard in different 250 mL flasks

- Take 50 mL of distilled water in a separate 250 mL flask
- Measure 50 mL of desired sample and pour into 250 mL flask
- Now if you have one sample to analyze, you'll have 6 flasks ready for digestion (1 blank, 1 sample and 4 standards)
- Add 1 drop phenolphthalein indicator in each. If a red color develops, add sulfuric acid solution until color just disappears.
- Add 1 mL of sulfuric acid digestion solution and 0.4 g of ammonium persulfate in each flask
- Boil gently for 30 to 40 minutes or until the final volume is 10 mL
- Allow it to cool, then add 1 drop of phenolphthalein indicator and titrate it with 1N sodium hydroxide until a faint pink color appears. The purpose is to neutralize the excess acid
- After this, make it up to 50 mL with distilled water. The digested sample is then further tested for total phosphate
- Now take 10 mL from each of the 6 flasks in a separate 25mL flasks
- Add 2 mL of Ammonium Molybdate-Metavanadate solution into it
- Wait for 15-20 minutes for color development
- Turn ON the UV/Vis Spectrophotometer (Analytic Jena, Specord 200 Plus)
- Click on INITIALIZE DEVICE
- Go to Wavelength selection window and select 470 nm
- Click on MODE and select Absorbance mode
- Pour the contents from the flask containing digested blank into a quartz cuvette
- Put it in the spectrophotometer's Reference slot
- Click on REFERENCE
- Now pour the remaining contents of this blank into another cuvette and put that cuvette in spectrophotometer's Measurement slot
- Click on Measure. After a few seconds, the absorbance will be displayed on the screen. This absorbance should be 0.00
- Now take the standard solution one by one in the cuvette and measure its absorbance in the Measurement slot. Don't remove or disturb the cuvette in the Reference slot as it's a double beam spectrophotometer
- From the absorbance values of the blank and standards, construct a calibration curve of concentration vs absorbance

- Now put the prepared sample into the cuvette and measure its absorbance
- From the calibration curve, note down its concentration against the absorbance

3.7.4 Power of hydrogen ions (pH)

pH is used to express the hydrogen ion concentration in a sample. The more acidic solution, the more hydrogen ion concentration and thus the lower pH value. In biological treatment of wastewater, pH must be maintained at specific level for optimum microbial activity.

pH is determined through a pH meter. This meter has a pH probe which has a combined glass electrode i.e. Sensing and Reference Cells. The sensing half-cell is a thin sensitive membrane separating the solution to be analyzed and a reference solution. Electric potential is developed due to liberation of hydrogen ions in the form of millivolts and difference between potential is used to record pH.

As the hydrogen ions concentration increase, the millivolts generated also increase. Therefore, a neutral solution will have 0 millivolts. An acidic solution will have positive value of millivolts generated and basic solution will have a negative value.

The meter is calibrated using 3 different buffers. A neutral buffer of pH 7, a basic buffer of pH 10.01 and an acidic buffer of pH 4.01. As we proceed with calibration, a graph between pH and millivolts generated is produced in the meter termed as slope of the meter.

The effective pH range for methanogens is from 6.5 to 7.5, with an optimal range of 6.8 to 7.2.

pH measurement using EUTECH pH 700 meter

- Turn ON the meter
- Rinse the probe with distilled water
- Press the CAL button to enter calibration mode
- Dip the probe into pH 7 buffer, wait for value to stabilize and press ENTER
- Remove the probe, rinse with distilled water and dip in pH 10 buffer. Again wait for value to stabilize and press ENTER. At this time, you'll see that a slope is shown for a few seconds on meter display

- Remove the probe again, rinse with distilled water and dip into pH 4 buffer. Wait for value to stabilize and press ENTER. Again updated slope will be displayed for few seconds and the meter will return to Measurement mode itself.
- If the meter doesn't go to Measurement mode itself, you have done something wrong and you'll have to repeat the whole procedure
- When meter is calibrated successfully, dip the probe into sample and note down the value from display when value is stabilized

3.7.5 Oxidation Reduction Potential (ORP)

ORP is the potentiometric measurement of all the oxidized and reduced species present in water bodies. It depends on the concentration of dissolved oxygen present in water. Greater quantity of oxygen mean greater oxidizing potential and thus higher ORP value. Besides oxygen, there may also be present some oxidizing species which will tend to increase the ORP value (Horne & Goldman, 1994). On the other hand, negative ORP value mean greater reducing potential and thus represent an anaerobic system. A good anaerobic system will thus have an ORP range of -300 to -450 mV

ORP is measured directly using an ORP meter or a pH meter with ORP electrode. The ORP probe consists of a Reference electrode consists of Silver or Silver Chloride system and a Sensing electrode which is made of noble element like Gold or Platinum. Such metals are resistant to chemical oxidations. Unlike pH meter, this meter can't be calibrated but it can be checked by 2 methods. The first is to short the electrodes of the probe and by doing so the value on the meter should be 0.5 mV. Alternatively, a standard solution can be used to check the difference in millivolts. The difference should not exceed ± 10 mV. These standard solutions can be Light's solution, ZoBell's solution or a Quinhydrone solution (Eaton et al., 2005).

Table 7: Potentials of Standard ORP solutions at 25^{o}C vs NHE

Zobell's Solution	428mV
Light's Solution	675mV
Quinhydrone at pH 4	482mV
Quinhydrone at pH 7	285mV

CHAPTER 4

RESULTS AND DISCUSSION

The results of all the experimental runs described in previous section are discussed here.

