

Optimization of Moving Bed Sequencing Batch Reactor for the Treatment of Oil Refinery Wastewater



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(2024)

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A thesis submitted to the National University of Sciences and Technology, Islamabad,

in partial fulfillment of the requirements for the degree of

**Master of Science in
Environmental Engineering**

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(2024)

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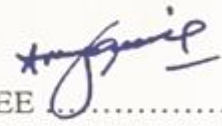
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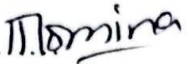
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To My Beloved Parents & Siblings,

And Respected Teachers

ACKNOWLEDGEMENTS

All praise to my lord, Allah Almighty, who bestowed me with enough patience and intellectual understanding in overall my research study. I am indebted to my supervisor Dr Sher Jamal Khan whose valuable advice, guidance and unwavering support throughout this journey made it possible to complete this Master research. I would like to show my appreciation to my GEC members Dr Muhammad Ali Inam & Dr Musharib Khan for their precious time and important feedback towards my research.

I would like to express my deepest gratitude to my parents and siblings for their constant prayers and support which helped me a lot in my toughest time of research. I deeply appreciate the academic staff of SCEE (IESE), especially the wastewater lab staff for their help during my experimental work. Also, I am so grateful to the MBR family for their technical assistance and unwavering support.

I would particularly like to give my special thanks to my lab fellows who gave me motivation and encouragement during the lab work. Finally, I would like to thank my hostel-mates: Kainat Amjad, Khadija Sharafdeen and Kanwal Anwar for making my Master journey very memorable.

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LIST OF SYMBOLS, ABBREVIATIONS AND ACRONYMS

ASP	Activated Sludge Process
NH ₄ -N	Ammonium-N
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
DO	Dissolved Oxygen
FR	Filling Ratio
HRT	Hydraulic Retention Time
MBBR	Moving Bed Biofilm Reactor
MBSBR	Moving bed Sequencing Batch Reactor
MLSS	Mixed Liquor Suspended Solids
MLVSS	Mixed Liquor Volatile Suspended Solids
ORW	Oil Refinery Wastewater
SBR	Sequencing Batch Reactor
SVI	Sludge Volume Index
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TSS	Total Suspended Solids

ABSTRACT

The oil refinery wastewater, distinguished by a high organic load and oil content, was effectively treated using a moving bed sequencing batch reactor (MBSBR). MBSBR integrates the benefits of both sequencing batch reactors (SBR) and moving bed biofilm reactor (MBBR) techniques, operated in sequencing batch mode, resulting in improved treatment performance. The performance of lab-scale aerobic MBSBR was investigated for an oil refinery wastewater with a chemical oxygen demand (COD) concentration of 810 ± 30 mg/L. The study aims to explore the performance of carrier media filling ratios (FRs) and hydraulic retention time (HRTs) on treating oil refinery wastewater as well as to determine the optimal value for both the parameters. The experimental investigation included optimization of parameters by adjusting the filling ratio ranging from 10 to 40% and hydraulic retention time from 6 to 24 h. The optimization was done by examining the organic pollutant concentration in the effluent. Higher media FR and HRT show higher pollutant removal efficiencies from wastewater. The COD, BOD, oil and Ammonium-N ($\text{NH}_4\text{-N}$) removal efficiencies were determined to be 88.43, 88.50, 86.21 and 88.72%, respectively at the optimum filling ratio of 30% and hydraulic retention time of 18 h. The morphology of biofilm shows that the biofilm thickness was larger at lower HRT as compared to higher HRT. Overall, this study highlights that a media filling ratio of 30% and HRT of 18 h gives the optimal treatment efficacy, providing a better understanding of how to treat oil refinery wastewater by using this hybrid treatment technology.

Keywords: Oil refinery wastewater; Moving bed sequencing batch reactor; Filling ratio; Hydraulic retention time; wastewater treatment

CHAPTER 1: INTRODUCTION

1.1 Background

Pakistan's oil refinery industry has played a major role in the country's economic growth and industrial development. Pakistan has five major oil refinery industries, such as Pak-Arab Refinery Limited (PARCO), Attock Refinery Limited (ARL), National Refinery Limited (NRL), Pakistan Refinery Limited (PRL), and Cnergyico Pakistan Limited (CPL), with a combined capacity of approximately 450,000 barrels of crude oil per day (bpd) (Finance Division - Government of Pakistan, 2023).

The oil refinery industry refines crude oil into various products such as naphtha, diesel, petrol, jet fuel, LPG, kerosene oil, furnace oil, and paving-grade asphalt. Pakistan's transport sector consumes 78.5% of petroleum products, while 10.8% of the total consumption is used by the power generation sector (Yousafzai, 2023). About 60 and 30% of diesel and motor gasoline demand, respectively, is being fulfilled by Pakistan's oil refinery industries (Petroleum Division, 2023).

The oil refinery industry refines crude oil into different petroleum products such as fuels, petrochemicals, and lubricants through various processes such as desalting, distillation, cracking, reforming, and alkylation (Speight, 2023). Extensive amounts of water are consumed by these refinery processes. To refine a barrel of crude oil, about 80-90 gallons of water are required for crude oil refinery production (Ezugbe & Rathilal, 2020).

Oil refining process generates wastewater approximately 0.4–1.6 times more than the quantity of crude oil being processed (Kumar et al., 2022). In 2021, 81 refineries releases

approximately half a billion gallons of wastewater each day (Louisa Markow et al., 2023). The refining process generates a large amount of wastewater that contains a variety of pollutants, which include oil and grease, suspended solids, ammonia, phenolic compounds, and various heavy metals. The specific composition of the wastewater varies according to the category of crude oil being refined and refining techniques being used.

The untreated wastewater from oil refineries can cause great environmental risks. Pollutants such as organic compounds and oils cause different health harm, such as gastrointestinal diseases, nausea, skin allergies, and infections in individuals exposed to them. Excessive untreated wastewater can lead to severe water pollution, which harms aquatic ecosystems and poses potential waterborne diseases to humans (Jory & Erdeni, 2024).

1.2 Problem statement

Oil refinery wastewater has a complex composition, making its treatment very challenging to meet stringent standard regulations for wastewater discharge or reuse. The toxic substances present in oil refinery wastewater not only damage aquatic ecosystems but also cause great health risks to human health. Conventional wastewater treatment systems often fail to handle oil refinery wastewater treatment processes. Many physical, biological, and chemical treatment processes such as dissolved air flotation (DAF), adsorption, oxidation, coagulation, and activated sludge processes are preferred to eliminate toxins from the wastewater (Asadi, 2018; C. Y. Cao & Zhao, 2012; EH et al., 2018; IWU, 2012; Jafarinejad et al., 2017; Jothinathan et al., 2021; Lee et al., 2020; Moneer et al., 2023; Qaderi et al.,

2018; Thakur et al., 2014) . In Pakistan, mostly activated sludge processes have been used to treat oil refinery wastewater (Irfan, 2009).

However, these processes often face challenges such as low treatment efficiency, high energy consumption, and sludge production. Therefore, there is a pressing need for more advanced and adaptable treatment solutions to address these complexities and mitigate environmental impacts.

1.3 Objectives of the study

The primary goal of this study was to evaluate the treatment efficiency of a moving bed sequencing batch reactor for oil refinery wastewater. The distinct objectives of this research study include:

- 1 Design and fabrication of a lab-scale set up of moving bed sequencing batch reactor (MBSBR).
- 2 Optimization of filling ratio (FR) in MBSBR to treat oil refinery wastewater.
- 3 Optimization of hydraulic retention time (HRT) at optimal FR in MBSBR to treat oil refinery wastewater.

1.4 Scope of the Study

This research study mainly focused on the treatment of oil refinery wastewater through a hybrid technology known as moving bed sequencing batch reactor (MBSBR). The synthetic wastewater employed, was characterized by COD, BOD, oil content, and Ammonium-N which are common characteristics of oil refinery wastewater. The key variables involve the effect of hydraulic retention time and media filling ratios on

wastewater treatment efficiency. The study's objective is to assess the performance of MBSBR in removing COD, oil, and ammonium-N from oil refinery wastewater. The study provides detailed analysis in optimizing MBSBR technology for greater treatment performance. This study contributes to the development of more constructive and sustainable wastewater treatment approaches for the oil refineries.

CHAPTER 2: LITERATURE REVIEW

2.1 Overview of petrochemical production process

The petrochemical industry converts crude oil into a wide range of chemicals, polymers, and fuels. This process begins with the extraction of crude oil from the earth's crust. When crude oil is transported to oil refineries, it gets skillfully refined and then converted into different fragments such as gasoline, diesel, kerosene, and jet fuel, which can power numerous daily life activities. The refinery process includes desalting, distillation, cracking, reforming, or alkylation (Speight, 2023). The description of the petrochemical refining process is discussed below:

2.1.1 *Desalting*

When crude oil is transferred to the refineries by pipelines or any other vehicles, it must meet strict regulations about oil and salt content because crude oil contains impurities like water, inorganic salts, suspended solids, and trace metals which often get dissolved in water which can contaminate crude stream. Failure to remove salt from the oil can lead to severe corrosion or scaling, especially in heater tubes. Desalting is a pivotal step to remove these contaminants which can reduce serious damage like equipment's plugging and fouling due to corrosion but can also prevent catalyst poisoning in a processing unit.

Desalting begins with the mixing of crude oil with washing water to ensure effective contact between them by using mixing valves or static mixers. After that, the mixture is separated into a vessel to separate the aqueous and organic phases. To address potential emulsion formation and water carryover, chemical demulsifiers are added. For further

reduction of water and salt content, an electric field is used which acts by fusing polar salty water droplets. Desalting is an essential step in refineries to meet strict requirements and mitigate the adverse effects of impurities on downstream processes (Bijani & Khamehchi, 2019). Desalting wastewater generated during the process comprises oil and grease, suspended solids, heavy metals, organic compounds, and sulfides (Ye et al., 2021).

2.1.2 Distillation

Fractional distillation is a primary method that separates the hydrocarbon contents of crude oil. Heated crude oil enters the distillation column, where lighter components ascend, and heavier compounds descend based on their boiling point. The resulting fractions enclose the column with gases at the top, followed by naphtha, kerosene, diesel, gas oil, and residue at the bottom. Afterward, these fractions are collected for further use or additional refining at different levels of the column. Refineries often use reflux systems to enhance the performance efficiency of distillation and assure product purity. The distillation products are obtained by controlling the temperature and pressure within the column (Taghipour et al., 2019).

2.1.3 Cracking

Cracking is a significant chemical process commonly used in oil refineries that decompose larger and more complex organic molecules such as long-chain hydrocarbons into smaller and lighter hydrocarbons that have high commercial or consumer value. It is a highly controlled process and produces alkanes and alkenes which are part of homologous series. Cracking involves two types of process, steam cracking and catalytic cracking.

Steam cracking yields high production of alkene by breaking hydrocarbons under extreme heat conditions typically between 800-900°C. The heat causes the larger hydrocarbon molecules to break down, that creates free radicals which get highly reactive with unpaired electrons. Ethylene, propylene, benzene, and butadiene are the products of steam cracking (Zhou et al., 2021).

Catalytic cracking weakens the carbon-carbon bonds of hydrocarbon and breaks it down into smaller components with the help of catalysts. The zeolite is used as a most common catalyst (Y. Liu et al., 2020). High-octane gasoline, diesel, and light olefins such as butene and propylene are the output of catalytic cracking.

2.1.4 Alkylation

Alkylation is a chemical procedure in which gaseous hydrocarbons are incorporated to produce high octane gasoline units. In this procedure, olefin (usually propylene and butylene) and iso-paraffin (i.e. isobutane) are combined with each other to achieve a larger chain molecule called alkylate. It produces high-quality gasoline that meets modern society's requirements (Zbuzek, 2014).

