COMPARATIVE ANALYSIS OF TERTIARY TREATMENT TECHNOLOGIES USED IN DECENTRALISED WASTEWATER TREATMENT SYSTEMS



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A FINAL YEAR PROJECT (FYP) REPORT SUBMITTED TO THE NATIONAL UNIVERSITY OF SCIENCES AND TECHNOLOGY IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF ENGINEERING IN ENVIRONMENTAL ENGINEERING

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APPROVAL SHEET

This is to certify that the contents and forms of thesis titled as "Comparative Analysis of Tertiary Treatment Technologies used in Decentralised Wastewater Treatment Systems" is the original work of author(s) and has been carried out under my direct supervision. I also certify that the thesis has been prepared under my supervision according to the prescribed format and I endorse its evaluation for the award of Bachelor of Engineering in Environmental Engineering Degree through the official procedures of the Institute.

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ABSTRACT

Pakistan is a developing country with a rapidly growing population, economic expansion, and a rise in industrial activity that is being challenged by an acute state of water scarcity. In urban areas, it is estimated that only 10% of all wastewater gets treated while the remaining portion is disposed of into land or surface water bodies without adequate treatment, causing pollution that is detrimental to ecological and human health. This can be seen in dense urban cities, where lack of adequate sanitation and safe disposal of wastewater causes faecal contamination of potable water and a high prevalence of water-borne diseases that are especially linked with high rates of child mortality. Since Pakistan is a signatory to United Nations Paris Agreement 2016, committed to fulfilling 17 Sustainable Development Goals (SDGs) by 2030, it has to treat 50% of its generated wastewater for SDG Indicator 6.3. So far, this has been an area of concern due to an overall failed implementation of centralized sewage treatment plants (CSTPs) as their operation and maintenance (O&M) requires large amounts of resources, and performance is not being monitored or evaluated.

Therefore, to investigate these performance gaps, a situational comparative analysis on CSTPs and DEWATS was performed. Following the findings of the situational analysis, a promising decentralized wastewater treatment technology of Anaerobic Filter (with newly developed Hybrid media) in combination with low-cost Woven Fibre Micro-Filtration Membrane (WFMF) membrane was proposed for the tertiary treatment (polishing) of wastewater. The removal efficiency and effectiveness of AF comprising of Hybrid media (PAC super-imposed on corrugated PVC pipes) – WFMF membrane was compared with AF (comprising of simple PVC filter bed) - WFMF Membrane. The performance of these two systems was assessed at lab scale and further analytical testing methods were performed to evaluate and compare the effluent parameters and check whether they comply with the wastewater reuse standards or not. The Flow rate (Q), Hydraulic Retention Time (H.R.T.) and water flux were set accordingly along with the fixed volume of ABR, AF, and storage tank. The end results showed that the AF (with Hybrid media) was able to achieve 96.84% of COD removal and 99.41% of turbidity removal. The AF (Hybrid media) alone removed up to 55.33% TP and 79.1% TKN. In the later stage a complete Cost Benefit Analysis for the two treatment systems was also performed followed by proposal of the most effective and cost viable solution for tertiary treatment of wastewater.

ACKNOWLEDGEMENTS

We would firstly like to express our utmost gratitude for Allah Almighty who gave us the strength and courage to complete our strenuous Final Year Project and gain maximum knowledge through it. We are thankful that this entire journey from gaining wisdom and achieving a higher purpose throughout these four years to being awarded with Bachelor's Degree in Environmental Engineering went really well and was truly a rewarding endeavour. None of this had been possible without His Mighty's bestowment of dedication in our hearts to remain motivated towards our goal.

We feel honoured to have Dr. Sher Jamal Khan as our project supervisor, who graced us with his expertise, mentoring, and support. Under his umbrella of leadership, and competency we remained steadfast towards our goal. During this entire period, he not only guided us academically but also kept counselling us and provided us with a safe space to talk and share openly. Working with Dr. Sher Jamal Khan has truly been an amazing experience and he is the reason why we had an unwavering determination to strive and achieve our ultimate purpose.

We are forever thankful to Engr. Aamir Khan for assisting us throughout the project work. His experience, knowledge and problem-solving skills benefited us a lot in our laboratory work. We were able to learn a lot from him and mastered to implement all those practical engineering techniques we learned during our degree related to our project domain.

Lastly, we feel indebted to our loved ones, our families and friends for believing in our capabilities, and for standing by us through every moment of hardship and joy. They were there to support us and help us regain our strength into working tirelessly and achieving big. We are thankful for their unprecedented love and prayers.

ABBREVIATIONS

ABR	Anaerobic Baffled Reactor
AF	Anaerobic Filter
HRT	Hydraulic Retention Time
SRT	Solids Retention Time
COD	Chemical Oxygen Demand
TKN	Total kjeldahl Nitrogen
TN	Total Nitrogen
ТР	Total Phosphorus
TSS	Total Suspended Solids
WFMF	Woven Fibre Micro-Filtration
PVC	Poly-Vinyl Chloride
PAC	Powdered Activated Carbon
GAC	Granular Activated Carbon
ТМР	Trans Membrane Pressure
MBR	Membrane Bio-Reactor
NEQS	National Environmental Quality Standards
MAF	Million Acre-Feet per Year
WWTP	Waste Water Treatment Plant
DEWATS	Decentralized Wastewater Treatment System
ORP	Oxidation Reduction Potential
DO	Dissolved Oxygen
AnMBRs	Anaerobic Membrane Bio-Reactor

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1.1. BACKGROUND

Global water crisis. Water scarcity. Thirst. Lack of clean water access. All these acronyms are pointing towards the fact that we are in a dire need of efficient water and waste-water treatment systems. Dirty water is the cause of around 485,000 deaths and about 785 million people lack access to clean water (Welfare, 2018). People are struggling, they need access to clean water for drinking, bathing, cooking and growing their food. Families and communities will be poverty stricken for centuries if they do not have access to clean and readily available water. Children are dropping out of school and parents are struggling to make ends meet (UN on World Water Day). Admittance to clean water makes a huge difference; it's a venturing stone to improvement. At the point when people get access to clean water, they're better able to practice hygiene and sanitation. Guardians can set aside their stresses over waterborne infections and absence of clean water access. They can water their harvests, livestock and improve their livelihoods. The availability of water resources is inherently connected to water quality, as the contamination of water sources might preclude different types of uses. Increased disposal of untreated sewage, joined with agrarian overflow and insufficiently treated wastewater from industry, have brought about the degradation of water quality all throughout the planet. On the off chance that latest things endure, water quality will keep on degrading throughout the next few decades, especially in developing countries situated in dry regions, further impacting human health and ecosystem, putting a strain on sustainable economic development and adding to water shortage

High income countries on average treat about 70% of the industrial and municipal wastewater they generate. This proportion drops to 38% in middle-income countries and further to 28% in lower income countries. In low-income countries, only 8% goes through treatment of any sort. These evaluations support the estimation that, globally, more than 80% of all wastewater is released without treatment which can also be an important source for extracting energy, essential nutrients and clean water (UN Water, 2017). In high-income countries, on the other hand, the primary motivation for advanced wastewater treatment is either to maintain environmental quality or to

provide an alternate water source in times of water scarcity. Nonetheless, due to a lack of economic, financial, technological, and institutional capabilities, the disposal of untreated wastewater is a common practise, particularly in agricultural countries.

Access to improved sanitation facilities can contribute essentially to reduce the health hazards. While 2.1 billion individuals gained access to these improved facilities since 1990, 2.4 billion still are deprived of these services and almost 1 billion people are bound to practice open defecation. However, it is not necessary that the improved sanitation conditions or public well-being can co-align with improved waste-water treatment. About 26% of urban and 34% of rural sanitation and WWT can reduce the human contact with excreta along the entire chain (reliefweb, 2017).

The discussion above concludes that the need for effective wastewater tertiary treatment technologies cannot be emphasized enough keeping in mind the current market trends, shares and recent developments. The global market on tertiary water and WWTS is forecasted to reach \$35.5 billion by 2027, at a CARG of 7.2% during the predicted period 2020-2027. The statistics for the year 2019 show that an estimated 3 billion to 10 billion gallons of untreated waste-water is released from STP annually. Roughly 23,000 to 75,000 sanitary sewers overflows take place every year (US, EPA). And the reasons connected to such rapid growth of the market is increasing population, industrialization and urbanization. The provision of safe water supplies and reduction of the environmental degradation has therefore become a challenging task. We are in a need of sustainable economy that focuses on reusing treated waste-water rather than simply discarding it. It can be used in various sectors as a potential resource after treatment and hence can benefit the society. The safe management of treated WW and sanitation has become even more important after the outbreak of deadly virus, COVID-19.

Pakistan reports 40% of the deaths due to drinking of contaminated water. The urbanization is increasing at an incredibly higher rate as compared to anywhere else in South Asia. Only 20% of the population has access to clean drinking water and about 40% get their water from contaminated sources, lacking access to any decent sanitation facilities. Pakistan over the years have turned from a water-surplus country to a water-deficient country. The water accessibility has reduced drastically from 1,299 m³ per capita in 1996-97 to 1,100 m³ per capita in 2006 and it is projected to lie under 700 m³ per capita by 2025. Therefore, use of waste-water for agriculture and irrigation has become a necessity. In Pakistan, industrial and domestic wastewater is disposed

of directly into the sewers, open channels and nearby water bodies. Except for Islamabad and Karachi, which treat only 8% of the generated wastewater before disposal, this effluent is not dealt with, and none of the urban communities have biological treatment facilities. Farmers value the wastewater used for irrigation because of its vital nutrient content (phosphorus, nitrogen), unwavering quality of supply and positive effects on agribusiness, families, households and monthly income.