4.1 Phase I: Performance of ABR (8, 10 &12 hrs HRT) and comparison of Kaldnes media with PVC media having 15 mm diameter and 25 mm length

In this phase of study, the ABR was run at 3 different HRTs i.e. 8, 10 and 12 hours. Each chamber of the ABR was initially fed with same amount of seed sludge so as the treatment performance of each successive chamber may be compared.

As for AF, one AF reactor was filled with Kaldnes media and the other one with PVC media having 25 mm length. Both these reactors were run separately at 12 hours of HRT to check the removal efficiencies of different pollutants.

4.1.1 pH and ORP during Phase I

The pH and ORP of the both the systems were monitored regularly. The average pH in the ABR reactor was found to be 7.3, in AF(Kaldness) 7.14 and in AF (PVC-25) 7.13. These pH values indicate that the systems were operating in a good anaerobic state because the pH for anaerobic reactors need to be in the range of 6.8 to 7.2 (Davis, 2010). Below or above this range, the pollutant removal and biogas production is affected. The time series of these reactors' pH can be observed in the following figure.

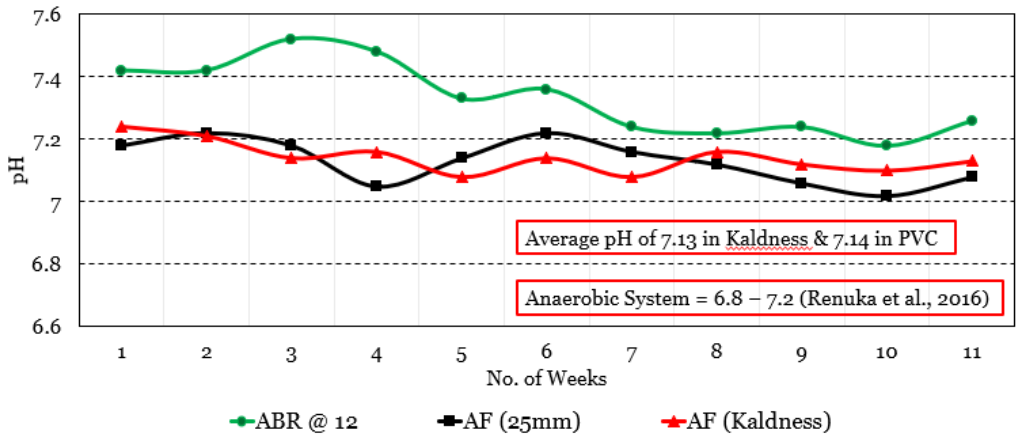

Figure 4-1: pH of different reactors during Phase I

It was found that with the passage of time, reactor moved towards more anaerobic state and hence the pH dropped to the optimum range of anaerobic conditions and after 2 months of operation, the pH became almost constant.

The ORP of the system was also monitored on regular basis. Anaerobic systems usually have negative ORP. Literature recommends ORP of -170 to -400 mV. The more the ORP is negative the better anaerobic conditions there are in a reactor because positive ORP means an environment capable of oxidation which is not desired in anaerobic reactors. Acclimatized anaerobic sludge from bottom of wetlands usually has an ORP of < -400mV (Test performed at lab using fresh sludge from wetlands near ISRA). In this case, the ORP was high at start of the reactor i.e. -200mV but when nitrogen purging was done and the system got acclimatized after 2 months of operation, the ORP dropped to -330mV. The average ORP of the 3 systems i.e. ABR, AF (Kaldnes) and AF (PVC-25) was found to be -337, -340 and -336mV, respectively. The time series of ORP for all 3 reactors is shown in following graph.

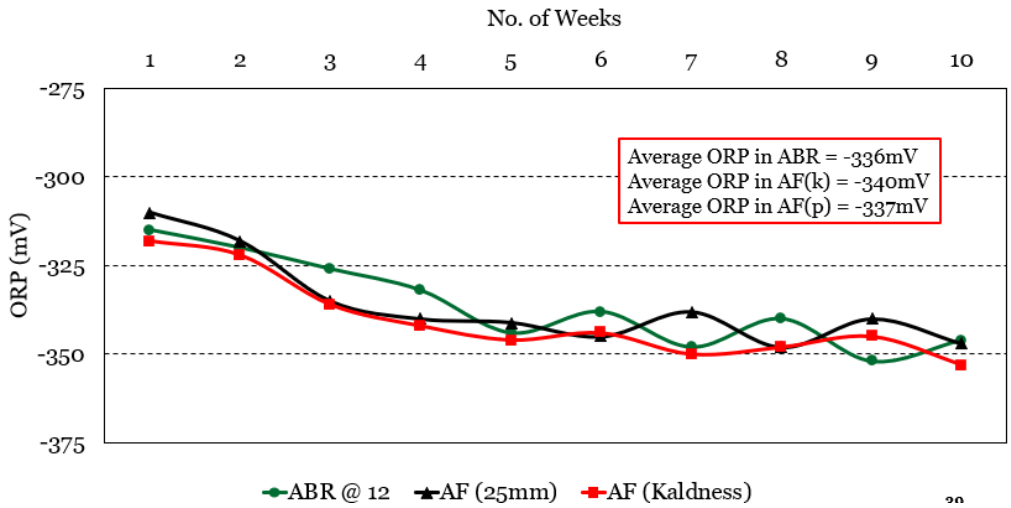

Figure 4-2: ORP of 3 Reactors during Phase I

4.1.2 Total Phosphorus removal during Phase I

Phosphorus removal in anaerobic reactors is usually less as compared to aerobic reactors. The removal of phosphorus is through Phosphorus Accumulating Organisms (PAOs). Such organisms use phosphorus as growth material (Sawyer et al., 2003). The phosphorus removal in ABR at 3 different HRTs of 8, 10 and 12 hours is shown is figure. The TP removal increased as we increased the HRT of the system. Such trend

was also been explained by Hahn and Figueroa who achieved 65% removal at HRT of 15 days (Hahn & Figueroa, 2015).