2.1.5 Reforming

Reforming is a rearranging process, where straight-chain alkanes convert into branch-chain isomers which have high octane numbers with the loss of a small molecule such as hydrogen. Usually, the Naphtha fraction is reformed into gasoline of high-octane number to enhance the fuel performance. Mostly, platinum-based catalysts supported on alumina are used in the reforming process of hydrocarbons which is why it can also be called

catalytic reforming. In reforming, aromatics are usually formed and process hydrogen gas as a by-product (Jalali et al., 2019).

2.1.6 Treatment

The treatment process, mainly hydrotreating, removes unwanted contaminants such as sulfur, nitrogen, and heavy metals from crude oil by binding them with hydrogen, absorbing them in separate columns, or using acids to eliminate them. After removing undesirable contaminants, the refined liquids are converted into gasoline, lubricants, kerosene, jet fuel, diesel fuel, heating oil, and petrochemical feedstocks essential for producing plastics and other everyday items (Bachmann et al., 2018). According to a previous report, about 80 – 90% of water provided to the oil refinery industry for their

processes is usually released as it is in the form of wastewater (Narayan Thorat & Kumar Sonwani, 2022). Figure 2.1 presents the process flow description of the oil refinery process.

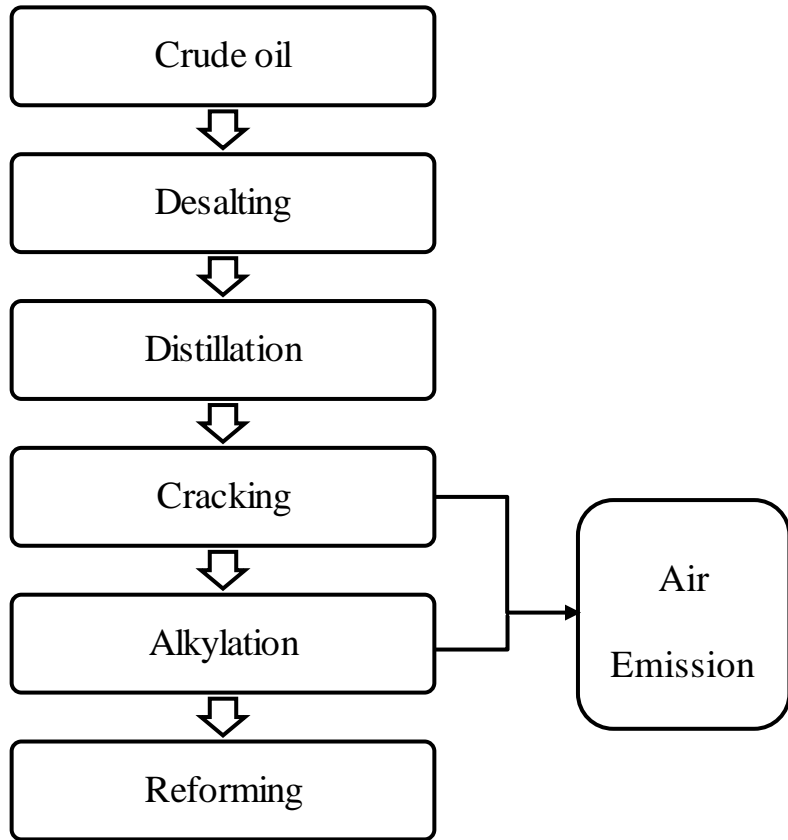


Figure 2.1 Flow chart of the oil refining process

2.2 Typical characteristics of oil refinery wastewater

The oil refinery industry emerges as one of the largest consumers of water due to its extensive usage in the production processes of various products, including liquefied gas, gasoline, furnace oil, kerosene, bitumen, etc. In addition, water is also used in cooling towers, boilers, steam drying, and cleaning, which finally becomes wastewater. The oil refinery industry discharged effluent contains a mixture of wastewater generated from various processes, and it also comprises pH, COD, BOD, suspended solids, oil and grease,

phenols, etc. For 1 ton of petroleum oil about 3.0-3.5 m³ petroleum refinery wastewater is produced (Younis et al., 2020). The wastewater from the petrochemical industry typically contains a range of pollutants, including hydrocarbons, heavy metals, toxic organic pollutants, sulfur, and refractory compounds (Kishor et al., 2021; B. Pratap et al., 2023; Singh et al., 2022). Petrochemical industry wastewater characteristics mentioned by various researchers are shown in the Table 2.1. Stringent national and international standards demand sustainable and environmentally friendly practices. Petroleum refinery industry has enhanced the implementation removal of treatment procedures to minimize the detrimental consequences of wastewater toxins on the environment.

Table 2.1: Synthetic oil refinery wastewater compositions

Parameters	References					
	(Ishak & Malakahmad, 2013)	(Ibrahim, 2015)	(Aziz & Fakhrey, 2016)	(Jia et al., 2018)	(Estrada-arriaga et al., 2019)	(Ahmad et al., 2023)
COD (mg/L)	743-1673	480	485	745.87	400	17,050 ± 418
BOD (mg/L)	205-448	195	155	-	-	560 ± 206
pH	7.50-9.41	8.0	7.74	7.78	6.8	6.42 ± 0.12
TDS (mg/L)	-		-	2000	-	-
TSS (mg/L)	280-340	315	-	200	78	19,000 ± 391
Oil and Grease (mg/L)	48-97	94	17.36	500	19	460 ± 59
Phenols (mg/L)	1.16-1.44	13.8	3.5	-	200	-

2.3 Oil refinery wastewater treatment technologies

A variety of physical and chemical treatments such as dissolved air flotation (DAF), adsorption, oxidation, coagulation, and advanced oxidation processes have been used to treat petrochemical industry wastewater (Asadi, 2018; Davarnejad et al., 2014; EH et al., 2018; Fahem & Abbar, 2020; IWU, 2012; Jothinathan et al., 2021; Khader et al., 2022; Lee et al., 2020; Moneer et al., 2023). Dissolved air flotation (DAF) capable of generating micro/nano-sized bubbles was used to remove contaminants of oily wastewater, resulting in the removal of 93.5% of the COD. The ability to manipulate bubble size proved critical, particularly in the efficient removal of oil particles that closely matched the size of the bubbles (Lee et al., 2020). Analysis of the COD absorption percentage, which reached 89.97% on hydrogen peroxide-modified carbon nanotubes revealed that the highest adsorption rates were achieved under specific conditions. The findings emphasized the importance of optimizing conditions for strengthening the efficiency of the adsorption process (Asadi, 2018). Powdered activated carbon (PAC), clinoptilolite natural zeolite (CNZ), and synthetic zeolite type X (XSZ) performance was studied in terms of COD, oil, and turbidity removal from produced water (PW) generated from oil production processes. PAC showed the highest removal efficiencies for oil (99.58%), and COD (95.87%) making it the most effective adsorbent (Khader et al., 2022). The performance of a combined microbubble-catalytic ozonation process (M-O₃/Fe/GAC) for treating petrochemical wastewater (PCW) was investigated. 88% COD removal was observed by the M-O₃/Fe/GAC ozonation process surpassing the micro-bubble and macro-bubble ozonation processes by 18% and 43%, respectively (Jothinathan et al., 2021). Three natural coagulants (Cicer arietinum seed, eggplant seed, and radish seed) performance was

compared in terms of removing turbidity, oil, and COD from the produced water. *Cicer arietinum*, eggplant, and radish seed showed 95.2, 92.18, and 93.48% COD removal efficiencies, respectively. Hence, *Cicer arietinum* seed was suggested as the optimal natural coagulant for COD removal (EH et al., 2018). Electrocoagulation (EC) with aluminum electrodes accomplished 74% for oil and grease, 76% for COD, and 49% for BOD treatment efficiency for oily wastewater proving to be an efficient and eco-friendly treatment method (Moneer et al., 2023). Another study used Electro-Fenton technique to compare the effectiveness of aluminum and iron plate electrodes for treatment of petroleum industry wastewater. The closure of the study demonstrated that iron electrode attained higher removal efficiencies for COD (67.3%) and color (71.58%) compared to the aluminum electrode, which had removal efficiencies of 53.94% for COD and 67.35% for color (Davarnajad et al., 2014). In a previous study, petroleum refinery wastewater was treated by the Electro-Fenton process. Porous graphite was used as an anode and cathode for batch electrochemical reactions. Different operating variables such as FeSO_4 and NaCl concentrations and time effect on COD removal from wastewater were explored. Under optimized parameters, 95.9% COD removal efficiency was accomplished (Fahem & Abbar, 2020). The performances of the oil refinery wastewater (ORW) treatment methods are briefed in the Table 2.2 shown below:

Table 2.2 Wastewater treatment technologies for oil refinery wastewater

ORW Treatment Methods	Wastewater Type	COD Removal (%)	References
Adsorption	Real ORW	89.97	(Asadi, 2018)
Coagulation	Synthetic petroleum produce water	95.2 (with Cicer arietinum seed) 92.18 (eggplant seed) 93.48 (radish seed)	(EH et al., 2018)
Electrocoagulation	Synthetic oily wastewater	76	(Moneer et al., 2023)
Ozonation	Real petrochemical wastewater	88 (microbubble-catalytic ozonation) 62 (microbubble ozonation)	(Jothinathan et al., 2021)
Dissolved air flotation	Synthetic oily wastewater	93.5	(Lee et al., 2020)
Adsorption	Synthetic oil produced water	95.87 (with powdered activated carbon)	(Khader et al., 2022)

		63.74 (with clinoptilolite natural zeolite) 80.32 (with synthetic zeolite type X)	
Electro-Fenton process	Real petroleum refinery wastewater	95.9	(Fahem & Abbar, 2020)

The physical and chemical processes for oil refinery wastewater have several drawbacks. One of the major disadvantages is frequent adsorbent replacement, leading to high operational costs. These processes require high energy consumption, making them less economical. They have limited efficiency in removing dissolved contaminants or emulsified oils. They also generated sludge, necessitating expensive additional treatment steps, and could result in the formation of harmful byproducts. So, biological methods can be chosen as a practical treatment approach for oil refinery wastewater treatment.

2.4 Biological Treatment for Oil Refinery Wastewater

Biological treatment technologies are one of the most extensively used methods worldwide. Organic components present in wastewater are biodegradable with the aid of micro-organisms. Many biological treatment technologies have opted for industrial wastewater treatment because they have high treatment potential, less harmful effects, and low economic cost (Y. Cao et al., 2017). Biological methods have a high capability of degrading organic matter into stable end products (Sanghamitra et al., 2021).