Besides these issues, one of the biggest dilemma Pakistan faces in this regard is that there appears to be no national policy in effort to support sustainable utilization of waste-water in this country. Problems of waste-water disposal roots to the economic disasters, failed implementation of the environmental laws and policies, lack of strategic plans and institutional failures. Therefore, although the laws and guidelines have been regulated with regards to proper treatment of waste-water, yet it is not being carried out due to the lack resources and competent labour.

To address this issue, our group has decided to work on tertiary treatment technologies to enhance the efficiency of Decentralized Waste-water Treatment system. The main motive behind our research is to propose an economical as well as effective solution to treat domestic waste-water generated in residential areas and communities. We plan to develop both practical and theoretical model of hybrid beds constructed using Activated Carbon and PVC pipes (20 mm in dia) and compare its' efficiency to GAC and membrane filters. The hybrid beds were designed as an alternative to pure AC and achieve high operational performance as well as removal efficiency giving it a competitive edge in the market.

The reasons why we opted for hybrid beds in DWTS is that it reduces the effect on environment and public health and increases the ultimate reuse of waste-water. It can promote the return of waste-water within the watershed of origin and evades the costly implementation of Centralized Treatment Systems. DWTS requires relatively low operational and maintenance cost and is suitable for rural areas and isolated communities. Considering that Pakistan is a developing and a water-scarce country, DWTS is the most suitable and cost-effective option that does not require an extensive sewerage network.

The proposed model is being used as Decentralized Treatment Systems installed in Saidpur and other housing communities. The use of hybrid beds would mould the system in such a way that there would be no need to use the natural wetlands and separate disinfection except if the waste-water is being used in households for bathing and in industries for cooling or heating.

To ensure our role towards sustainability, we went through the 2030 Agenda for Sustainable Development; it presents a more extensive set of objectives for water besides water supply and sanitation, there are a number of water-related goals. Improve water quality by 2030 by decreasing pollution, eliminating dumping, limiting the release of harmful chemicals and materials, splitting the proportion of untreated wastewater, and expanding recycling and safe reuse around the world, according to SDG Target 6.3. Because Pakistan has such a poor level of wastewater treatment, there is a pressing need for technological advancements and safe water reuse options to help achieve Target 6.3, which is important for achieving the overall agenda. Therefore, alongside tertiary treatment of waste-water, we also plan on recycling the treated waste-water for horticulture, irrigation, households and other purposes accordingly to incorporate the factor of sustainable development in our project.

The need for effective tertiary treatment as well as reuse of treated waste-water can also be acknowledged by going about the facts and figures that shows the disposal of waste-water in Pakistan without adequate treatment. According to the latest estimates, the total quantity of waste-water produced in Pakistan is 962,335 million gallons including 674,009 million gallons from municipal and 288,326 million gallons from industrial use. The total amount of waste-water discharged into the rivers is 392,511 million gallons (UNIDO, 2000). Further estimates show that 2,000 million gallons of sewage is being disposed of into the local water bodies every day (Pak-SCEA 2006).

> Sector wise estimated waste-water production in Pakistan:

SR. NO.	SOURCE	VOL	UME
		10 ⁶ m ³ y ⁻¹	Percent%
1	Industrial	395	6
2	Commercial	266	5
3	Urban Residential	1,628	25
4	Rural residential	3,059	8

Table1. Wastewater production in Pakistan. Source: Pakistan's Wetland ActionPlan, 2000, prepared by NNCW and WWF

5	Agriculture	1,036	16
TOTAL		6,414	100

> Waste-water produced Annually by cities (WB-CWRAS Paper 3, 2005)

Table 2. Annual wastewater production in cities. Source: Master Plan for Urban Wastewater (Municipal and Industrial) Treatment Facilities in Pakistan. Final Report, Lahore: Engineering, Planning and Management Consultants, 2002)

CITY	URBAN POPULATION	TOTAL WASTEWATER	% OF TOTAL	% OF TREAT	RECEIVI NG
	(1998 CENSUS)	PRODUCED		ED	WATER
		(10 ⁶ M ³ /Y)			BODY
					River Ravi,
					irrigation
LAHORE	5,143,495	287	12.5	0.01	canals,
					vegetable
					farms
					River Ravi,
FAISALABAD	2,008,861	129	5.6	25.6	River
					Chenab
					and
					vegetable
					farms
					SCARP
GUJRANWAL	1,132,509	71	3.1	-	drains,
A					vegetable
					farms
				-	River Soan
RAWALPINDI	1,409,768	40	1.8		and
					vegetable
					farms
				-	
SHEIKHUPUR	870,110	15	0.7		SCARP
A					drains

	1			-	River
MULTAN	,197,384	66	2.9		Chenab,
					irrigation
					canals and
					farms
				-	River Ravi,
SIALKOT	713,552	19	0.8		irrigation
					canals and
					farms
KARACHI	9,339,023	604	26.3	15.9	Arabian
					Sea
					River
HYDERABAD	1,166,894	51	2.2	34.0	Indus,
					irrigation
					canals and
					SCARP
					drains
PESHAWAR	982,816	52	2.3	36.2	Kabul
					River
OTHER	19,475,588	967	41.8	0.7	-
TOTAL	43,440,000	2,301	100.0	7.7	-
URBAN					

1.2. PROBLEM STATEMENT

Pakistan once a water-surplus country is now suffering from severe water scarcity due to the growing population at an annual rate of 2%, along with rapid and un-regulated urbanization. The efficient utilization of other resources of water has therefore become necessary for Pakistan to meet its growing demand. Wastewater is one of the greatest sources which can be used in various sectors post treatment since it contains 99% of water. According to an official source, 4.36 BCM wastewater is generated annually out of which only 10 % wastewater is treated which leads to \$380 million loss annually for the country along with 250,000 death of children from waterborne diseases (Hooper and Austen, 2013).

For country like Pakistan where expensive technology for wastewater treatment is not viable due to economic constraints, enhanced and improvised decentralized wastewater treatment systems proves to be quite effective for developing countries saving them from extensive sewerage networks, technical, operational and maintenance problems and providing a cost-effective alternative for wastewater treatment.

1.3. OBJECTIVES

- I. To investigate the removal efficiency of a hybrid media AF (PAC-coated PVC), and to compare its effluent characteristics with simple PVC media AF
- II. To analyse the treatment performance of a low-cost flat sheet membrane.
- III. To perform cost-benefit analysis and propose the most optimal treatment technology

1.4. SCOPE OF THE FINAL YEAR PROJECT

The principal aim of our project was to evaluate the treatment performance of a hybrid filter packing material and compare it with the removal efficiencies achieved by simple PVC filter bed in combination with flat-sheet membrane. The hybrid filter material was expected to enhance the secondary treatment performance of the AF by providing carbon adsorption and anaerobic biological degradation simultaneously. This was achieved by selecting an optimal corrugated PVC pipe length and diameter and coating the outside of the pipes with PAC slurry. Thus, PAC was superimposed over individual filter packing, and it provided an adequate surface area for adsorption of soluble refractory organics and heavy metals in the wastewater. The hollow inside of the PVC pipes allowed sufficient space for the growth of biofilm which degrades the organic materials in the wastewater. In this way, the hybrid nature of the filter material was realized. The focus of the project was to optimize the workability of the hybrid media in the anaerobic filters (AF) of decentralized wastewater systems in Pakistan. Additionally, the project also investigated the enhanced efficiency and feasibility of employing membrane treatment in place of anaerobic filters, thus eliminating the need for an intermediate treatment step altogether. By enhancing wastewater treatment in the anaerobic filter through innovative hybrid media or by thoughtful substitution of membrane technology in the place of anaerobic filters, the need for constructed wetlands (CW) for polishing domestic wastewaters was appreciably reduced. This considerably reduced the vacant land requirements for sewage treatment facilities for newly developing and expanding municipalities.

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

Pakistan has recently been identified as a nation undergoing water scarcity due to exhausting ground and surface water reservoirs, predominant drought conditions, and shifting of freshwater to more persistent domestic as well as industrial uses. Therefore, the hunt for nonconventional water resources for irrigation and other uses has become an area of prime focus. These other sources include wastewater which is already generated in ample amounts across the country. Currently, 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 1,299m³ to 1,100m³ in 2006. It is further projected that the water availability will be decreased further leaving less than 700m³ per capita by 2025 (Murtaza and Zia, 2012)

2.1.1. Secondary and Tertiary treatment systems:

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 Physio-Chemical Treatment. The Biological treatment technologies are wider spread due to its efficiency in removal of organic contaminants. These Biological technologies are again classified into Aerobic Treatment and Anaerobic Treatment. Aerobic treatment is the treatment that involves the utilization of oxygen throughout the process to convert organic matter into CO_2 and H_2O . On the other hand, an-aerobic treatment as the name implies is a microbial degradation process of organics that take place in the absence of oxygen followed by the production of methane gas. However, 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 odour problems (Aamir Khan, 2019)

For the study, Anaerobic Baffled Reactor (ABR) was first operated at three different HRT of 8, 10, and 12 hours. The treated effluent from the ABR was then fed into two parallel Anaerobic Filters (AF) followed by polishing through a flat sheet Woven Fibre Micro-Filtration Membrane (WFMF). In the first stage, the treatment performances of Kaldnes media and simple PVC media (having diameter of 15 mm and length of 25 mm) were compared. In the second stage, the length of the PVC pipe was modified to 20 mm and its treatment performance was subsequently compared to the PVC pipe of length 15mm to determine which size is considered the most optimum for the operation of full-scale plant. In the third and the final stage, the inlet to the membrane tank and it permeate were tested for the COD removal and turbidity. The TMP of the membrane was also monitored side by side.