At 8 hours of HRT, system only achieved 13% of TP removal but as the HRT was increased to 12 hours, TP removal reached 22%. TP removal further increased in the Anaerobic Filter because of the greater biomass concentration in the biofilms. The overall TP removal using Kaldnes media in the filter was observed to be 46% whereas it was found to be 40% for PVC media with 25 mm length.

The phosphorus removal is due to biomass formation of the anaerobic microbes. Now the biomass formation in anaerobic reactors is less due to longer SRTs and thus the removal efficiency is less as compared to aerobic reactors in which biomass formation and sludge wastage is more.

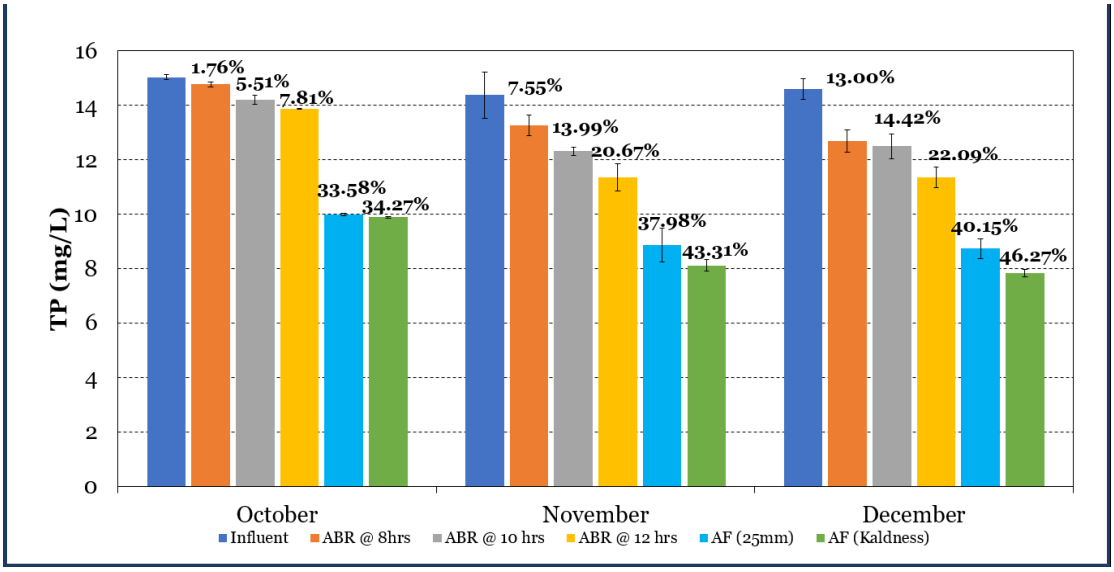

Figure 4-3: TP Removal in Phase 1

4.1.3 TKN Removal in Phase I

Nitrogen is a nutrient used by microbes for growth and it's removal in anaerobic processes is relatively less as compared to Aerobic processes (Show & Lee, 2017). In this study, Nitrogen removal like TP was dependent on HRT and biomass of the system. As the HRT was increased from 8 to 12 hours in ABR, the removal improved from 20 to 50%. And when the treated water was further treated in AF at an additional HRT of 12 hours, the performance further improved to 74% in Kaldness media filter and 68% in PVC-25 filter. It can be seen from these results, that as the surface area for biofilm is increased in case of Kaldnes media, the removal efficiency is also more.

Martha J. Hahn in her study reported TKN removal of 73% at HRT of 15 days in ABR-Pond system (Hahn & Figueroa, 2015).