2.5 Types of biological treatment

2.5.1 Activated sludge process

Activated sludge process (ASP) uses aeration and biological flocs (bacteria and protozoa) for biodegradation of organic matter and suspended solids present in wastewater. The process starts with air injecting into the wastewater which allows the microorganism to

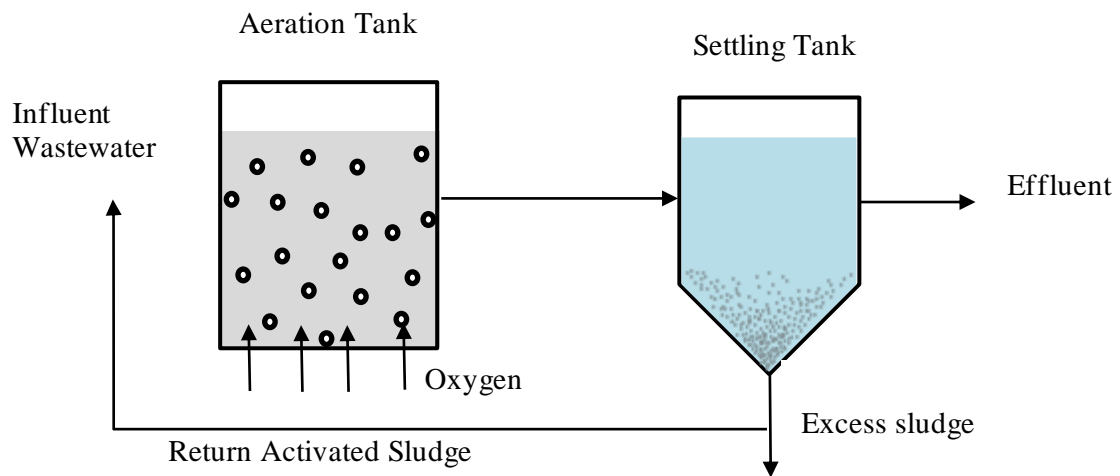


Figure 2.2 Schematic Illustration of Activated Sludge Process

degrade organic matter. Afterward, the wastewater is transferred to the settling tank in which separation of sludge and treated wastewater occurs. Some amount of the settled sludge is recycled while others are removed for further treatment and disposal (Siatou et al., 2020).

Oil refinery contaminated wastewater was treated by un-modified ASP and $\text{Fe}_3\text{O}_4/\text{silica}$ nanocomposite-modified ASP. The performance comparison shows that the modified ASP showed 85.17% COD removal efficiency while the un-modified ASP showed the lowest COD removal efficiency which was 40.22% (Zabermawi & Bestawy, 2024). In a previous

study, an activated sludge process was opted to treat petroleum refinery wastewater. About 94-95% COD and 85-87% TOC removal efficiency was achieved (Santo et al., 2013).

2.5.2 *Sequencing batch reactor*

A sequencing batch reactor (SBR) is a better replacement for the conventional activated sludge process for organic pollutants and nitrogen removal from wastewater. SBR can treat wastewater by alternating aerobic and anoxic phases within a single tank which enhances its treatment efficiency. In this technology, all the treatment phases operated sequentially in the same tank. SBRs require less space because they perform all treatment stages such as fill, react, settle, draw, and idle in one single reactor, reducing the extra need for reactors (Ng et al., 2021). Figure 2.3 unveils the schematic diagram of SBR process.

Sequencing Batch Reactors (SBRs) were well-suited for industrial wastewater treatment due to their effectiveness in removing organic matter and nutrients, their durability, and their ease of implementation (Mace & Mata-Alvarez, 2002). In another study, Thakur and his coworkers (2014) treated petroleum refinery wastewater with the help of SBR technology. 80 and 83% COD and TOC removal efficiencies were achieved, which was considered maximum. However, they suggested that 77 and 79% removal efficiencies for COD and TOC, respectively were optimum at HRT of 0.83 day (Thakur et al., 2014). Another study recommended that biological methods, especially sequencing batch are one

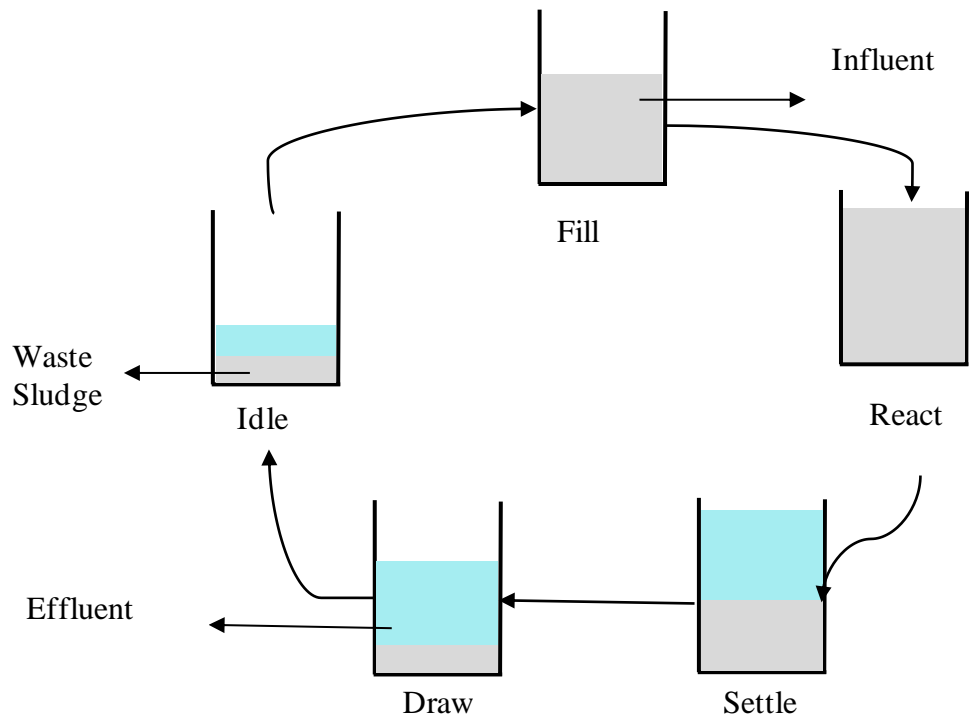


Figure 2.3 Schematic Diagram of Sequencing Batch Reactor

of the most favorable wastewater treatment methods for the petroleum industry. SBR easily adapts to different treatment levels such as secondary, advanced secondary, nitrification, denitrification, and nutrient removal (Jafarinejad et al., 2017).

2.5.3 Moving bed biofilm reactor

Moving bed biofilm reactor (MBBR) is a biological treatment that degrades organic components from the wastewater with the help of attached biomass. Moving bed biofilm reactor is an alteration of the activated sludge process and biofilter process which reduces the disadvantages of conventional biological treatment system (Gzar et al., 2021). The system contains suspended carrier media which promotes the growth of microorganisms on them and forms a biofilm layer. Aeration is provided to the system to ensure the media

movement. Different compositions and structures of carrier media such as polyethylene (Ali & Aziz, 2024; Banerjee et al., 2024; Tolêdo et al., 2024), polypropylene (Abu Bakar et al., 2020; J. Liu et al., 2019), polyurethane foams (Nhut et al., 2020; Sandip & Kalyanraman, 2019), and haydite (Zhao et al., 2019) can be used for industrial wastewater treatment. Moving bed biofilm reactors had preference over conventional systems because it efficiently removes organic compounds and NH_3 from wastewater in minimum time and less space. MBBR has been selected to treat municipal wastewater (Iliopoulou et al., 2023; V. Pratap et al., 2024), industrial wastewater which contains phenolic compounds (Aslam et al., 2024; Kuc et al., 2022), textile dye (Madan et al., 2023), and pesticides (Gaioto et al., 2023). The structural description of an MBBR is presented in the Figure 2.4.

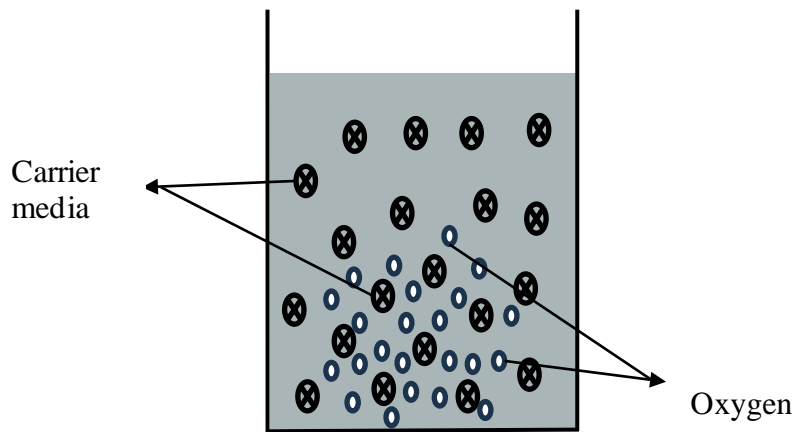


Figure 2.4 Schematic of Moving Bed Biofilm Reactor

The performance of lab-scale MBBR with K3 carriers was examined for industrial wastewater treatment at HRT of 3, 5, 8, and 12 hours. At an HRT of 8 hours, 80% COD removal efficiency was found to be the optimum HRT (Majid, 2019). The comparative

study on petrochemical wastewater treatment revealed that the MBBR achieved higher COD removal proficiency i.e., 85.75% and showed good shock loading resistance in comparison to ASP (C. Y. Cao & Zhao, 2012). The series configuration MBBR was used to treat petroleum wastewater. After treating the wastewater, 97% COD removal efficiency was achieved at 23 hr HRT which was considered the highest. The study also stated that increasing retention time and filling ratio enhances wastewater treatment efficiency (Qaderi et al., 2018). Aziz and his friends did a comparative examination of SBR and MBBR treatment efficiencies for treating residential wastewater with the ambition of reuse. The study ensured that the MBBR showed exceptional removal efficiency as compared to the SBR (Aziz et al., 2020).

The difference in oxygen transfer efficiency between fine bubble and coarse bubble aeration was investigated. The tests showed that the fine bubble aeration ensures good mixing of carrier media but had low oxygen transfer efficiency. It suggested that bacteria presence can enhance the oxygen transfer which can improve the treatment efficiency (Collivignarelli et al., 2019). The lab-scale anaerobic moving bed bioreactor (An-MBBR) and anaerobic hybrid reactor (UAHR) performance was compared. The study outcome showed that the UAHR had better COD removal capability as well as greater biogas generation than the An-MBBR for desizing textile wastewater treatment (Shahzad et al., 2021). However, MBBR has disadvantages like it has large operational costs and high aeration requirements for wastewater treatment (Safwat, 2019).

2.6 Hybrid biological treatment

Researchers have shown keen interest in hybrid treatment which exhibits both suspended and attached growth processes based upon its advantages that resolve the issues of previous biological systems.

2.6.1 Moving bed sequencing batch reactor

Moving bed sequencing batch reactor (MBSBR) integrates the benefit of both SBR and MBBR treatment technologies which enhance the treatment efficiency of the wastewater

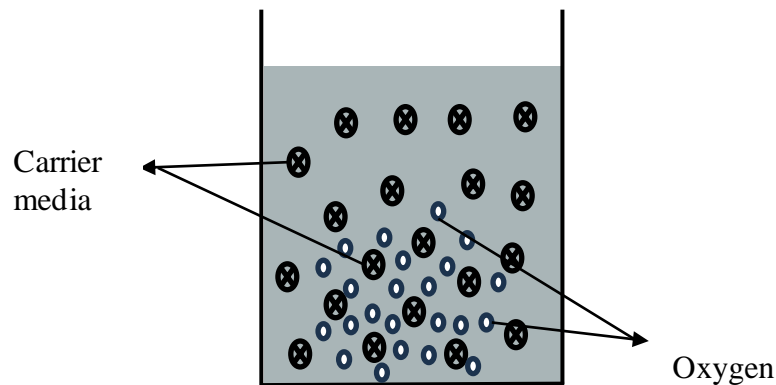


Figure 2.5 Schematic of Moving Bed Sequencing Batch Reactor

treatment technologies. In this system, carrier media is used which provides enough surface area for microorganisms to grow and form a biofilm layer which helps in the biodegradation of organic matter present in wastewater. The system operates in a sequential cycle: fill (influent enters the system), react (aeration and contaminant degradation), settle (solids settle), draw (treated effluent removed), and idle. MBSBR provides high-efficiency treatment because it provides enough surface for microbial activity. Its compact design makes it ideal for installations in space-constrained areas.