2.1.2. Performance of WFMF membrane and PVC filter media (20mm length):

The results below showed that the filter media constructed using 20mm PVC pipes gave a COD removal efficiency of 85% whereas the filters media constructed using 15mm PVC pipes gave a removal efficiency of 86%. However, 20mm PVC is considered the most optimal because 15mm PVC although giving better results can lead to shape distortion of media in the bottom layers of the filter due to its small size, hence it should be avoided. As for the tertiary treatment, the membrane had a threshold value of 20kPa after which its fouling occurred. It was found out that membrane was able to remove 99% of suspended solids and reduced the turbidity up to 3.77 NTU giving an overall efficiency of 98%.



Figure 1. COD removal in stage 2. Source: (Aamir Khan, 2019)



Figure 2. COD removal in Stage 2. Source (Aamir Khan, 2019)



Figure 3. Turbidity removal in Stage 3. Source: (Aamir Khan, 2019)

2.2. Treating NUST Domestic Wastewater using Decentralized Wastewater Treatment System

The two main objectives of this research were to compare the treatment efficiency of filter media comprising of PVC pipes having 20mm length with filter media comprising of PVC pipes of 25mm length and secondly to assess the performance of pure GAC filter media. Domestic wastewater from NUST was preferred for the experimentation. A combination of Anaerobic Baffled Reactor (ABR) and Anaerobic Filter (AF) was used. The setup was further equipped with Membrane Filter (MF) in later stages to enhance removal efficiencies of various parameters. The system was designed at an HRT of 24 hours and a flow rate of 1.75 L/hr, which was maintained by using a peristaltic pump. The inlet pipe was connected from the NUST MBR plant to the setup. The inlet pipe entered the 100-liter storage tank. From there, the wastewater was pumped to ABR through peristaltic pump. The wastewater in ABR flowed through different compartments and finally entered AF. Inside the AF, wastewater moved against gravity, encountering biofilm development onto the filter media. The effluent then entered the membrane tank for further treatment.

2.2.1. Treatment efficiency of Granular Activated Carbon (GAC) filter media:

The results for the GAC filter were the most important part of the experiment. TSS values for influent ranged from 95-100 mg/L with an average value of 98 mg/L. However, after treatment through ABR and AF (GAC), TSS values dropped to 12-14 mg/L with an average value of 13 mg/L giving a removal efficiency of 86.7%.



Figure 4. Removal efficiency of TSS from GAC filter media. Source: (Treating NUST's Domestic Wastewater using DWTS, 2020)

TP values for influent ranged from 15-17 mg/L with an average value of 16 mg/L. However, after treatment through ABR and AF (GAC), TP values dropped to 5-6 mg/L with an average value of 5.4 mg/L giving a removal efficiency of 66%.



Figure 5. Removal efficiency of TSS from GAC filter media. Source: (Treating NUST's Domestic Wastewater using DWTS, 2020)

TKN values for influent ranged from 37-40 mg/L with an average value of 38 mg/L. However, after treatment through ABR and AF (GAC), TKN values dropped to 28-29 mg/L with an average value of 28.5 mg/L giving a removal efficiency of 25.7%. The enhanced removal efficiency using GAC accounted for its high adsorption capacity for dissolved organic nitrogen. Although, the physical adsorption capacity of GAC degrades with time since the filter media becomes exhausted, but biofilm-based degradation for dissolved organics and particulate matter improves.



Figure 6. Removal efficiency of TSS from GAC filter media. Source: (Treating NUST's Domestic Wastewater using DWTS, 2020)

COD values for influent ranged from 335-350 mg/L with an average value of 343 mg/L. However, after treatment through ABR and AF (GAC), COD values dropped to 95-98 mg/L with an average value of 97 mg/L giving a removal efficiency of 72%. This led to another conclusion that GAC was a better filter media compared to PVC media (20 mm length) which showed removal of 61% only.



Figure 7. Removal efficiency of TSS from GAC filter media. Source: (Treating NUST's Domestic Wastewater using DWTS, 2020)

The pH and ORP of the treated effluent were also measured. It had an average pH of 7.43 which complied with the NEQS (pH: 6 - 9). A good anaerobic system has an ORP range of -300 to -450 mV. When PVC media (20 mm length) was replaced with AF (GAC), conditions again became aerobic for AF, ORP was increased to 22 mV. However, with the passage of time, anaerobic conditions prevailed and ORP gradually dropped to -268 mV. For ABR, conditions continued in anaerobic phase since there was no change in operational condition of ABR.

GAC filter media proved to be the most optimal solution however, the major disadvantage was that it is costly and needs subsequent regeneration of GAC.

2.3. Trace organic compounds' persistence and elimination in centralized and decentralized wastewater treatment systems

The retention capacity of organic compounds in treated wastewater from Decentralized Wastewater Treatment Systems (DEWATS) and centralized Wastewater Treatment Plants (WWTPs) is becoming a matter of concern because of their ability to cause potential damage to both the environment and human health. For this research, a Non-Targeted Analysis (NTA) technique was used with extensive gas chromatography (two-dimensional) in combination with time-of-flight mass spectrometry to evaluate the concentration and frequency of persistent and removed organic compounds in the centralized WWTPs located in USA and South Africa as well as in DEWATS located in South Africa. As in whole, it was assessed that the removal efficiency of chemical compounds for both the plants were similar to the concentration of compounds present in the inlet and the outlet stream of each treatment plant. However, the DEWATS which employs the anaerobic treatment process has been identified to have a higher Hydraulics Retention Time (HRT) as well as Solids Retention Time (SRT) and was able to remove additional 13 chemical components displaying a greater reduction capacity in normalized peak areas compared to the two centralized WWTPs. Further, out of the total 111 common compounds temporarily detected in all three inlet streams,11 remained in all replicas, including 5 compounds that were not detected previously in WW systems. No visible differences were found between the physical and chemical properties of the retained and extracted compounds, however, significant differences could be seen between some of the molecular descriptors. These obtained results hold important applications for the treatment of trace organic chemicals in decentralized and centralised WWTPs and subsequent control monitoring of new compounds present in outlet stream of WWTP. (Mladenov, et al., 2022)

2.4. Contribution of Microparticles in membrane fouling in an anaerobic baffled reactor from acidogenesis to methanogenesis phases

The primary impurity responsible for fouling in Anaerobic Membrane Bioreactors (AnMBRs) was categorised as microparticles ranging in size from 0.45 μ m to 10 μ m. The properties and fouling capacity of micro-particles are well understood in completely mixed and single-stage reactors, but the presence of microparticles in multi-stage anaerobic bioprocesses remains a mystery. The composition and fouling potential of microparticles were investigated using a lab-scale anaerobic baffled reactor having four compartments namely (C1-C4). The microparticles contained an increasing percentage of total organics in the top supernatant but a decreasing

concentration from C1 to C4, according to photometric analysis. Dead-end and longterm filtration experiments demonstrated that C1's top supernatant had a significantly higher fouling potential than C2-C4. The fouling rate was positively connected with the biomass accumulation rate, and the supernatant microparticles significantly accumulated in the form of cake layers for each compartment (68-95 percent of the total organics), notably the fraction of 1-5 µm. A considerable bio-phase separation occurred between C1 (acidogenesis) and C2-C4 (methanogenesis) based on reactor performance. In the supernatant microparticles, hydrolytic and fermentative bacteria from the families Enterobacteriaceae, Veillonellaceae, and Streptococcaceae were prevalent, notably in C1, which explains a connection with biomass accumulation rate and subsequent fouling. The microparticles in the acidogenesis phase exhibited a higher fouling potential, as evidenced by the above findings. In conclusion, the findings imply that acidification and pre-hydrolysis with feedstocks, as well as the construction of AnMBRs using membrane units and multi-phase anaerobic processes, may be useful to fouling control. (Yang, et al., 2022)

2.5. Evaluating the performance of an anaerobic baffled reactor for pre-treating black water: Potential application in rural China

Black water can be described as water that is highly concentrated with human waste, however it only accounts for a small percentage of home sewage. The possibility of use of a modified form of anaerobic baffled reactor (ABR) for pre-treating black water in rural China was investigated. The classification of microbial structure was also looked into to see whether it had any application potential. The ABR's configuration and structure was changed in response to its application demand. After comparing the HRTs of 24, 36, 48, and 72 h, a hydraulic retention time (HRT) of 48 h was identified as the optimal HRT. The ABR was able to achieve an average removal efficiency of 14.21% for ammonium nitrogen (NH4-), 94.05% for COD, 32.54% for TP, and 28.78% for TN throughout 112 days of continuous operation. After a 60-day continuous operation, samples from three separate compartments were taken for archaeal and bacterial community research using 16S rRNA. The ABR contained lot of bacteria responsible for causing microbial degradation as а well as methanogenic archaea. The bacterial compositions of the three samples were assessed and it was found out that they had identical classes of genus levels and phylum, however the percentages of bacteria varied between compartments. Archaea

distribution demonstrated a pattern of succession with flow direction. In general, the ABR performs well under a 48-hour HRT and has a lot of potential for practical use. (Zha, et al., 2019)

2.6. Elimination of micropollutants from municipal wastewater by adsorption on powdered activated carbon and separation by innovative precoat filtration

Organic micropollutants are removed from wastewater treatment plant (WWTP) effluent, which protects aquatic ecosystems in receiving water bodies. Advanced wastewater treatment with activated carbon adsorption is a promising technology. To reduce leakage of Powdered Activated Carbon (PAC) and desorption of dangerous compounds in the environment, the use of PAC necessitates a second separation stage. Preliminary PAC separation using precipitation and sedimentation in conjunction with a sand filter has proven to be effective thus far. Filtering separators in the form of candle filters are a space-saving alternative, although they have not yet been deployed on a large scale. The study entails looking into the process design in terms of hydraulic characteristics, retention, and an effective filtration mechanism. Following preliminary testing with a pressurised filter cell (PFC), a scale-up to a pilotscale candle filter is carried out under optimal conditions. The use of a cellulose precoat layer is required to protect the fabric from obstruction, to retain the PAC, and to sustain the flow. Cellulose with a medium fibre length and a C = 1 kgm 2 applied quantity appears to be suitable for obtaining high quality permeate throughput and clear filtrate. The combination of precipitants (FeCl3) and sedimentation lowers the total suspended solids (TSS) concentration of the wastewater PAC suspension, increasing throughput by a factor of seven. The candle filter precoat experiments reveal a complex superposition of the dominant filtration mechanism. Clogging filtration is the most common type of filtration at first. Separation within the precoat, as well as the formation of a thin cake of PAC particles, become increasingly important during filtration (Morsch, Möhlendick, Süsser, & Nirschl, 2021).