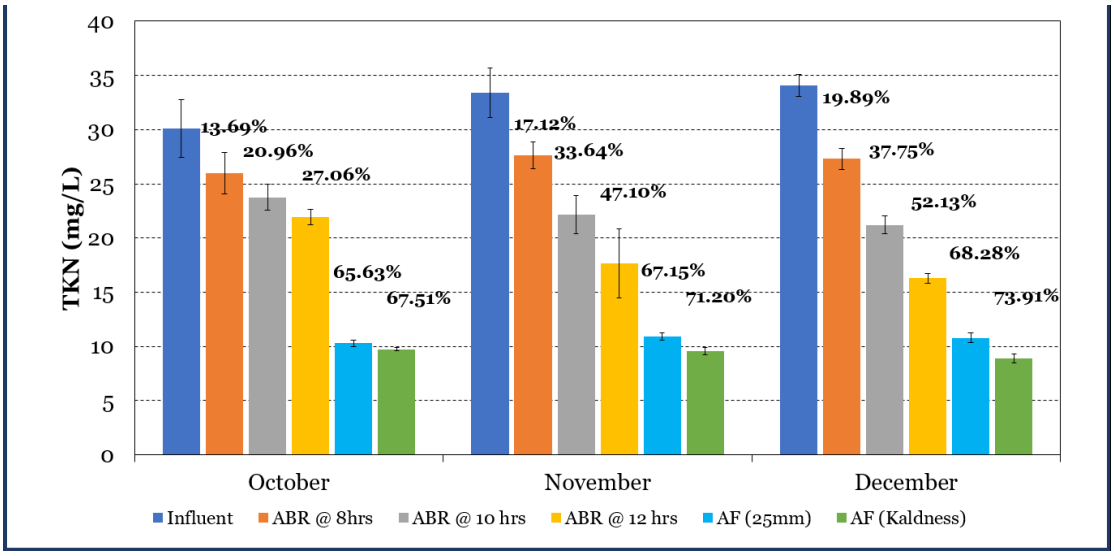

Figure 4-4: TKN Removal efficiency in Stage I

4.1.4 COD Removal in Phase I

The most important part of wastewater treatment is COD removal to reduce the organic load for reuse or discharge. Anaerobic processes usually have better COD removal as compared to Aerobic processes. Renuka and co-workers in 2016, worked on ABR-AF system and reported COD removal of 90% at HRT of 40 hours (Renuka et al., 2016).

In our study, the COD removal in ABR was observed to be 66, 70 and 74% at HRT of 8, 10 and 12 hours. This removal increased to 87% in Kaldnes filter and 84% in PVC-25 filter. The detailed trends of COD removal can be observed in the following figure.

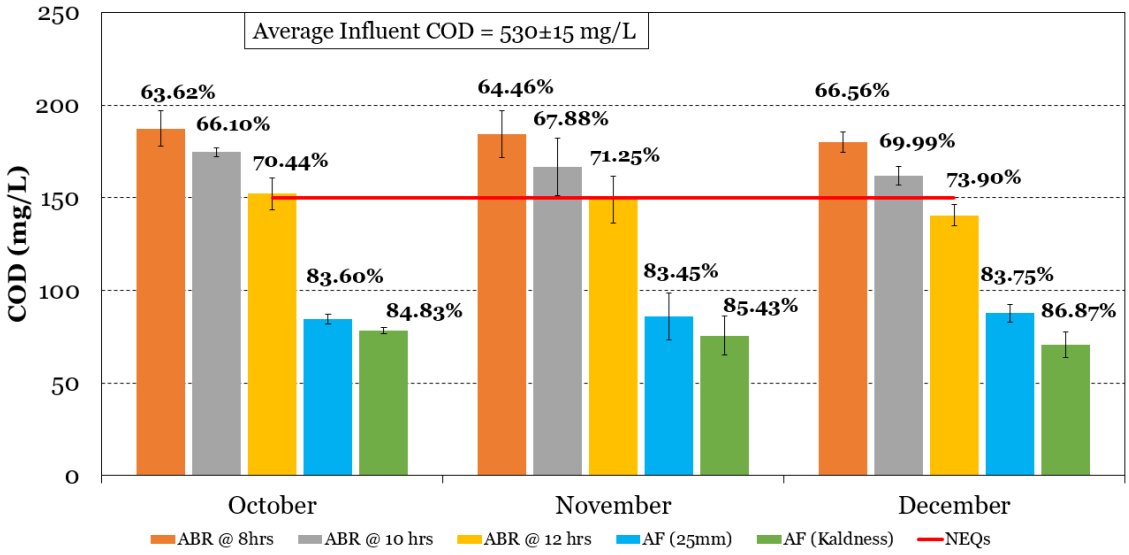

Figure 4-5: COD Removal in Phase I

4.2 Phase II: Performance of ABR (8, 10 &12 hrs HRT) and Comparison of PVC media 15 mm length and 20 mm length

In this phase of study, the performance of filter having 20 mm length media was compared with the filter having 15 mm length media.

4.2.1 pH and ORP during Phase II

In this phase, ABR was already acclimatized in the previous phase but AF was set-up and therefore, its pH and ORP were higher at start but gradually reduced as anaerobic conditions were achieved.