In summary, MBSBR delivers a competent, effective and space-saving solution for wastewater treatment, combining the advantages of both SBR and MBBR technologies to achieve exceptional performance.

CHAPTER 3: METHODOLOGY

The experimental approach for the oil refinery wastewater treatment is discussed in this chapter. The overview of experimental work in this research study followed two main phases (as shown in Figure 3.1). In the first phase, MBSBR performance was evaluated in terms of media filling ratios while different HRT impacts on treatment were investigated in the second phase.

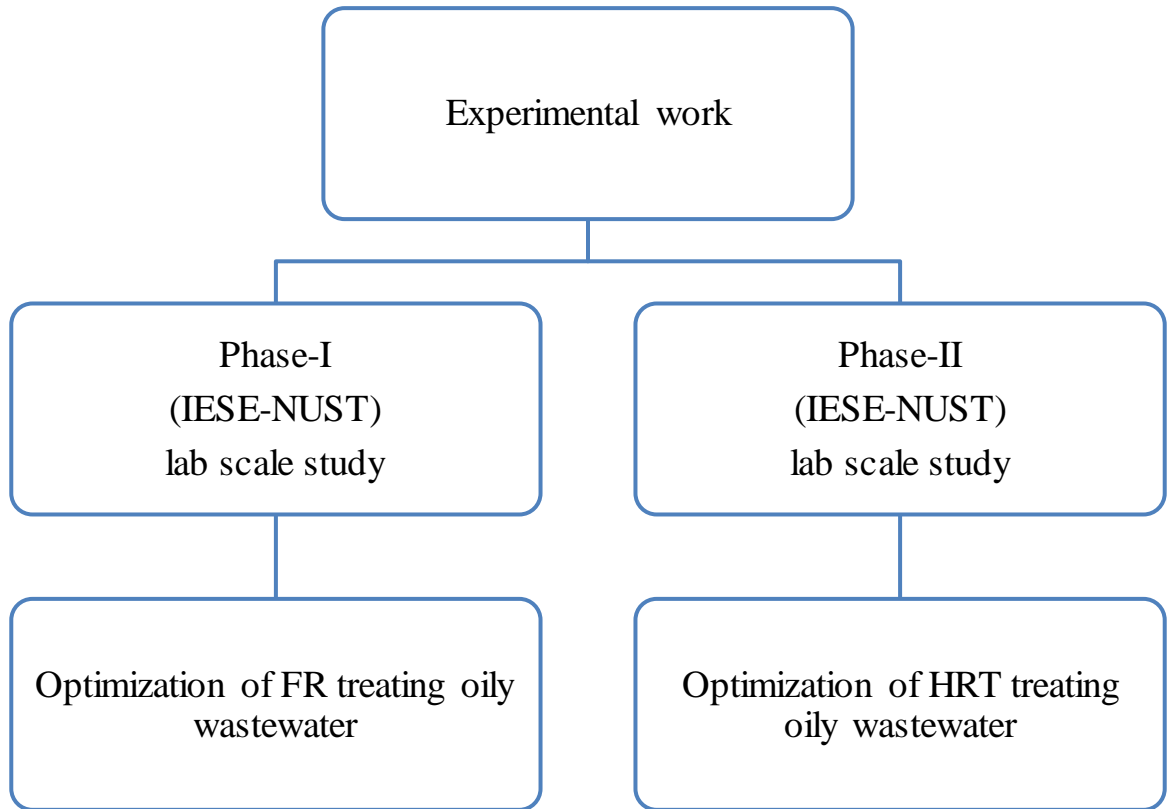


Figure 3.1 Two main phases of experimental work

3.1 Wastewater Characteristics

Oil refinery wastewater requiring treatment was obtained from an oil refinery company located in Islamabad, Pakistan. The wastewater was collected in sample bottles and taken to the lab where its composition was investigated. Afterward, the wastewater was distinguished by a COD concentration differing from 780 to 840 mg/L. Thus, the synthetic petroleum refinery wastewater recipe was prepared according to the analysis of real wastewater.

For aerobic wastewater treatment, the COD:N:P ratio was set to 100:5:1 (Q. Liu et al., 2022). In the synthetic wastewater recipe, the carbon, nitrogen, and phosphorus concentrations were sourced from phenol, ammonium chloride (NH_4Cl), and potassium dihydrogen phosphate (KH_2PO_4), respectively. The synthetic recipe is displayed on the Table 3.1. The pH of wastewater was adjusted in the range of 7.5-8.0 by adding sodium bicarbonate (Wang et al., 2021).

Table 3.1 Synthetic wastewater recipe

Chemicals	Chemical formula	Concentration (mg/L)
Phenol	C_6H_5OH	340
Sodium Bicarbonate	$NaHCO_3$	500
Ammonium Chloride	NH_4Cl	154.8
Potassium Dihydrogen Phosphate	KH_2PO_4	35.6
EDTA	$C_{10}H_{16}N_2O_8$	1.5
Potassium iodide	KI	0.03
Iron Chloride	$FeCl_3$	1.5
Cobalt Chloride	$CoCl_2$	0.15
Zinc Chloride	$ZnCl_2$	0.12
Copper Sulphate	$CuSO_4$	0.03
Boric Acid	H_3BO_3	0.15
Sodium Molybdate	Na_2MoO_4	0.15

The synthetic oil refinery wastewater was prepared according to the recipe deduced from the characteristics of the real wastewater. The synthetic wastewater characteristics is outlined in Table 3.2, that mimics the typical contaminants found in actual wastewater.

Table 3.2 Synthetic Wastewater Composition

Parameters	Unit	Synthetic Wastewater
Chemical Oxygen Demand (COD)	mg/L	810 ± 30
Biological Oxygen demand (BOD)	mg/L	400 ± 30
Oil	mg/L	11.97 ± 10
Ammonia-N	mg/L	54 ± 10
pH	-	7.5

3.2 Experimental Set-up

The illustrative image of a lab scale MBSBR system is portrayed in Figure. 3.2 and a pictorial view of the system consisting of a feed tank, reactor, and effluent tank is shown in the Figure 3.3. The reactor had an effective volume of 10 L with a working volume of 8 L. The synthetic wastewater was filled in the reactor from the inlet tank with the help of a peristaltic pump (Masterflex, 77200-60, Cole Parmer, USA). Fine air was injected with the help of an air diffuser connected to the air pump (RS-608, RS Electrical, Pakistan) placed

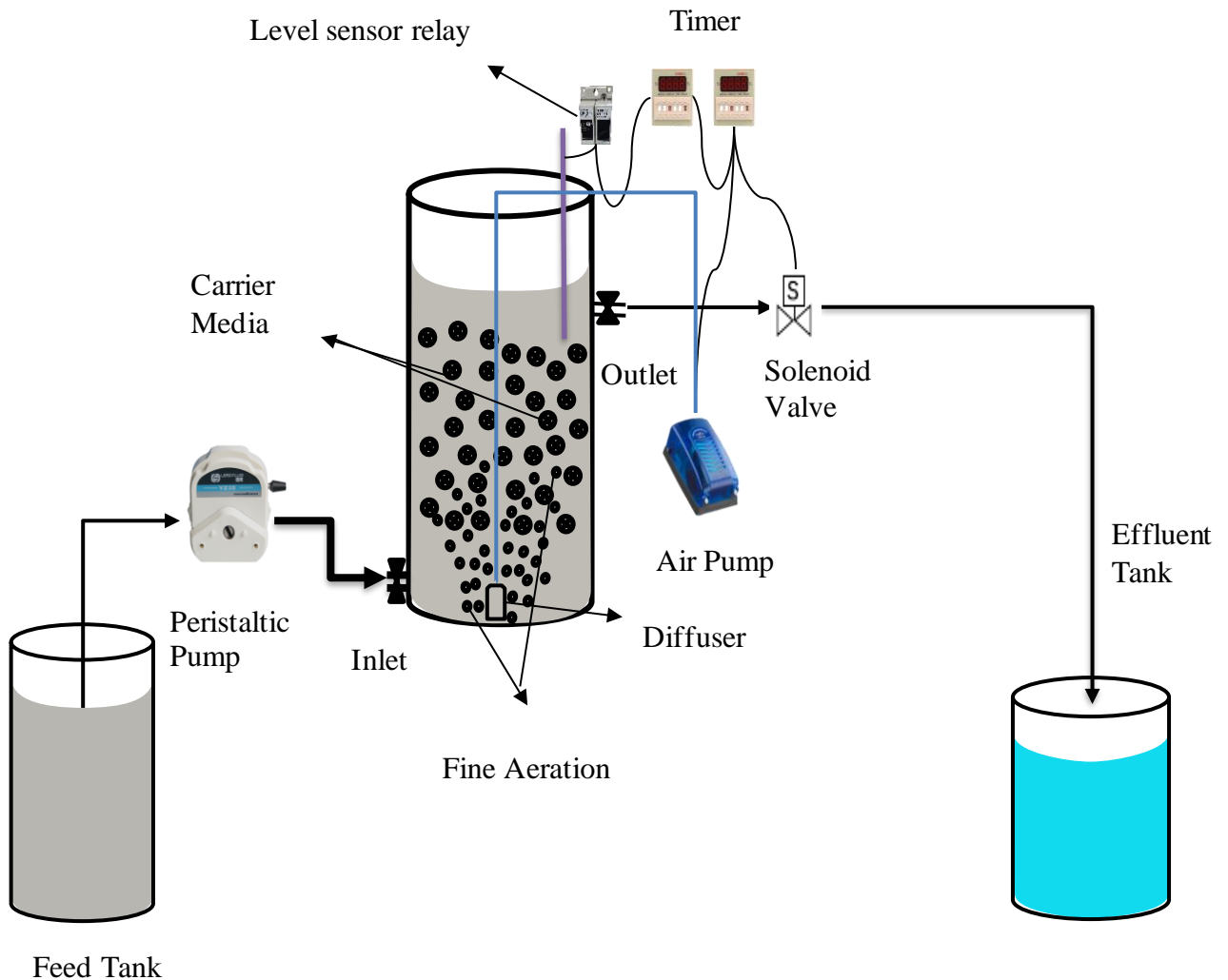


Figure. 3.2 Process Flow Diagram of Moving Bed Sequencing Batch Reactor

at the reactor bottom which promoted the synthetic wastewater circulation and carrier media movement throughout the experiment procedure. The solenoid valve (ST-SA012B105E-380AC, JP Fluid Control, USA) connected to an outlet pipe was only opened during the effluent discharge time into the tank after treatment. The timers (DH48S-S, OMRON, Japan) were connected to a solenoid valve and the air pumps for balancing the reaction and settling time of the system. The reactor was operated in cyclic mode including filling, reacting, settling, and decant phases. The settling time was fixed for 90 min so that the suspended sludge could settle down during that period. After the settling phase, the effluent was discharged while the influent was introduced during the filling phase, a duration of 30 min. The timers played an integral part in synchronizing the operational phases. The liquid level controller (LLC-101X, MICRO MAX, Iran) located at the top of the reactor helped maintain the water level of the system. The whole treatment process was conducted according to the selected HRT. The DO level was maintained in the range of 2.0 - 4.0 mg/L (Aimale-Troy et al., 2024). A high-density polythene carrier (MBBR19, Nihhao, China) with a specific surface area of 550 m²/m³ was used as a media for the treatment process.



Figure 3.3 Pictorial view of lab-scale MBSBR

3.3 Experimental Conditions

The lab-scale MBSBR was operated in three stages:

- a) Start-up and acclimatization,
- b) Media filling ratio optimization, and

c) Hydraulic retention time optimization.