Table 3. Literature Review on DWTS and treatment using Hybrid filter media:

Author(s)	Research Title	Findings
My Thi Tra Ngo, et al.	Performance of flat sheet	The Nano filtration
	Nano filtration membrane	membrane has high

	system for secondary wastewater effluent.	potential to produce high quality recycled water with TOC removal greater than 93%.
Aušra Mažeikienė	Improving small scale wastewater treatment plant using filtering tertiary treatment unit.	Tertiary treatment unit filled with Filtrate P, capable of removing Phosphorus up to 91.8% and SS up to 99.4%
Diana Bernal, et al.	Key criteria for considering decentralization in municipal wastewater management.	DWTS are feasible for developing countries due to minimum energy, operational, maintenance costs and greater reuse potential.
Natalie Mladenov	Persistence and removal of trace organic compounds in centralized and decentralized wastewater treatment systems.	Wastewater effluent samples revealed that 11 compounds were persistent (P) and 67 were fully removed by centralized and decentralized treatment
J. Yang, et al.	Ultrafiltration as tertiary treatment for municipal wastewater reuse	UF as tertiary treatment for municipal wastewater concludes that permeate

had high quality and could be reused for nonpotable purposes as it met WHO and French guidelines for reuse.

Direct filtration for the treatment of the coagulated domestic sewage using flat-sheet ceramic membranes FSCM was effective in removal of SS, organics, TP and other pollutants from municipal wastewater. Precoagulation was effective as it mitigated the membrane-fouling problem.

Zhao, et al.

3.CHAPTER THREE: MATERIALS AND METHODS

3.1. Lab Scale Setup

A combination of 3 units that make up the Decentralized Wastewater Treatment Plant were designed and utilized at lab scale to carry out this research. The three units are Anaerobic Baffled Reactor, Anaerobic Filter and Membrane Tank.

3.1.1. Anaerobic Baffled Reactor

A lab scale ABR was fabricated with acrylic sheets. The overall thickness of the ABR was 6 mm. The ABR consisted of six chambers, each chamber had a gas outlet and a sampling port of 6 mm diameter. The main purpose of the sampling port was to check the performance of each chamber.

The total volume of ABR was 23 liters with effective volume of about 21 liters. Free Board of 2 liters was provided at the top.



Figure 8: Lab Scale ABR Design AutoCAD



Figure 9: ABR for Lab Scale Study

3.1.2. Anaerobic Filter Design

Similarly, anaerobic filter was also fabricated with the acrylic sheets having thickness of about 6 mm. The total volume of the reactor was 16.5 liters with effective volume of 15.33 liters. The total depth of the system was 35.5 cm (14 inches) with media depth of about 30.5 cm. The media used in the study was a Hybrid Media formed by coating Powdered Activated Carbon (PAC) on the outer surface of locally available corrugated tubes with diameter of 20 mm (Pipes used for collection of distilled water in ACs). The PVC media was cut into 15 mm length pieces as PVC media with 15 mm length and 20 mm diameter gives the maximum removal efficiency (Aamir Khan, 2019). A mesh having pore size of 12 mm was installed at the bottom of the media bed. It was placed 2.54 cm (1 inch) above the bottom of the reactor. Another mesh with pore size of 6 mm was placed at the top of the media bed just below the effluent line to prevent sludge escape into the effluent line. The purpose of both of these meshes was to prevent

fluidization.





Figure 10: Lab Scale AF AutoCAD



Figure 11: AF for Lab Scale Study

3.1.2.1. Preparation of Hybrid Media

The Hybrid Media was prepared by coating Powdered Activated Carbon on the outer surface of the locally available corrugated PVC pipe (pipe for collection of distilled water in ACs). For this purpose a binder **polyvinylidene difluoride** (PVDF) and a solvent N-N-Dimethyl was used. 220mL of N-N-Dimethyl was taken in which 12g of PVDF and 100g of PAC was added. Then the mixture was stirred at 300 rpm for 3 hours. After 3 hours a homogeneous solution was obtained. PVC pipes were cut in specific dimensions of 20 mm diameter and 15 mm length. Each piece of PVC pipe was covered with a thin layer of prepared slurry. The covered PVC pipe pieces were than oven dried for 24 hours at 60°C.





Figure 12: Virgin PVC Pipes

Figure 13: PVC coated with PAC



Figure 14: AF filled with Hybrid Media

3.1.3. Membrane Tank and Module Design

A 5 liter membrane tank was designed with acrylic sheets. Feed water was provided to the tank from above with the port located 1.5 inch below the opening. For discharge of excess water another port is provided parallel to the feed port. A sample port was also provided at the bottom.

The membrane used was a Flat Sheet Woven Fiber Microfiltration Membrane (WFMF). The flat sheet WFMF membrane modules consist of four key elements: a PVC frame, a permeate outlet which allows permeate to flow out of the module, two sheets of Woven Fabric membrane glued to either side of the frame, and two spacers between the sheets of fabric to keep the two membranes apart and allow fluid to flow into the module.

The module has a length of 15 cm and width of 10.5 cm with effective dimensions being 11 cm and 7.5 cm respectively. The effective dimension in this case refers to the dimension of the membrane area on which filtration process occurs. The membrane had a pore size of $1 - 3 \mu m$. The effective surface area surface area of the membrane was 0.0165 m² and the operational flux was 6 LMH which was maintained with a peristaltic pump throughout the experiment.

Table 4: Membrane Specifications

MEMBRANE SPECIFICATIONS

PORE SIZE	1-3 µm
OPERATIONAL FLUX	6 LMH
MAXIMUM ALLOWED TMP	30 KPa
EFFECTIVE SURFACE AREA	0.0165 m ²
OPERATION MODE	Dead End – Outside IN
CONFIGURATION	2 sheets pasted on both sides of the
	Module



Figure 15: Membrane Tank

3.2. Process Flow

Our study had two process flows each of them are described below.

3.2.1. System Design 1

Synthetic wastewater from a 50 liter Plastic Tank was provided to the ABR with the help of a peristaltic pump (Longer Precision Measuring Instrument China, BT300-2J). The feed water first entered the first chamber of the ABR and came in contact with the acclimatized anaerobic sludge present at the bottom. The feed water then rose up to enter the second chamber and water passed through all the six chambers following the same pattern till the feed water reached the effluent port. The treated wastewater effluent was then pumped to the AF. The AF was provided with flow from the bottom. The AF was gradually filled with the treated effluent from the ABR which came in contact with the PAC and the biofilm formed at the hollow center of the PVC. The treated wastewater was finally collected at the top. The treated effluent from the AF was then fed into the membrane tank. Permeate was collected from the top of the module with the help of a peristaltic pump. A water trapper and a TMP meter (Data Logging Manometer, as per Scientific USA, 15 psi) was installed with the permeate

line to check the TMP across membrane. Permeate was then tested for different parameters of COD, TKN and TP.



Figure 16: Process Flow Diagram of System Design 1



Figure 17: Experimental Setup System Design 1

3.2.2. System Design 2

Now in order to compare the efficiency of the hybrid media with media bed that consisted of PVC pipes of 15 mm diameter and 20 mm length. For this purpose we
had to mimic a pervious study "*Decentralized Wastewater Treatment System* (*DEWATS or DWTS*) for Medium Strength Domestic Wastewater". This study showed that the PVC pipe with dimensions of 20 mm length and 15 mm diameter with an HRT of 12 hours gave the maximum removal efficiency of COD, TKN and TP.

As the time was limited a synthetic feed of AF having PVC bed (length: 20 mm, dia: 15 mm) was prepared by using the recipe in section 3.4.1.2.

Then the synthetically prepared feed was tested and passed through the WFMF membrane. The membrane was operated with this feed for three runs that is till the membrane reached its limit of 30 KPa. The membrane clogged after an interval of three days after which it was chemically and physically cleaned.