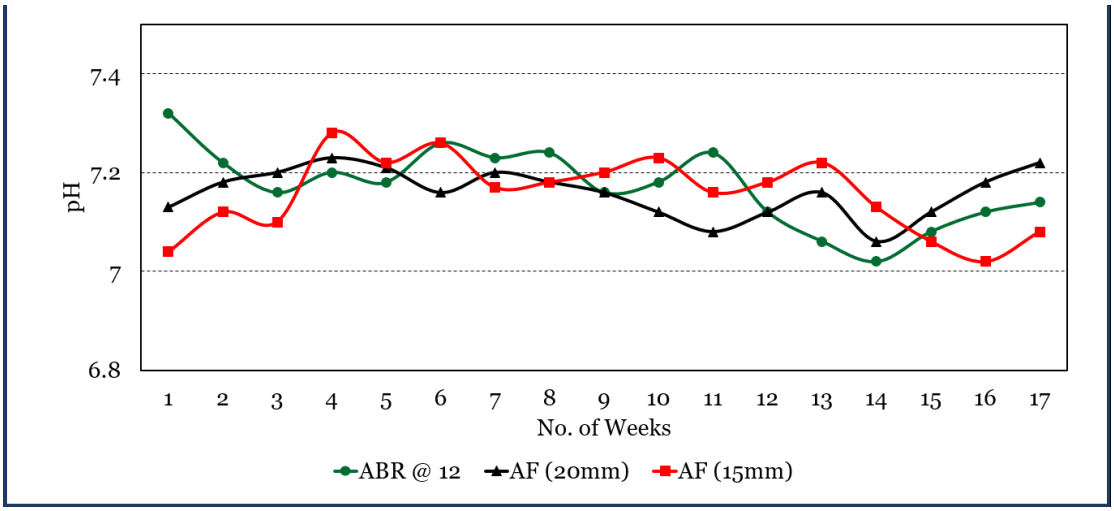

Figure 4-6: pH in Phase II

It can be observed from the figure that average pH in ABR was 7.17 whereas, in AF reactors, the average pH was found to be 7.15

The ORP of the ABR remained constant due to acclimatized anaerobic state whereas, the newly set AF reactors showed gradual reduction in the ORP with the passage of time. The performance of the filter improved as the ORP came down to -300 mV.

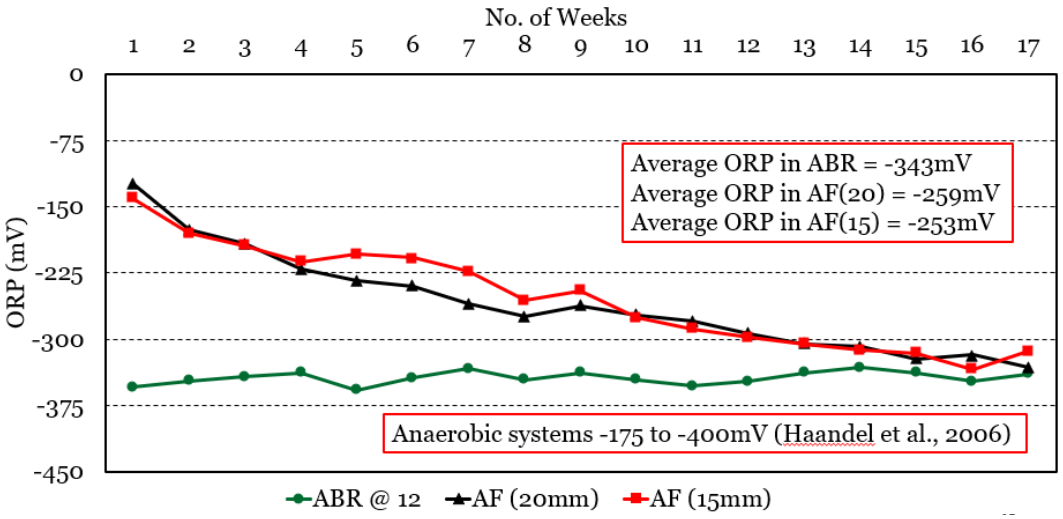

Figure 4-7: ORP during Phase II

4.2.2 Total Phosphorus removal during Phase II

The TP removal efficiency can be seen in the following figure. The TP removal increased when the media length was reduced due to the high surface area and thus larger biomass concentration in the attached biofilms.

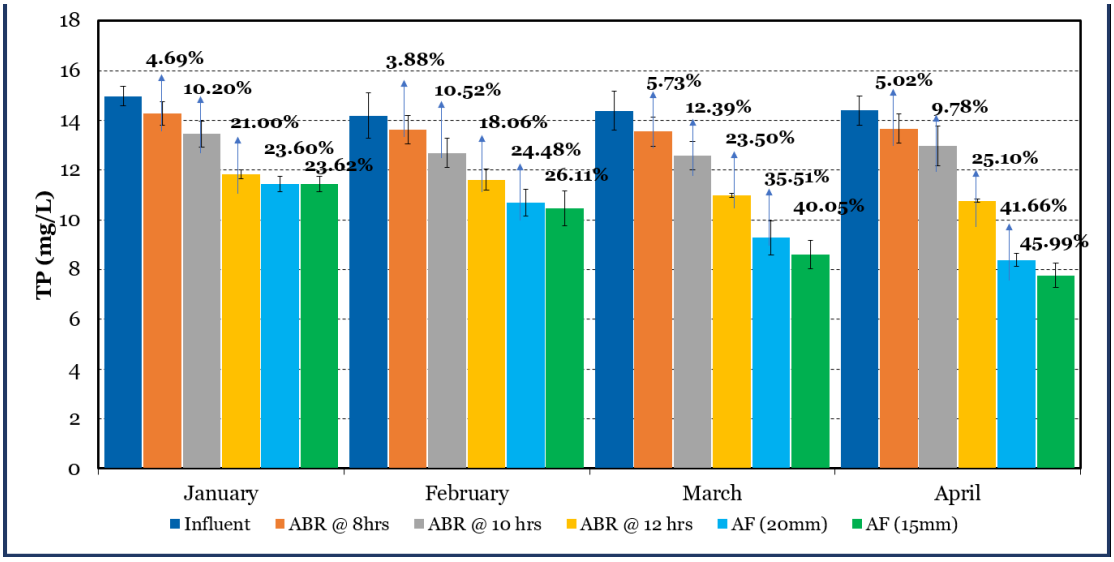

Figure 4-8: TP Removal in Phase II

The TP removal efficiency of 15 mm media was found to be 46% which is the same achieved through the Kaldnes media filter earlier in phase I. The removal efficiency with 20 mm PVC media was found to be 42% which seems significant if treated water is to be re-used for agriculture purposes (refer to chapter 2 for reuse standards).