The study begins with the acclimatization of sludge, targeting to optimize the filling ratio of carriers and hydraulic retention time within the Moving Bed Sequencing Batch Reactor (MBSBR) system to enhance the efficiency of treating synthetic oil refinery wastewater. Seed sludge for the acclimatization was acquired from the Membrane Bioreactor (MBR) plant operational at the National University of Sciences and Technology (NUST) in Islamabad, Pakistan.

The reactor was operated in sequential batch mode to treat synthetic oil refinery wastewater. The mixed liquor suspended solid (MLSS) concentration was kept between 3000-4000 mg/L. During the acclimatization phase, synthetic wastewater was fed to increase the adaptation rate of microorganisms to it. After microorganisms get adapted to synthetic wastewater, they start growing on the surface area of carrier media present in the reactor. After the 1st biofilm layer formation on the carrier media, the reactor was run for 20 days at HRT of 24 h until it achieved a steady state. The treatment operation was carried out in sequential phases such as fill and draw (30 min), react (22 h), and settle (90 min). Afterward, the carrier media filling ratio was then gradually raised to 20%, 30%, and 40% at HRT of 24 h for 20 days each, respectively. COD, ammonium-N ($\text{NH}_4\text{-N}$), and oil removal were chosen as key indicators to check the performance of the MBSBR reactor. The carrier media filling ratio was optimized after evaluation of its pollutant removal performance from effluent at different ratios.

Following the optimization of the carrier media filling ratio, the reactor was run at different hydraulic retention times of 18, 12, and 6 hours, each for a duration of 20 days. During

these trials, the sequential operational phases were changed based on the individual HRTs to determine the optimal retention time for maximum treatment efficiency. This systematic approach enabled a inclusive evaluation of the reactor's performance across different factors, finally leading to the identification of the most effective HRT for the treatment process.

The performance results collected under all operational conditions helped in the identification of optimal HRT. Table 3.3 shows the summarized operating conditions of moving bed sequencing batch reactor for oil refinery wastewater treatment.

Table 3.3 Operation conditions of MBSBR

Operating Conditions		
Parameters	Phase-I	Phase-II
Hydraulic Retention Time (HRT)	24 h	(6, 12, 18, 24) h
Filing Ratio (FR)	(10, 20, 30, 40) %	30%
pH	7.5 – 8.0	7.5 – 8.0
Temperature	35 °C	35 °C
MLSS	3000-4000 mg/L	3000-4000 mg/L
MLVSS/MLSS	0.65 - 0.75	0.65 - 0.75

DO	2-4 mg/L	2-4 mg/L
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3.4 Analytical Methods

Effluent fractions were taken from the MBSBR to inquire its treatment efficiency. The treated effluent was analyzed for chemical oxygen demand (COD), ammonium-N, and oil content at intervals of two days, while pH and temperature measurements were recorded daily. COD removal was calculated using the Closed Reflux Method according to the guidelines set by the American Public Health Association (APHA et al., 2017). Biological Oxygen Demand (BOD) of the effluent was measured with a Respirometric BOD Meter (BD600, Lovibond Water Testing and Colour Measurement, Germany).

Ammonium nitrogen (NH₄-N) levels were assessed using an automatic distillation unit (UDK-149, VELP Scientifica, Italy). Mixed liquor suspended solids (MLSS) were quantified following standard methods (APHA et al., 2017). pH and temperature were measured using a pH meter (HI-5221, Hanna Instruments Ltd., UK). The oil content was determined using the Gravimetric method, with n-Hexane as the extraction solvent (US Environmental Protection Agency, 2023).

Additionally, the surface morphology of the biofilm on the carrier media was analyzed using a Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX) analyzer (JSM-6490A, JEOL, Japan). The sludge volume index (SVI) was calculated with the help of the given formula below (Tesh, 2021):

$$SVI \left(\frac{mL}{g} \right) = \frac{\text{Settled sludge volume} \left(\frac{mL}{L} \right)}{MLSS \left(\frac{mg}{L} \right)} \times 1000mg/g \quad (3.1)$$

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Start-up and Acclimatizing Stage

The sludge acclimatization was done by filling the MBSBR reactor with 30% seed sludge at the bottom while the top portion of the reactor was filled with 10% carrier media. Synthetic wastewater was supplied to the reactor in batch mode, sustaining HRT of 24 h. The acclimatization phase was operated until the biofilm was developed on the carrier media and COD removal achieved a steady state. MLSS concentration in suspension was measured 2.2 g/L at the start of the acclimatization phase.

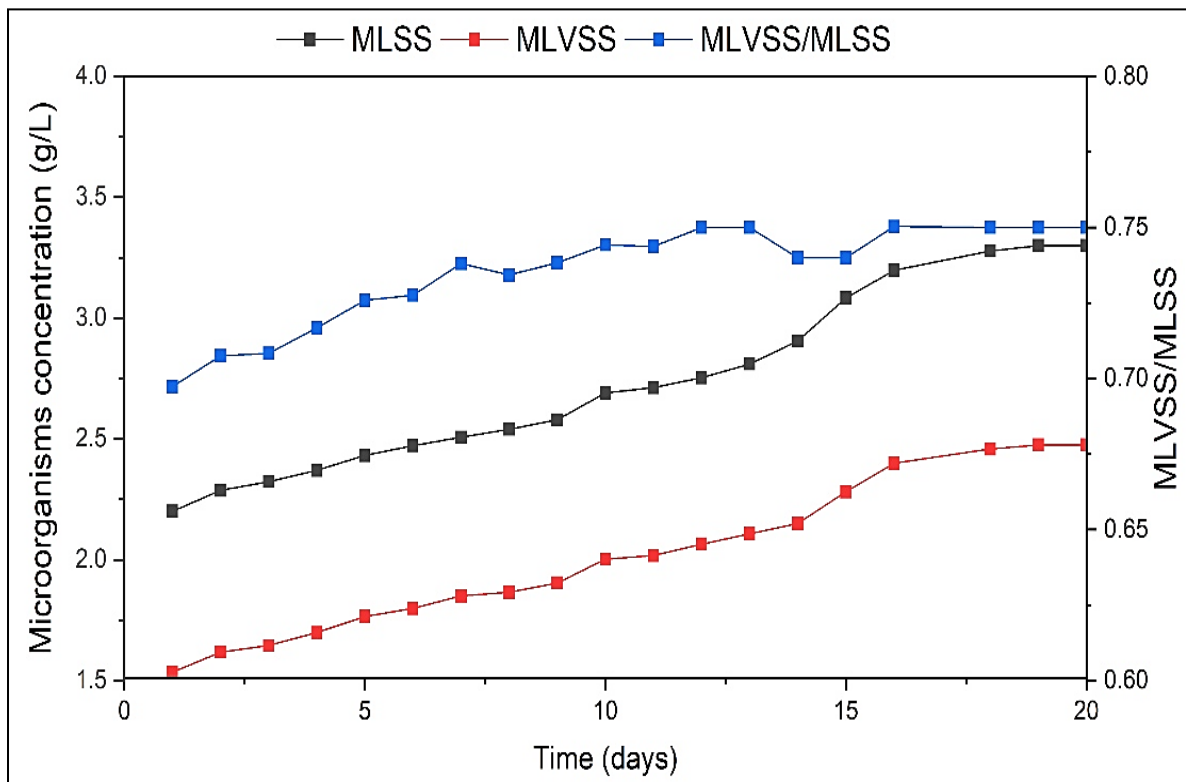


Figure. 4.1 Variations of MLSS, MLVSS concentration, and MLVSS/MLSS ratio against time

Initially, the MLSS increase rate was low because of the low pH as depicted from the Figure. 4.1 The presence of acidic components such as phenol causes the low pH of wastewater because the buffer takes time to stabilize the pH. Afterwards, the pH was increased and fell within the extent of 6.5 – 8.0 which is an ideal condition for microbial growth (Ramanadham et al., 2013).

As the biofilm layer matured and the system stabilized, the MLSS value increased to 3.3 g/L. The change in MLSS shows the growth and maturation of biofilm which is requisite for effective biological treatment of wastewater. The MLVSS/MLSS ratio ranging from 0.7 - 0.75 exhibits the high concentration of volatile solids that expedite the efficient organic pollutant degradation from wastewater. A previous study reported that MLVSS/MLSS maintained at 0.75 showed not only a significant increase in the active biomass but also removed organic pollutants from the wastewater effectively (Cai et al., 2020). Furthermore, the sludge volume index calculated was 119.39 mL/g indicating better sludge settlement (Metcalf et al., 2003).

Overall, the high MLVSS/MLSS ratio and low SVI exhibit better settling characteristics of sludge and consequently enhance the wastewater treatment efficiency. This improved settleability corresponds with stable microbial activity, which led to the enhancement of the treatment process's effectiveness.

4.2 Effect of Filling Ratio

4.2.1 Organic Removal

4.2.1.1 Oil and COD removal

The oil content in effluent gradually increases with the increase in media filling ratio, as exhibited in the Figure. 4.2(a). The oil removal capability was found to be 80.36% at 10% FR and 24 h HRT, which proved to be the lowest. The reason for low efficiency is that less surface area is available for microorganisms to grow on them. Hence, it led to a decrement in the growth of microorganisms that are responsible for breaking down hydrocarbons from oil into less toxic contaminants such as CO₂ and water, reducing the ability to remove oil content from wastewater.

At a constant HRT of 24 h, the removal efficiencies increase from 84.54% to 90.64% as the media FR changes from 10% to 20% and then to 30%. The oil removal efficiency was calculated to be 93.48% at a filling ratio of 40%. At FR of 30%, plastic carrier media in MBBR removed 94.10% of oil, while BIOAQUA carrier media removed 77.05% from the oil refinery wastewater (Ali & Aziz, 2024).

A higher filling ratio provides enough surface area for microbial growth that allows maximum interaction between microorganisms and wastewater, which enhances the oil degradation rate. Therefore, a 30% filling ratio was selected as the optimum filling ratio to remove oil from oil refinery wastewater.

The effect of four filling ratios, i.e., 10, 20, 30, and 40%, on COD removal efficiency is outlined in Figure. 4.2(b). All experiments were performed at the same HRT of 24 h. The

results, as represented in the figure, demonstrated that the COD removal efficiencies were 82.3, 88.39, 91.06, and 94.80% for media FR of 10, 20, 30, and 40%, respectively.

Another study concluded that a higher carrier filling ratio (such as 50%, 60% or 70%) showed less COD removal efficiency as compared to a lower filling ratio, i.e., 30% and 40%. At a higher media filling ratio, no rapid and uniform movement was observed because of poor contact time between substrate and biofilm carrier media (Kamble & Shaha, 2020). Also, it does not give enough space for media recirculation, which often causes collisions between carrier media. This collision leads to less biofilm growth, specifically on the outer surface of the carrier media.

Lower filling ratios provide more space for greater media movement, enhancing the interaction between the biofilm and wastewater substrate. This promoted better biofilm development and improved COD removal efficiency. The findings underscore the importance of maintaining an optimal filling ratio for ensuring optimal reactor performance.