Figure 18: Process Flow Diagram of System Design 2



Figure 19: Experimental Setup System Design

3.3. Equipment used in the Study

- I. Peristaltic Pump (Longer Precision Measuring Instrument China, BT300-2J)
- II. TMP Meter (Data Logging Manometer 15psi, Sper Scientific USA)
- III. COD Thermo-reactor (Velp ECO-25)
- IV. pH meter (Eutech pH700, USA)
- V. ORP meter (Hanna HI83141, USA)
- VI. UV-Vis Spectrophotometer (Specord 200, Analytik Jena, Germany)
- VII. TKN Analyzer (DK-6 Digestor, SMS Scrubber, JP Water Pump & UDK-149 Distillation unit, Velp Scientifica Italy)
- VIII. Turbidimeter

3.4. Seeding

For the startup of the ABR seed sludge was used. The sludge was collected from wetlands located at the back of ISRA apartments at NUST H-12 Campus. The sludge was collected from a depth of 12 feet to ensure that it was anaerobic. Each compartment of the ABR was fed with approximately 10% by volume of seed sludge. The anaerobic sludge had an ORP of -310mV which ensures that it was anaerobic.

3.4.1. Synthetic Wastewater

3.4.1.1. For System Design 1:

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 5: Synthetic Wastewater Recipe for Design 1

CHEMICALS

CONCENTRATION (MG/L)

DEXTROSE (C ₆ H ₁₂ O ₆)	520.00
AMMONIUM CHLORIDE (NH₄CL)	133.66
POTASSIUM DI HYDROGEN PHOSPHATE (KH₂PO₄)	17.15
CALCIUM CHLORIDE (CACL ₂)	4.87
MAGNESIUM SULFATE (MGSO4)	4.87
FERRIC CHLORIDE (FECL ₃)	0.50
SODIUM HYDROGEN CARBONATE (NAHCO₃)	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

3.4.1.2. For System Design 2:

The average concentration of the effluent from AF having PVC Bed (length: 20mm, dia: 15mm) 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. The same chemicals used in design 1 were used in design 2.

Table 6: Synthetic Wastewater Recipe for Design 2

CHEMICALS CONCENTRATION (MG/L)

DEXTROSE (C ₆ H ₁₂ O ₆)	79.56
AMMONIUM CHLORIDE (NH ₄ CL)	42.78
POTASSIUM DI HYDROGEN	11.61
PHOSPHATE (KH2PO4)	
CALCIUM CHLORIDE (CACL ₂)	0.974
MAGNESIUM SULFATE (MGSO4)	0.974
FERRIC CHLORIDE (FECL ₃)	0.1

SODIUM HYDROGEN CARBONATE (NAHCO ₃)	16.0
COBALT CHLORIDE (COCL ₂)	0.01
ZINC CHLORIDE (ZNCL ₂)	0.01
NICKLE CHLORIDE (NICL ₂)	0.01

3.5. Experimental Runs

This research was carried out in two runs for the purpose of comparison between tertiary treatment technologies that is between conventional PVC media bed and newly formed hybrid media bed in AF. Anaerobic conditions were ensured through regular monitoring of pH and ORP during all the phases.

3.5.1. Phase 1:

It consisted of ABR, AF with Hybrid Bed and WFMF membrane. This phase was of about 3 months with two months of prior acclimatization. The setup started in January 2022 followed by two months of acclimatization of ABR. After two months of acclimatization the system was rum for another three months from March, 2022 to May, 2022. The ABR was run at an HRT of 12 hours whereas the AF with an HRT of 12 hours. The AF was filled with the hybrid media that was prepared as discussed in section 3.1.2.1.

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. The ABR was given continuous flow of feed water at 2 rpm.

The AF (Hybrid Bed) feed was fed into the Membrane tank. Membrane module submerged in the reactor having surface area of 0.0165 m² was operated at a flux of 6 LMH and 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. The membrane was given three runs. Each run ended when the membrane reached its limit of 30 KPa. Each run took about 5 days to complete. After each run the membrane was physically and chemically cleaned. After each run the treated effluent from the membrane was tested twice for COD and Turbidity.

3.5.2. Phase 2:

Second phase consisted of ABR, AF with virgin PVC media having dimensions 20 mm diameter and 15 mm length followed by WFMF membrane. A study in 2019 on "Decentralized Wastewater Treatment System (DEWATS or DWTS) for Medium Strength Domestic Wastewater" was carried out at IESE, NUST that followed the same process flow as our phase 1. As the study could not be repeated again for the purpose of comparison between virgin PVC media bed performance and PAC coated PVC media bed in AF due to time constraints a synthetic feed of exactly same composition as treated effluent from the AF with virgin PVC media was prepared in the lab by using the results from the study. The synthetic feed was prepared once a week. The synthetic AF feed was fed into the Membrane tank and permeate was collected through a peristaltic pump. The membrane was given three runs. Each run ended when the membrane reached its limit of 30 KPa. Each run took about 5 days to complete after which the membrane was physically and chemically cleaned. For each run the membrane effluent was tested twice for COD and Turbidity.

3.5.3. Phase 3:

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 0.0165 m² was operated at a flux of 6 LMH and 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. The membrane was given three runs. Each run ended when the membrane reached its limit of 30 KPa. Each run took about 5 days to complete. After each run the membrane was physically and chemically cleaned.

3.6. Membrane Setup, Resistance and Cleaning

To evaluate the performance of the WFMF membranes pure water flux experiment was performed.

In order to check any leakages in membrane rejection performance of the membrane in the filtration of limestone solution containing particles of 5 µm was observed.

3.6.1. Pure water flux experiments

Pure water flux experiments involve filtering distilled water using membranes, at different pump speeds. The procedure was as follows:

- > Fill the feed vessel with distilled water.
- Place the membrane pack in the feed vessel and leave it for 10 minutes to allow the pressure within the membrane modules to equalize
- Set the pump to a particular speed, and then let it run for 10 minutes before taking any measurements. This will allow it to stabilize.
- Thereafter, measure the time taken to collect a certain volume of water and the corresponding pressure drop (ΔP) across the membrane using a stopwatch and a pressure gauge, respectively. Repeat this procedure at different pump speeds. The volume of permeate collected should be constant at the various pump speed
- > The pure water fluxes of the membranes are then calculated using Equation 1.

Equation 1

$$J_o = \frac{v}{A \times \Delta t}$$

where J_0 is the pure water flux (L/m² h or LMH), v is the volume of water collected (L), A is the effective area of the membranes (m²), and Δt is the time taken to collect the water (h).

Pure water flux curves can then be plotted using the calculated flux and the ΔP .

The pure water flux results can also be used to calculate the intrinsic membrane resistance. Equation 2 shows an expression used to calculate this resistance.

Equation 2

$$\boldsymbol{R}_{\boldsymbol{m}} = \frac{\Delta \boldsymbol{P}}{\mu \boldsymbol{J}_{o}}$$

Where, R_m is the intrinsic membrane resistance (m⁻¹), ΔP is the pressure drop across the membrane during water filtration (Pa), μ is the water viscosity (Pa.s) and J_o is the pure water flux.

3.6.2. Testing of Module

Before running any experiments, the membrane was tested for any leakages for this purpose a limestone solution having 5µm particle size was passed through the membrane. The following procedure was followed.

- Fill the feed vessel with distilled water. Set the pump to a certain speed and allow it run for 30 minutes. This will ensure that any starch on the membrane fabric is washed out.
- Maintain the set pump speed
- > Replaced the distilled water with limestone solution of desired concentration.
- Run the pump at the set speed. During the filtration process, measure the permeate turbidity at a 3-minute interval.
- Stir the feed to maintain a homogeneous solution. In addition, add more feed continuously to the vessel so as to maintain a constant feed level.
- Continue with filtration process until the permeate turbidity levels off and becomes constant. If a pump speed of 100 rev/min is chosen, the permeate turbidity should level off to a value of less than 0.5 NTU within the first 10 minutes. If the permeate turbidity is high than 1 NTU and keeps on fluctuating, then there is a possibility of leakage

3.6.3. Filtration experiments

The feed filtration process using the WFMF membrane was carried out as described below:

- > First, carry out the pure flux experiment as described in section 1.3.1.
- Then set the pump to a particular pump speed that will be used in the feed filtration process.
- Transfer the membrane pack to a tank containing the feed and leave it for 10 minutes to allow the pressure within the modules to equalize.
- > At the earlier set pump speed, start the filtration process. Then measure the permeate turbidity, permeate volume collected, and the pressure drop (ΔP) across the membranes after every 5 minutes until the end of the filtration process.

Throughout the filtration process, add the feed continuously to the tank so as to maintain a constant feed level. The permeate volume and ΔP results are then used to calculate filtration fluxes and resistances. The filtration fluxes and resistances at pre-defined intervals were calculated using Equation 3 and Equation 4, respectively. Thereafter, resistance profiles were generated.

Equation 3

$$J = \frac{v}{A \times \Delta t}$$

Where, J is the permeate flux (LMH), v is the volume of permeate collected (L), A is the effective area of the membrane (m²), and Δt is the time taken to collect the permeate (h).

Equation 4

$$\boldsymbol{R}_t = \frac{\Delta \boldsymbol{P}}{\mu \boldsymbol{J}_o}$$

Where, R_t is the membrane resistance during the filtration process (m⁻¹), ΔP is the pressure drop across the membrane during filtration process (Pa), μ is the permeate viscosity (Pa.s), and J is the permeate flux (LMH).

3.6.4. Cleaning of the Membrane

Fouled WFMF membranes can cleaned using various methods. These methods include water scouring, i.e. simple rinsing under tap water, air scouring, backwashing, brushing and finally sodium hypochlorite soak. Among the mentioned techniques, it is only hypochlorite soak followed by brushing that can restore the fouled membranes to their original permeability. To completely restore fouled WFMF membranes, the following was done:

- First, rinse the fouled membranes under tap water to remove any visible fouling layer.
- Second, soak the rinsed modules in 0.03% hypochlorite (also known as 'jik') solution, and let them stay in the solution overnight.
- Third, rinse and brush the membranes to remove any remaining foulants. Repeat the rinsing process twice or thrice.