4.2.3 TKN removal during Phase II

The TKN removal in both the AF reactors is shown in following figure.

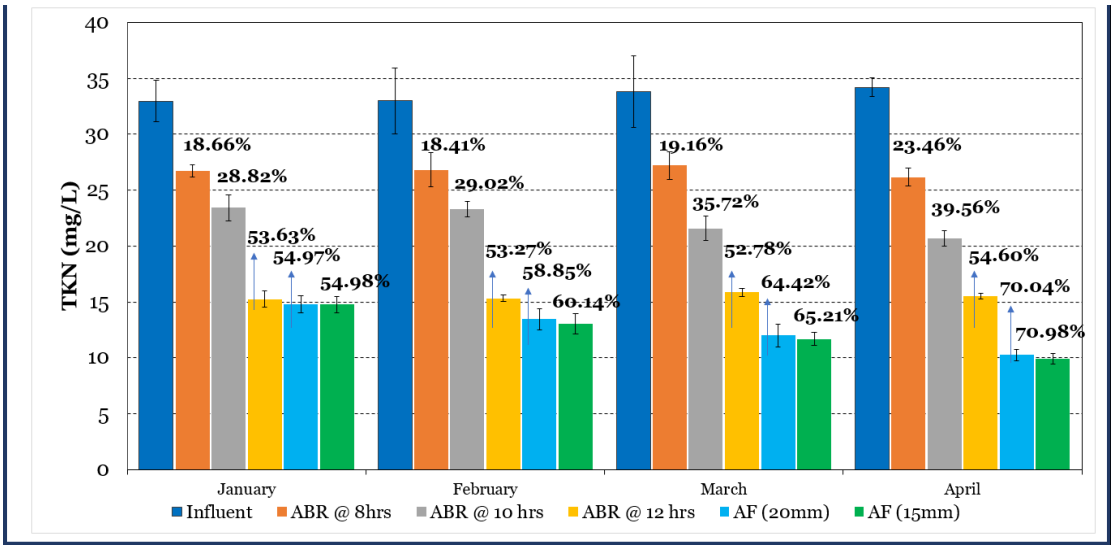

Figure 4-9: TKN Removal in Phase II

The removal efficiency of both the media i.e. 20 mm and 15 mm was found to be almost the same. Any further reduction in size is not necessary as media structure may get

distorted after that. It can be deduced from this figure that 20 mm media filter can achieve the optimal removal efficiency.

If replacement of Kaldnes media is concerned, from results of both phases it can be observed that the conventional media can easily be replaced by 15 mm PVC media as both these media have same removal efficiencies.

4.2.4 COD removal during Phase II

The COD removal in this phase is shown in the following figure.

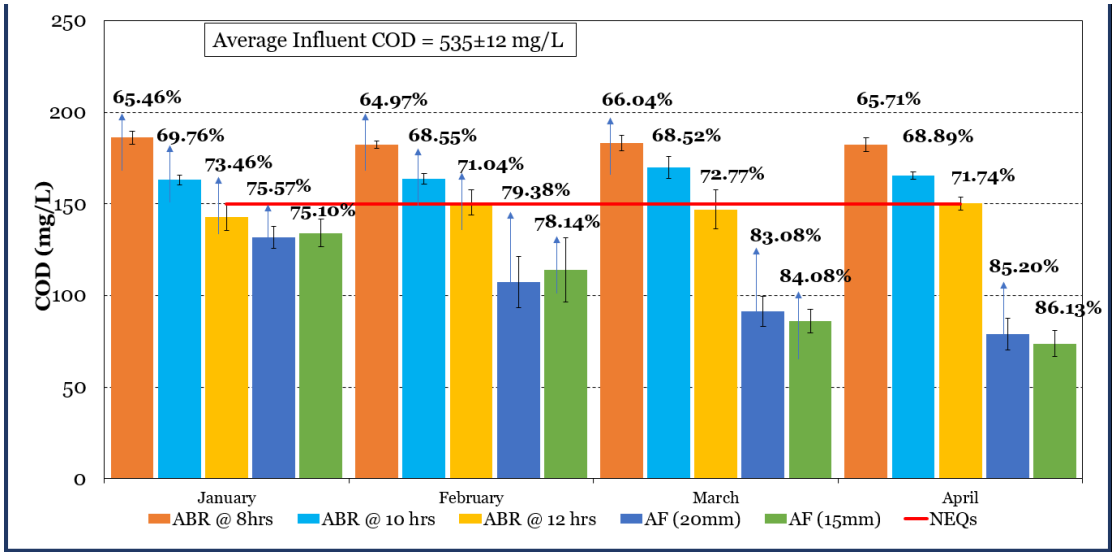

Figure 4-10: COD Removal Phase II

From the figure, it can be observed that enough COD removal was achieved in both these filters. The removal efficiency of 20 mm was found to be 85% whereas in 15 mm filter it was 86%. It can be concluded that 20 mm media can be used in AF reactors for optimal performance.

4.3 Phase III: COD and Turbidity removal in Membrane reactor

In this phase, COD and TKN removal efficiencies of the overall system were determined to choose a suitable option for polishing of the AF effluent if generated water has to be reused (refer to chapter 2 for reuse standards). The TMP through the membrane was also monitored regularly to check the fouling rate of the membrane.