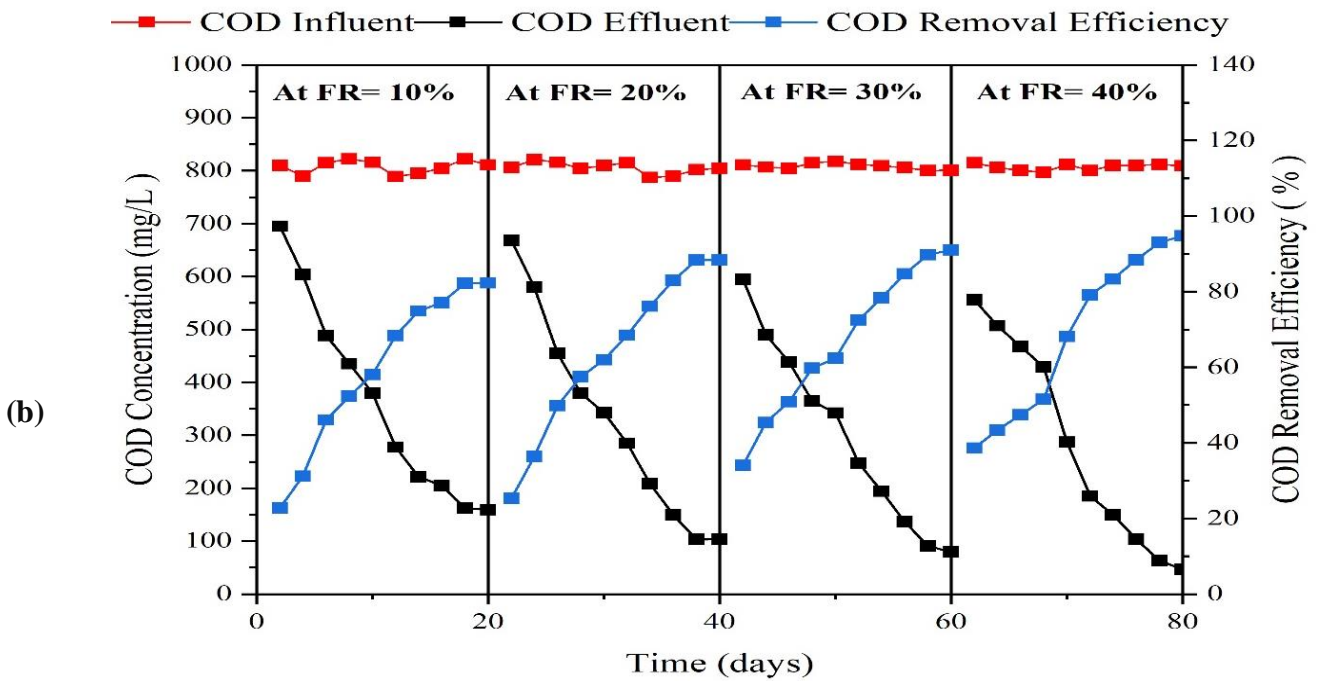
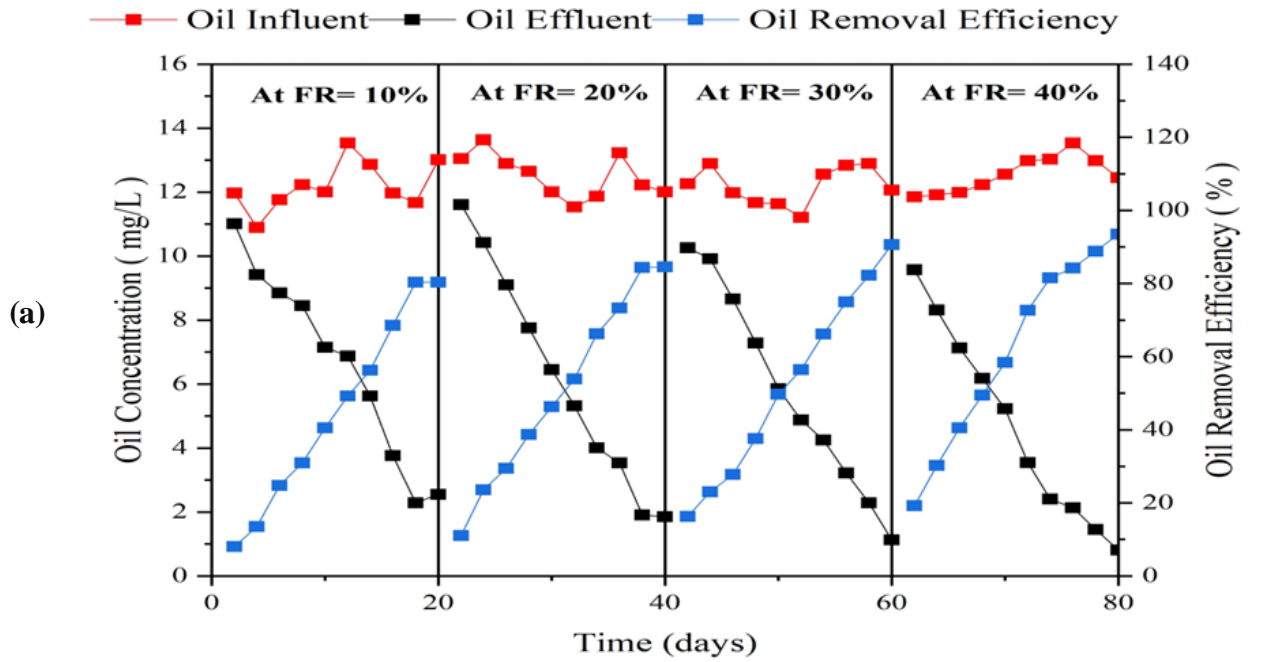


Figure. 4.2 (a) Oil and (b) COD concentration in effluent at different filling ratios such as 10%, 20%, 30%, and 40% and their respective oil removal efficiency.

4.2.1.2 BOD Removal

The Figure 4.3 shows that the MBSBR showed greater BOD removal efficiency at higher media FR than at lower FR. When the media filing ratio increases from 10 to 20, 30, and

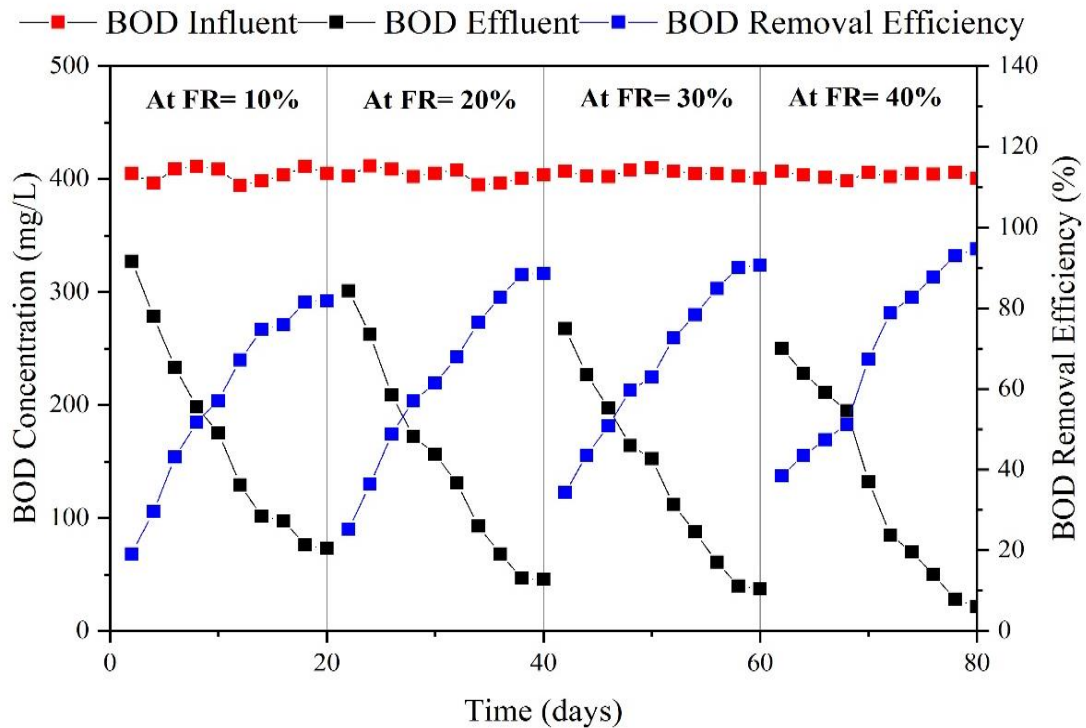


Figure 4.3 BOD concentration in effluent and its removal efficiency at different filing ratio such as 10, 20, 30, and 40%, respectively.

40%, the BOD removal efficiency was measured to be 81.89, 88.6, 90.6 and 95.6% respectively. A low filing ratio provides less area for microorganisms to biodegrade organic matter present in wastewater. The BOD/COD ratio is equal to or greater than 0.5, which shows that microorganisms can biologically degrade organic matter (Metcalf et al., 2003; Shokoohi et al., 2017).

A larger volume of media offers better retention of biomass resulting in providing stable microbial communities that are responsible for effective organic matter degradation (Phanwilai et al., 2020).

4.2.2 Nutrient Removal

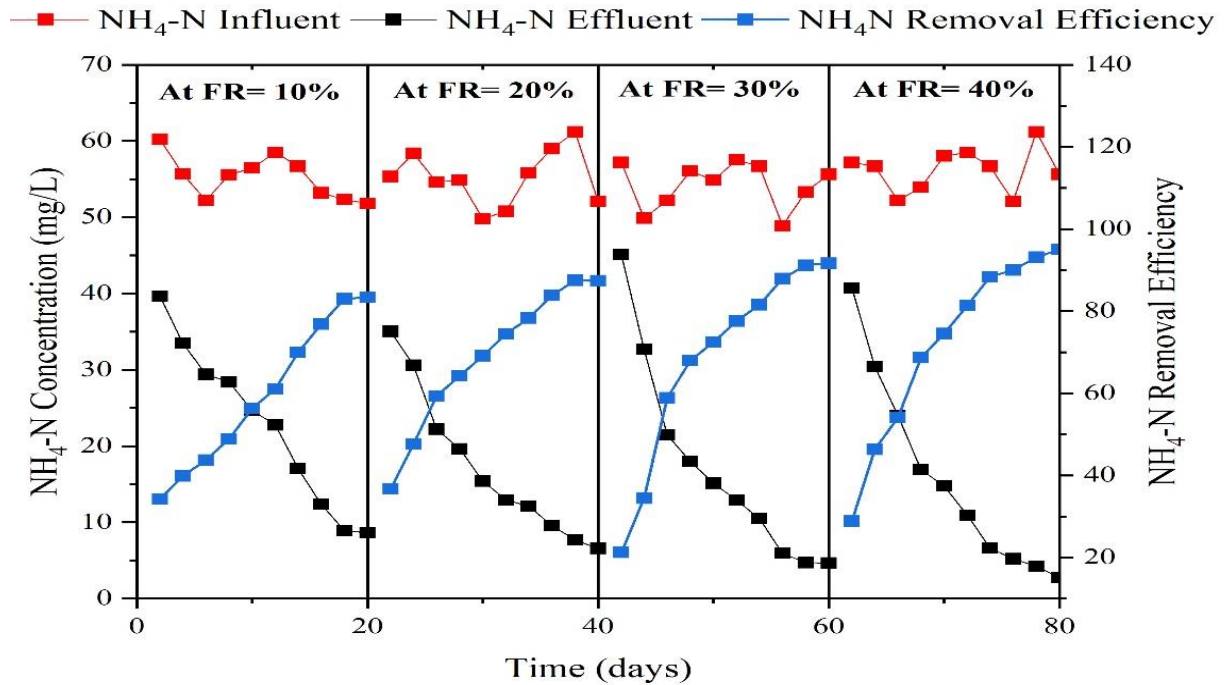


Figure. 4.4 NH₄-N concentration in effluent at different filling ratios such as 10%, 20%, 30%, and 40% and their respective NH₄-N removal efficiency

As displayed in Figure. 4.4, Ammonium-N elimination rates are higher at FR of 30 and 40% as compared to lower FR (10 and 20%). The highest removal efficiency was measured as 95%, followed by 91.64, 87.40, and 83.36% at 40, 30, 20, and 10%, respectively. The large number of carrier media provides adequate surface area for nitrifying bacteria growth. Because nitrifying bacteria use ammonium-N as a food source and decreases its concentration in oil refinery wastewater. Another study stated that more carrier media provide large mass transfer areas and sufficient oxygen supply to nitrify bacteria, which enhances the ammonia removal capability of the wastewater (Zhao et al., 2019). Based on these findings, a filling ratio of 30% was selected as the optimal FR because it not only

provides oil, COD, BOD, and NH₄-N removal efficiencies above 90% but also minimizes the system operational cost to treat oil refinery wastewater.