- Finally, measure the pure water fluxes of the membranes to see if the original permeability has been restored.
- If the original permeability has not been restored, rinse and brush the membranes again. Then repeat the pure water flux experiment.
- This cleaning process is usually carried out at the end of every filtration process.

For interval cleaning, i.e. in between filtration process, water scouring can be used. Water scouring simple involves cleaning the membranes under a tap or using water from a hose pipe.

3.7. Analytical methods & their removal mechanisms

In this study 5 parameters were regularly monitored. pH and ORP were regularly monitored to maintain anaerobic state within the system at all times. To check the performance of the hybrid bed in AF COD, TP and TKN were determined to check removal efficiency of hybrid bed and to draw comparison between the PVC Bed and Hybrid Bed. Turbidity and COD were checked after each run of the membrane to check the performance of a low cost WFMF membrane.

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 CO2 and H2O 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 H2O and CO2 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.

aC₆H₁₂O₆ + b O₂ -----> x CO₂ + y H₂O

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 CO2 and H2O 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 (H2SO4) 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). 45 Chemicals, Glassware & Equipment required for COD analysis.

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/ Digester

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

$N_1.V_1$ (Std. Potassium Dichromate Reagent) = $N_2.V_2$ (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).
- 2. 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).
- 4. 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^oC and timer for 2 hours.
- 6. After 2 hours of digestion, remove the vials from the digester and place them in test tube rack.
- 7. 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 colour is still yellowish, pour the contents into titration flask and add 2-3 drops of Ferroin indicator.
- 9. Now start adding FAS from burette drop wise to it. A blue colour will be achieved which is due to Cr⁺³ ions but ignore this and continue adding titrant until a reddish-brown colour 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.

N₁.V₁ (Std. Potassium Dichromate Reagent) = N₂.V₂ (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°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
- Bulb Condenser
- 800mL Kjeldahl Flask or any Round bottom flask that can withstand temperature up to 400°C
- 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^oC 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 H₂SO₄

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:

N1V1 = N2V2

0.1N * V1 = 0.02N * 1000mL

V1 = 200mL

Procedure

- 1. Take 100 ml of sample in a 500 ml volumetric flask and dilute it to 300 ml with distilled water.
- 2. Add 50 ml of digestion reagent
- Heat it inside a fume hood at 360 to 400°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.
- 4. Cool the flask and dilute the solution to 300 ml with distilled water
- 5. Adjust the pH using 0.1N NaOH and phenolphthalein as indicator
- 6. 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-Na₂S₂O₃ into the Kjeldahl flask. Quickly connect the bulb condenser to the flask so that ammonia loss can be prevented.
- 8. 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.

- 9. 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.
- 10. As distillation is started, the color of the boric acid solution will start changing from purple to green.
- 11. In the end, take the mixed indicator solution and titrate it against 0.02N Sulfuric acid until the first pink shade appears.
- 12. Repeat the same procedure with 100 ml of blank. Calculate TKN using the following equation

$$TKN \left(\frac{mg}{L} \right) = \frac{(A-B)x \, 280}{mL \, of \, sample}$$

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

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 H₂SO₄

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

- 1. Take 20mL sample into Velp Digestion tube
- 2. Add 2 tablets of Kjtabs VCM A00000274
- 3. If you don't have Kjeldahl tabs, then add 7g Potassium Sulfate and 0.2g Copper Sulfate
- 4. Add 20mL concentrated sulfuric acid into it
- 5. Turn ON the DK-6 digestor
- 6. Put the Steel cap on tubes and turn ON the JP Recirculating pump
- Heat the tubes at 160°C for 30min, then at 260°C for 30min, then at 360°C for 30min and finally at 420°C for 30min.
- 8. Wait for digestion to complete
- 9. Turn ON the UDK-149 distillation unit and wait for Pre-Heating to complete.
- 10. Put the digested sample tube in distillation unit.
- 11. Add 40mL distilled water and 25mL 30% NaOH into the tube by selecting the appropriate program.

- 12. Add 25mL 4% Boric Acid in titration flask and put it at the end of distillation pipe.
- 13. Start the distillation process at 50% steam power for 5min.
- 14. After distillation is complete, take the flask, add 0.5mL Mixed Indicator and titrate against 0.02N H₂SO₄.
- 15. Calculate TKN using the following equation

TKN (mg/L) = ((A-B)x 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 molybdophosphate 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 $KH_2PO_4 = \frac{31}{39+2+31+64} \times 220$
- > 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. HCI. 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

- 1. Prepare Phosphorus concentrations of 5 mg/L, 10 mg/L, 15 mg/L and 20 mg/L from the stock Phosphate solution.
- 2. Take 50 mL of each standard in different 250 mL flasks.
- 3. Take 50 mL of distilled water in a separate 250 mL flask.
- 4. 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).
- 6. 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.
- 8. 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.
- 10. After this, make it up to 50 mL with distilled water. The digested sample is then further tested for total phosphate.
- 11. Now take 10 mL from each of the 6 flasks in a separate 25mL flasks.
- 12. Add 2 mL of Ammonium Molybdate-Metavanadate solution into it.
- 13. Wait for 15-20 minutes for color development.
- 14. Turn ON the UV/Vis Spectrophotometer (Analytic Jena, Specord 200 Plus).
- 15. Click on INITIALIZE DEVICE.
- 16. Go to Wavelength selection window and select 470 nm.
- 17. Click on MODE and select Absorbance mode.
- 18. Pour the contents from the flask containing digested blank into a quartz cuvette.
- 19. Put it in the spectrophotometer's Reference slot.
- 20. Click on REFERENCE.
- 21. Now pour the remaining contents of this blank into another cuvette and put that cuvette in spectrophotometer's Measurement slot.
- 22. Click on Measure. After a few seconds, the absorbance will be displayed on the screen. This absorbance should be 0.00.
- 23. 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.
- 24. From the absorbance values of the blank and standards, construct a calibration curve of concentration vs. absorbance.
- 25. Now put the prepared sample into the cuvette and measure its absorbance.
- 26. 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

- 1. Turn ON the meter.
- 2. Rinse the probe with distilled water.
- 3. Press the CAL button to enter calibration mode.
- 4. Dip the probe into pH 7 buffer, wait for value to stabilize and press ENTER.
- 5. 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.
- 7. If the meter doesn't go to Measurement mode itself, you have done something wrong and you'll have to repeat the whole procedure.
- 8. 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 means 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 \pm 10 mV. These standard solutions can be Light's solution, ZoBell's solution or a Quinhydrone solution (Eaton et al., 2005).

ZOBELL'S SOLUTION	428MV
LIGHT'S SOLUTION	675mV
QUINHYDRONE AT PH 4	482mV
QUINHYDRONE AT PH 7	285mV

Table 7: Potentials of Standard ORP solutions at 25°C vs NHE

4. CHAPTER FOUR: RESULTS AND DISCUSSION

In this chapter, we will be assessing the performance efficiency of the following secondary and tertiary treatment systems:

- 1. ABR \implies AF (virgin PVC filter media) and ABR \implies AF (Hybrid media).
- ABR → AF (virgin PVC filter media) → WFMF membrane and ABR → AF (Hybrid media) → WFMF membrane

The treatment technology which is considered the most optimal in terms of cost, efficiency and effectiveness is then proposed on the basis of the results drawn.

4.1. Phase I: Performance efficiency of ABR combined with AF composed of Virgin PVC bed

The effluent from the ABR was fed into the AF. The AF is the filter bed made up of layers of corrugated PVC pipes having length of 20mm and diameter of 15mm arranged in the reactor. Both ABR and AF were run at an HRT of 12 Hrs. The effluent from the AF was then tested for the following parameters:

- 1. Total Phosphorus (TP)
- 2. Total kjeldahl Nitrogen (TKN)
- 3. COD
- 4. pH and ORP
- 5. Turbidity

4.1.1. Results for the removal of Total Phosphorus during Phase I:

The incorporation of phosphate into TSS and subsequent removal from these solids is how phosphorous is removed from wastewater. Phosphorous can be found in chemical precipitates as well as biological solids (such as microorganisms). In anaerobic zone, the removal of phosphorus occurs through Phosphorus Accumulating Organisms (PAOs). Such organisms use phosphorus as a growth material (Goodfellow et al., 2004).

The HRT of both ABR and AF was set at 12 hours because according to previous studies, TP removal increases as the HRT of the system increases. The trend was also explained by Hahn and Figueroa who achieved 65% TP removal at HRT of 15

days (Hahn & Figueroa, 2015), 22% at an HRT of 12 hours and only 13% TP removal at an HRT of 8 hours.

The ABR-AF effluent was tested for TP twice a week for three months (March, April and May). The mean value of the tested parameter for each month was used to plot the final graph. The Figure 20. Below shows the results and the removal efficiency of TP.



Figure 20. TP removal in ABR-AF (PVC media) during Phase I

The TP removal increased gradually because of the formation of biofilm of anaerobic microbes which accounts for increased accumulation of biomass. The trend in the graph shows that the concentration of phosphorus in the influent feed water which was 15 mg/L was reduced to 10 mg/L after passing through the ABR. And this value was further reduced to 7.7 mg/L after passing through the AF (PVC media). The overall removal efficiency of TP was 49.33%.

However, this removal percentage is still less than that of the aerobic systems where phosphorus removal is comparatively higher than anaerobic systems. It is because in anaerobic systems the accumulation of biomass on biofilm is less due to longer SRT lesser sludge wastage (Aamir Khan, 2019).