4.3.1 COD removal through Membrane

The COD removal of the membrane system is shown in the following graph. The Blue line with square markers represents removal efficiency of membrane only whereas the Green line with diamond markers represents the overall COD removal efficiency of the complete system starting from ABR through AF and finally MF Membrane.

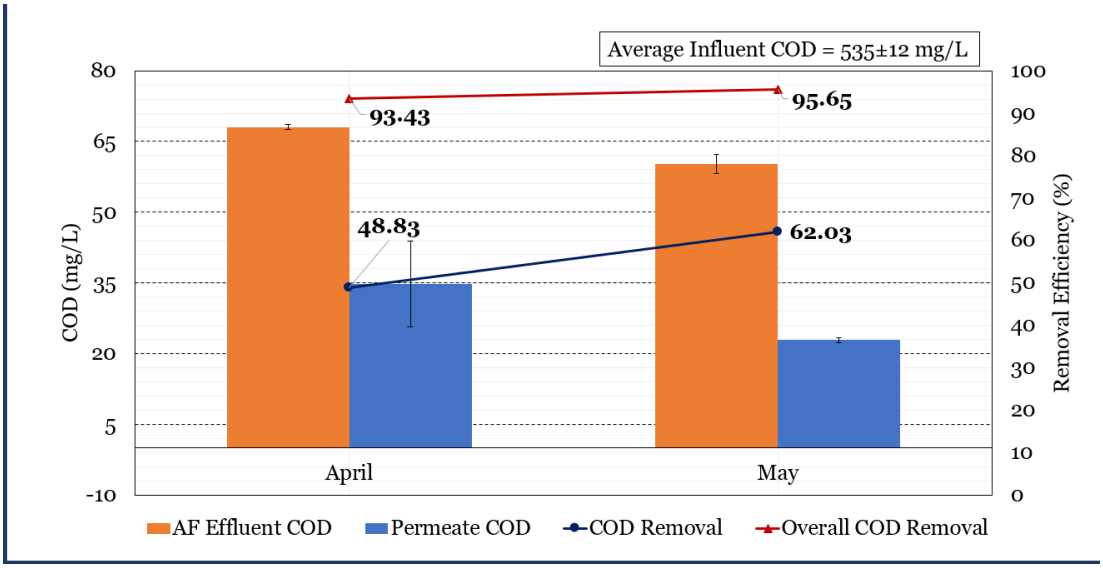

Figure 4-11: COD removal in phase III

It can be observed that the combined system achieved COD removal of 95% at HRT of 24 hours and membrane flux of 6LMH with effluent concentration of 22 mg/L of COD.

COD removal of 96% at an operational flux of 30LMH was reported by Ratanatamskul and his co-workers in their study conducted in 2015 where they used a Hollow Fiber membrane having pore size of 0.4 microns for polishing of the effluent (Ratanatamskul et al., 2015).

4.3.2 Turbidity Removal through Membrane system

The suspended solids or turbidity removal of the overall system is shown in the following graph. The red line shows the removal efficiency of membrane only whereas the blue line shows the overall Turbidity removal of the system and it was found that using a MF membrane with 1-3 µm pore size can effectively remove almost all the suspended solids present in the raw influent.

The overall removal efficiency was observed to be 98% with an effluent turbidity of 3.77 NTU. Suspended solids removal efficiency of 99% was reported using a 0.4 µm Hollow Fiber membrane (Ratanatamskul et al., 2015)

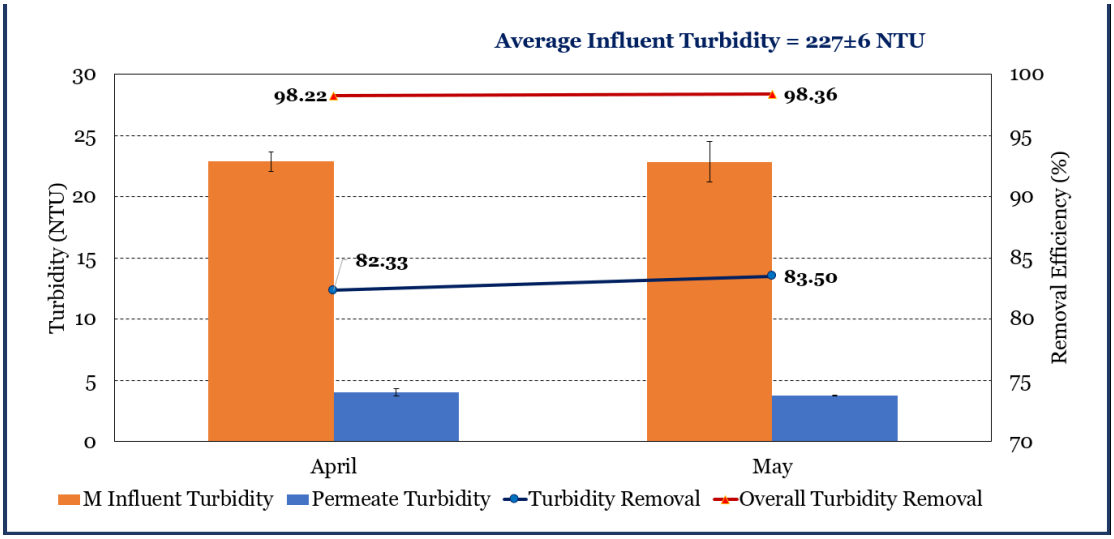

Figure 4-12: Turbidity Removal

4.3.3 TMP Profile of the Membrane

The membrane system was run for 2 months and the fouling behavior was observed through data logging manometer. Plot of the recorded TMP is shown in the following figure.