4.3 Effect of Hydraulic Retention Time

4.3.1 Organic Removal

4.3.1.1 Oil and COD Removal

Figure. 4.5 (a) highlights the impact of hydraulic retention time on oil removal potency. The oil removal potential was monitored to be 90.64% at a media filling percentage of 30% and HRT of 24 h. However, as the HRT was shortened, the oil removal efficiency gradually started decreasing. The oil removal efficiency declined from 90.64% to 86.21, 78.61, and 68.42% as HRT lowered from 24 h to 18 h, 12 h, and then to 6 h respectively. Another study on hospital wastewater treatment through MBBR concluded that oil removal efficacy was found to be maximal at the HRT of 24 h (Shokohi et al., 2018). An increase in HRT provides a longer time for microorganisms that degrade organic matter from wastewater under aerobic conditions, resulting an increment in oil removal performance (Zheng, 2016).

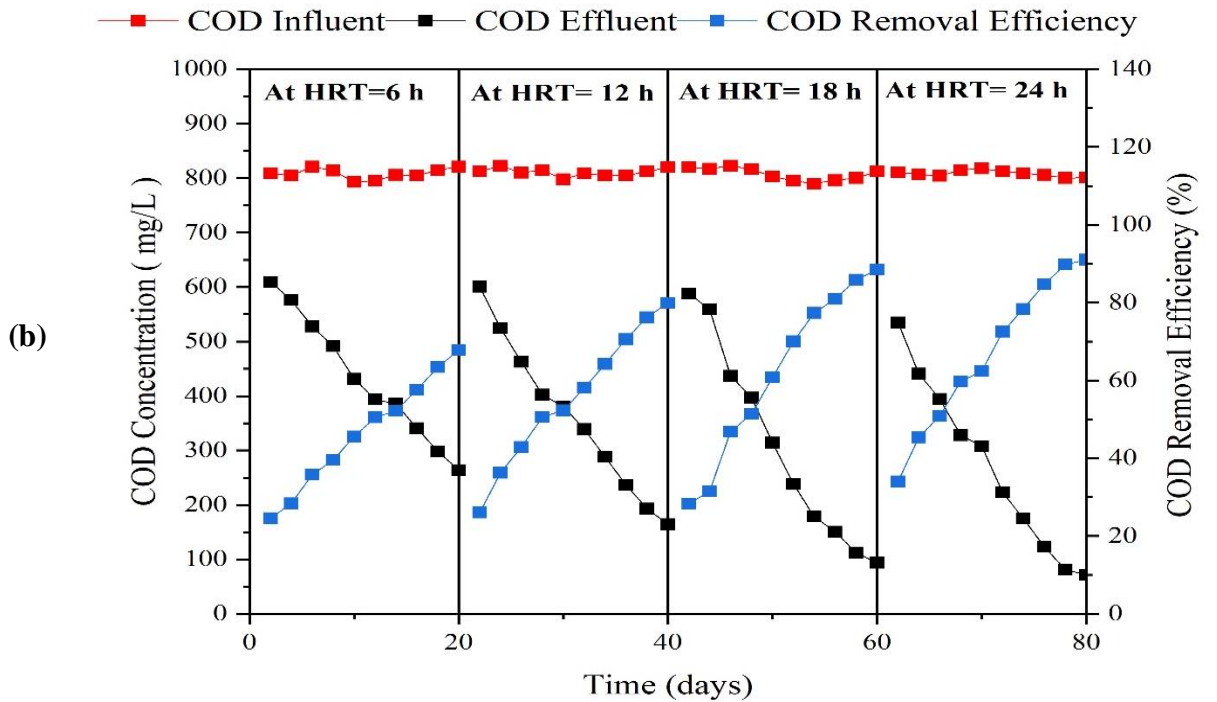
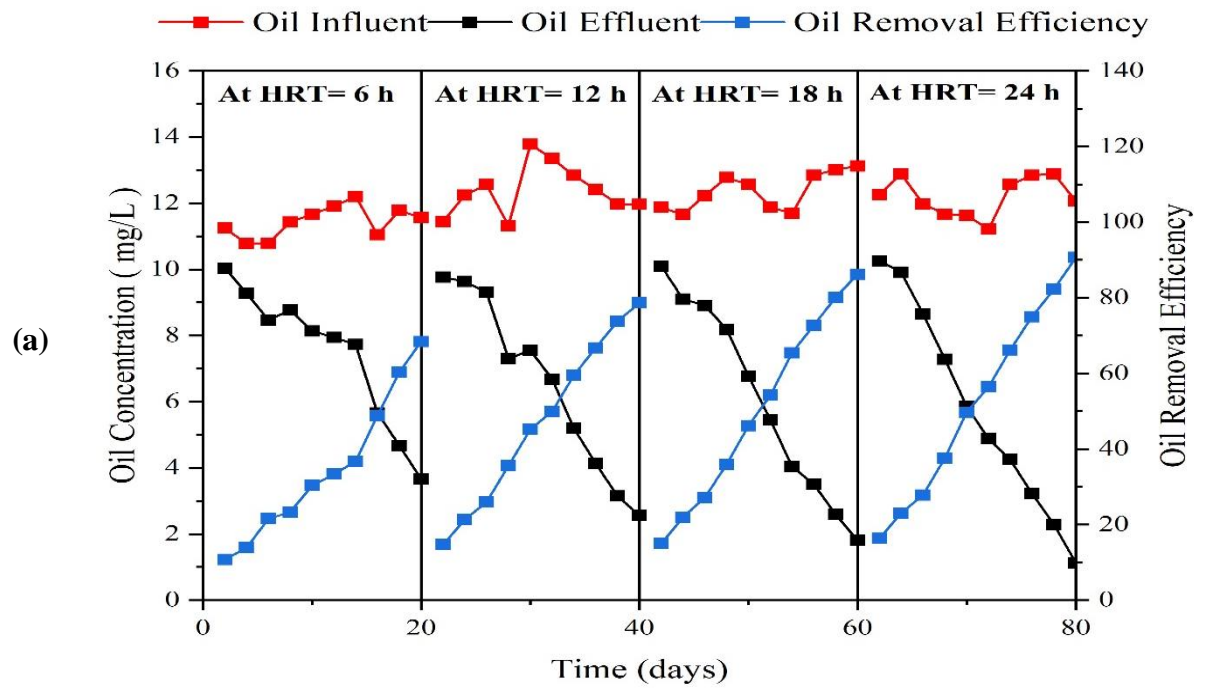


Figure. 4.5 (a) Oil and (b) COD concentration in effluent and its removal efficiency at different HRTs such as 6 h, 12 h, 18 h, and 24 h, respectively

The study evaluates the HRT influence on COD removal of reactor-based wastewater at constant loading of COD = 810 mg/L. Figure. 4.5(b) presents that COD removal rates vary in the effluent with varying HRTs. COD concentration in the effluent is lowest at an HRT of 24 h as compared to other HRTs. The highest COD removal efficiency was recorded to be 91.06% at 24 h HRT. The COD removal efficiency was detected to be 88.43, 79.95, and 67.84% at HRT of 18, 12, and 6 h, respectively. COD removal efficiency gradually decreased with the reduction in HRT from 24 to 6 h. This trend can be noticed in Figure. 4.5(b), highlighting the inverse relationship between HRT and COD reduction efficiency.

At lower HRT, microorganisms have insufficient contact time with carrier media to effectively remove organic matter through biological treatments (Abedinzadeh et al., 2018). Similarly, another study noted that COD removal efficiency maximizes because microorganisms get enough time to degrade organic matter with the increase in HRT (Abyar et al., 2017). These results emphasize the importance of optimizing HRT to achieve high COD removal efficiency in MBSBR treatment techniques. Long retention time provides sufficient time for microbial activity, leading to better organic pollutant degradation and enhanced overall wastewater treatment performance.

4.3.1.2 BOD Removal

The Figure 4.6 shows the BOD removal from wastewater at different HRTs. The BOD removal efficiency increased from 67.40 to 90.66% at an HRT of 6 to 24 h. 88.50% BOD removal efficiency was measured at an HRT of 18 h. At higher HRT, microorganisms get enough time to approach organic constituents present in wastewater and break down it into non-toxic and stable substances (Shokoohi et al., 2017).

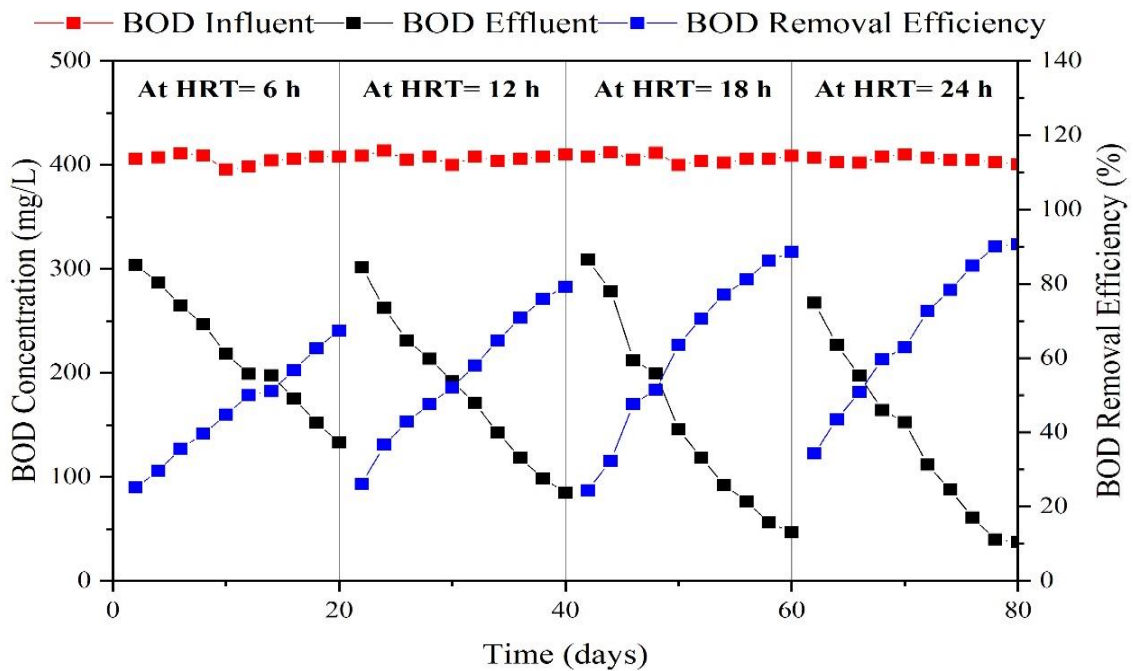


Figure 4.6 BOD concentration in effluent at different HRTs such as 6, 12, 18, and 24 h and their respective BOD removal efficiency

The complete degradation of a contaminant can only be done in longer retention time, as compared to a shorter retention time, because it has the inverse effect on the biological system (Nakhli et al., 2014). In another study, the effect of different HRTs was investigated on wastewater pollutant removal efficiencies through the MBBR system, and 18 h was

suggested by authors as the optimal HRT to remove wastewater micropollutants (Jiang et al., 2018).