4.1.2. Results for the removal of Total Kjeldahl Nitrogen during Phase I:

Nitrification and denitrification are the biological processes that are responsible for the removal of nitrogen from wastewater. Denitrification is the conversion of nitrate to nitrogen gas by heterotrophic bacteria in the absence of oxygen using organic matter as a carbon source and nitrate as an electron acceptor (Assefa et al., 2019). The

factors that affect the performance activity of nitrifying bacteria and determine the rate of nitrification process include: environmental factors (such as temperature, pH, microbial population, nitrogen conc. and organic carbon content) as well as operational. Increasing population of of nitrifying bacteria also accounts for the effectiveness of wastewater treatment process further optimising the elements impacting the nitrogen removal process. The temperature ranging from 15 to 21° C exhibits stable nitrification performance for both lab scale and full-scale WWTP, which could be attributable to the high activity of nitrifying bacteria. On the other hand, lower temperatures hinder the action of nitrifying bacteria, resulting in a reduced nitrification activity in the winter. As a result, increasing sludge age in cold temperatures necessitates a larger MLSS concentration to maintain stable nitrification performance. The sludge age also affects the temperature. It is discovered that if the sludge is aged 20 days above, it reduces the negative impacts of low temperature on nitrifying activity. MLSS concentration was discovered to be one of the most important parameters determining nitrification performance in WWT using this correlation technique. (Komorowska-Kaufman et al., 2006)

The removal of nitrogen hence depends upon the concentration of biomass and HRT. Like TP, TKN removal increases with the increase in HRT. The ABR-AF effluent was tested for TKN twice a week for three months (March, April and May). The mean value of the tested parameter for each month was used to plot the final graph. The Figure 21 shows the results and the removal efficiency of TKN.



Figure 21. TKN removal in ABR-AF (PVC media) during Phase I

The TKN removal increased gradually because of the formation of biofilm which accounts for increased biomass concentration. Secondly, TKN removal also depends on HRT, for 12 hours of HRT in ABR, the amount of TKN was 15.5 mg/L (having removal efficiency of 64.5%) but when the ABR effluent was further treated in AF at an additional HRT of 12 hours, the amount of TKN further reduced to 10.5 mg/L giving a removal efficiency of 70% at the end.

4.1.3. Results for the removal of Chemical Oxygen Demand during Phase I:

COD is one of the most important parameters for assessing the quality of treated wastewater because COD is mainly responsible for causing pathogenic pollution and toxicity in wastewater. A high COD value indicates an increased level of oxidizable organic matter and reduced amount of DO. In anaerobic treatment COD removal takes place by the action of anaerobic bacteria to convert organic molecules in wastewater to methane gas, CO₂ and additional biomass. The microbial degradation is preferred because the biofuel produced can be used as an alternative energy source. Anaerobic processes usually have better COD removal as compared to Aerobic processes.

Studies show that the COD removal also increase with an increase in HRT. For our system, the ABR-AF effluent was tested for COD twice a week for three months (March, April and May). The mean value of the tested parameter for each month was used to plot the final graph. The Figure 22 shows the results and the removal efficiency of COD with time.



Figure 22. COD removal in ABR-AF (PVC media) during Phase I

The bar graph shows that the amount of COD in ABR effluent stream was almost 150 mg/L which was further reduced to 75.1 mg/L after passing through the AF and the reason is the formation of biofilm on the PVC filter media which breaks down the COD into its main components. The green trend line shows that the COD removal efficiency in starting was 78.12% which increased up to 85.04% at the end, hence showing the performance efficiency of AF (PVC media).

4.1.4. pH and ORP of the system during Phase I:

pH is a precise measurement of the concentration of hydrogen ions in a solution, and ORP is a relative measure of chemicals that cannot distinguish one from another. ORP is usually used to assess the working conditions of sludge in ABR. The ideal values of ORP for anaerobic digestion lies in the range of -100 mV to -300 mV as for the pH, the value greater than 6.8 indicates optimal operating conditions

The pH and ORP of the both the systems i.e.; ABR and AF were monitored regularly. The average pH value in the ABR reactor was found to be 7.3 whereas in AF (simple PVC media) the pH value was 7.14 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). The reason identified is that the value above or below this range, the biogas production and pollutant removal is affected. ABR and AF effluents was tested separately for pH twice a week for three months (March, April and May). The mean value of the tested parameter for each week was used to plot the final graph. The Figure 23 shows the pH trend over the period of three months:



Figure 23. pH of ABR and AF (PVC media) during Phase I

It can be seen that with passing time, reactor moved towards anaerobic condition which explains why 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 as mentioned before. According to the literature, an ORP of -170 to -400 mV is considered ideal. The higher the negative value of ORP, the better the prevalence of anaerobic conditions in the reactor. Positive ORP value means an environment capable of oxidation which is not preferable for anaerobic treatment. Acclimatized anaerobic sludge from bottom of wetlands usually has an ORP of < - 400mV but since we collected the sludge from the bottom of the constructed wetland near the ISRA apartments, 2 months were needed to acclimatize the sludge in the reactor and bring the system in operational conditions. The measured ORP in the early stages lied in the range of -180 mV to -200 mV but when nitrogen purging was done (to maintain anaerobic conditions) and the system got acclimatized after 2 months of operation, the ORP dropped to -330mV. The average ORP of the 2 systems i.e. ABR, AF (PVC media) -326 mV. The Figure 24 shows the ORP trend over the period of three months:



Figure 24. ORP of ABR and AF (PVC media) during Phase I

4.1.5. Turbidity removal in AF (PVC media) during Phase I:

Turbidity is the cloudiness and haziness of wastewater caused due to prescience of suspended, dissolved and colloidal solids, mainly suspended solids are responsible. Turbidity in wastewater can be removed in secondary treatment through ABR by the process of settling and decanting of solids and particulate matter. About 80-90% of SS are removed in ABR reactor due to increased SRT and HRT of 12 hours. However,

some suspended and colloidal solids still remain persistent and are removed in the later stages by AF and further by membrane (tertiary treatment if required).

The AF effluent was tested for turbidity using turbidimeter twice a week for three months (March, April and May). The mean value of the tested parameter for each week was used to plot the final graph. The Figure 25 shows the turbidity trend over the period of three months:



Figure 25. Turbidity of AF (PVC media) during Phase I

The graph shows that with time, turbidity removal increases. In the first run i.e.; the month of March, the removal efficiency was 82.61% but enhanced up to 84.35% by the end of May which is a fairly good value. The reason that can be attributed to this high removal efficiency is the formation of biofilm on PVC filter bed that retains any suspended solids and colloidal solids present in the incoming stream. The remaining colloidal and dissolved solids can be removed further in the tertiary treatment.

4.2. Phase II: Performance efficiency of ABR combined with AF composed of Hybrid media (PAC coated on PVC)

The effluent from the ABR in the second phase was fed into the AF (Hybrid media). The AF here is the filter bed made up of layers of corrugated PVC pipes having length of 20mm and diameter of 15mm coated with Powdered Activated Carbon (PAC) arranged in the reactor. Both ABR and AF were run at an HRT o 12 Hrs. The effluent from the AF was then tested for the following parameters:

- 1. Total Phosphorus (TP)
- 2. Total Kjeldahl Nitrogen (TKN)
- 3. COD
- 4. pH and ORP
- 5. Turbidity

4.2.1. Results for the removal of Total Phosphorus during Phase II:

The HRT of both ABR and AF was set at 12 hours because according to previous studies, TP removal increases as the HRT of the system increases. The ABR-AF (hybrid) effluent was tested for TP twice a week for three months (March, April and May). The mean value of the tested parameter for each month was used to plot the final graph. The Figure 26 shows the results and the removal efficiency of TP.



Figure 26. TP removal in ABR-AF (Hybrid media) during Phase II

It can be seen that the TP removal increased gradually because of the formation of biofilm of anaerobic microbes which accounts for increased accumulation of biomass. The removal efficiency is also much higher than simple PVC AF because of the external coating of PAC on PVC that causes adsorption of Phosphorus hence reducing the overall concentration of phosphorus. The trend in the graph shows that the concentration of phosphorus in the influent feed water which was 15 mg/L was reduced to 11 mg/L after passing through the ABR. And this value was further reduced to 6.3 mg/L after passing through the AF (PVC media). The overall removal efficiency of TP at the end of process was 55.33%.

4.2.2. Results for the removal of Total Kjeldahl Nitrogen during Phase II:

The removal of nitrogen hence depends upon the concentration of biomass and HRT. Like TP, TKN removal increases with the increase in HRT. The ABR-AF effluent was tested for TKN twice a week for three months (March, April and May). The mean value of the tested parameter for each month was used to plot the final graph. The Figure 27 shows the results and the removal efficiency of TKN.



Figure 27. TKN removal in ABR-AF (Hybrid media) during Phase II

The TKN removal increased gradually because of the formation of biofilm which accounts for increased biomass concentration. The removal efficiency is also much higher than simple PVC AF because of the external coating of PAC on PVC that causes adsorption of Nitrogen hence reducing its overall concentration. Secondly, TKN removal also depends on HRT, for 12 hours of HRT in ABR, the amount of TKN was 15.5 mg/L (having removal efficiency of 72.2%) but when the ABR effluent was further treated in AF at an additional HRT of 12 hours, the amount of TKN further reduced to 6.5 mg/L giving a removal efficiency of 79.71% at the end of the process.

4.2.3. Results for the removal of Chemical Oxygen Demand (COD) during Phase II:

Studies show that the COD removal also increase with an increase in HRT. For our system, the ABR-AF effluent was tested for COD twice a week for three months (March, April and May). The mean value of the tested parameter for each month was



used to plot the final graph. The Figure 15 shows the results and the removal efficiency of COD with time.