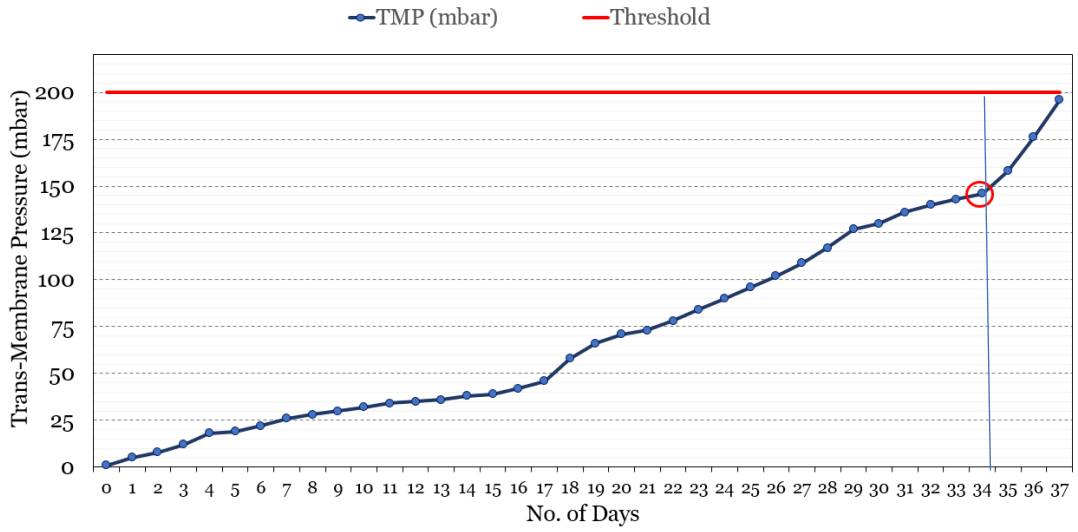

Figure 4-13: TMP Profile

A threshold limit of 200 mbar or 20 kPa was set for TMP rise. TMP of 200 mbar represents fouled membrane. It can be seen from the figure that the membrane performed very well in removing the suspended solids without much rise of TMP. Gradually with time the TMP increased because of formation of cake layer on membrane surface. It can be observed from the figure, that after 35^{th} day of operation, the TMP began to rise abruptly thus indicating a TMP jump. The membrane then achieved the threshold within 2 days of the start of TMP jump and fouled on 37^{th} day of run which means an MF membrane with 1-3 µm pore size can produce better results with less fouling rates as compared to 0.4 µm membrane.

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

From the work done and results of all 3 phases of this study, it can be concluded that:

- ABR with 12 hours of HRT in combination with AF with 12 hours of HRT produces better results in terms of COD, Phosphorus and Nitrogen removal and thus the product water can be easily reused or discharged
- The overall 24 hours of HRT produced water with effluent COD of around 75mg/L but if reuse is not the aim, then ABR can be set for 10 hours of HRT which will result in effluent COD of around 120mg/L and thus can be disposed-off in receiving water bodies
- The conventional Kaldnes media can be replaced by locally available PVC corrugated pipe pieces as the removal efficiency of both these is almost the same
- As the length of PVC media was decreased from 25 to 20 and then to 15 mm, the treatment efficiency also increased because of the increased surface area for biomass attachment
- Although the 15 mm length PVC media produced best results as compared to other two sizes, but such small size also leads to shape distortion in the bottom layers of filter with the passage of time due to increased weight in the filter due to which such small size media should be avoided and the 20 mm size should be used for optimal performance in the filter
- If water reuse is desired, an additional pre-treatment unit must be installed at the end of AF. Microfiltration Woven Fiber Membrane with pore size of 1-3 µm can be used for this post-treatment/polishing purpose
- In this study, the membrane system was run for 37 days producing high quality effluent with average COD of 22mg/L and average Turbidity of 3.7 NTU without fouling. After this filtration time, the membrane need to be cleaned physically and will be ready for next filtration run
- As the fouling rate is less as compared to conventional UF membranes, therefore WFMF membrane serves best alternative for polishing with very little energy requirement

Based on the results and conclusions, recommendations for future work are:

- A problem was encountered during ABR-AF operation that after certain rise of water level in AF, the water starts exerting back pressure on ABR due to which ABR overflows. To prevent such overflow, there should be a level difference of 10 cm between successive compartments of ABR. Such arrangement will allow the water to flow to the AF uninterruptedly
- Seed sludge from bottom of pre-installed wetlands may be used for inoculation to ensure quick start-up and acclimatization
- For re-use, polishing through WFMF membrane and then disinfecting through Chlorine is recommended
- Research need to be carried out to study the effect of OLR on Biogas production rate
- Influence of temperature or seasonal variations in treatment performance need to be evaluated
- Treatment performance through real domestic wastewater need to be carried out as real sewage contain much more complex organics
- PVC is a plasticizer and research need to be carried out to determine any escape of such material in the effluent

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