4.3.2 Nutrient Removal

Nitrification is a process to remove ammonia from wastewater but the whole process takes plenty of time due to the prolonged development rate of nitrifying bacteria. The $\text{NH}_4\text{-N}$ removal rate drops from 91.64% to 67.80% by lowering HRT from 24 h to 6 h, as demonstrated in Figure. 4.7. The MBSBR exhibited a removal efficiency of 88.72 % at an

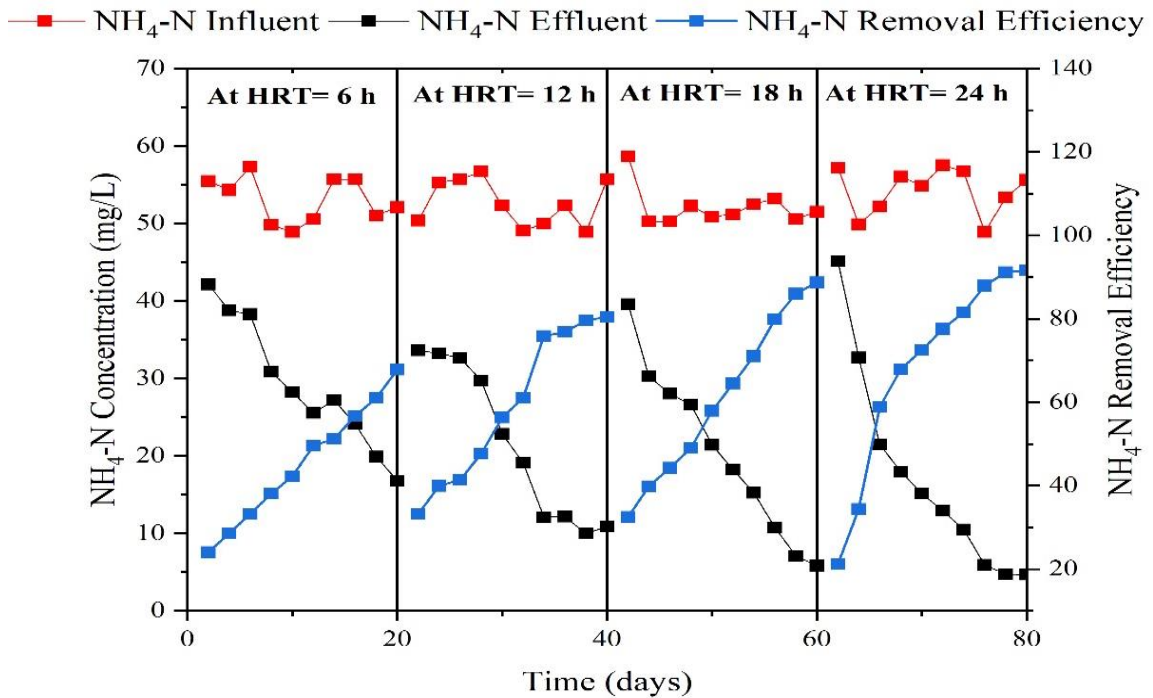


Figure. 4.7 $\text{NH}_4\text{-N}$ concentration in effluent at different HRTs such as 6, 12, 18, and 24 h and their respective $\text{NH}_4\text{-N}$ removal efficiency

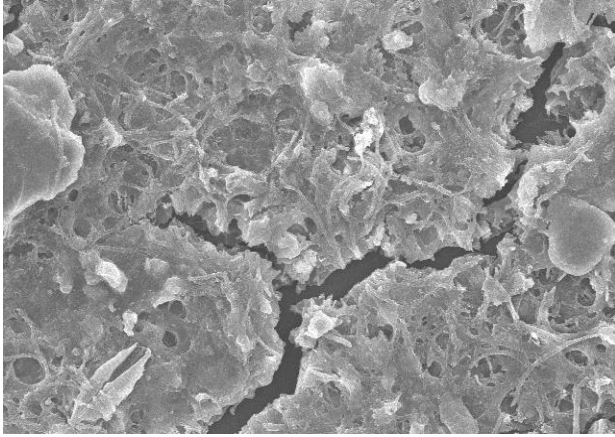
HRT of 18 h. Nitrification is a two-step procedure in which, for initial step, nitrifying bacteria converts $\text{NH}_4\text{-N}$ to nitrite, and then, for the second step, it is converted to nitrate. Therefore, the overall process needs higher retention time to achieve better outcomes. The

ammonia degradation rate decreases at lower HRT due to less reaction time between microorganisms and wastewater and an incomplete nitrification process (Sandip & Kalyanraman, 2019).

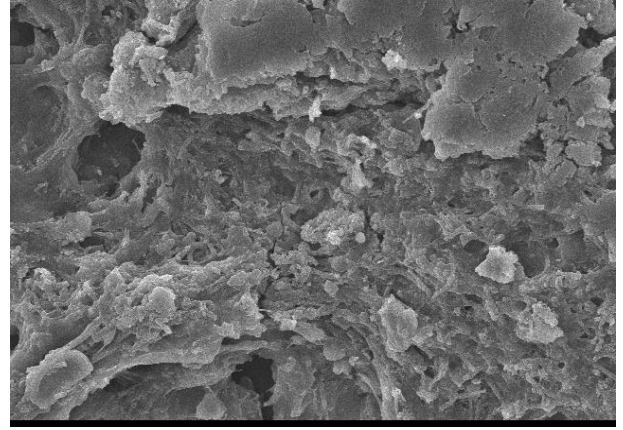
Longer HRT provides *Nitrosomonas* and *Nitrobacter* bacteria with enough reaction time, which eventually results in a higher $\text{NH}_4\text{-N}$ removal rate (Sayara et al., 2021). After investigating the performance of MBSBR on organic and nutrient removal, 18 h was selected as the optimum HRT to treat petrochemical wastewater because the difference in removal efficiency is minimal between HRT of 18 h and 24 h. Also, shorter HRT reduces reactor volume which automatically lowers the operational cost while maintaining high removal efficiency and provides greater operational flexibility in practical terms.

4.4 Scanning Electron Microscopy Observation

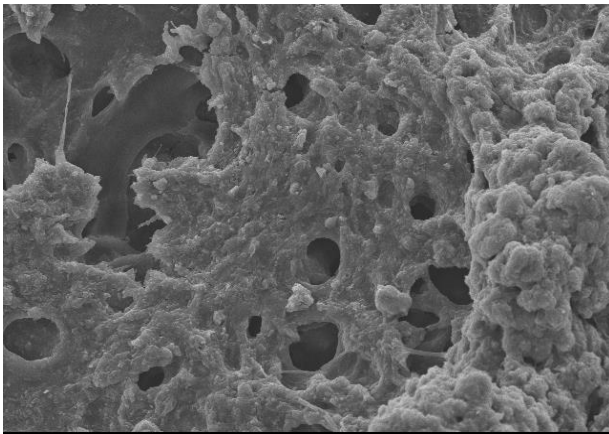
Scanning Electron Microscopy (SEM) analysis of thick and dense biofilm present on carrier media at several filling ratios is displayed in the Figure. 4.8. As media FR increases, the removal efficiency also escalates because mass transfer between microorganisms and nutrients doesn't get disrupted. The biofilm thickness gradually decreases with the increase in media FR, which contributes to providing the best outcome, i.e. removing many contaminants from wastewater, because higher media FR facilitates better mass transfer exchange. From the Figure. 4.8, we conclude that biofilm mostly consists of bacillus, cocci, filamentous bacteria, and extra polymeric substances at all filling ratios (Zhao et al., 2019).



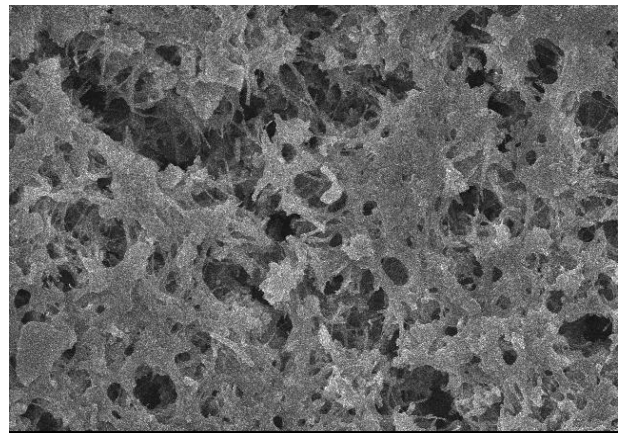
(a)



(b)



(c)



(d)

Figure. 4.8 SEM Analysis of carrier media at filing ratio (a) 10% (b) 20% (c) 30% and (d) 40%

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The moving bed sequencing batch reactor (MBSBR), a hybrid biological treatment technology, was used in this study due to its capability and effectiveness in treating industrial wastewater, particularly from oil refineries. MBSBR combines the interests of both attached growth and suspended growth processes by providing them with easy access to wastewater treatment. This study investigated the influence of various media filling ratios and hydraulic retention times (HRTs) in treating oil refinery wastewater using MBSBR. These operational parameters were varied to investigate their impact on the COD, BOD, NH₄-N, and oil content removal from the wastewater.

The study concluded that increasing both media FR and HRT significantly enhanced the removal efficiencies of the pollutants, which typically improved the wastewater treatment performance. The enhanced treatment performance was primarily due to improved conditions for microbial growth and activity. The carrier media plays a vital role in this process because a higher media FR ratio provides enough available surface area for biofilm growth. Also, longer HRT provides more time for microorganisms to interact with wastewater. These two factors work simultaneously to enhance the system removal efficiency.

The MBSBR system achieved 88.43, 88.50, 84.54, and 88.72% removal efficiency for COD, BOD, oil, and NH₄-N, respectively at HRT of 18 h and media FR of 30%. Hence, 18 h HRT and 30% of media FR were selected as optimal operating conditions. Because it

is easier to operate and lowers the operational cost in practical applications. MBSBR technology can easily adapt to changes in filling ratio, HRT, and other operating conditions, ensuring consistent wastewater treatment performance. Overall, the conclusion of this study suggested that the MBSBR system is highly effective for the oil refinery wastewater treatment. As industries continue to face stringent environmental regulations and the need for effective wastewater management, technologies like MBSBRs are a feasible option for sustainable and effective treatment solutions.

5.2 Recommendations

According to the findings of this study, there are many areas where further research could be done to enhance the understanding and application of moving bed sequencing batch reactor (MBSBR) technology for the treatment of any type of industrial wastewater. Future research should focus on sustainable practices, such as utilizing biodegradable and eco-friendly carrier media such as coconut shells and promoting the use of renewable energy sources to power the treatment facility. While this study has demonstrated high removal efficiencies for oil, COD, BOD, and $\text{NH}_4\text{-N}$ under specific conditions, it is crucial to understand how the system performs over extended periods, especially when exposed to continuous variations in influent characteristics. Monitoring the microbial community over time and assessing the resilience of the biofilm under these conditions would provide valuable insights into the long-term operational stability of the system. Secondly, future studies should use real oil refinery wastewater to check the efficacy of the semi-pilot scale MBSBR to achieve promising results and monetary value promoting full-scale plants. New studies should be focused on the integration of MBSBR with other advanced treatment technologies, such as membrane bioreactors (MBR) or advanced oxidation processes (AOP), to accomplish higher removal efficiencies and address a broader range of pollutants. In summary, future research should focus on the long-term performance, optimization of operational parameters, biofilm control, integration with advanced treatment technologies, and the economic feasibility of MBSBR systems. These studies will help to further refine the technology, making it an even more effective and adaptable solution for the treatment of petrochemical and other industrial wastewaters.

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