Figure 28. COD removal in ABR-AF (Hybrid media) during Phase II

The bar graph shows that the amount of COD in ABR effluent stream was almost 150 mg/L which was further reduced to 50 mg/L after passing through the AF and the reason is the formation of biofilm inside the PVC filter media which breaks down the COD into its main components. This hybrid media also consists of an external coating of PAC which leads to adsorption of additional particulates. The blue trend line shows that the COD removal efficiency in the starting was 82.8% which increased up to 90.0% at the end of the process, hence showing the performance efficiency of AF (hybrid media).

4.2.4. pH and ORP of the system during Phase II:

The pH and ORP of the both the systems i.e.; ABR and AF (hybrid media) were monitored regularly. The average pH value in the ABR reactor was found to be 7.3 whereas in AF (simple PVC media) the pH value was 7.12 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

ABR and AF effluents was tested separately for pH twice a week for three months (March, April and May). The mean value of the tested parameter for each week was used to plot the final graph. The Figure 29 shows the pH trend over the period of three months:



Figure 29. pH of ABR and AF (Hybrid media) during Phase II

The ORP of the system was also monitored on regular basis. The ORP of the system was monitored for three weeks. The average ORP of the 2 systems i.e. ABR, AF (PVC media) was measured to be -326 mV. The negative value indicates prevalence of ideal anaerobic conditions.



Figure 30. ORP of ABR-AF (Hybrid media) during Phase II

4.2.5. Turbidity removal in AF (Hybrid media) during Phase II:

The AF effluent was tested for turbidity using turbidimeter twice a week for three months (March, April and May). The mean value of the tested parameter for each week was used to plot the final graph. The Figure 31 shows the turbidity trend over the period of three months:



Figure 31. Turbidity removal in AF (Hybrid media) during Phase II

The graph shows that with time, turbidity removal increases. In the first run, the removal efficiency was 91.07% but enhanced up to 93.86% in the third run which is a fairly good value. The reason that can be attributed to this high removal efficiency is the formation of biofilm on PVC filter bed that retains any suspended solids and colloidal solids present in the incoming stream. The remaining colloidal and dissolved solids can be removed further in the tertiary treatment.

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 WFMF membrane was run separately with both types of AF to assess the performance of membrane in terms of fouling rate and backwashing rate. The TMP through the membrane was also monitored regularly to check the fouling rate of the membrane.
4.3.1. COD removal through Membrane system:

The COD removal of the membrane system is shown in the following graph. The blue line represents removal efficiency of membrane with AF (PVC media) and the red line with represents the COD removal efficiency with AF (Hybrid media). Whereas, the green line gives the overall COD removal efficiency of the system.



Figure 32. COD removal in Membrane system during Phase III

> For AF (PVC media) – Membrane:

It can be observed that the combined system at HRT of 12 hours and membrane flux of 6LMH gave effluent concentration of 24 mg/L of COD and removal efficiency of 68.39% at the end of the process.

> For AF (Hybrid media) – Membrane:

It can be observed that AF (Hybrid media) combined with Membrane gave an overall COD removal of 69.27% at an HRT of 12 hours and membrane flux of 6LMH with effluent concentration of 15 mg/L. The overall COD removal enhanced from 94.8% to 96.84% which clearly shows that performance of membrane with AF (Hybrid media) is better than the performance with AF (PVC media). And the reason is the extra coating of PAC on PVC which causes adsorption of the particles.

4.3.2. Turbidity removal through Membrane system:

The suspended solids or turbidity removal of the overall system is shown in the Figure 33. The green line shows the removal efficiency of membrane with AF (PVC media), blue line shows the removal efficiency of membrane with AF (Hybrid media) and the

red line shows the overall Turbidity removal of the system and it was found that using a low-cost WFMF membrane having a pore size of 1-3 μ m can effectively remove almost all the suspended solids present in the raw influent



Figure 33. Turbidity removal in Membrane system during Phase III

From the graph, it can be seen that the turbidity removal efficiency of AF (PVC media) – Membrane which was 84.35% after the third run enhanced up to 93.86% using AF (Hybrid media) – Membrane system with an effluent turbidity of 1.4 NTU. However, a slight fall in the overall removal efficiency can be seen in the third run because the same membrane was used for the testing of both systems and hence its threshold capacity also reduced with time.

4.3.3. TMP Profile of the Membrane system:

The membrane system was run for 3 months and the fouling behaviour was observed through data logging TMP meter. Plot of the recorded TMP is shown in the Figure 34.



Figure 34. Measured TMP of the Membrane system during Phase III

A threshold limit of 300 mbar or 30 kPa was set for TMP rise. TMP of 300 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. However, gradually with time the TMP increased because of formation of cake layer on membrane surface. It can be observed from the figure, that after 35th 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 37th day of run which means an MF membrane with 1-3 μ m pore size can produce better results with less fouling rates as compared to 0.4 μ m membrane.

The TMP performance of the membrane system was enhanced up to 70% when connected with AF (Hybrid media) in comparison with AF (PVC media)

4.4. Cost Benefit Analysis

A cost-benefit analysis compares the predicted or estimated costs and benefits (or opportunities) connected with a project choice to see if it makes sense from a business standpoint. When we performed Cost Benefit Analysis the Hybrid Medias cost was about 94 Rs. Per litre while for only PVC med the cost was 53 Rs. Per litre. Although the cost if Hybrid Bed is high but its removal efficiency is quiet high as compared to the PVC bed. Also, the return of investment of hybrid media will be less thus making it a better option for use in AF.

Low cost WFMF membrane is preferable as its removal efficiency was high and the cost is low as compared to the conventional Microfiltration membrane. The results are shown in the Table 8.

PVC BED AF		HYBRID BED AF		
MATERIALS	Cost	Materials	Cost	
PCV PIPE		PVC Pipe	10 Rs. Per Foot	
		Binder (PVDF)	60 Rs. Per Kg	
	10 Rs. Per Foot	Solvent (N-N-	100 Rs. Per litre	
		dimethyl)		
		PAC	6000 Rs. Per Kg	
PVC PIPE USED		PVC Pipe used	80 Feet, 800 Rs.	
AND COST		Binder (PVDF)	12 g, o.6 Rs.	
	80 Feet , 800 Rs.	used	220ml, 22 Rs.	
		Solvent (N-N-		
		dimethyl)	100g, 600 Rs.	
		PAC		
TOTAL PRICE FOR	800 Rs.	Total Price for 15	1422.6 Rs	
15 LITRES		litre		
PRICE FOR 1	53.33 Rs.	Price per litre	94.84 Rs.	
LITRES				
REMOVAL		Removal		
EFFICIENCY OF	85%, 70%, 49%	Efficiency of COD,	90 %, 55 %, 80%	
COD, TKN AND TP		TP, TKN		
CONVENTIONAL	40,000 Rs.	WFMF Cost	18,000 Rs.	
MEMBRANE COST				
COD, TURBIDITY				
REMOVAL		Removal		
EFFICIENCY	68%, 84%	Efficiency	94 %, 69 %	
THROUGH		Turbidity, COD		
MEMBRANE				

Table 8: Cost Benefit Analysis

5. CHAPTER FIVE: CONCLUSION AN RECOMMENDATIONS

5.1. CONCLUSION:

From the work done and results of all the three phases it can be concluded that the concentration of COD, TKN and TP considerably reduced when hybrid media was used in the AF as compared to PVC media in the AF. The table below summarizes the concentrations thus showing a better comparison between the two medias.

PARAMETERS	INFLUENT	EFFLUENT	EFFLUENT	NEQS
		PVC MEDIA AF	HYBRID	
			MEDIA AF	
TP	15 mg/L	7.6 mg/L	6.7 mg/L	
TKN	34 mg/L	10.2 mg/L	6.9 mg/L	
COD	522 mg/L	78.1 mg/L	52.2 mg/L	150 mg/L
TURBIDITY	230 NTU	3.6 NTU	1.35 NTU	<5 NTU
PH	7.3	7.15	7.0	6-9

Table 9. Summary of Concentrations

- Hybrid Media was tested to be the most optimal filter media giving 90, 80, and 55% removal of COD, TKN and TP respectively in comparison with virgin PVC media which 85, 70, and 49% removal of COD, TKN and TP respectively and Granular Activated Carbon media. It can therefore replace these two types of filter media.
- The removal efficiency of the low-cost WFMF membrane was enhanced in combination with hybrid media, producing high quality effluent with average COD of 16.5 mg/L and turbidity of 1.35 NTU thus reducing the overall fouling rate.
- The removal efficiency of the WFMF membrane showed that it can be an effective alternative to constructed wetlands for post-treatment polishing of treated wastewater.

- Treated wastewater (permeate) from the membrane can be re-used for landscaping, construction and other domestic purposes.
- If water reuse is desired, an additional pre-treatment unit must be installed at the end of AF. Microfiltration Woven Fibre Membrane with pore size of 1-3 µm can be used for this post-treatment/polishing purpose.
- The Cost Benefit Analysis of the treatment technologies performed showed that the Hybrid filter media although relatively costly offers a greater set of benefits and applications.
- Treated wastewater from DWTS can be used for non-potable applications such as landscaping, horticulture, fire fighting etc. after going through membrane filtration.

5.2. RECOMMENDATIONS:

- To ensure uninterrupted and a full gravity flow, some level difference must be provided between different compartments of ABR.
- 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 of in receiving water bodies.
- 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.
- 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.
- Vacuum Distillation should be performed in drying the hybrid media rather than oven drying it at 60°C for 24 hours.

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